Supplementary Information

Photo-catalyst-free Photomediated Pinacol Coupling of Ketones/Aldehydes by Formate at Room Temperature

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1.General Experimental Information

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Both alkenes are purchased from Energy Chemical.

NMR spectra were obtained on a JNM-ECZ600R/S1 spectrometer. NMR data were obtained for ¹H at 400 MHz and 600 MHz, and for ¹³C at 100 MHz. The ¹H NMR (400 MHz, 600 MHz) chemical shifts Chemical shifts were measured relative to CDCl₃ or DMSO-*d*₆ as the internal references (CDCl₃: δ = 7.26, DMSO-*d*₆: δ = 2.50). The ¹³C NMR (100 MHz) chemical shifts were given using CDCl₃ as the internal references (CDCl₃: δ = 77.16). ESI HRMS was recorded on a Waters SYNAPT G2 and Water XEVO G2 Q-ToF. The 365nm light were obtained on PLH-18CU. TLC was performed on glass-backed silica plates. Column chromatography was performed on silica gel (300-400 mesh), eluting with petroleum ether and ethyl acetate. The yields are based on isolated compounds after purification.

2. General procedure for the synthesis of products

A 15 mL Schlenk tube equipped with a magnetic stirring bar was charged with ketone (0.1 mmol), HCOOK (0.2 mmol), EtOH (4 mL). The mixture was then stirred at room temperature under air or nitrogen atmosphere and irradiated with a 30 W of 365 nm LEDs light for 12 h (**Figure S1**). After completion of the reaction, the reaction mixture was diluted with EtOAc and then concentrated in vacuo, and the resulting residue was purified by column chromatography on silica gel or preparative TLC (EtOAc/PE, etc.) to afford the corresponding products.

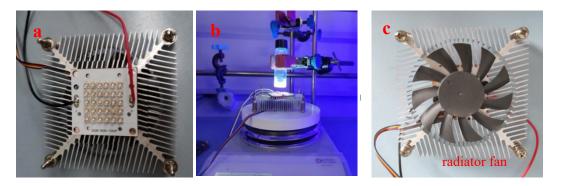


Figure S1 Reaction equipment : a. Assemble the lamp plate and heat sink. Use purchased 30 W UV LEDs with radiator. b. The distance between the lamp plate and the heat sink should be 0.5 to 1cm. c. The back of the radiator fan.

3. Procedure for gram scale reaction

The 250 mL quartz round bottom flask, charged with a magnetic stir bar, was vacuumed and backfilled with argon for three times. ketone (20 g), HCOOK (2 equiv), and EtOH (160 mL), were added into flask sequentially under the protection of Argon. Then the flask was sealed connected to balloon to release the gas generated during the reaction and stirred at room temperature under light (365 nm, 30 W) for 8-72 h (**Figure S2**). After completion, the reaction mixture was diluted with EtOAc and then concentrated in vacuo, and the resulting residue was purified by recrystallization from EtOAc to afford the pinacol product.



Figure S2 Gram scale reaction equipment.

4. Light source information.

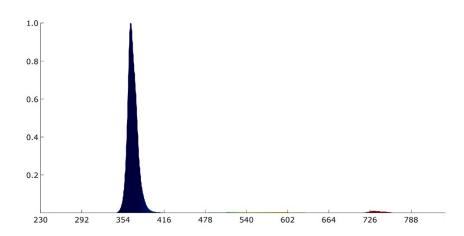


Figure S3 The UV spectrum used in this work (365 nm)

5. Reduction potential of aldehydes and ketones.

Aldehydes and ketones	Reduction potential	References
O ₂ N O	-0.86 V	Synlett 2016 , 27, 714–723
	-2.06 V	Synlett 2016, 27, 714–723
MeO	-1.07 V	LangesChemistryHandbookVersion13 th
	1.40.11	Synlett 2016, 27, 714–723
NC	-1.42 V	Chemical Society 1986, 108, 7727-7738.
	-1.66 V	Synlett 2016, 27, 714–723
F ₃ C 0	-1.58 V	Chemical Society 1986, 108, 7727-7738.
Ph	-1.72 V	Synlett 2016 , 27, 714–723
	-1.73 V	Synlett 2016 , 27, 714–723
	-1.85 V	Synlett 2016, 27, 714–723
0 T	-1.82 V	Chem. Sci., 2022, 13,5973–5981
CI ²	-1.75 V	Chemical Society 1986, 108, 7727-7738.
	-1.93 V	Synlett 2016, 27, 714–723
	-0.94 V	LangesChemistryHandbookVersion13th
	-1.89 V	Chem. Commun., 2010, 46, 2730–2732
	-2.11 V	Chem. Commun., 2018, 54, 11017
0	-1.94 V	Synlett 2016, 27, 714–723
0	-2.28 V	Synlett 2016, 27, 714–723
	-2.24 V	Synlett 2016, 27, 714–723
0 ''	-2.11 V	Synlett 2016, 27, 714–723
	-1.85 V	Chem. Sci.,2019, 10,10937
	-2.1 V	Nat. Commun. 2021, 12, 3306.
	-1.91 V	Synlett 2016 , 27, 714–723
0	-1.74 V	Synlett 2016, 27, 714–723
F ₃ C	-1.90 V	<i>Chemical Society</i> 1986 , 108, 7727-7738.

 Table S1
 Some reduction potential of aldehydes and ketones reported by previous works.

O ₂ N O	-0.93 V	Synlett 2016 , 27, 714–723
Q	-2.16 V	Synlett 2016, 27, 714–723
	-2.19 V	Chemical Society 1986 , 108, 7727-7738.
	-2.23 V	Synlett 2016 , 27, 714–723

6. The effect of water on the reaction.

	(Y) <u> </u>	<, 365 nm LED H₂O, r.t., Air	HO 2a
Entry	H ₂ O	Volume	Yield (%)
1	1 eq	0.015 ul	99
2	2 eq	0.030 ul	99
3	4 eq	0.060 ul	99
4	8 eq	0.120 ul	99
5	10 eq	0.150 ul	99
6	20 eq	0.300 ul	97
7	40 eq	0.450 ul	96
8	100 eq	1.5 mL	95

 Table S2
 The effect of water on the reaction ^{a,b}

General reaction conditions (unless otherwise stated) : a 0.1 mmol of 1a, 0.2 mmol of HCOOK, 4 mL of EtOH, 30 W LEDs (365 nm), at air atmosphere and room temperature for 6 h.^b Yields determined by NMR analysis.

7. The effect of the amount of HCOOK on the reaction

o 1a	HCOOK, 365 nm LED EtOH, H ₂ O, r.t., Air	
Entry	HCOOK	Yield (%)
1	1 eq	90
2	2 eq	99
3	4 eq	99
4	6 eq	99

 Table S3
 The effect of the amount of HCOOK on the reaction ^{a,b}

General reaction conditions (unless otherwise stated) : ^a 0.1 mmol of **1a**, 0.2 mmol of HCOOK, 4 mL of EtOH, 30 W LEDs (365 nm), at air atmosphere and room temperature for 6 h. ^b Isolated Yields.

8. HR-Mass spectrum of mechanism study.

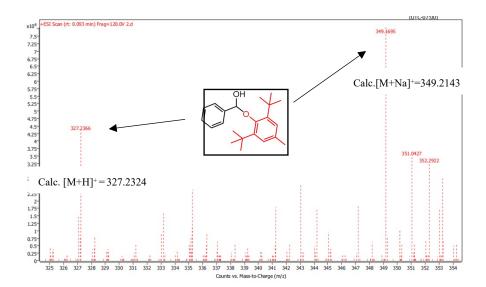


Figure S4. HR-Mass spectrum

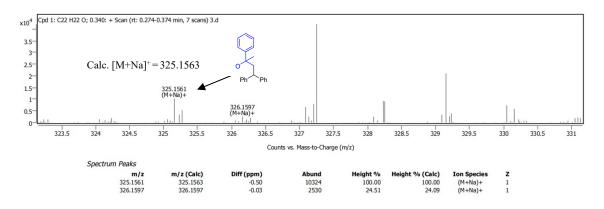
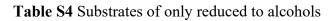
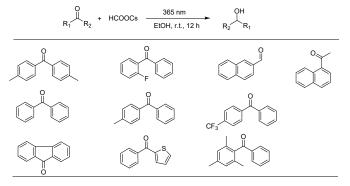


Figure S5. HR-Mass spectrum

9. Substrates of only reduced to alcohols.





10. The study of selectivity.

General reaction conditions: 0.1 mmol of **1a**, 0.2 mmol of HCOOM, 4 mL of solvent, 30 W LEDs (365 nm), at air atmosphere for 12 h. Results were determined by NMR analysis.

1a HCOOM, 365 nm LED EtOH, r.t., Air,12 h 2a			
Entry	HCOOM	meso:dl	Yield (%)
1	HCOONH ₄	1:1.22	60
2	HCOONa	1:1.16	97
3	НСООН	1:1.24	90
4	(HCOO) ₂ Ca	1:1.13	95
5	HCOOK	1:1.18	99

Table S5The effect of HCOOM on the selectivity.

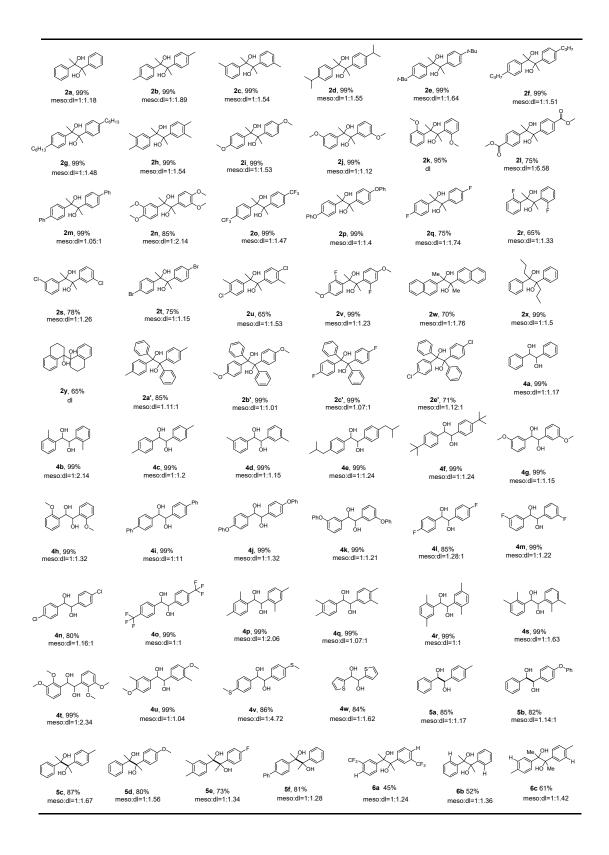
Table S6The effect of solvent on the selectivity.

	HCOOK, 365 nm LED Solvent, r.t., Air, 12 h		
Entry	Sovent	meso:dl	Yield (%)
1	DMSO	1:1.45	99
2	MeOH	1:1.16	99
3	EtOH	1:1.18	99
4	iPrOH	1:1.12	99
5	EtOH/DMSO (1:1)	1:1.38	99
6	EtOH/H ₂ O (2:1)	1:1.30	90
7	EtOH/DCM (1:1)	1:1.07	88
8	EtOH/HFIP (1:1)	1:1.02	93
9	EtOH/DMF (1:1)	1:1.37	90

Table S7The effect of temperature on the selectivity.

ĺ	HCOOK, 3 EtOH, A	ir, 12 h	HO 2a
Entry	T(°C)	meso:dl	Yield (%)
1	-78	-	No reaction
2	-40	1:1.11	56
3	0	1:1.17	94
4	20	1:1.18	99
5	40	1:1.14	99
6	60	1:1.09	99

11. The selectivity of products.



12. GC analyzes the gas of the reaction.

The template reaction stirred for 8 hours, then extract gas (1 mL) from reaction vial for GC analysis. Agilent 7890A (Thermo, USA), detector: TCD, temperature 150°C, column: HP-PLOT/Q 30 m X 0.32 mm X 20 um, inject temperature 150°C, Oven Column 70°C, He, 8min/L.

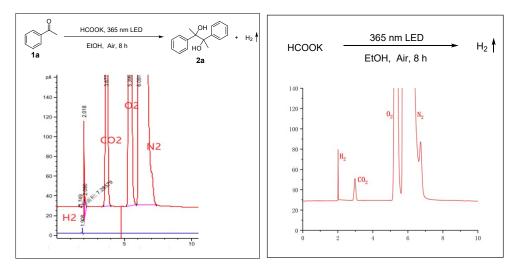
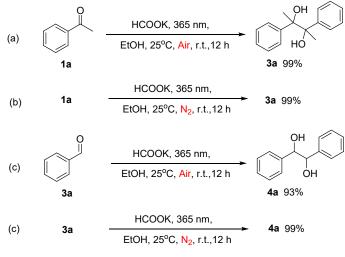


Figure S6. GC analyzes the gas of the reaction.

13. The effect of oxygen on the reaction

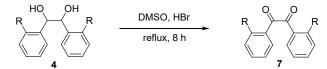
The reaction doesn't require oxygen. Oxygen is not a necessary condition for the reaction, and the reaction can proceed without the involvement of oxygen. Oxygen is not required for the production of carbon dioxide radical anion.



Scheme S1 The effect of oxygen on the reaction

14. Procedure for synthetic application.

Synthsis of 7a and 7h: The synthesis of 7a and 7h followed the procedure of previous work : Euro. J. Org. Chem. 2004, 3040



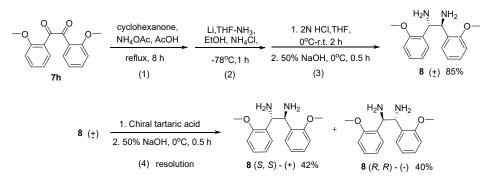
20 g of **4** was added to a 500-mL round-bottomed flask, and 250 mL of DMSO and 40 mL of HBr (40%) added. The mixture was headed to 120 °C. After the mixture was stirred for 3 h, the mixture was poured into 1 L of vigorously stirred water. The mixture was left overnight to cool to room temperature, the solid collected by filtration, washed 4 times with water, and dried under reduced pressure to afford **7** as a brown solid. This product was further purified by recrystallization from hexane/EtOAc. dried under reduced pressure to afford **7** as the white solid.

Benzil 7a, 18.8 g 92%, white solid, mp 105–106°C. ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.00 (dt, *J* = 7.4, 1.3 Hz, 4H), 7.72 – 7.64 (m, 2H), 7.54 (td, *J* = 7.7, 1.7 Hz, 4H); ¹³C NMR (100 MHz, DMSO):δ 194.70, 135.02, 133.15, 130.04, 129.16.

1,2-bis(2-methoxyphenyl)ethane-1,2-dione 7h, 19.3 g 95%, white solid, 131–132°C. ¹H NMR (400 MHz, DMSO-*d*₆): δ 8.10 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.59 (ddd, *J* = 8.3, 7.3, 1.8 Hz, 2H), 7.14 (td, *J* = 7.5, 1.0 Hz, 2H), 6.97 (dd, *J* = 8.4, 1.0 Hz, 2H), 3.61 (s, 6H);¹³C NMR (100 MHz, DMSO): δ 192.57, 160.48, 135.65, 130.55, 123.56, 121.46, 112.60, 55.98.

Synthsis of (<u>+</u>)-1,2-bis(2-methoxyphenyl)ethane-1,2-diamine:

The synthesis of **8** followed the procedure of previous works : Org. Synth **1993**, 71, 22; J. Am. Chem. Soc. **2006**, 128, 14212-14213.



(1) A 500 mL, three-necked, round-bottomed flask equipped with a mechanical stirrer and a reflux condenser is charged with 200 mL of glacial acetic acid, 20 g (74 mmol) of 7 h, 40 g of ammonium acetate and 80 mL (0.77 mol) of cyclohexanone. The mixture is stirred and heated at reflux temperature for 3 h and then, while hot, poured into 1 L of vigorously stirred water. The mixture is left overnight to cool to ambient temperature, the crystals are collected by filtration, washed 4 times with 150 mL of water, crushed in a mortar and dried under reduced pressure to

give a pale yellow solid. The solid was used directly in the next step without further purification.

(2) A 500 mL, four-necked, round-bottomed flask equipped with a mechanical stirrer, thermometer and dry ice condenser is charged with the solid product of last step 25 g (0.250 mol). The flask is flushed with argon, and 100 mL of tetrahydrofuran is added. The mixture is stirred until all solids dissolve, cooled to -78° C (dry ice/acetone bath) and treated with a stream of gaseous ammonia until the volume of liquid increases by about 300 mL. One of the side necks is then equipped with a solids addition funnel and 2.4 g (1.00 mol) of lithium is slowly introduced by cutting the wire with scissors in a gentle stream of argon. The rate of lithium addition is such that the temperature does not rise above -65° C. Following the addition of lithium, the mixture is stirred for 30 min and 10 mL of ethanol is slowly added. The mixture is stirred for an additional 20 min and 25 g of ammonium chloride is added. The cooling bath is removed, the mixture is allowed to warm to 0°C, 100 mL of water is carefully introduced, and the phases are separated. The aqueous phase is washed 3 times with 200 mL of ether and the combined organic extracts are washed with brine, dried over anhydrous sodium sulfate, filtered and concentrated with a rotary evaporator to about 100 mL.

(3) The solution is transferred to a 500 mL, one-necked, round-bottomed flask equipped with a mechanical stirrer, cooled to 0°C and treated with 100 mL of 2 N aqueous hydrochloric acid. The biphasic mixture is vigorously stirred at ambient temperature for 1 h, 150 mL of water is added and phases are separated. The organic phase is washed with 100 mL of water and the combined aqueous phases are extracted with 150 mL of dichloromethane. The aqueous solution is then carefully treated with 100 mL of 2 N aqueous sodium hydroxide and the mixture is extracted 3 times with 150 mL of methylene chloride. The combined organic extracts are washed with brine, dried over anhydrous sodium sulfate, and filtered. Removal of volatile material under reduced pressure (water aspirator) gives 17 g (yield of three steps 85%) of racemic diamine as a pale yellow solid. M.p: 85-87 °C; ¹H NMR (300 MHz, CDCl₃): δ 7.22 (dd, *J* = 7.5, 1.8 Hz, 2H), 7.10 (td, *J* = 7.8, 1.8 Hz, 2H), 6.81 (td, *J* = 7.5, 1.2 Hz, 2H), 6.74 (dd, *J* = 8.2, 1.2 Hz, 2H), 4.45 (s, 2H), 3.76 (s, 6H); ¹³C NMR (100 MHz, CDCl₃): 156.85, 132.17, 128.00, 127.54, 120.22, 110.30, 55.39, 55.17.

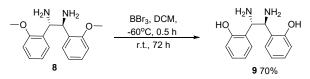
(4) (1S,2S)-1,2-bis(2-methoxyphenyl)ethane-1,2-diamine:

A 500 mL round-bottomed flask was charged with 24.3 g (0.09 mol) of (d,l)-8 and 150 mL ethanol. The mixture was heated to 70 °C, and a homogeneous solution of L-(+)-tartaric acid (13.4 g, 0.09 mol) in 150 mL ethanol was added. The tartrate salts precipitate immediately, and the mixture was cooled to ambient temperature. The solid were collected by filtration, washing twice with ethanol, and dried under reduced pressure. The tartrate salts were recrystallized twice using ethanol/water to give 16.9 g of colorless crystals. The salt was transferred to a 500 mL, round-bottomed flask and suspended in 150 mL water. After the mixture was cooled to 0-5 °C, under vigorously stirring, 15 mL of 50% aqueous NaOH was added dropwise followed by 150 mL dichloromethane, and stirring is continued for 1 h. The aqueous phase was extracted with CH₂Cl₂ (100 mL × 3). The combined organic phase was washed with brine and dried over MgSO₄. Removal of the volatile material under reduced pressure gave (S,S)-**8** as a yellowish solid (10.2 g, yield: 42%), $[\alpha]_D^{20} = -54.9$ °(c = 0.5, CHCl₃).

(1R,2R)-1,2-bis(2-methoxyphenyl)ethane-1,2-diamine:

The filtrates from all crystallizations are combined and the solvent is evaporated under vacuum. The residual is transferred to a 500 mL, round-bottomed flask, and suspended in 150 mL of water, After the mixture was cooled to 0-5 °C, under vigorously stirring, 15 mL of 50% aqueous NaOH was added dropwise followed by 150 mL dichloromethane, and stirring is continued for 1 h. The aqueous phase was extracted with CH_2Cl_2 (50 mL × 3). The combined organic phase was washed with brine and dried over MgSO₄. Removal of the volatile material under reduced pressure gave enriched (R,R)-8 as yellowish solid. This material was treated in the same manner as described for the opposite enantiomer to give 9.7 g of white solid, yield: 40 %, [α]_D²⁰ = +54.1° (c = 0.5, CHCl₃).

Synthesis of 2,2'-((1S,2S)-1,2-diaminoethane-1,2-diyl)diphenol 9



A solution of **8** (5 g 13.4 mmol) in dry CH₂Cl₂ (50 mL) was cooled to -78°C and then BBr₃(5.2 mL, 52 mmol) was added. After the mixture was stirred for 30 min at -78°C, the cooling bath was removed and the stirring was continued for additional 24 h. The reaction was quenched by addition of water (10 mL), and CH₂Cl₂ was removed under the reduced pressure. The aqueous solution was neutralized by addition of aqueous NaHCO₃, and then extracted with EtOAc (50 mL × 3). The organic layer was washed with brine, and dried over Na₂SO₄. After removal of the solvent, the residue was purified by column chromatography to give 3.1 g of **9** as a light yellow solid: **70%**, ¹H NMR (400 MHz, DMSO-*d*₆): δ 6.98 (td, *J* = 7.6, 1.8 Hz, 2H), 6.81 (dd, *J* = 7.6, 1.7 Hz, 2H), 6.68 (dd, *J* = 8.0, 1.3 Hz, 2H), 6.55 (td, *J* = 7.4, 1.3 Hz, 2H), 6.02 (s, 4H), 4.12 (s, 2H), 3.42 (s, 2H); ¹³C NMR (100 MHz, DMSO): δ ¹³C NMR (101 MHz, DMSO) δ 156.44, 128.95, 128.93, 127.69, 117.88, 116.33, 58.46.

15. Determination of the reaction quantum yield

Determination of the light intensity at 365 nm: According to the procedure work (*Chem. Sci.* **2015**, *6*, 5426-5434). *J. Am. Chem. Soc.* **2021**, *143*, 8987-8992. Hatchard, C. G.; Parker, C. A. Proc. Roy. Soc. (London) 1956, A235, 518–536. Pure Appl. Chem. 2004, 76, 2105–2146. Chemical Actinometry. Handbook of Photochemistry, 3rd Ed; Taylor & Francis Group, LLC. Boca Raton, FL, 2006, 601–616)

The photon flux of the LED was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (88 mg) in 30 mL of a 0.05 M H₂SO₄ solution. And A buffered solution of 1,10- phenanthroline was prepared by dissolving 1,10-phenanthroline (2 mg) and sodium acetate (11.25 g) in 50 mL of a 0.5 M solution H₂SO₄. Both solutions were stored in the dark. To determine the photon flux of the LEDs, the ferrioxalate solution (2.0 mL) was placed in a cuvette and irradiated for 30 s at $\lambda_{max} = 365$ nm. After

irradiation, the phenanthroline solution (0.35 mL) was added to the cuvette and the mixture and stired in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A nonirradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated using eq 1.

$$mol \, Fe^{2\,+} = \frac{V \cdot \Delta A}{l \cdot \varepsilon} \quad (1)$$

where V is the total volume (0.00235 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, 1 is the path length (1.00 cm),

and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm (11,100 L mol⁻¹ cm⁻¹). The photon flux can be calculated using eq 2.

photo
$$flux = \frac{mol Fe^{2+}}{\Phi \cdot t \cdot f} =$$
(2)

where Φ is the quantum yield for the ferrioxalate actinometer (1.21 for a 0.006 M solution at λ = 365 nm), f (0.999) is the fraction of light absorbed at λ = 365 nm by the ferrioxalate actinometer. (Hatchard, C. G.; Parker, C. A. Proc. Roy. Soc. (London) 1956, A235, 518–536.) **t** is the irradiation time (30 s).

The photon flux was calculated (average of three experiments) to be 6.94×10⁻⁸ einsteins s⁻¹

Determination of the reaction quantum yield:

The reaction was carried out in a reaction tube under standard conditions: acetophenone **1a** (0.1 mmol), potassium formate **2a** (0.2 mmol, 2 eq) and 2 mL EtOH were added to a reactor containing a magnetic stirrer. The mixture was stirred under nitrogen atmosphere while irradiated by 365 LEDs for 30 min. The crude yield of the product **3a** was determined by ¹H NMR based on a 1,3,5-trimethoxybenzene standard and the final yield was 50% (average of three experiments).

The reaction quantum yield (Φ) was determined using eq 4. Where the photon flux is 6.94×10^{-8} einsteins s⁻¹ (determined by actinometry as described above), **t** is the reaction time (1800 s) and **f** is the fraction of incident light absorbed by the reaction mixture, determined using eq 3. An absorption spectrum of the reaction mixture gave an absorbance value of 0.857 at 365 nm (A = 0.857 indicating that the fraction of light absorbed is f = 0.861).

$$f = 1 - 10^{-A(365 \text{ nm})} \quad (3)$$
$$\Phi = \frac{Mol \, product}{flux \cdot t \cdot f} \quad (4)$$

The reaction quantum yield (Φ) was thus determined to be $\Phi = 0.469$

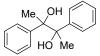
$$\Phi = \frac{Mol \ product}{flux \cdot t \cdot f} = \frac{(0.1 \times 10^{-3} \text{mol} \times 0.5)}{(0.1 \times 10^{-3} \text{mol} \times 0.5)/(6.94 \times 10^{-8} \text{einsteins s}^{-1} \times 1800 \text{s} \times 0.861) = 0.469}$$

16. Compound Characterizations

The products 2a¹, 2b², 2c³, 2i⁴, 2j³, 2l⁵, 2m⁶, 2o⁷, 2p⁸, 2q⁸, 2r⁹, 2s³, 2t⁶, 2v⁸, 2w¹, 2x⁹, 2y⁸, 2z⁸, 3a¹⁰,

 $3b^{8}$, $3c^{10}$, $4a^{1}$, $4b^{11}$, $4c^{8}$, $4d^{10}$, $4f^{1}$, $4q^{3}$, $4p^{11}$, $4g^{12}$, $4i^{3}$, $4t^{13}$, $4l^{3}$, $4m^{14}$, $4n^{14}$, $4o^{9}$, $4w^{3}$, $5f^{8}$, $5d^{15}$, $5i^{8}$, $6b^{8}$, $6c^{8}$ are consistent with published ones.

2,3-Diphenylbutane-2,3-diol (2a)



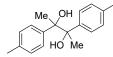
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.18

¹H NMR (400 MHz, CDCl₃): δ 7.26 – 7.09 (m, 10H), 2.72 (s, 1H), 2.41 (s, 1H), 1.55 (s, 2H), 1.46 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 143.87, 143.50, 127.44, 127.28, 127.25, 127.16, 127.09, 127.07, 127.00, 126.91, 78.91, 78.66, 25.11, 24.94.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₈O₂ [M+Na]⁺: 265.125, Found: 265.1195.

2,3-Di-p-tolylbutane-2,3-diol (3b)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.89

¹H NMR (400 MHz, CDCl₃): δ 7.16 – 6.90 (m, 8H), 2.71 (s, 1H), 2.30 (s, 4H), 2.29 (s, 3H), 1.49 (s, 2H), 1.41 (d, *J* = 3.3 Hz, 4H)

¹³C NMR (100 MHz, CDCl₃): δ 141.06, 140.71, 136.65, 136.50, 128.12, 127.96, 127.42, 126.99, 78.90, 78.65, 25.33, 25.15, 21.10, 21.06.

HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₂ [[M+Na]⁺: 293.1518, Found: 293.1513.

2,3-Di-m-tolylbutane-2,3-diol (2c)

OH Me. HO Me

The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.54.

¹H NMR (400 MHz, CDCl₃): δ 7.15 – 7.09 (m, 2H), 7.09 – 7.01 (m, 3H), 7.01 – 6.96 (m, 3H), 2.57 (s, 1H), 2.45 – 2.31 (m, 1H), 2.29 (s, 4H), 2.27 (s, 3H), 1.54 (s, 3H), 1.45 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 143.83, 143.51, 143.47, 136.80, 136.78, 136.76, 136.59, 136.58, 136.56, 128.39, 128.36, 128.34, 127.99, 127.95, 127.82, 127.69, 127.25, 127.09, 124.66, 124.62, 124.60, 124.17, 124.14, 79.00, 78.97, 78.77, 78.73, 25.20, 25.10, 21.70, 21.68. HRMS (ESI, m/z): Calcd for C₁₈H₂₂O₂ [[M+Na]⁺: 293.1518, Found: 293.1512.

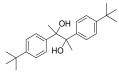
2,3-Bis(4-isopropylphenyl)butane-2,3-diol (2d):

ОН С

The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso: dl* =1:1.55. ¹H NMR (400 MHz, CDCl₃): δ 7.24 – 6.90 (m, 8H), 2.81 (dq, *J* = 13.4, 6.8 Hz, 2H), 2.50 (s, 1H), 2.11 (s, 1H), 1.44 (s, 2H), 1.38 (s, 4H), 1.16 (dd, *J* = 7.1, 5.3 Hz, 12H); ¹³C NMR (100 MHz, CDCl₃): δ 147.63, 147.53, 141.44, 141.10, 127.40, 126.99, 125.42, 125.31, 78.88, 78.60, 33.75, 33.73, 25.28, 25.21, 24.16, 24.13, 24.08.

HRMS (ESI, m/z) : Calcd for $C_{22}H_{30}O_2$ [M+Na]⁺: 349.2144, Found: 349.2134.

2,3-Bis(4-(tert-butyl)phenyl)butane-2,3-diol (2e)



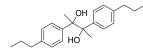
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.64.

¹H NMR (400 MHz, CDCl₃): δ 7.32 – 7.12 (m, 8H), 2.62 (s, 1H), 2.19 (s, 1H), 1.51 (s, 2H), 1.46 (s, 4H), 1.31 (d, *J* = 6.1 Hz, 18H).

¹³C NMR (100 MHz, CDCl₃): δ 149.90, 149.79, 141.07, 140.74, 127.11, 126.70, 124.28, 124.20, 78.79, 78.51, 34.50, 34.46, 31.52, 31.49, 25.24.

HRMS (ESI, m/z): Calcd for C₂₄H₃₄O₂ [M+Na]⁺:377.2457, Found: 377.2455.

2,3-Bis(4-propylphenyl)butane-2,3-diol (2f)



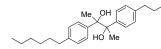
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.51.

¹H NMR (400 MHz, CDCl₃): δ 7.17 – 7.08 (m, 4H), 7.07 – 6.98 (m, 4H), 2.56 (q, *J* = 7.1 Hz, 4H), 1.63 (qd, *J* = 7.6, 6.2 Hz, 4H), 1.55 (s, 2H), 1.47 (s, 4H), 0.93 (td, *J* = 7.4, 5.7 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃): δ 141.49, 141.33, 141.25, 140.95, 128.80, 128.61, 128.50, 128.32, 127.50, 127.36, 127.34, 126.90, 78.96, 78.73, 38.16, 37.70, 37.64, 26.64, 25.21, 25.15, 24.59, 24.52, 24.34, 14.04, 13.96, 13.88.

HRMS (ESI, m/z) : Calcd for $C_{22}H_{30}O_2Na$ [M+Na]⁺: 349.2144, Found: 349.2134.

2-(4-Hexylphenyl)-3-(4-pentylphenyl)butane-2,3-diol (2g)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.48.

¹H NMR (400 MHz, CDCl₃): δ 7.10 (dd, *J* = 12.7, 8.1 Hz, 4H), 7.06 – 6.96 (m, 4H), 2.57 (q, *J* = 7.2 Hz, 5H), 2.29 (d, *J* = 6.6 Hz, 1H), 1.58 (dq, *J* = 12.9, 6.1, 5.4 Hz, 5H), 1.53 (s, 2H), 1.46 (s, 3H), 1.39 – 1.24 (m, 13H), 1.08 – 0.68 (m, 7H).

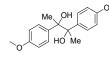
¹³C NMR (100 MHz, CDCl₃): δ 141.73, 141.69, 141.57, 141.54, 141.23, 140.94, 128.63, 127.42,

127.36, 127.28, 126.96, 126.92, 78.96, 78.73, 35.64, 35.59, 31.91, 31.89, 31.53, 31.50, 31.47, 29.15, 29.08, 25.19, 25.13, 25.11, 22.79, 14.25. HRMS (ESI, m/z) : Calcd for C₂₇H₄₀O₂Na [M+Na]⁺: 419.2926, Found:419.2916.

2,3-Bis(3,4-dimethylphenyl)butane-2,3-diol (2h)

The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso: dl* = 1:1.54. ¹H NMR (400 MHz, CDCl₃): δ 7.16 – 7.02 (m, 5H), 6.99 (dd, *J* = 7.9, 2.1 Hz, 1H), 2.67 (s, 1H), 2.29 (d, *J* = 5.0 Hz, 6H), 2.26 (d, *J* = 5.4 Hz, 6H), 1.56 (s, 2H), 1.49 (s, 4H). ¹³C NMR (100 MHz, CDCl₃): δ 141.47, 141.12, 135.37, 135.23, 135.15, 135.12, 128.81, 128.72, 128.52, 128.42, 124.99, 124.50, 78.83, 78.57, 25.37, 25.26, 20.01, 19.98, 19.43, 19.39. HRMS (ESI, m/z) : Calcd for C₂₀H₂₆O₂Na [M+Na]⁺: 321.1831, Found: 321.1822.

2,3-Bis(4-methoxyphenyl)butane-2,3-diol (2i)



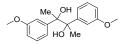
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.53.

¹H NMR (400 MHz, CDCl₃): δ 7.20 – 7.08 (m, 4H), 6.86 – 6.71 (m, 4H), 3.83 (s, 4H), 3.81 (s, 2H), 2.61 (s, 1H), 2.33 (s, 1H), 1.53 (d, *J* = 36.0 Hz, 6H).

¹³C NMR (100 MHz, CDCl₃): δ 158.66, 158.56, 136.22, 135.89, 128.68, 128.25, 112.68, 112.53, 78.83, 78.61, 77.48, 77.36, 77.16, 76.84, 55.32, 25.29, 25.13.

HRMS (ESI, m/z) : Calcd for $C_{18}H_{22}O_4Na$ [M+Na]⁺: 325.1416, Found:325.1410.

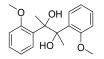
2,3-Bis(3-methoxyphenyl)butane-2,3-diol (2j)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.12.

¹H NMR (400 MHz, CDCl₃): δ 7.13 (t, *J* = 8.1 Hz, 2H), 6.85 (dt, *J* = 7.9, 1.3 Hz, 1H), 6.77 (d, *J* = 7.6 Hz, 5H), 3.69 (s, 4H), 3.65 (s, 3H), 2.88 (d, *J* = 1.1 Hz, 2H), 1.54 (s, 3H), 1.47 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 158.83, 158.64, 145.68, 145.29, 128.22, 128.06, 120.09, 119.51, 113.51, 113.13, 112.70, 112.58, 78.91, 78.90, 78.66, 55.19, 55.18, 25.13, 24.99. HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₄Na [M+Na]⁺: 325.1416, Found:325.1410.

2,3-Bis(2-methoxyphenyl)butane-2,3-diol (2k)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (95% yield). dl

¹H NMR (400 MHz, CDCl₃): δ 7.08 (ddd, J = 8.7, 7.3, 1.7 Hz, 2H), 6.98 – 6.86 (m, 2H), 6.74 (td, J = 7.6, 1.2 Hz, 2H), 6.65 (dd, J = 8.3, 1.2 Hz, 2H), 5.41 (s, 2H), 3.40 (s, 6H), 1.57 (s, 6H). ¹³C NMR (100 MHz, CDCl₃): δ 158.03, 132.23, 129.93, 128.39, 120.35, 111.35, 82.54,55.49, 24.63. HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₄Na [M+Na]⁺: 325.1416, Found:325.1413.

Dimethyl 4,4'-(2,3-dihydroxybutane-2,3-diyl)dibenzoate (2l)

The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (75% yield). *meso:* dl = 1:6.58

¹H NMR (400 MHz, CDCl₃): δ 7.94 – 7.81 (m, 4H), 7.28 – 7.15 (m, 4H), 3.92 (s, 5H), 3.91 (s, 1H), 2.66 (s, 2H), 1.60 (s, 1H), 1.53 (s, 6H).

¹³C NMR (100 MHz, CDCl₃): δ 167.12, 148.50, 129.17, 128.70, 128.60, 127.57, 127.22, 78.98, 52.25, 24.89.

HRMS (ESI, m/z) : Calcd for C₂₀H₂₂O₆Na [M+Na]⁺: 381.1314, Found: 381.1311.

2,3-Di([1,1'-biphenyl]-4-yl)butane-2,3-diol (2m)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1.05:1.

¹H NMR (400 MHz, DMSO-*d*₆) δ 7.76 – 7.53 (m, 8H), 7.53 – 7.38 (m, 6H), 7.38 – 7.20 (m, 4H), 5.10 (s, 1H), 5.00 (s, 1H), 1.57 (s, 3H), 1.38 (s, 3H).

¹³C NMR (100 MHz, DMSO-*d*₆): δ146.57, 140.14, 138.44, 128.84, 127.15, 126.51, 126.29, 125.87, 67.78, 25.82.

HRMS (ESI, m/z) : Calcd for C₂₈H₂₆O₂ Na [M+Na]⁺: 417.1831, Found:417.1830.

2,3-Bis(3,4-dimethoxyphenyl)butane-2,3-diol (2n)

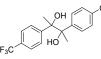
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (85% yield). *meso:* dl = 1:2.14.

¹H NMR (400 MHz, CDCl₃): δ 6.84 – 6.61 (m, 6H), 3.84 (dd, *J* = 4.6, 1.2 Hz, 6H), 3.71 (dd, *J* = 16.4, 1.3 Hz, 6H), 2.49 (s, 2H), 1.58 (d, *J* = 1.2 Hz, 2H), 1.48 (d, *J* = 1.3 Hz, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 148.00, 147.49, 136.26, 119.95, 111.38, 109.72, 78.89, 55.89, 55.78, 55.75, 24.99.

HRMS (ESI, m/z) : Calcd for C₂₀H₂₆O₆Na [M+Na]⁺: 385.1627, Found: 385.1620.

2,3-Bis(4-(trifluoromethyl)phenyl)butane-2,3-diol (20)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. colorless oil (99% yield). *meso:* dl = 1:1.47.

¹H NMR (400 MHz, CDCl₃): δ 7.49 (dd, *J* = 8.6, 2.5 Hz, 4H), 7.40 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.1 Hz, 2H), 2.77 (s, 1H), 2.40 (s, 1H), 1.55 (s, 3H), 1.50 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 147.85, 147.29, 147.27, 130.17, 129.85, 129.65, 129.53, 129.33, 129.20, 127.89, 127.61, 125.67 (d, *J*= 4 Hz), 124.33 (m, *J* = 80 Hz), 122.96 (d, *J*= 4 Hz), 122.94, 78.75, 78.43, 25.24, 24.86.

HRMS (ESI, m/z) : Calcd for $C_{18}H_{16}F_6O_2Na [M+Na]^+$: 401.0952, Found: 401.0922.

2,3-Bis(4-phenoxyphenyl)butane-2,3-diol (2p)

PhO

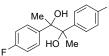
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. colorless oil (99% yield). *meso:* dl = 1:1.4.

¹H NMR (400 MHz, CDCl₃): δ 7.34 – 7.27 (m, 4H), 7.20 – 7.12 (m, 4H), 7.11 – 7.04 (m, 2H), 7.02 – 6.94 (m, 4H), 6.91 – 6.81 (m, 4H), 2.62 (s, 1H), 2.44 (s, 1H), 1.59 (s, 2H), 1.50 (s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 157.38, 157.21, 156.37, 156.15, 138.88, 138.46, 129.85, 129.82, 128.92, 128.53, 123.39, 123.27, 118.99, 118.96, 118.93, 118.79, 117.72, 117.44, 117.39, 78.81, 78.60, 25.16, 25.06.

HRMS (ESI, m/z) : Calcd for C₂₈H₂₆O₄Na [M+Na]⁺: 449.1729, Found: 449.1720.

2,3-Bis(4-fluorophenyl)butane-2,3-diol (2q)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. white solid (75% yield). *meso:* dl = 1:1.94.

¹H NMR (400 MHz, CDCl₃): δ 7.16 (dd, *J* = 8.7, 5.5 Hz, 2H), 7.11 (dd, *J* = 8.4, 5.5 Hz, 2H), 6.90 (dt, *J* = 11.9, 8.7 Hz, 4H), 2.57 (s, 2H), 1.56 (s, 2H), 1.47 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 162.11 (dd, J= 245 Hz), 139.62 (d, J= 3 Hz), 139.24 (d, J=3 Hz), 129.21 (d, J= 8 Hz), 128.82 (d, J= 8 Hz), 114.07 (dd, J= 28 Hz), 78.73, 78.47, 25.29, 25.02. HRMS (ESI, m/z) : Calcd for C₁₆H₁₆F₂O₂Na [M+Na]+: 301.1016, Found: 301.1011.

2,3-Bis(2-fluorophenyl)butane-2,3-diol (2r)



The title compound was synthesized according to the general procedure. The product was purified

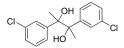
by flash column chromatography. White solid (65% yield). *meso:* dl = 1:1.33.

¹H NMR (400 MHz, CDCl₃): δ 7.45 (td, J = 8.1, 1.9 Hz, 1H), 7.30 – 7.05 (m, 4H), 7.00 – 6.91 (m, 2H), 6.86 (ddd, J = 13.1, 8.1, 1.3 Hz, 1H), 3.27 – 3.11 (m, 1H), 3.08 (s, 1H), 1.76 (d, J = 2.2 Hz, 3H), 1.64 (t, J = 2.2 Hz, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 160.72 (dd, *J*= 251 Hz), 130.36, 130.32, 130.21, 130.11, 130.07, 129.45 (dd, *J*= 9 Hz), 123.74 (d, *J*= 3 Hz), 123.37 (d, *J*= 3 Hz), 116.46, 116.21, 115.96, 79.86 (d, *J*= 4 Hz), 79.74 (d, *J*= 4 Hz) 24.60, 24.58, 24.54, 24.52, 24.17, 24.15, 24.10, 24.08.

HRMS (ESI, m/z) : Calcd for $C_{16}H_{16}F_2O_2Na [M+Na]^+$: 301.1016, Found: 301.1011.

2,3-Bis(3-chlorophenyl)butane-2,3-diol (2s)



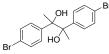
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. colorless oil (78% yield). *meso:* dl = 1:1.26.

¹H NMR (400 MHz, CDCl₃): δ 7.32 (t, *J* = 1.8 Hz, 1H), 7.26 (dddt, *J* = 7.6, 5.9, 3.8, 1.6 Hz, 3H), 7.23 – 7.10 (m, 3H), 7.01 (dt, *J* = 7.9, 1.5 Hz, 1H), 2.34 (s, 2H), 1.56 (s, 3H), 1.50 (s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 145.99, 145.43, 133.64, 133.55, 128.65, 128.52, 127.81, 127.56, 127.51, 127.31, 125.68, 125.33, 78.66, 78.34, 25.23, 24.89.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₆Cl₂O₂Na [M+Na]⁺: 333.0425, Found: 333.0422.

2,3-bis(4-bromophenyl)butane-2,3-diol (2t)



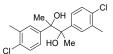
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. colorless oil (75% yield). *meso:* dl = 1:1.15.

¹H NMR (400 MHz, CDCl₃): δ 7.28 (dt, *J* = 9.1, 2.3 Hz, 5H), 7.17 (ddd, *J* = 6.0, 4.3, 2.1 Hz, 5H), 7.14 – 7.08 (m, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 6.97 – 6.92 (m, 2H), 2.21 (s, 4H), 1.59 – 1.20 (m, 12H).

¹³C NMR (100 MHz, CDCl₃): δ 143.92, 143.56, 142.91, 142.44, 130.51, 130.43, 130.35, 130.29, 129.40, 129.34, 129.01, 127.57, 127.50, 127.45, 127.41, 127.39, 127.29, 127.24, 127.20, 127.06, 127.04, 121.59, 121.39, 79.01, 78.75, 78.63, 78.36, 25.25, 25.20, 25.09, 24.85.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₆Br₂O₂Na [M+Na]⁺: 422.9395, Found: 422.9350.

2,3-Bis(4-chloro-3-methylphenyl)butane-2,3-diol (2u)

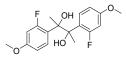


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (65% yield). *meso:* dl = 1:1.53.

¹H NMR (400 MHz, CDCl₃): δ 7.27 – 7.11 (m, 2H), 7.11 – 6.96 (m, 3H), 6.92 (dd, J = 8.4, 2.4 Hz, 1H), 2.59 (s, 1H), 2.32 (s, 3H), 2.30 (d, J = 6.2 Hz, 3H), 1.56 – 1.50 (m, 2H), 1.48 – 1.42 (m, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 142.39, 141.92, 134.80, 134.68, 133.39, 133.23, 130.21, 129.87,

127.96, 127.82, 126.43, 126.01, 25.26, 25.00, 20.28, 20.25. HRMS (ESI, m/z) : Calcd for C₁₈H₂₀Cl₂O₂Na [M+Na]⁺: 361.0738, Found: 361.0730.

2,3-Bis(2-fluoro-4-methoxyphenyl)butane-2,3-diol (2v)



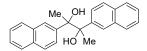
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.23.

¹H NMR (400 MHz, CDCl₃): δ 7.25 (t, *J* = 9.2 Hz, 1H), 6.93 (t, *J* = 9.2 Hz, 1H), 6.57 (dd, *J* = 8.9, 2.6 Hz, 1H), 6.43 (dt, *J* = 12.0, 2.7 Hz, 2H), 6.34 (dd, *J* = 14.5, 2.6 Hz, 1H), 3.71 (d, *J* = 0.9 Hz, 3H), 3.67 (d, *J* = 0.9 Hz, 3H), 2.95 (s, 2H), 1.63 (d, *J* = 2.3 Hz, 3H), 1.55 - 1.47 (m, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 160.46 (m, *J*= 244 Hz), 160.25 (t, *J*= 12 Hz), 130.79 (d, *J*= 6 Hz), 130.59 (d, *J*= 6 Hz), 122.35 (d, *J*= 4 Hz), 122.25 (d, *J*= 3 Hz), 109.43 (d, *J*= 3 Hz), 109.10 (d, *J*= 3 Hz), 109.09, 102.16, 101.87, 101.58, 79.57 (d, *J*= 4 Hz), 79.46 (d, *J*= 5 Hz), 55.61, 55.57, 24.62, 24.59, 24.55, 24.53, 24.19, 24.13.

HRMS (ESI, m/z) : Calcd for $C_{18}H_{20}F_2O_4Na \ [M+Na]^+$: 361.1228, Found: 361.1220.

2,3-Di(naphthalen-2-yl)butane-2,3-diol (2w)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (70% yield). *meso:* dl = 1:1.76;

¹H NMR (400 MHz, CDCl₃): δ 7.85 (d, *J* = 1.9 Hz, 1H), 7.83 – 7.73 (m, 3H), 7.72 – 7.69 (m, 2H), 7.66 (dd, *J* = 8.7, 4.3 Hz, 2H), 7.50 – 7.36 (m, 5H), 7.27 (dd, *J* = 8.7, 1.9 Hz, 1H), 2.91 – 2.64 (m, 1H), 2.37 (s, 1H), 1.68 (s, 2H), 1.62 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): 141.66, 141.14, 132.81, 132.68, 132.60, 132.51, 128.46, 128.44, 127.50, 126.85, 126.60, 126.11, 126.03, 126.02, 125.94, 125.65, 79.39, 79.03, 25.72, 25.42.
HRMS (ESI, m/z) : Calcd for C₂₄H₂₂O₂Na [M+Na]⁺: 365.1518, Found: 365.1515.

4,5-Diphenyloctane-4,5-diol (2x)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.5.

¹H NMR (400 MHz, CDCl₃): δ 7.22 (h, *J* = 10.1, 6.9 Hz, 10H), 2.60 (s, 1H), 2.24 (dq, *J* = 7.7, 4.0 Hz, 1H), 2.01 (ddd, *J* = 14.9, 9.4, 3.7 Hz, 1H), 1.63 (dtt, *J* = 21.2, 13.3, 3.6 Hz, 2H), 1.25 – 1.03 (m, 2H), 0.94 – 0.59 (m, 8H) °

¹³C NMR (100 MHz, CDCl₃): δ 141.89, 141.00, 128.31, 127.63, 127.44, 127.23, 126.95, 126.76, 81.83, 37.95, 37.49, 17.01, 16.84, 14.66, 14.64.

HRMS (ESI, m/z) : Calcd for C₂₀H₂₆O₂Na [M+Na]⁺: 321.1831, Found:321.1830.

3,3',4,4'-Tetrahydro-[1,1'-binaphthalene]-1,1'(2H,2'H)-diol (2y)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (65% yield). *dl*.

¹H NMR (400 MHz, CDCl₃): δ 8.17 (dd, J = 7.6, 1.8 Hz, 2H), 7.26 – 7.15 (m, 4H), 7.08 (d, J = 7.4 Hz, 2H), 3.21 (s, 2H), 2.72 (q, J = 3.0 Hz, 1H), 2.68 (q, J = 3.1 Hz, 1H), 2.53 (ddd, J = 16.3, 11.3, 5.5 Hz, 2H), 1.64 (tdd, J = 14.3, 6.4, 3.6 Hz, 5H), 1.57 (q, J = 3.8, 3.1 Hz, 1H), 1.26 (ddd, J = 14.5, 12.1, 5.0 Hz, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 140.68, 138.52, 129.21, 129.19, 129.00, 127.35, 126.53, 36.59, 31.29, 20.22.

HRMS (ESI, m/z) : Calcd for C₂₀H₂₂O₂Na [M+Na]⁺: 317.1518, Found:317.1510.

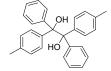
1,1,2,2-Tetraphenylethane-1,2-diol (2z):



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield).

¹H NMR (400 MHz, CDCl₃): δ 7.46 – 7.31 (m, 10H), 7.23 (qd, *J* = 4.2, 1.7 Hz, 10H), 3.11 (s, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 144.32, 128.75, 127.41, 127.07, 83.17. HRMS (ESI, m/z) : Calcd for C₂₆H₂₂O₂Na [M+Na]⁺: 389.1518, Found:389.1511.

1,2-Diphenyl-1,2-di-p-tolylethane-1,2-diol (2a')



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (85% yield). *meso:* dl = 1.11:1.

¹H NMR (400 MHz, CDCl₃): δ 7.36 – 7.29 (m, 4H), 7.22 – 7.11 (m, 10H), 6.98 (t, *J* = 7.8 Hz, 4H), 3.02 (s, 2H), 2.30 (s, 3H), 2.28 (s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 144.64, 141.41, 136.72, 136.62, 130.46, 128.78, 128.73, 128.67, 128.63, 128.62, 128.36, 128.19, 128.16, 127.36, 127.34, 126.94, 126.87, 83.09, 21.11.
HRMS (ESI, m/z) : Calcd for C₂₈H₂₆O₂Na [M+Na]⁺: 417.1831, Found: 417.1830.

1,2-Bis(4-methoxyphenyl)-1,2-diphenylethane-1,2-diol (2b')

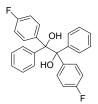
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.01.

¹H NMR (400 MHz, CDCl₃): δ 7.30 (ddt, J = 5.8, 2.5, 1.4 Hz, 4H), 7.19 – 7.11 (m, 10H), 6.68 (dd, J = 9.0, 7.5 Hz, 4H), 3.73 (s, 3H), 3.72 (s, 3H), 2.97 (s, 2H).

¹³C NMR (100 MHz, CDCl₃): δ δ 158.41, 158.35, 144.68, 136.52, 130.02, 129.96, 128.72, 128.65, 128.49, 128.00, 127.47, 127.31, 126.89, 126.83, 126.50, 113.95, 112.64, 82.99, 75.84, 55.33, 55.19, 55.17.

HRMS (ESI, m/z) : Calcd for C₂₈H₂₆O₄Na [M+Na]⁺: 449.1729, Found: 449.1722.

1,2-Bis(4-fluorophenyl)-1,2-diphenylethane-1,2-diol (2c')



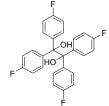
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1.07:1.

¹H NMR (400 MHz, CDCl₃): δ 7.43 – 7.32 (m, 2H), 7.32 – 7.09 (m, 12H), 6.84 (td, *J* = 8.8, 2.1 Hz, 4H), 2.96 (s, 1H), 2.94 (s, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 163.19, 160.73, 139.81 (d, *J*= 3 Hz), 130.43 (d, *J*= 8 Hz), 130.39, 128.37, 128.29, 115.67, 115.46, 114.56, 114.35, 82.73.

HRMS (ESI, m/z) : Calcd for $C_{26}H_{20}F_2O_2Na$ [M+Na]⁺: 425.1329, Found: 425.1321.

1,1,2,2-Tetrakis(4-fluorophenyl)ethane-1,2-diol (2d')



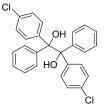
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield).

¹H NMR (400 MHz, CDCl₃): δ 7.32 – 7.24 (m, 8H), 6.95 – 6.84 (m, 8H), 2.90 (s, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 144.04, 143.82, 130.60 (d, *J*= 9 Hz), 128.53 (d, *J*= 6 Hz), 127.65, 127.51, 127.39, 114.32, 114.11, 82.94.

HRMS (ESI, m/z) : Calcd for $C_{26}H_{18}F_4O_2Na$ [M+Na]⁺: 461.1141, Found: 461.1131.

1,2-Bis(4-chlorophenyl)-1,2-diphenylethane-1,2-diol (2e')



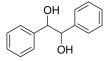
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (71% yield). *meso:* dl = 1.12:1.

¹H NMR (400 MHz, CDCl₃): δ 7.45 – 7.37 (m, 2H), 7.33 – 7.24 (m, 5H), 7.24 – 7.11 (m, 11H), 2.99 (s, 1H), 2.96 (s, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 143.68, 143.38, 142.88, 142.85, 133.18, 132.99, 130.32, 130.29, 130.25, 128.80, 128.74, 128.64, 128.53, 128.50, 128.43, 127.76, 127.70, 127.66, 127.62, 127.56, 127.52, 82.92, 82.88.

HRMS (ESI, m/z) : Calcd for C₂₆H₂₀Cl₂O₂Na [M+Na]⁺: 457.0738, Found: 457.0735.

1,2-Diphenylethane-1,2-diol (4a)



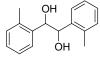
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.17.

¹H NMR (400 MHz, CDCl₃): δ 7.18 (hept, J = 3.3 Hz, 3H), 7.14 – 7.03 (m, 5H), 6.98 (qd, J = 3.6, 1.9 Hz, 2H), 4.68 (s, 1H), 4.53 (d, J = 1.6 Hz, 1H), 3.40 – 2.99 (m, 1H), 2.76 – 2.27 (m, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 140.00, 139.83, 128.51, 128.23, 128.17, 128.08, 128.07, 127.95, 127.21, 127.08, 79.17, 78.09.

HRMS (ESI, m/z) : Calcd for $C_{14}H_{14}O_2Na$ [M+Na]⁺: 237.0892, Found:237.0890.

1,2-Bi-o-tolylethane-1,2-diol (4b)



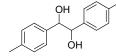
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:2.14.

¹H NMR (400 MHz, CDCl₃): δ 7.49 (dd, *J* = 7.8, 1.5 Hz, 2H), 7.24 – 7.18 (m, 1H), 7.13 – 7.05 (m, 4H), 7.02 (td, *J* = 7.5, 1.5 Hz, 2H), 6.98 (dd, *J* = 5.2, 3.7 Hz, 1H), 6.85 – 6.80 (m, 2H), 5.07 (s, 1H), 4.84 (s, 2H), 3.01 (s, 2H), 2.05 (s, 3H), 1.56 (s, 6H).

¹³C NMR (100 MHz, CDCl₃): δ 138.15, 138.10, 136.14, 135.96, 130.20, 130.06, 127.74, 127.72, 127.32, 126.86, 126.06, 125.96, 74.66, 73.27, 19.17, 18.79.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₈O₂Na [M+Na]⁺:265.1204, Found:265.1201.

1,2-Di-p-tolylethane-1,2-diol (4c)



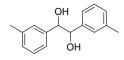
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.2.

¹H NMR (400 MHz, CDCl₃): δ 7.13 – 7.00 (m, 4H), 6.97 – 6.91 (m, 4H), 4.64 (s, 1H), 4.55 (s, 1H), 2.56 – 2.28 (m, 1H), 2.26 (s, 3H), 2.21 (s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 137.90, 137.58, 137.16, 137.14, 129.10, 128.94, 127.18, 126.99, 78.90, 78.16, 21.29, 21.26.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₈O₂Na [M+Na]⁺: 265.1204, Found:265.1201.

1,2-Di-*m*-tolylethane-1,2-diol (4d):



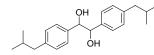
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.15.

¹H NMR (400 MHz, CDCl₃): δ 7.11 (t, *J* = 7.5 Hz, 1H), 7.06 – 6.91 (m, 5H), 6.86 (d, *J* = 2.0 Hz, 1H), 6.78 (dt, *J* = 7.6, 1.6 Hz, 1H), 4.59 (s, 1H), 4.51 (s, 1H), 2.82 (s, 2H), 2.23 (s, 3H), 2.18 (s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 140.14, 140.10, 137.84, 129.04, 128.72, 128.69, 128.31, 128.12, 128.09, 127.96, 127.65, 124.41, 124.16, 78.94, 78.35, 78.32, 21.56, 21.51.

HRMS (ESI, m/z) : Calcd for $C_{16}H_{18}O_2Na \ [M+Na]^+$: 265.1205, Found: 265.1201.

1,2-Bis(4-isobutylphenyl)ethane-1,2-diol (4e):



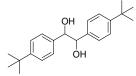
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.24.

¹H NMR (400 MHz, CDCl₃): δ 7.05 (d, J = 8.2 Hz, 2H), 6.99 (d, J = 8.1 Hz, 2H), 6.90 (s, 4H), 4.66 (s, 1H), 4.54 (s, 1H), 2.91 (s, 1H), 2.37 (d, J = 7.2 Hz, 2H), 2.33 (dd, J = 7.2, 1.5 Hz, 2H), 2.29 – 2.09 (m, 1H), 1.74 (dhept, J = 16.5, 6.8 Hz, 2H), 0.84 – 0.75 (m, 12H).

¹³C NMR (100 MHz, CDCl₃): δ 141.70, 141.39, 137.41, 137.29, 129.10, 128.90, 126.98, 126.78, 79.20, 78.14, 45.25, 45.19, 30.33, 30.31, 22.45, 22.42, 22.34.

HRMS (ESI, m/z) : Calcd for C₂₂H₃₀O₂Na [M+Na]⁺: 349.2144, Found: 349.2141.-

1,2-Bis(4-(tert-butyl)phenyl)ethane-1,2-diol (4f)



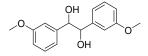
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.24.

¹H NMR (400 MHz, CDCl₃): δ 7.32 – 7.26 (m, 2H), 7.19 (dq, *J* = 8.7, 2.0 Hz, 4H), 7.03 (dd, *J* = 8.3, 1.8 Hz, 2H), 4.61 (d, *J* = 2.1 Hz, 2H), 2.59 (s, 2H), 1.24 (s, 9H), 1.21 (s, 9H).

¹³C NMR (100 MHz, CDCl₃): δ 151.32, 150.86, 137.44, 130.18, 127.04, 126.60, 125.55, 125.47, 125.19, 78.35, 78.19, 34.70, 34.62, 31.48, 31.46.

HRMS (ESI, m/z) : Calcd for C₂₂H₃₀O₂Na [M+Na]⁺: 349.2144, Found: 349.2141.

1,2-Bis(3-methoxyphenyl)ethane-1,2-diol (4g)



The title compound was synthesized according to the general procedure. The product was purified

by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.15.

¹H NMR (400 MHz, CDCl₃): δ 7.10 (td, J = 7.9, 1.6 Hz, 1H), 7.03 (td, J = 7.9, 1.4 Hz, 1H), 6.74 – 6.68 (m, 2H), 6.68 – 6.62 (m, 2H), 6.61 – 6.53 (m, 2H), 4.65 (d, J = 1.3 Hz, 1H), 4.52 – 4.45 (m, 1H), 3.59 (dt, J = 9.0, 1.2 Hz, 6H), 2.83 – 2.71 (m, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 159.53, 159.42, 141.70, 141.52, 129.23, 129.17, 119.57, 119.40, 113.96, 113.94, 113.70, 112.46, 112.39, 78.91, 77.96, 55.30, 55.26, 55.25.

HRMS (ESI, m/z) : Calcd for $C_{16}H_{18}O_4Na [M+Na]^+$: 297.1103, Found: 297.1101.

1,2-bis(2-methoxyphenyl)ethane-1,2-diol (4h)

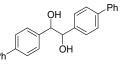
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.32.

¹H NMR (400 MHz, CDCl₃): δ 7.26 – 7.14 (m, 4H), 6.89 (dtd, *J* = 11.6, 7.5, 1.1 Hz, 2H), 6.79 (ddd, *J* = 19.2, 8.6, 1.2 Hz, 2H), 5.29 (s, 1H), 5.07 (s, 1H), 3.68 (s, 3H), 3.66 (s, 3H), 3.55 (s, 1H), 3.20 (s, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 156.97, 156.92, 128.66, 128.56, 128.50, 128.44, 128.27, 128.24, 120.46, 110.25, 110.22, 74.35, 73.42, 73.41, 55.27, 55.23.

HRMS (ESI, m/z) : Calcd for $C_{16}H_{18}O_4Na \ [M+Na]^+$: 297.1103, Found: 297.1101.

1,2-Di([1,1'-biphenyl]-4-yl)ethane-1,2-diol (4i)



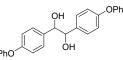
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (85% yield). *meso:* dl = 1:11.

¹H NMR (400 MHz, DMSO- d_6): δ 7.68 – 7.60 (m, 8H), 7.58 – 7.49 (m, 8H), 7.44 (t, J = 7.7 Hz, 8H), 7.37 – 7.31 (m, 4H), 7.31 – 7.17 (m, 8H), 5.41 (s, 4H), 4.71 (d, J = 2.1 Hz, 4H).

¹³C NMR (100 MHz, DMSO-*d*₆): δ 141.81, 139.99, 138.43, 128.89, 127.83, 127.23, 126.48, 125.59, 77.08.

HRMS (ESI, m/z) : Calcd for C₂₆H₂₂O₂Na [M+Na]⁺: 389.1517, Found: 389.1520.

1,2-Bis(4-phenoxyphenyl)ethane-1,2-diol (4j)

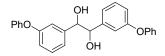


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.32.

¹H NMR (400 MHz, CDCl₃): δ 7.23 (ddd, *J* = 8.5, 7.4, 6.0 Hz, 4H), 7.12 (d, *J* = 8.6 Hz, 2H), 7.03 – 6.97 (m, 4H), 6.94 – 6.83 (m, 6H), 6.81 – 6.77 (m, 2H), 4.71 (s, 1H), 4.56 (s, 1H), 2.96 (s, 1H), 2.34 (s, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 157.23, 157.19, 157.02, 134.84, 134.65, 129.90, 129.87, 128.67, 128.55, 123.50, 123.43, 119.03, 118.91, 118.66, 118.64, 78.93, 27.05.

1,2-Bis(3-phenoxyphenyl)ethane-1,2-diol (4k)



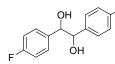
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.21.

¹H NMR (400 MHz, CDCl₃): δ 7.23 – 7.10 (m, 6H), 6.98 (td, *J* = 7.3, 1.3 Hz, 2H), 6.89 – 6.68 (m, 9H), 6.63 (t, *J* = 1.9 Hz, 1H), 4.73 (s, 1H), 4.49 (d, *J* = 1.8 Hz, 1H), 2.97 (s, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 157.19, 157.16, 157.12, 157.04, 141.86, 141.58, 130.07, 129.86, 129.74, 129.57, 123.34, 123.33, 121.94, 119.30, 118.87, 118.77, 118.60, 117.80, 117.60, 79.10, 77.51.

HRMS (ESI, m/z) : Calcd for C₂₆H₂₂O₄Na [M+Na]⁺: 421.1416, Found: 421.1414.

1,2-Bis(4-fluorophenyl)ethane-1,2-diol (4l)



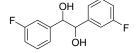
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (85% yield). *meso:* dl = 1.28:1.

¹H NMR (400 MHz, CDCl₃): δ 7.29 – 7.21 (m, 2H), 7.14 – 7.03 (m, 4H), 7.03 – 6.95 (m, 2H), 5.46 (dd, J = 2.7, 1.4 Hz, 1H), 5.32 (dd, J = 3.1, 1.5 Hz, 1H), 4.65 – 4.60 (m, 1H), 4.58 (dd, J = 3.2, 1.3 Hz, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 165.95 (m, *J*= 240 Hz), 144.35 (d, *J*= 3 Hz), 143.47 (d, *J*= 3 Hz), 134.22 (dd, *J*= 13 Hz), 119.26 (d, *J*= 2 Hz), 119.05 (d, *J*= 2 Hz), 81.87, 81.42.

HRMS (ESI, m/z) : Calcd for C₁₄H₁₂F₂O₂Na [M+Na]⁺: 273.0703, Found: 273.0701.

1,2-Bis(3-fluorophenyl)ethane-1,2-diol (4m)

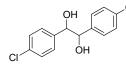


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.22.

¹H NMR (400 MHz, CDCl₃): δ 7.23 (dtd, *J* = 21.8, 7.4, 5.5 Hz, 2H), 7.04 – 6.86 (m, 5H), 6.85 – 6.79 (m, 1H), 4.85 (q, *J* = 2.1 Hz, 1H), 4.66 (q, *J* = 2.7 Hz, 1H), 2.68 (s, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 164.04, 161.60, 142.31 (d, *J*= 7 Hz), 142.16 (d, *J*= 7 Hz), 129.81 (t, *J*= 8 Hz), 122.75 (d, *J*= 3 Hz), 115.27, 115.24, 115.06, 115.03, 114.01 (d, *J*= 10 Hz), 78.53. HRMS (ESI, m/z) : Calcd for C₁₄H₁₂F₂O₂Na [M+Na]⁺: 273.0703, Found: 273.0701.

1,2-Bis(4-chlorophenyl)ethane-1,2-diol (4n)



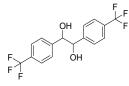
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (80% yield). *meso:* dl = 1.16:1.

¹H NMR (400 MHz, DMSO- d_6): δ 7.38 – 7.30 (m, 2H), 7.30 – 7.24 (m, 4H), 7.17 – 7.12 (m, 2H), 5.58 – 5.52 (m, 1H), 5.47 – 5.40 (m, 1H), 4.68 (d, J = 2.6 Hz, 1H), 4.61 (d, J = 3.5 Hz, 1H).

¹³C NMR (100 MHz, DMSO-*d*₆): δ 141.90, 141.06, 131.25, 129.14, 128.94, 127.28, 127.23, 76.38, 76.15.

HRMS (ESI, m/z) : Calcd for C₁₄H₁₂Cl₂O₂Na [M+Na]⁺: 305.0112, Found:305.0109.

1,2-Bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol (40)



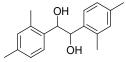
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.

¹H NMR (400 MHz, CDCl₃): δ 7.43 (t, *J* = 9.0 Hz, 4H), 7.30 – 7.04 (m, 4H), 4.85 (s, 1H), 4.63 (s, 1H), 3.05 (s, 1H), 2.51 (s, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 143.46, 143.30, 130.47 (m, *J*= 53 Hz), 127.45 (d, *J*= 6 Hz), 125.50, 125.44, 125.42, 125.38, 125.34, 125.30, 125.27, 125.23, 125.20, 125.16, 122.80, 122.73, 78.52, 77.48, 77.32, 77.16, 76.84.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₂F₆O₂Na [M+Na]⁺: 373.0639, Found: 373.0639.

1,2-Bis(2,4-dimethylphenyl)ethane-1,2-diol (4p)



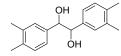
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:2.06.

¹H NMR (400 MHz, CDCl₃): δ 7.49 (d, *J* = 7.9 Hz, 4H), 7.31 – 7.23 (m, 2H), 7.09 – 6.98 (m, 6H), 6.95 (d, *J* = 1.9 Hz, 2H), 6.83 – 6.73 (m, 4H), 5.13 (s, 2H), 4.93 (s, 4H), 2.33 (s, 6H), 2.28 (s, 12H), 2.20 (s, 6H), 1.71 (s, 12H).

¹³C NMR (100 MHz, CDCl₃): δ 137.45, 137.31, 136.22, 135.79, 135.56, 135.30, 131.04, 127.20, 126.99, 126.76, 126.69, 74.55, 73.68, 21.16, 21.11, 19.29, 18.92.

HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₂Na [M+Na]⁺: 293.1517, Found:293.1522.

1,2-Bis(3,4-dimethylphenyl)ethane-1,2-diol (4q)

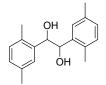


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1.07:1.

¹H NMR (400 MHz, CDCl₃): δ 7.03 – 6.97 (m, 2H), 6.95 (d, *J* = 7.8 Hz, 1H), 6.89 – 6.83 (m, 2H), 6.74 (dd, *J* = 7.7, 2.0 Hz, 1H), 4.52 (s, 1H), 4.51 (d, *J* = 1.9 Hz, 1H), 2.54 (s, 2H), 2.16 (s, 6H), 2.11 (s, 4H), 2.10 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 137.90, 137.80, 136.75, 136.69, 136.42, 136.13, 129.77, 129.48, 128.49, 128.09, 124.80, 124.47, 78.51, 78.31, 19.91, 19.86, 19.61, 19.56.

1,2-Bis(2,5-dimethylphenyl)ethane-1,2-diol (4r):



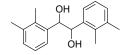
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso:* dl = 1:1.

¹H NMR (400 MHz, CDCl₃): 7.35 (d, *J* = 1.9 Hz, 1H), 7.10 (s, 1H), 6.92 (d, *J* = 1.2 Hz, 2H), 6.85 (dd, *J* = 7.7, 1.9 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 5.03 (s, 1H), 4.86 (s, 1H), 2.24 (s, 3H), 2.22 (s, 3H), 2.12 (s, 3H), 1.60 (s, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 138.26, 137.91, 135.73, 135.39, 133.38, 132.72, 130.25, 130.20, 128.68, 128.54, 127.73, 127.31, 74.49, 73.91, 21.23, 21.18, 18.97, 18.45.

HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₂Na [M+Na]⁺: 293.1517, Found:293.1522.

1,2-Bis(2,3-dimethylphenyl)ethane-1,2-diol (4s):

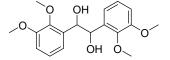


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso: dl* = 1:1.63.

¹H NMR (400 MHz, CDCl₃): δ 7.37 (dd, J = 7.8, 1.5 Hz, 1H), 7.14 (dd, J = 9.9, 5.5 Hz, 1H), 7.07 – 6.97 (m, 3H), 6.94 (dd, J = 7.6, 1.5 Hz, 1H), 5.14 (s, 1H), 4.91 (d, J = 1.2 Hz, 1H), 3.00 (s, 2H), 2.17 (s, 2H), 2.01 (d, J = 6.3 Hz, 6H), 1.45 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 138.36, 138.05, 136.69, 136.64, 135.01, 134.66, 129.50, 129.28, 128.67, 125.66, 125.50, 125.29, 125.02, 124.54, 74.91, 74.06, 27.04, 20.87, 20.74, 14.85, 14.35. HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₂Na [M+Na]⁺: 293.1517, Found:293.1522.

1,2-Bis(2,3-dimethoxyphenyl)ethane-1,2-diol (4t)

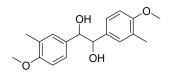


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (99% yield). *meso: dl* =1:2.34.

¹H NMR (400 MHz, CDCl₃): δ 6.93 – 6.85 (m, 2H), 6.80 (dt, *J* = 7.9, 2.4 Hz, 2H), 6.73 (ddt, *J* = 10.2, 8.1, 1.8 Hz, 2H), 5.07 (s, 1H), 5.02 (s, 1H), 3.80 – 3.70 (m, 8H), 3.70 – 3.65 (m, 4H), 3.20 (s, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 152.43, 146.92, 146.71, 134.42, 133.97, 133.94, 124.11, 124.09, 124.00, 120.23, 120.18, 112.03, 111.98, 73.89, 73.86, 73.78, 60.92, 60.90, 60.78, 55.86, 55.81. HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₆Na [M+Na]⁺: 357.1314, Found:357.1312.

1,2-Bis(4-methoxy-3-methylphenyl)ethane-1,2-diol (4u)



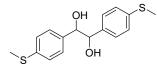
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (80% yield). *meso:* dl = 1:1.04.

¹H NMR (400 MHz, CDCl₃): δ 7.15 (d, *J* = 6.8 Hz, 2H), 6.98 (d, *J* = 2.2 Hz, 1H), 6.91 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.86 – 6.76 (m, 1H), 6.69 (d, *J* = 8.3 Hz, 1H), 4.63 (s, 1H), 4.61 (s, 1H), 3.85 (s, 3H), 3.81 (s, 3H), 2.92 (s, 1H), 2.24 (s, 3H), 2.18 (s, 3H), 2.08 (s, 1H);

¹³C NMR (100 MHz, CDCl₃): δ δ 157.48, 157.30, 131.48, 129.26, 127.47, 125.63, 110.38, 109.61, 78.63, 77.48, 77.16, 76.84, 55.49, 55.45, 16.43, 16.35.

HRMS (ESI, m/z) : Calcd for C₁₈H₂₂O₄Na [M+Na]⁺: 325.1415, Found: 325.1442.

1,2-bis(4-(methylthio)phenyl)ethane-1,2-diol (4v)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (86% yield). *meso:* dl = 1:4.72.

¹H NMR (400 MHz, DMSO-*d*₆): δ 7.18 (q, *J* = 8.4 Hz, 2H), 7.12 – 6.97 (m, 6H), 5.34 (s, 1H), 4.57 (s, 2H), 3.39 (s, 1H), 2.44 (d, *J* = 12.3 Hz, 6H).

¹³C NMR (100 MHz, DMSO-*d*₆): δ 140.06, 139.06, 136.06, 135.91, 127.99, 127.92, 127.84, 125.20, 125.14, 125.07, 77.03, 76.57, 14.98, 14.82.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₈O₂S₂Na [M+Na]⁺: 329.0646, Found: 329.0645.

1,2-Di(thiophen-2-yl)ethane-1,2-diol (4w)

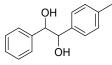
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (84% yield). *meso:* dl = 1:1.62.

¹H NMR (400 MHz, CDCl₃): δ 7.21 (dd, J = 5.0, 1.3 Hz, 1H), 7.19 – 7.16 (m, 2H), 6.95 (dd, J = 3.6, 1.2 Hz, 1H), 6.90 (dd, J = 5.0, 3.5 Hz, 1H), 6.83 (dd, J = 5.1, 3.5 Hz, 2H), 6.75 (dd, J = 3.6, 1.2 Hz, 2H), 5.05 (s, 1H), 4.97 (s, 2H), 3.02 (s, 1H), 2.50 (s, 1H).

¹³C NMR (100 MHz, CDCl₃): δ 143.04, 142.67, 126.78, 126.75, 126.16, 125.99, 125.79, 125.55, 75.09, 74.57.

HRMS (ESI, m/z) : Calcd for $C_{10}H_{10}O_2S_2Na$ [M+Na]⁺: 249.0020, Found:249.001.

1-Phenyl-2-(p-tolyl)ethane-1,2-diol (5a)



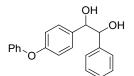
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (85% yield). *meso:* dl = 1:1.17.

¹H NMR (400 MHz, CDCl₃): δ 7.32 (td, J = 5.1, 2.1 Hz, 2H), 7.28 – 7.18 (m, 5H), 7.18 – 7.09 (m, 6H), 7.08 – 6.99 (m, 5H), 4.83 – 4.70 (m, 2H), 4.64 (dt, J = 8.9, 3.1 Hz, 2H), 2.83 (s, 4H), 2.37 (d, J = 2.2 Hz, 3H), 2.33 (s, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 140.12, 140.02, 139.82, 137.73, 137.71, 137.44, 137.12, 137.03, 136.84, 128.97, 128.93, 128.84, 128.19, 128.16, 128.11, 127.99, 127.88, 127.83, 127.22, 127.19, 127.16, 127.12, 127.08, 127.00, 126.97, 79.12, 79.04, 78.91, 78.82, 78.03, 78.00, 77.98, 21.24, 21.22.

HRMS (ESI, m/z) : Calcd for C₁₅H₁₆O₂Na [M+Na]⁺: 251.1048, Found: 251.1032.

1-(4-phenoxyphenyl)-2-phenylethane-1,2-diol (5b)



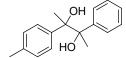
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (82% yield). *meso: dl*=1.14:1

¹H NMR (400 MHz, CDCl₃): δ 7.13 (d, J = 15.6 Hz, 4H), 7.07 – 7.00 (m, 2H), 6.96 (d, J = 8.6 Hz, 3H), 6.85 (t, J = 10.2 Hz, 3H), 6.79 – 6.66 (m, 3H), 4.66 – 4.61 (m, 1H), 4.45 (d, J = 5.5 Hz, 1H), 3.63 (s, 2H).

¹³C NMR (100 MHz, CDCl₃): δ 157.14, 156.95, 156.84, 139.71, 134.81, 134.52, 129.80, 129.77, 128.63, 128.54, 128.12, 127.94, 127.17, 127.09, 123.38, 123.34, 118.90, 118.83, 118.79, 118.52, 118.45, 79.24, 78.81, 78.71, 77.97, 77.92, 77.49, 77.42.

HRMS (ESI, m/z) : Calcd for $C_{20}H_{18}O_3Na C_{24}H_{20}O_2Na [M+Na]^+$: 329.1154, Found: 329.1146.

2-phenyl-3-(p-tolyl)butane-2,3-diol (5c)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (87% yield). *meso:* dl = 1:1.67.

¹H NMR (400 MHz, CDCl₃): δ 7.10 (dddd, *J* = 12.5, 9.2, 4.9, 2.6 Hz, 5H), 7.04 – 6.98 (m, 1H), 6.97 – 6.89 (m, 3H), 2.68 (s, 1H), 2.38 (s, 1H), 2.21 (d, *J* = 5.8 Hz, 3H), 1.50 – 1.39 (m, 3H), 1.39 – 1.25 (m, 5H).

¹³C NMR (100 MHz, CDCl₃): δ 143.90, 143.54, 140.99, 140.64, 136.51, 136.44, 136.35, 136.29, 127.96, 127.83, 127.81, 127.47, 127.39, 127.19, 127.08, 127.07, 127.03, 126.98, 126.96, 126.94, 126.82, 126.77, 78.91, 78.88, 78.83, 78.80, 78.65, 78.61, 26.96, 25.13, 25.11, 25.02, 25.00, 24.94, 24.89, 24.87, 24.81, 21.01, 20.98.

HRMS (ESI, m/z) : Calcd for $C_{17}H_{20}O_2Na$ [M+Na]⁺: 279.1361, Found:279.1353.

2-(4-methoxyphenyl)-3-phenylbutane-2,3-diol (5d)

The title compound was synthesized according to the general procedure. The product was purified

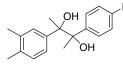
by flash column chromatography. Colorless oil (80% yield). meso: dl =1:1.56.

¹H NMR (400 MHz, CDCl₃): δ 7.28 – 7.04 (m, 14H), 6.76 (dd, J = 8.9, 6.9 Hz, 4H), 3.79 (d, J = 6.7 Hz, 6H), 2.57 (d, J = 12.5 Hz, 2H), 2.27 (d, J = 9.9 Hz, 1H), 1.56 (d, J = 7.4 Hz, 5H), 1.48 (d, J = 11.1 Hz, 7H).

¹³C NMR (100 MHz, CDCl₃): δ 158.74, 158.65, 144.05, 143.70, 136.09, 135.74, 128.72, 128.25, 127.48, 127.40, 127.27, 127.12, 127.08, 126.97, 112.74, 112.58, 79.14, 78.83, 78.72, 78.54, 55.35, 25.36, 25.23, 25.15, 25.10.

HRMS (ESI, m/z) : Calcd for C₁₇H₂₀O₃Na [M+Na]⁺: 295.1310, Found:295.1301.

2-(3,4-dimethylphenyl)-3-(4-fluorophenyl)butane-2,3-diol (5e)

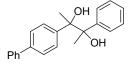


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (73% yield). *meso:* dl = 1:1.34.

¹H NMR (400 MHz, CDCl₃) δ 7.16 – 6.96 (m, 2H), 6.92 (q, *J* = 5.9, 4.9 Hz, 2H), 6.82 (dddd, *J* = 19.5, 8.8, 6.8, 4.2 Hz, 3H), 2.33 (s, 2H), 2.16 – 2.13 (m, 3H), 2.11 (t, *J* = 4.4 Hz, 3H), 1.51 – 1.39 (m, 3H), 1.36 (d, *J* = 9.0 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃) & 163.24, 163.20, 163.14, 163.09, 160.80, 160.77, 160.70, 160.65, 141.37, 141.17, 141.02, 140.83, 139.82, 139.60, 139.36, 139.21, 135.42, 135.37, 135.27, 135.23, 135.16, 135.07, 135.04, 129.30, 129.22, 129.17, 129.09, 128.84, 128.75, 128.72, 128.70, 128.62, 128.60, 128.56, 128.44, 128.36, 128.29, 124.91, 124.79, 124.43, 124.39, 114.04, 113.98, 113.88, 113.83, 113.78, 113.67, 113.58, 78.79, 78.63, 78.58, 78.54, 25.25, 25.20, 25.16, 25.11, 25.05, 24.91, 24.84, 19.91, 19.89, 19.34, 19.29.

HRMS (ESI, m/z) : Calcd for C₁₈H₂₁FO₂Na [M+Na]⁺: 311.1424, Found:311.1422. 2-([1,1'-biphenyl]-4-yl)-3-phenylbutane-2,3-diol (5f)

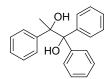


The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (81% yield). *meso:* dl = 1:1.28.

¹H NMR (400 MHz, CDCl₃) δ 7.43 (d, J = 7.0 Hz, 2H), 7.37 – 7.23 (m, 4H), 7.23 – 7.14 (m, 2H), 7.14 – 6.97 (m, 6H), 2.69 (s, 1H), 2.45 (s, 1H), 1.54 – 1.25 (m, 6H).

¹³C NMR (150 MHz, CDCl₃) δ 143.93, 143.56, 143.08, 142.69, 140.82, 140.78, 139.82, 139.75, 139.68, 128.88, 128.86, 128.03, 127.98, 127.62, 127.56, 127.53, 127.49, 127.43, 127.39, 127.37, 127.35, 127.30, 127.24, 127.20, 127.15, 127.13, 127.10, 127.05, 126.99, 126.04, 126.00, 125.98, 125.91, 125.86, 79.02, 78.99, 78.96, 78.92, 78.77, 78.74, 78.70, 78.67, 25.34, 25.26, 25.25, 25.20, 25.14, 25.08, 25.03. HRMS (ESI, m/z) : Calcd for $C_{22}H_{22}O_2Na$ [M+Na]⁺: 341.1517, Found: 341.1524.

1,1,2-Triphenylpropane-1,2-diol (5g)



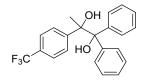
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (75% yield).

¹H NMR (400 MHz, CDCl₃): δ 7.52 (dt, J = 7.7, 2.7 Hz, 2H), 7.40 (dq, J = 7.2, 2.9, 2.5 Hz, 2H), 7.19 (ddt, J = 8.8, 6.7, 1.8 Hz, 1H), 7.12 (dq, J = 6.9, 2.5, 2.0 Hz, 3H), 7.04 (tq, J = 5.2, 2.9 Hz, 7H), 2.82 (d, J = 11.4 Hz, 1H), 2.57 – 2.33 (m, 1H), 1.56 (t, J = 2.7 Hz, 3H).

¹³C NMR (100 MHz, CDCl₃): δ 144.62, 143.96, 143.86, 143.82, 128.73, 128.54, 128.47, 128.34, 128.27, 127.68, 127.61, 127.57, 127.46, 127.42, 127.35, 127.20, 127.09, 127.05, 127.01, 126.94, 126.65, 82.18, 80.42, 26.84.

HRMS (ESI, m/z) : Calcd for $C_{21}H_{20}O_2Na$ [M+Na]⁺: 327.1361, Found: 327.1365.

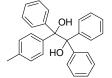
1,1-Diphenyl-2-(4-(trifluoromethyl)phenyl)propane-1,2-diol (5h)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (80% yield).

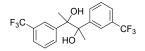
¹H NMR (400 MHz, CDCl₃) δ 7.57 – 7.47 (m, 2H), 7.46 – 7.37 (m, 2H), 7.36 – 7.28 (m, 4H), 7.21 – 7.17 (m, 3H), 7.09 (td, *J* = 5.2, 2.0 Hz, 3H), 2.73 (s, 1H), 2.46 (d, *J* = 12.1 Hz, 1H), 1.62 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 149.10, 144.32, 143.80, 143.58, 128.76, 128.32, 128.29, 127.94, 127.81, 127.72, 127.49, 127.42, 127.41, 127.09, 124.14 (m, *J*= 4 Hz), 82.49, 80.19, 27.13. HRMS (ESI, m/z) : Calcd for C₂₂H₁₉F₃O₂Na [M+Na]⁺: 395.1235, Found:395.1233.

1,1,2-triphenyl-2-(p-tolyl)ethane-1,2-diol (5i)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid 83% yield). *dl* : *meso* = 1:0 ¹H NMR (400 MHz, CDCl₃): δ 7.21 (dq, *J* = 7.1, 3.3 Hz, 6H), 7.05 (ddq, *J* = 7.4, 4.9, 2.7 Hz, 11H), 6.87 (dt, *J* = 5.8, 2.9 Hz, 2H), 2.92 (s, 2H), 2.17 (p, *J* = 2.6 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 144.60, 144.53, 144.43, 144.40, 144.33, 141.32, 136.74, 136.68, 136.59, 128.75, 128.73, 128.65, 128.62, 128.19, 128.15, 127.41, 127.36, 127.32, 127.07, 127.02, 126.96, 126.92, 126.85, 83.17, 83.07, 21.10. HRMS (ESI, m/z) : Calcd for C₂₇H₂₄O₂Na [M+Na]⁺: 403.1675, Found: 403.1648

2,3-bis(3-(trifluoromethyl)phenyl)butane-2,3-diol (6a)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. Colorless oil (45% yield). *meso:* dl = 1:1.24.

¹H NMR (400 MHz, CDCl₃): δ 7.60 – 7.45 (m, 3H), 7.44 – 7.30 (m, 5H), 2.46 (s, 2H), 1.64 (s, 3H), 1.57 (d, *J* = 1.5 Hz, 4H).

¹³C NMR (100 MHz, CDCl₃): δ 144.55, 144.15, 130.59, 130.19, 129.88 (d, *J*= 3 Hz), 129.56 (d, *J*= 3 Hz), 127.72, 127.67, 125.50, 125.48, 124.12, 124.08, 123.95, 123.91, 123.88, 122.79, 122.77, 78.55, 78.34, 24.87, 24.58.

HRMS (ESI, m/z) : Calcd for $C_{18}H_{16}F_6O_2Na [M+Na]^+$: 401.0952, Found: 401.0951.

2,3-diphenylbutane-2,3-diol (6b)



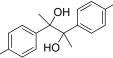
The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (52% yield). *meso:* dl = 1:1.36.

¹H NMR (400 MHz, CDCl₃): δ 7.36 – 7.15 (m, 10H), 2.80 – 2.28 (m, 2H), 1.62 (s, 2H), 1.54 (s, 3H).

¹³C NMR (101 MHz, CDCl₃): δ 143.79, 143.43, 127.36, 127.21, 127.17, 127.08, 127.02, 126.99, 126.92, 126.83, 78.83, 78.59, 25.03, 24.86.

HRMS (ESI, m/z) : Calcd for C₁₆H₁₈O₂Na [M+Na]⁺: 265.1205, Found: 265.1195.

2,3-di-p-tolylbutane-2,3-diol (6c)



The title compound was synthesized according to the general procedure. The product was purified by flash column chromatography. White solid (61% yield). *meso:* dl = 1:1.42.

¹H NMR (400 MHz, CDCl₃): δ 7.26 – 7.16 (m, 2H), 7.15 – 7.07 (m, 6H), 2.76 – 2.46 (m, 2H), 2.36 (d, *J* = 4.8 Hz, 6H), 1.57 (s, 3H), 1.50 (s, 4H).

¹³C NMR (100 MHz, CDCl₃):8 141.07, 140.72, 136.55, 136.40, 128.05, 127.89, 127.41, 126.99, 78.87, 78.63, 25.26, 25.07, 21.05, 21.02.

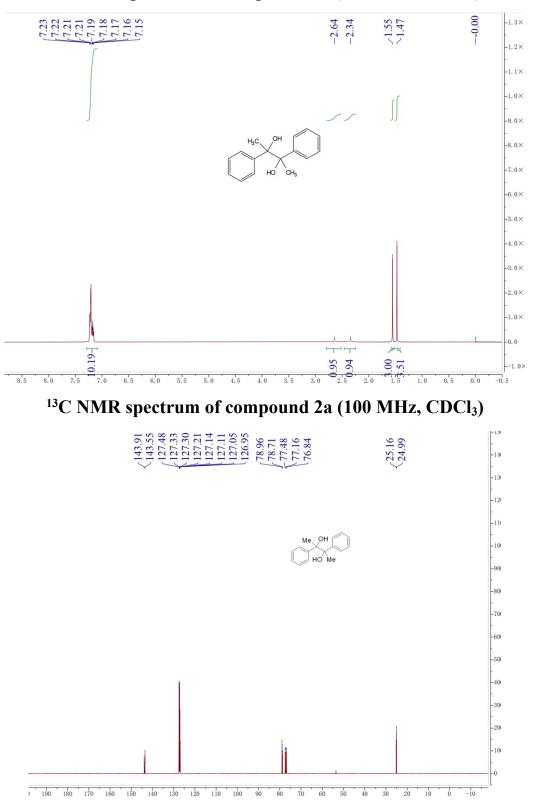
HRMS (ESI, m/z): Calcd for $C_{18}H_{22}O_2Na [M+Na]^+$: 293.1518, Found: 293.1513. 3,3-diphenylpropanoic acid (11)



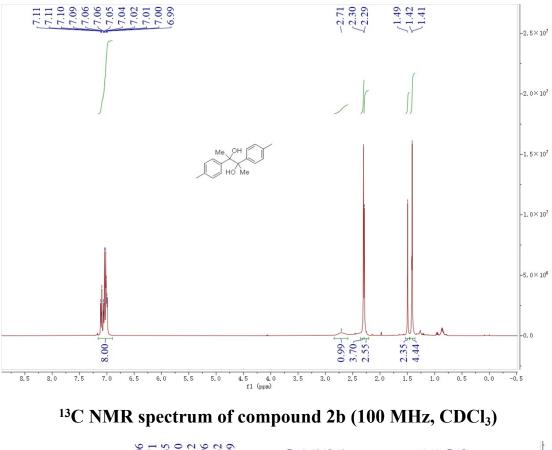
¹H NMR (400 MHz, CDCl₃): δ 7.23 – 7.10 (m, 10H), 4.46 (t, *J* = 4.1 Hz, 1H), 3.02 (d, *J* = 3.0 Hz, 2H).

¹³C NMR (150 MHz, CDCl₃): δ 177.53, 143.39, 128.77, 127.76, 126.78, 46.78, 40.52.

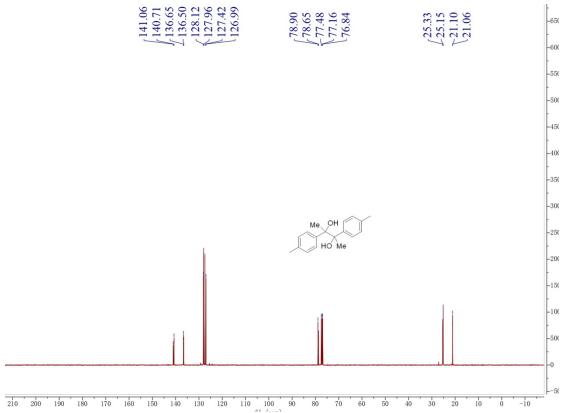
17. NMR Spectra

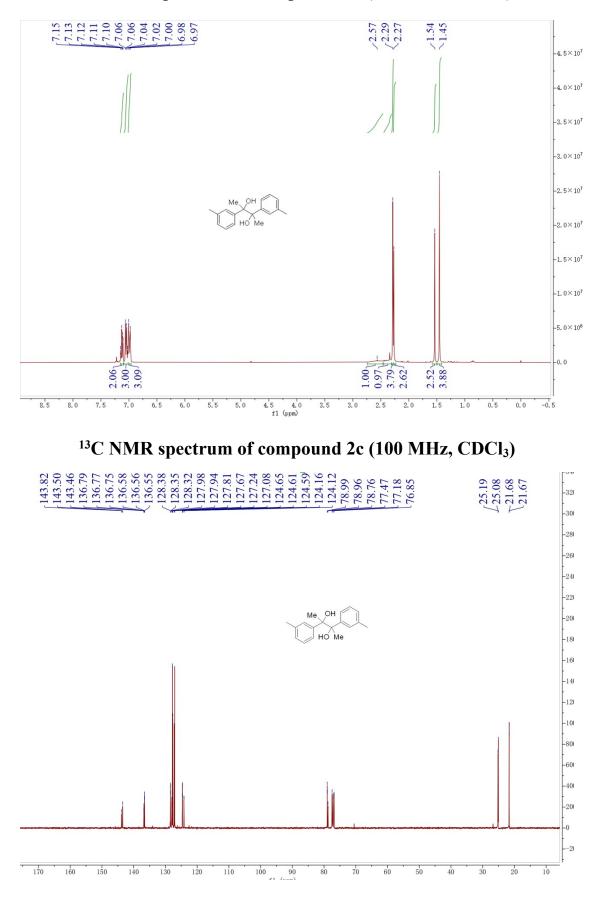


¹H NMR spectrum of compound 2a (400 MHz, CDCl₃)

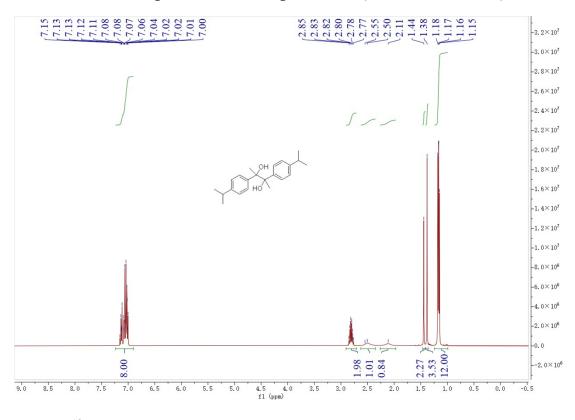


¹H NMR spectrum of compound 2b (400 MHz, CDCl₃)



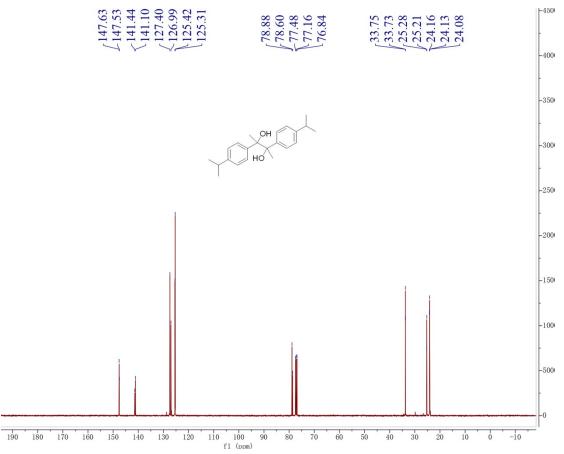


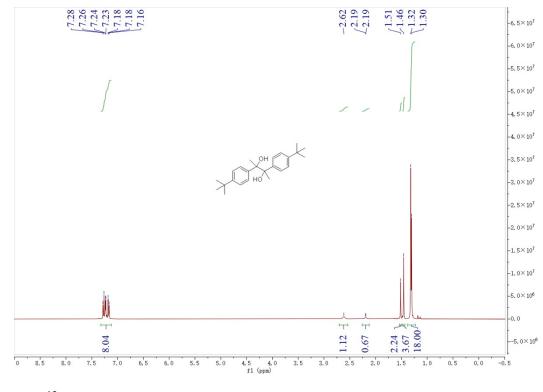
¹H NMR spectrum of compound 2c (400 MHz, CDCl₃)



¹H NMR spectrum of compound 2d (400 MHz, CDCl₃)

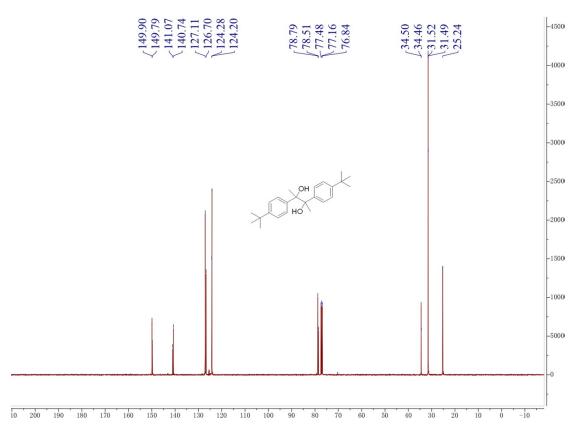
¹³C NMR spectrum of compound 2d (100 MHz, CDCl₃)

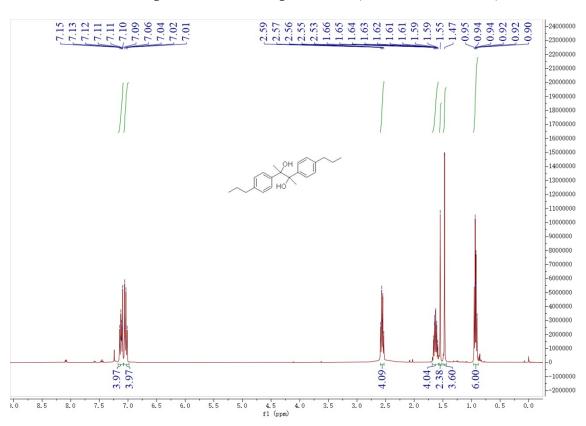




¹H NMR spectrum of compound 2e (400 MHz, CDCl₃)

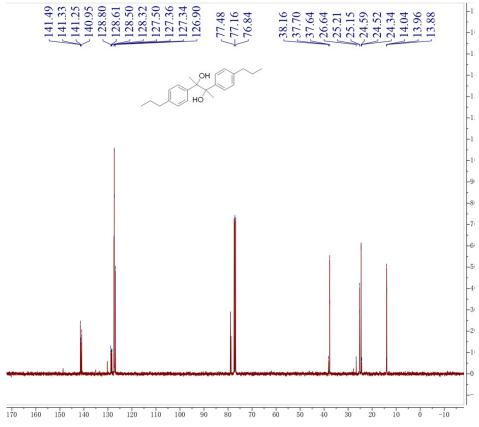
¹³C NMR spectrum of compound 2e (100 MHz, CDCl₃)

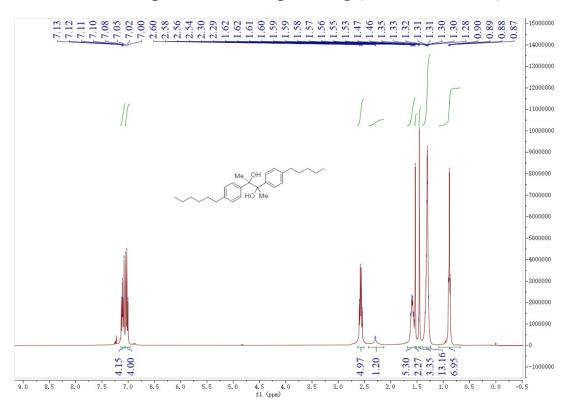




¹H NMR spectrum of compound 2f (400 MHz, CDCl₃)

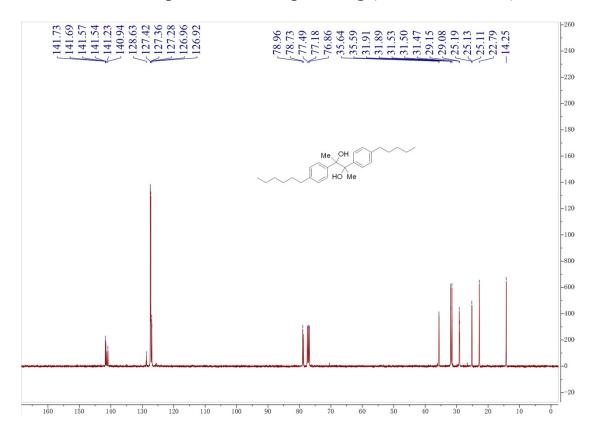
¹³C NMR spectrum of compound 2f (100 MHz, CDCl₃)

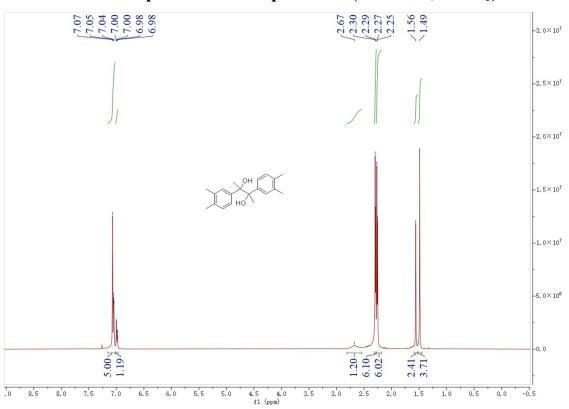




¹H NMR spectrum of compound 2g (400 MHz, CDCl₃)

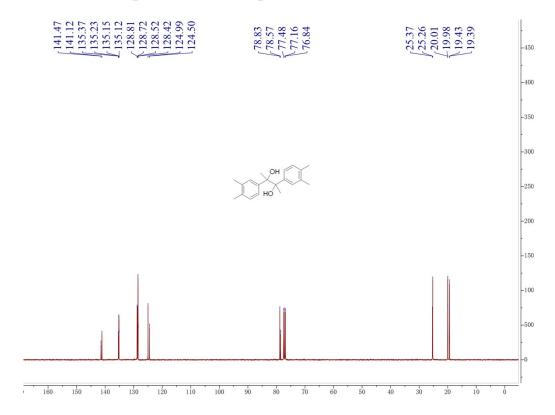
¹³C NMR spectrum of compound 2g (100 MHz, CDCl₃)

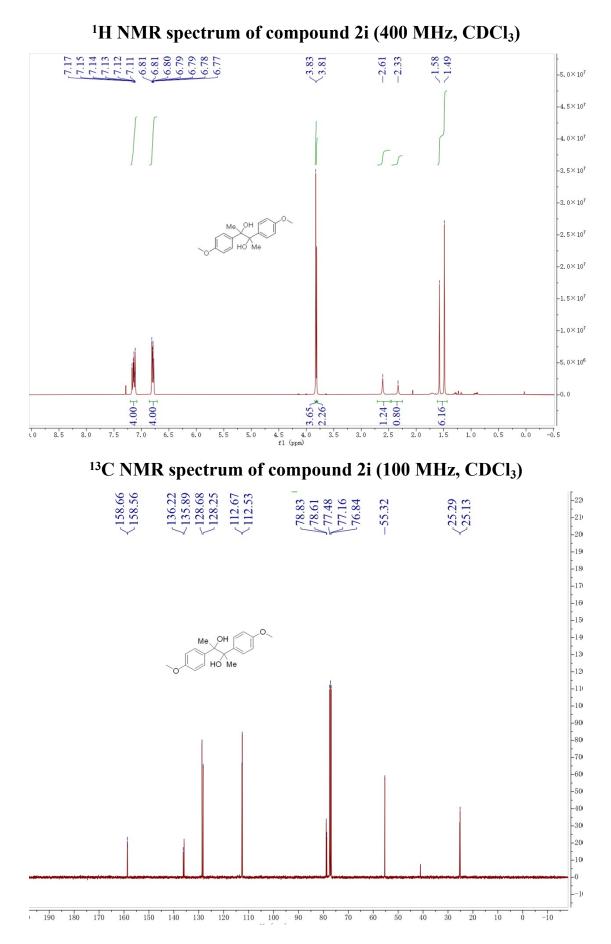




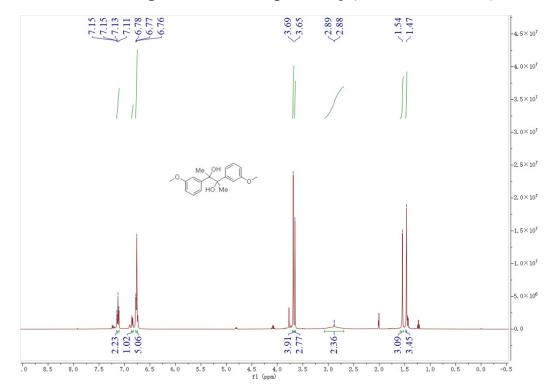
¹H NMR spectrum of compound 2h (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 2h (100 MHz, CDCl₃)



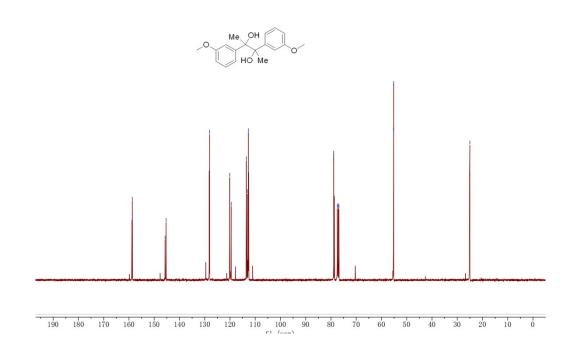


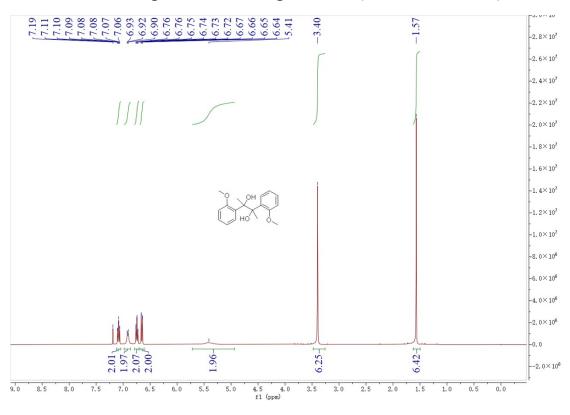




¹³C NMR spectrum of compound 2j (100 MHz, CDCl₃)

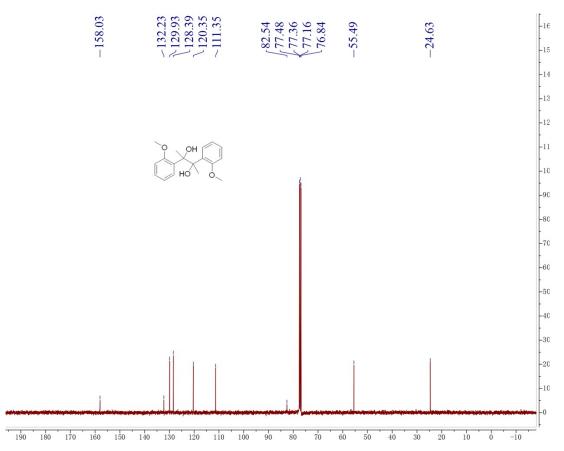
52 24 111111111111111111111111111111111111	∞ ∞	5.68 8.22 8.22 9.51 9.51 9.51 3.13 3.13 3.13 3.13 3.13 3.13 3.13 1.66 .66 .66 .66 .19 .10 .10 .10 .10 .10 .10 .10 .10 .10 .10	13 99
	15	22007088811111202044	25

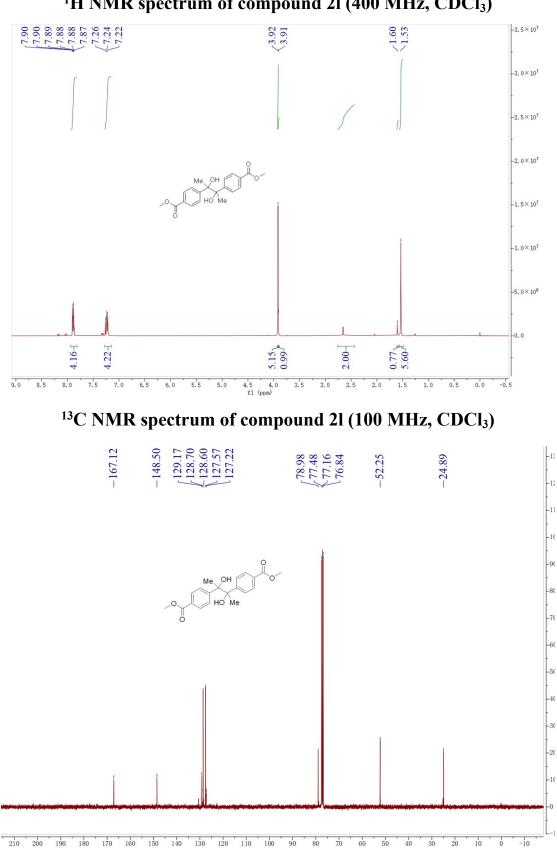


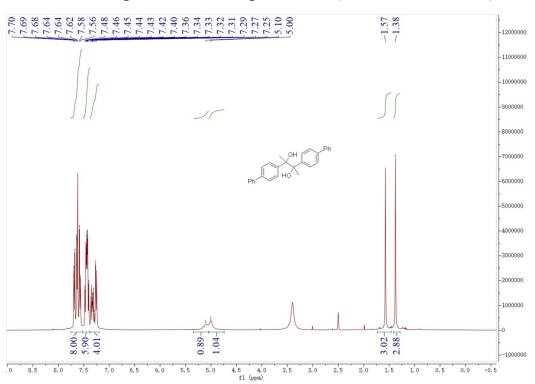


¹H NMR spectrum of compound 2k (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 2k (100 MHz, CDCl₃)

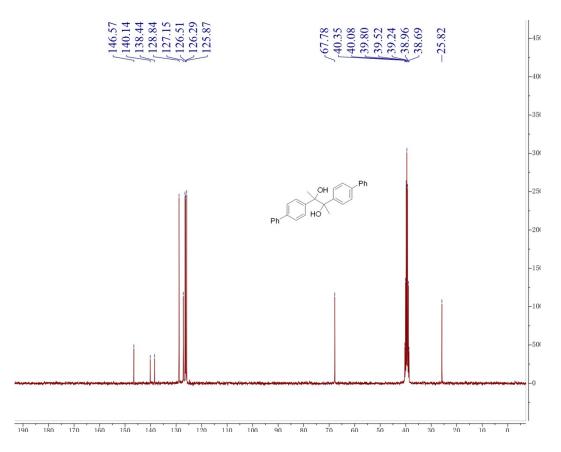


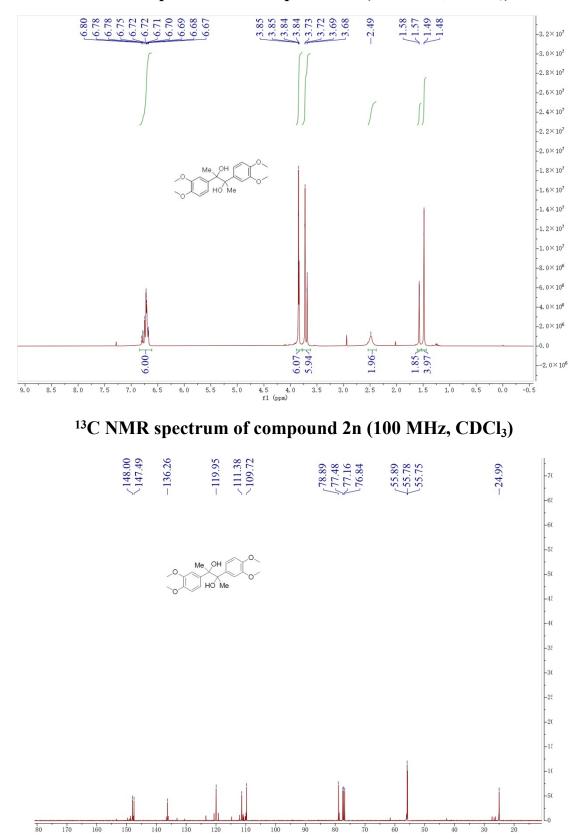




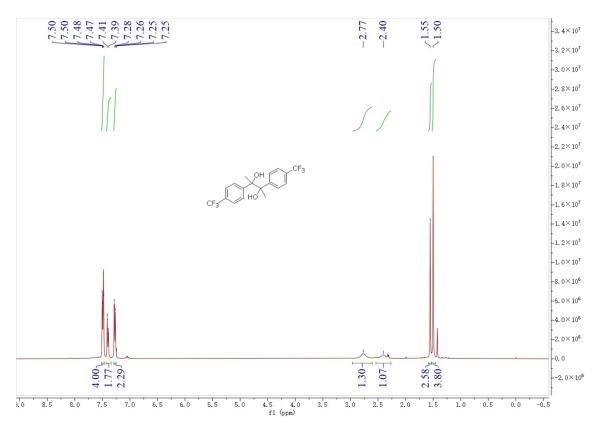
¹H NMR spectrum of compound 2m (400 MHz, DMSO-*d*₆)

¹³C NMR spectrum of compound 2m (100 MHz, DMSO-*d*₆)



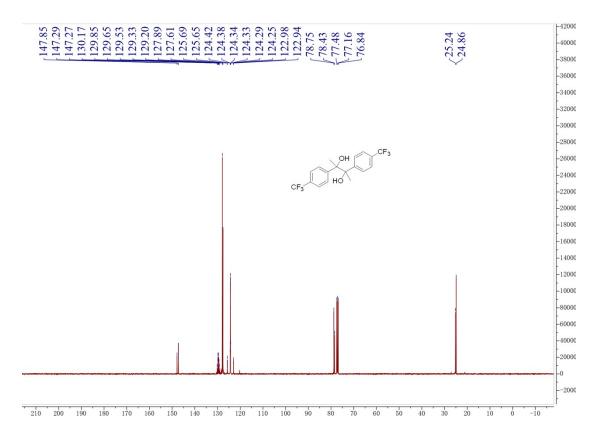


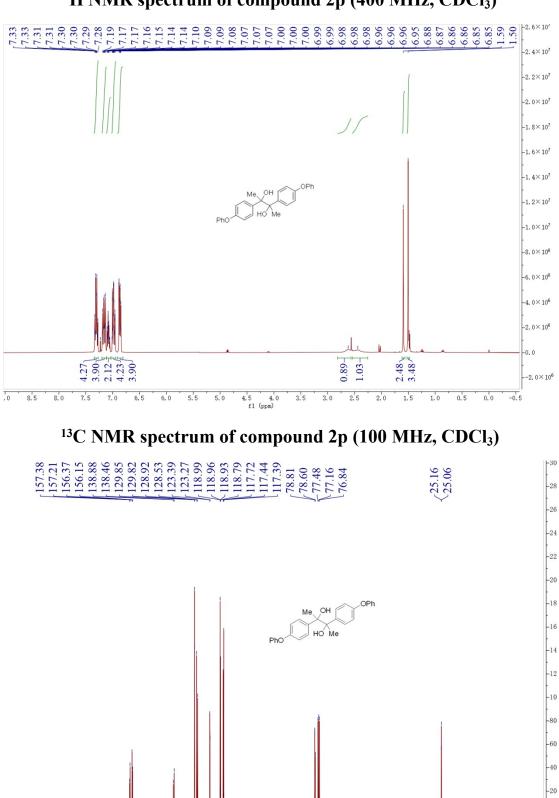
¹H NMR spectrum of compound 2n (400 MHz, CDCl₃)



¹H NMR spectrum of compound 20 (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 20 (100 MHz, CDCl₃)





¹H NMR spectrum of compound 2p (400 MHz, CDCl₃)

40 30

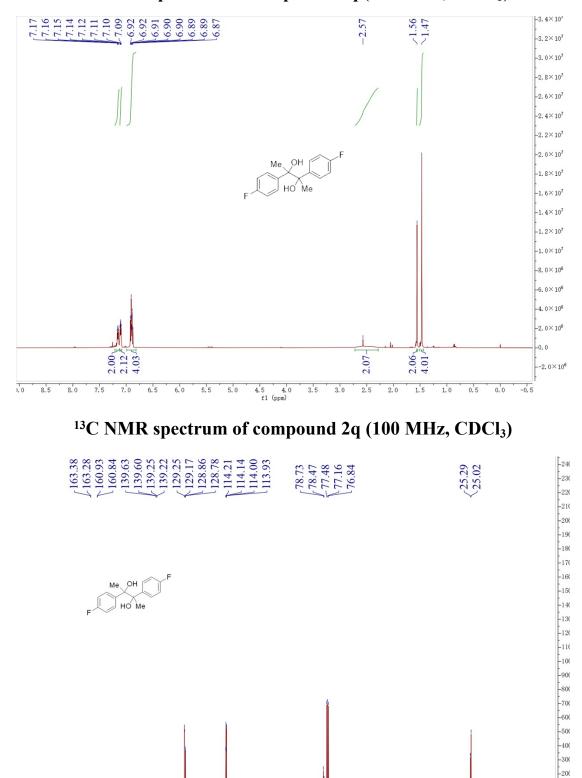
-10

100 90

10 200

190 180 170 160 150

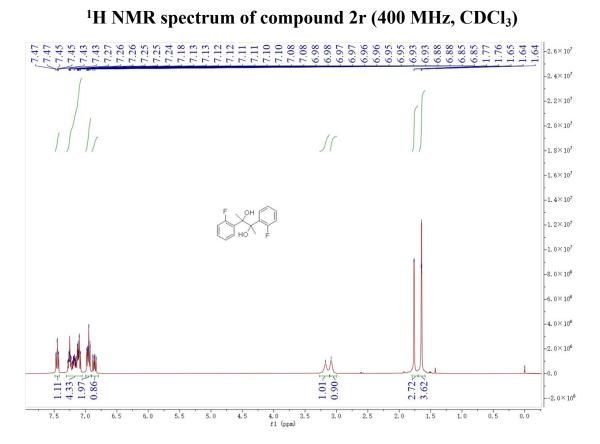
-0



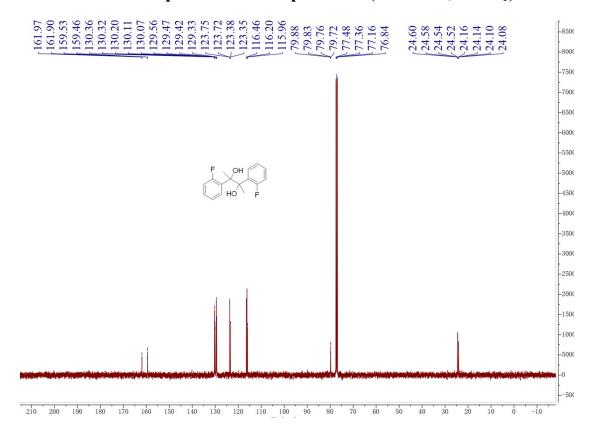
¹H NMR spectrum of compound 2q (400 MHz, CDCl₃)

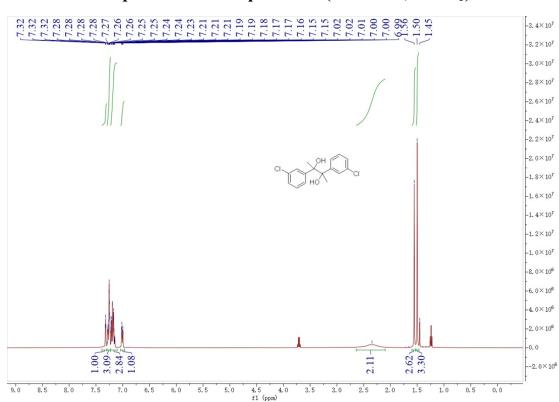
90 180

-10(-0 --1(--2(



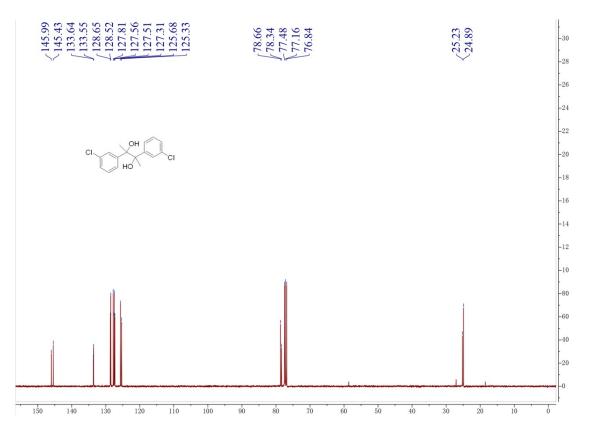
¹³C NMR spectrum of compound 2r (100 MHz, CDCl₃)

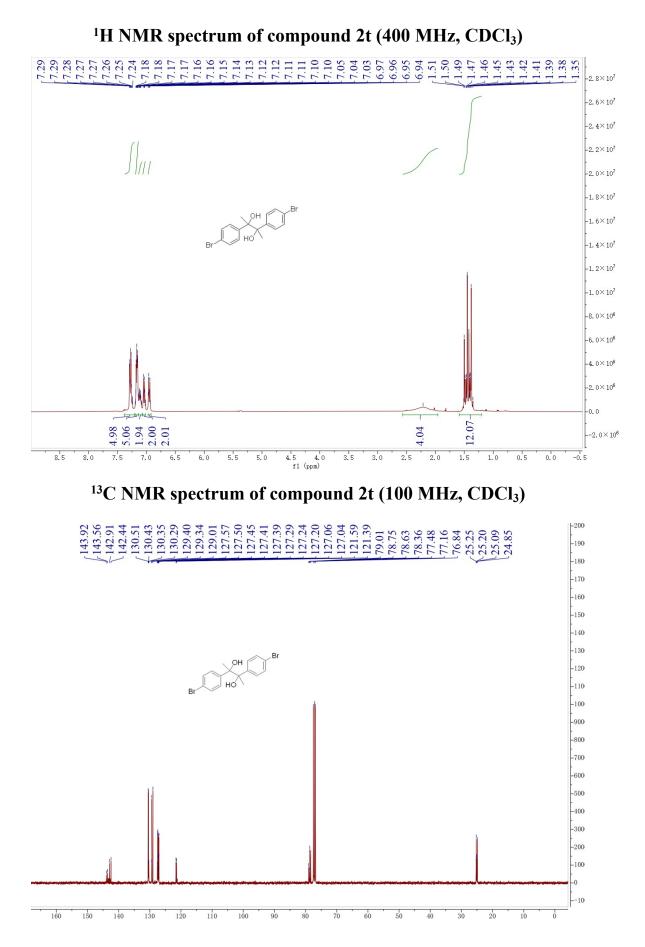


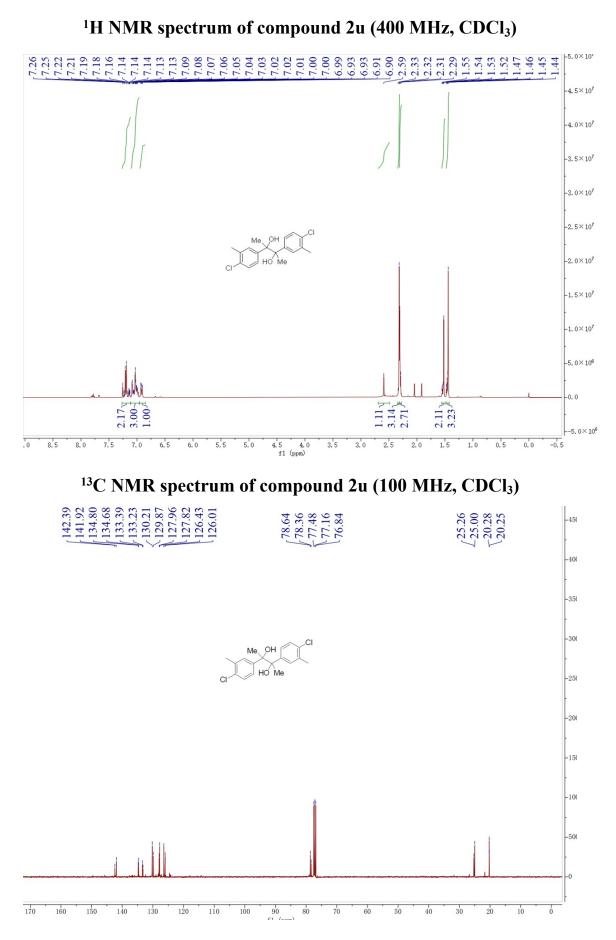


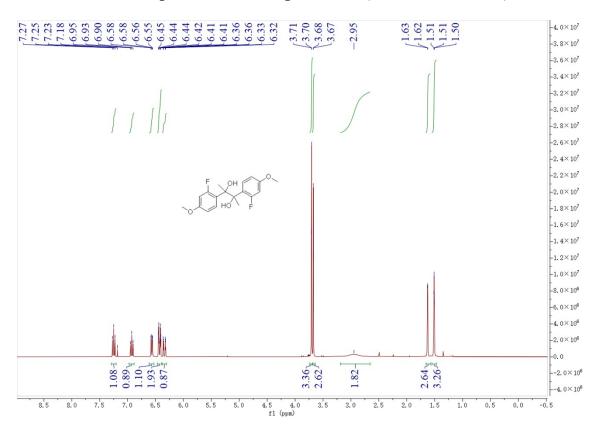
¹H NMR spectrum of compound 2s (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 2s (100 MHz, CDCl₃)



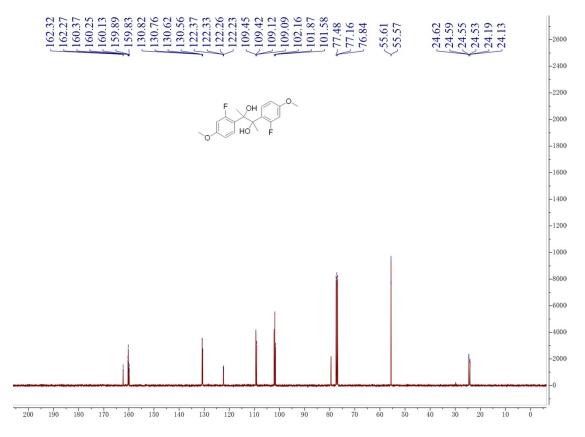


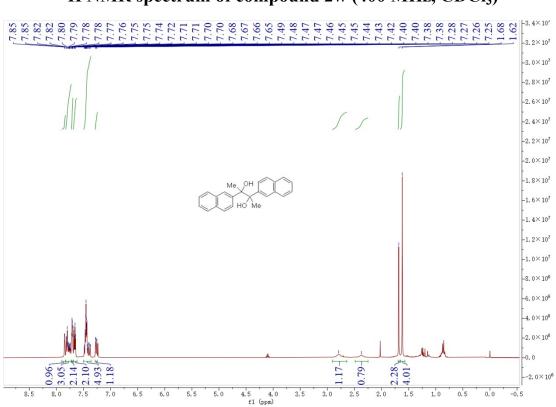




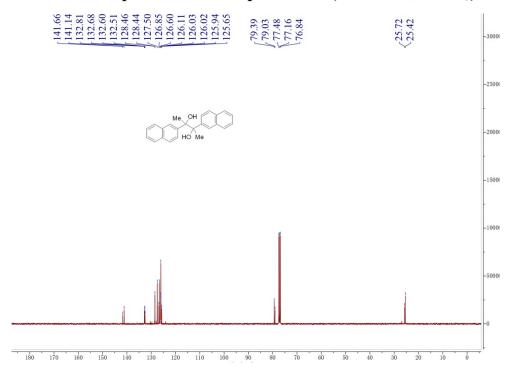
¹H NMR spectrum of compound 2v (400 MHz, CDCl₃)

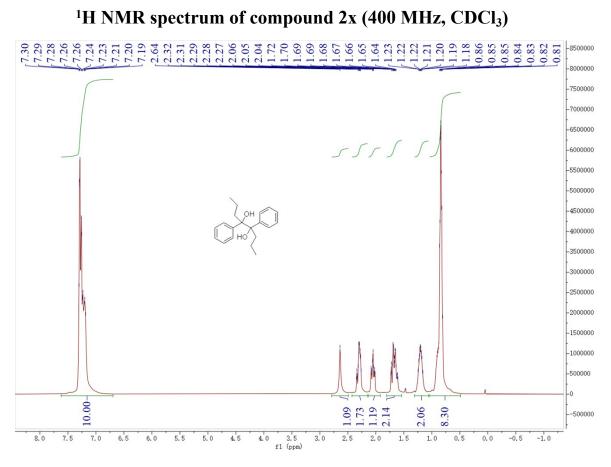
¹³C NMR spectrum of compound 2v (100 MHz, CDCl₃)



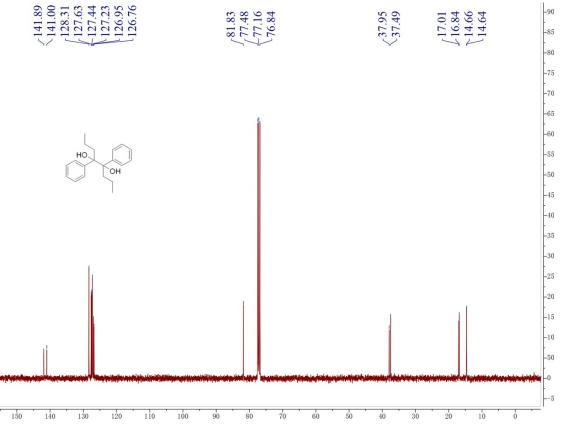


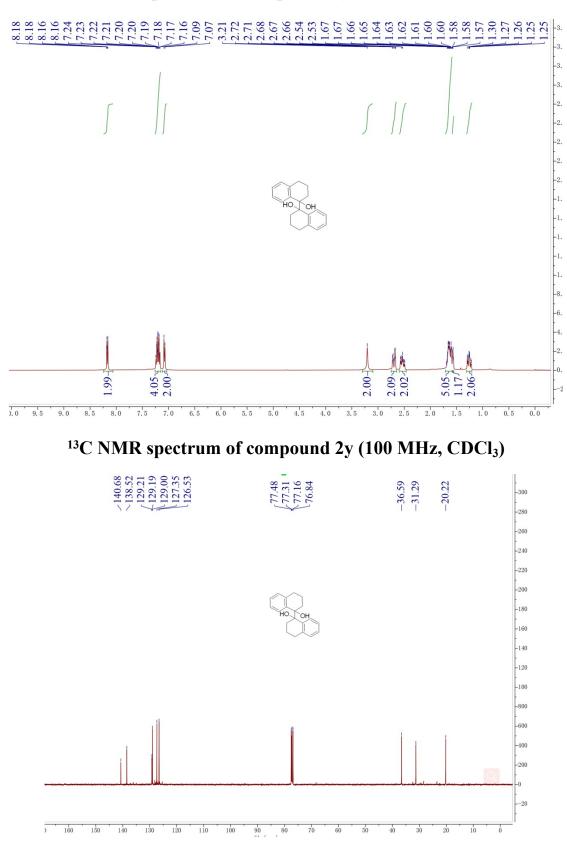
¹³C NMR spectrum of compound 2w (100 MHz, CDCl₃)



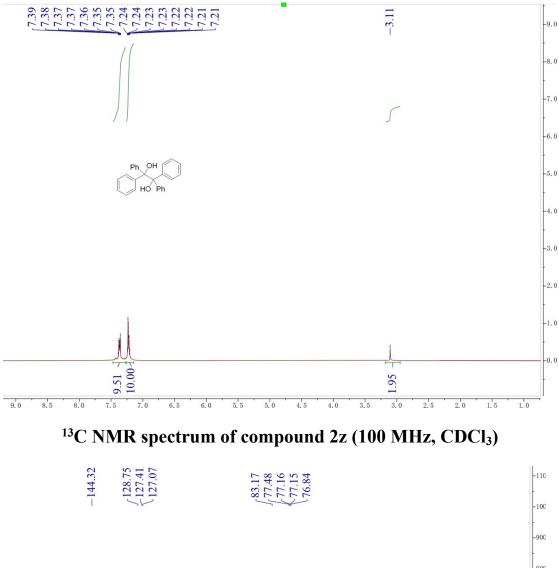


¹³C NMR spectrum of compound 2x (100 MHz, CDCl₃)

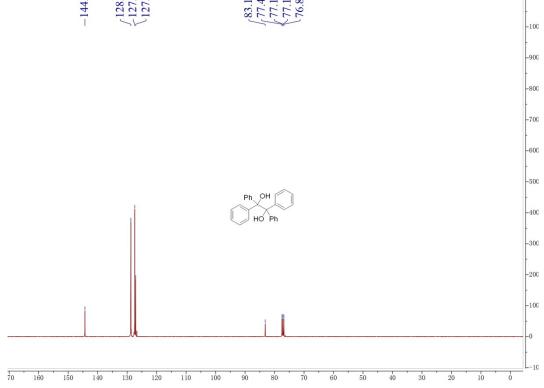


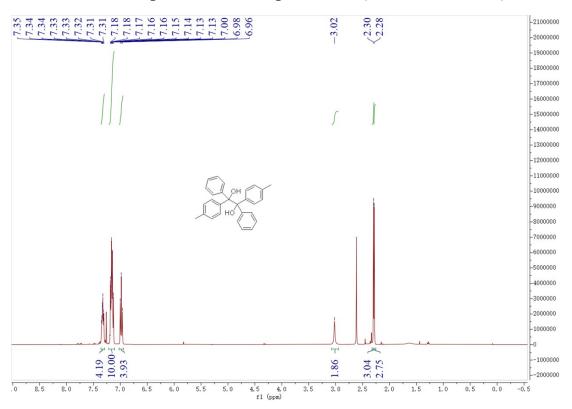


¹H NMR spectrum of compound 2y (400 MHz, CDCl₃)



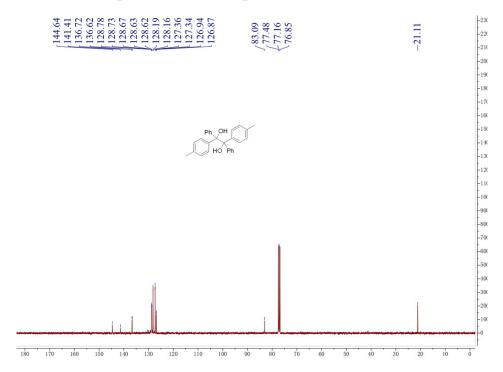
¹H NMR spectrum of compound 2z (400 MHz, CDCl₃)

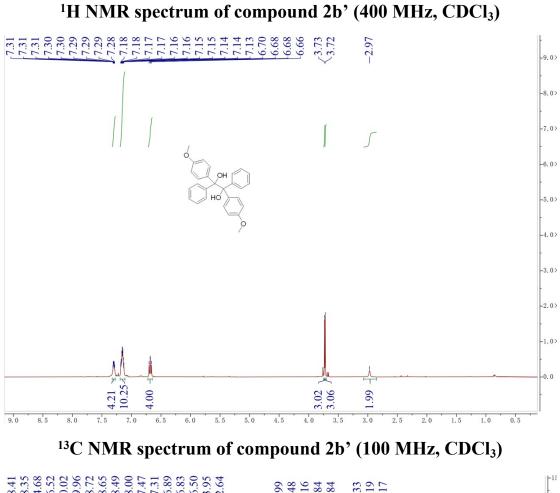


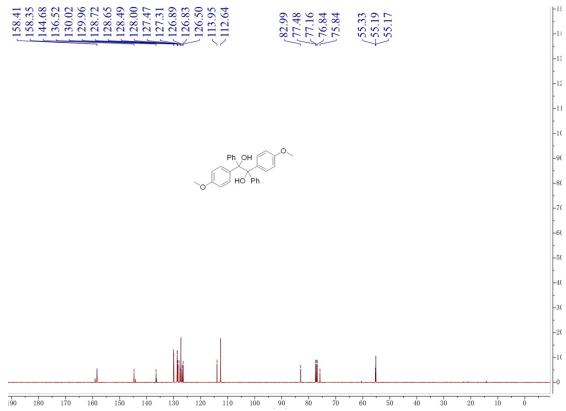


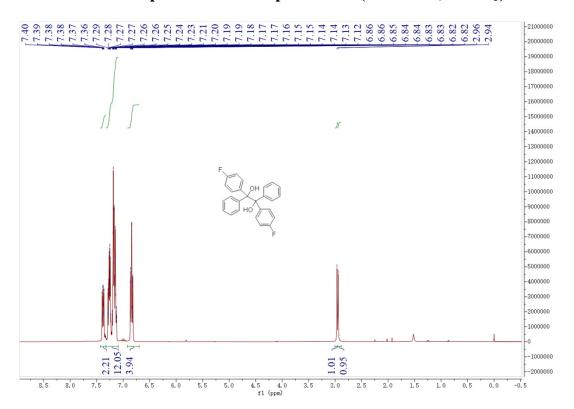
¹H NMR spectrum of compound 2a' (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 2a' (100 MHz, CDCl₃)



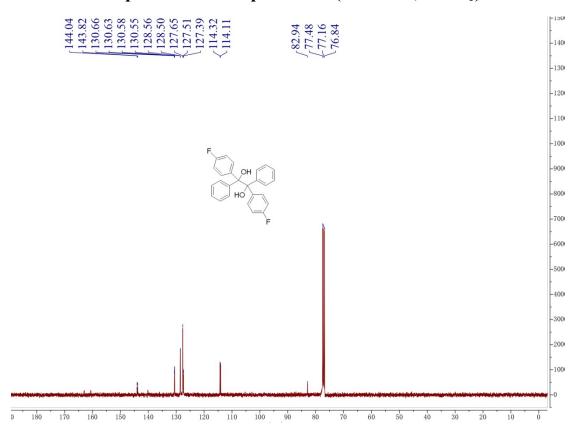


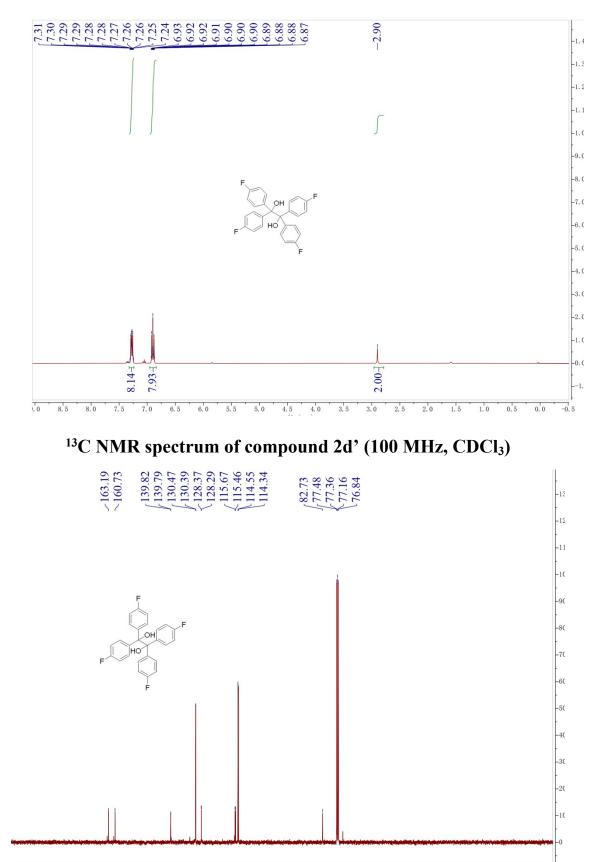




¹H NMR spectrum of compound 2c' (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 2c' (100 MHz, CDCl₃)





¹H NMR spectrum of compound 2d' (400 MHz, CDCl₃)

90

80 70 60

50 40

30 20

10

0

110 100

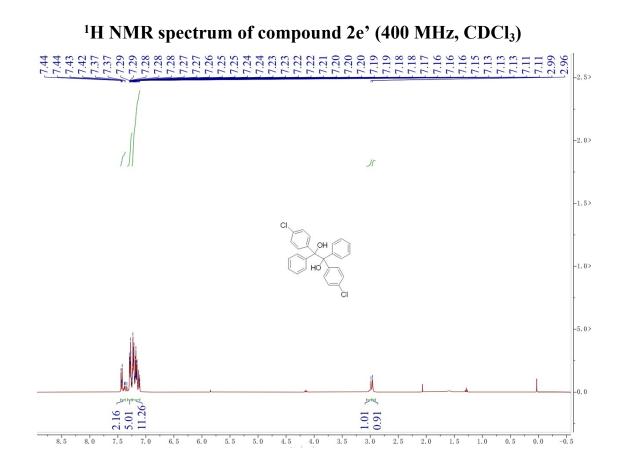
120

0 190

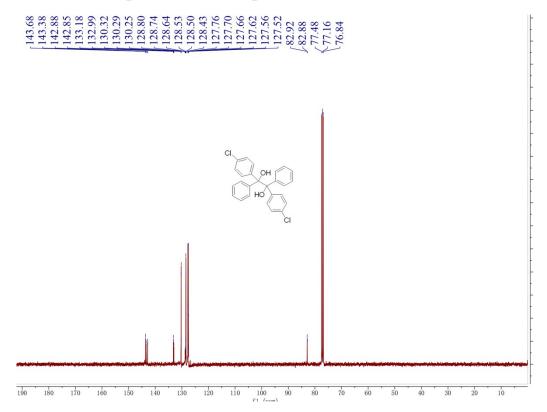
180

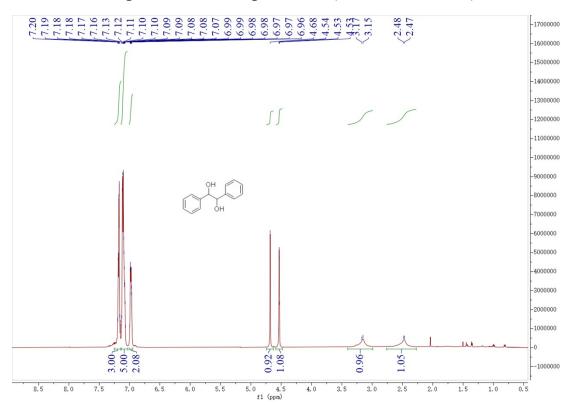
170 160

150 140 130



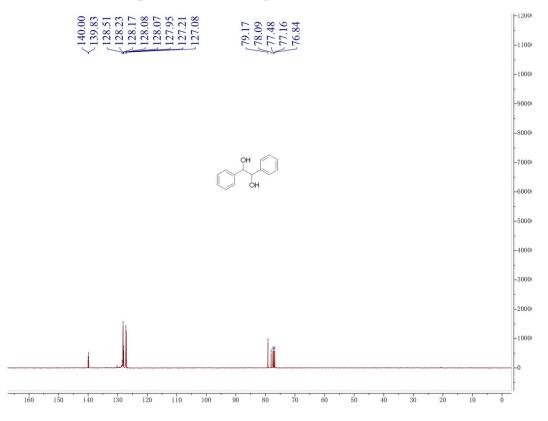
¹³C NMR spectrum of compound 2e' (100 MHz, CDCl₃)

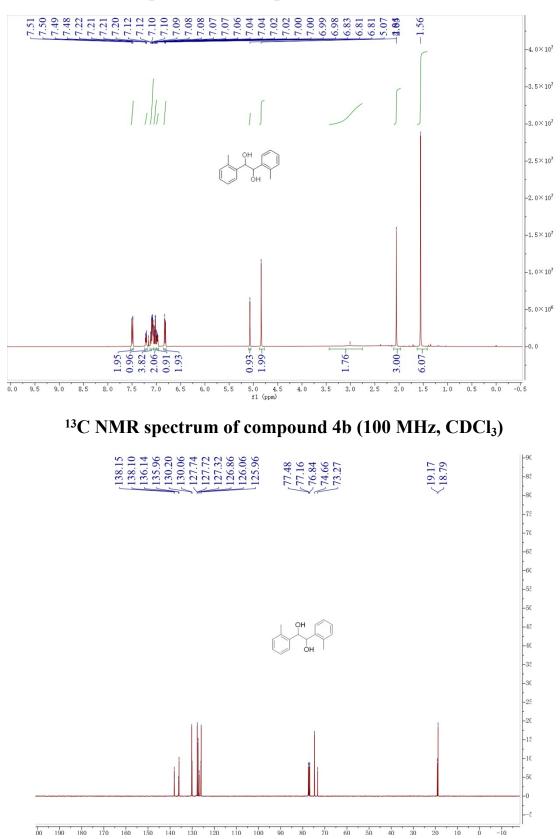




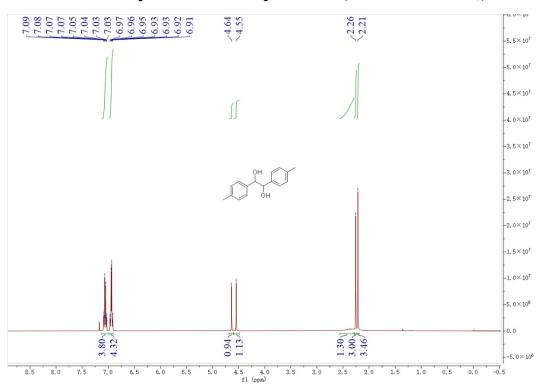
¹H NMR spectrum of compound 4a (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 4a (100 MHz, CDCl₃)



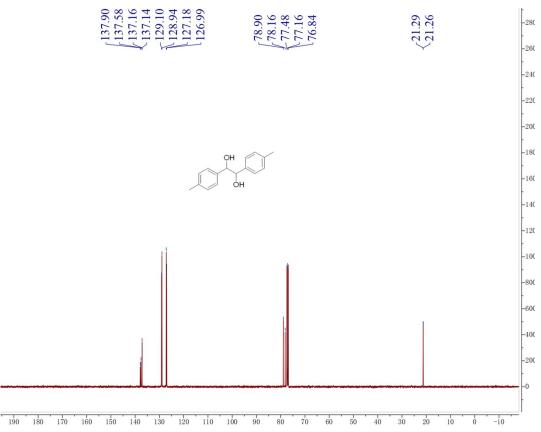


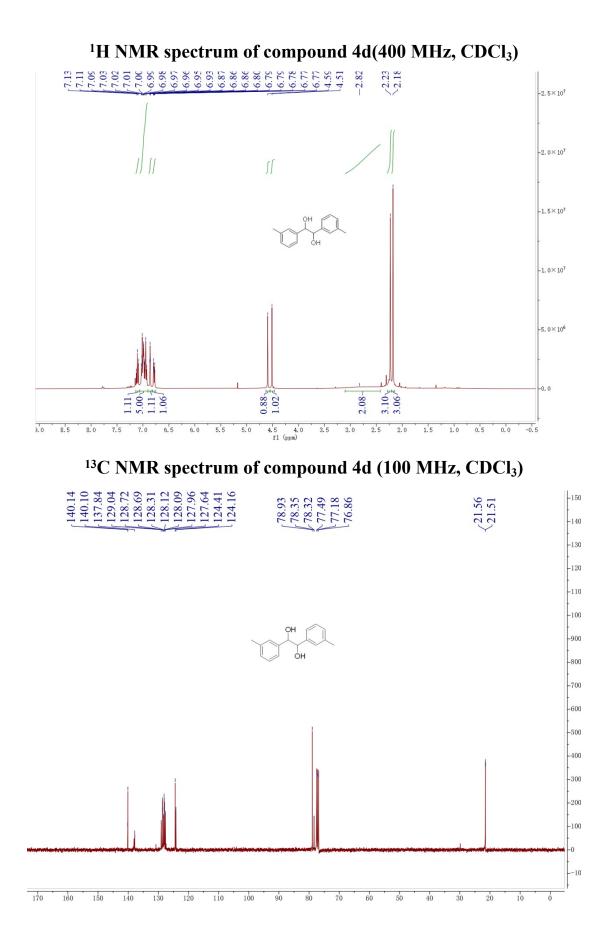
¹H NMR spectrum of compound 4b (400 MHz, CDCl₃)

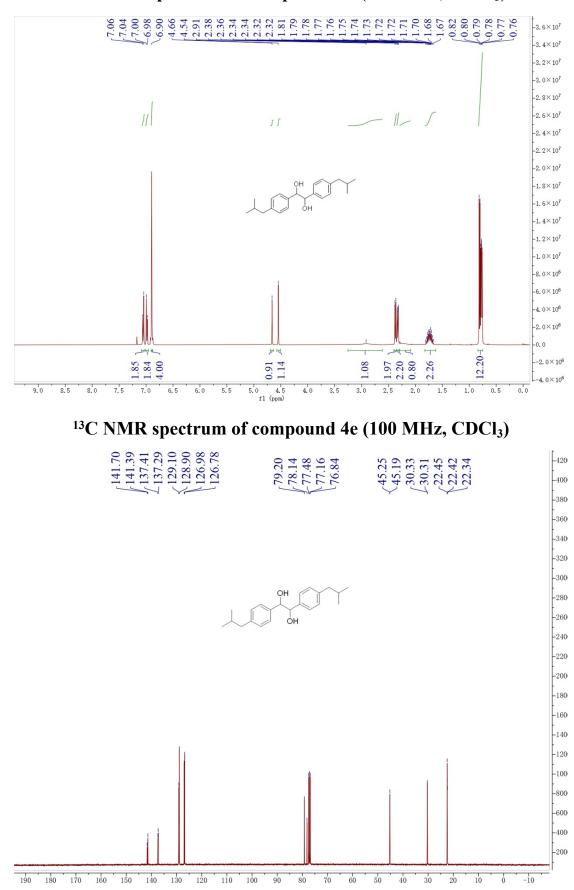


¹H NMR spectrum of compound 4c (400 MHz, CDCl₃)

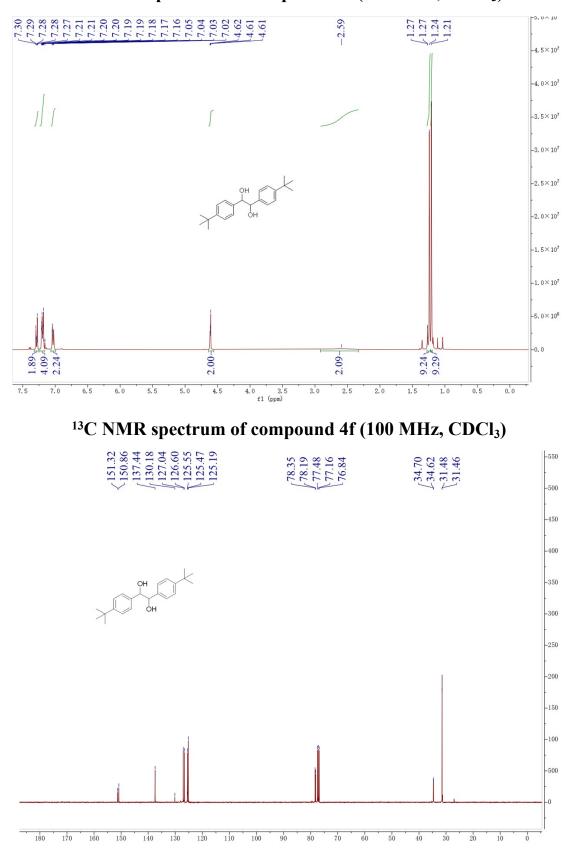
¹³C NMR spectrum of compound 4c (100 MHz, CDCl₃)



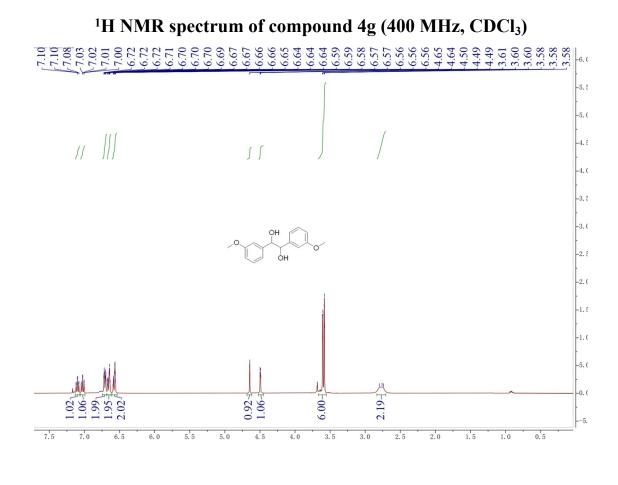




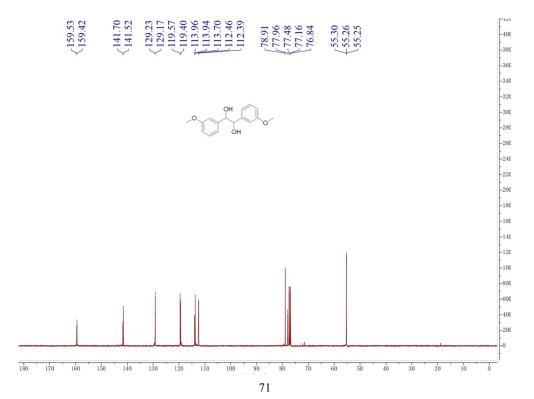
¹H NMR spectrum of compound 4e (400 MHz, CDCl₃)

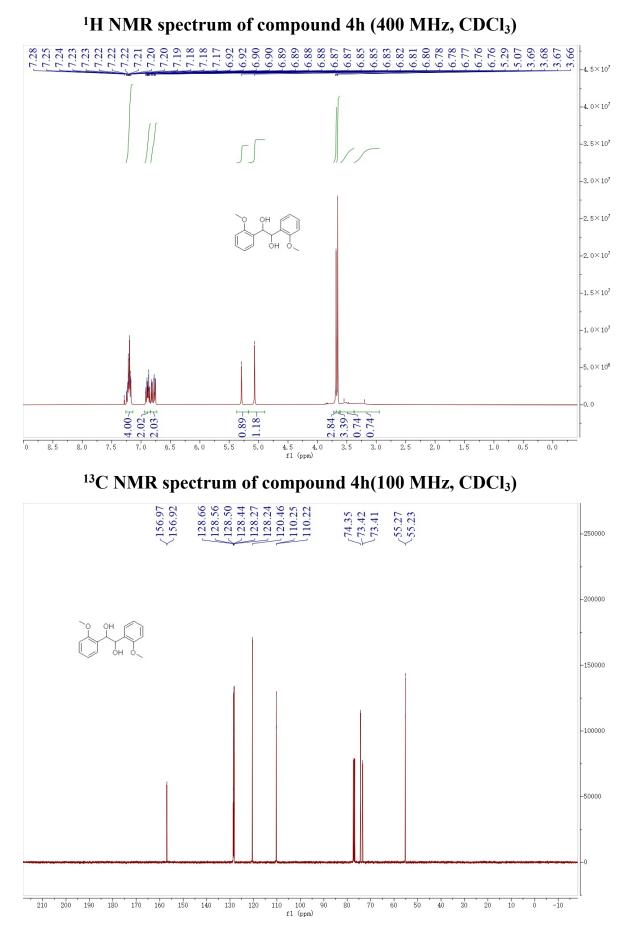


¹H NMR spectrum of compound 4f (400 MHz, CDCl₃)

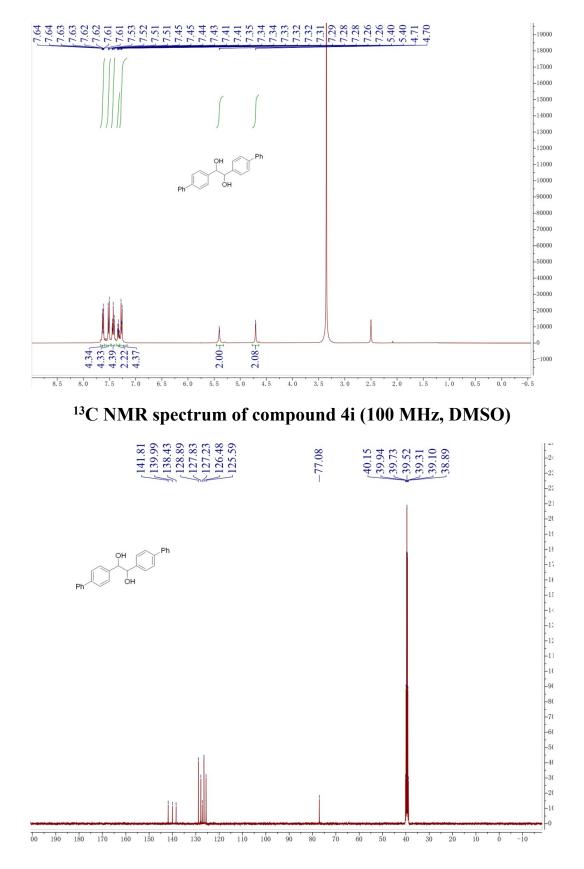


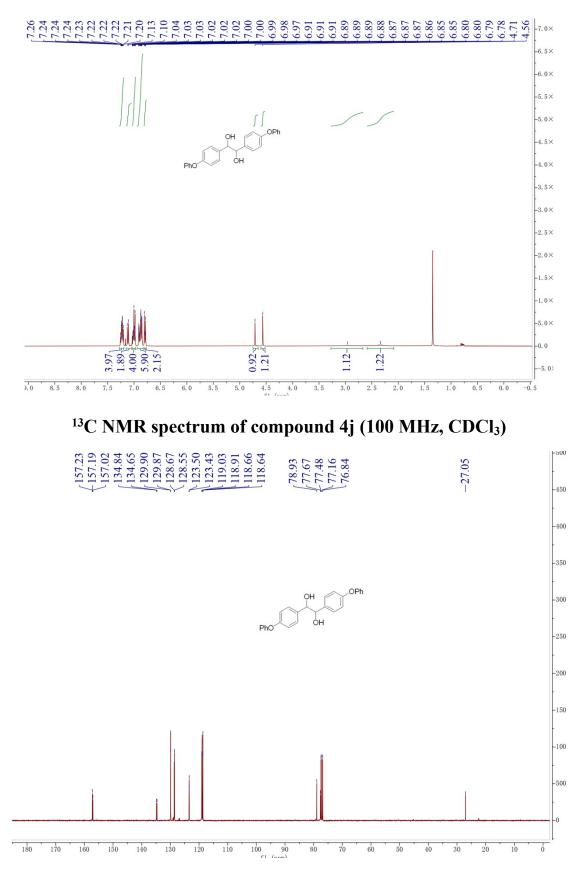
¹³C NMR spectrum of compound 4g (100 MHz, CDCl₃)



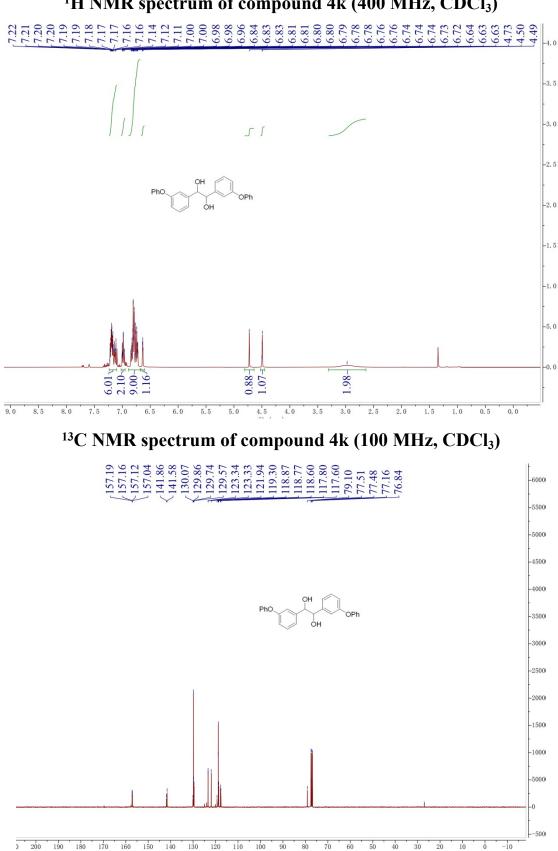




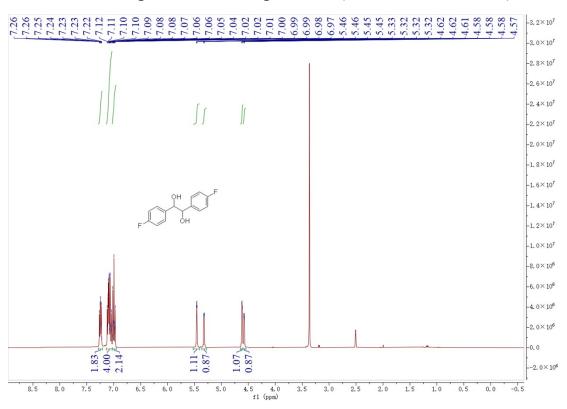




¹H NMR spectrum of compound 4j (400 MHz, CDCl₃)

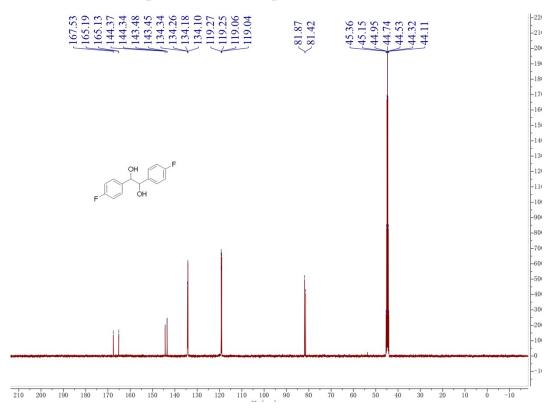


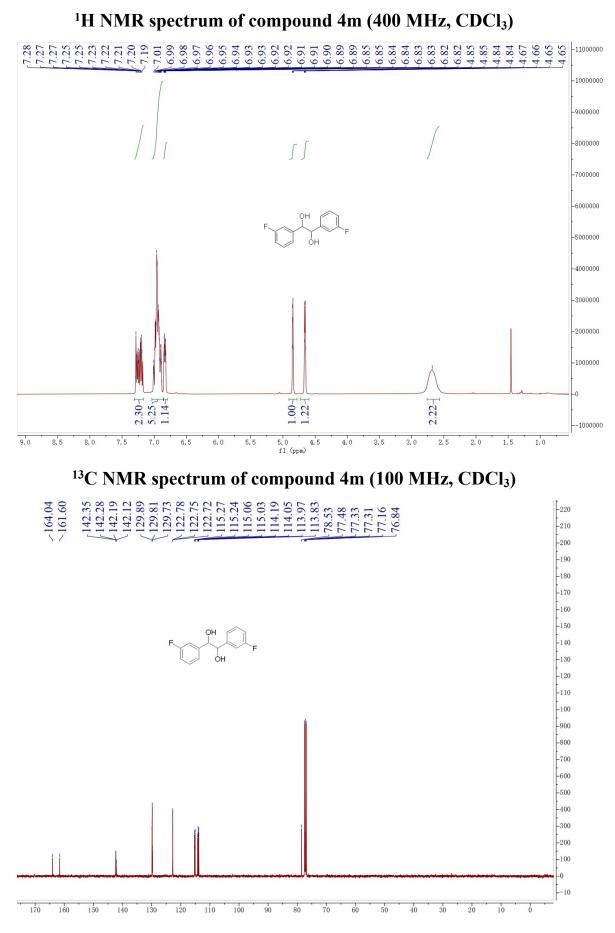
¹H NMR spectrum of compound 4k (400 MHz, CDCl₃)

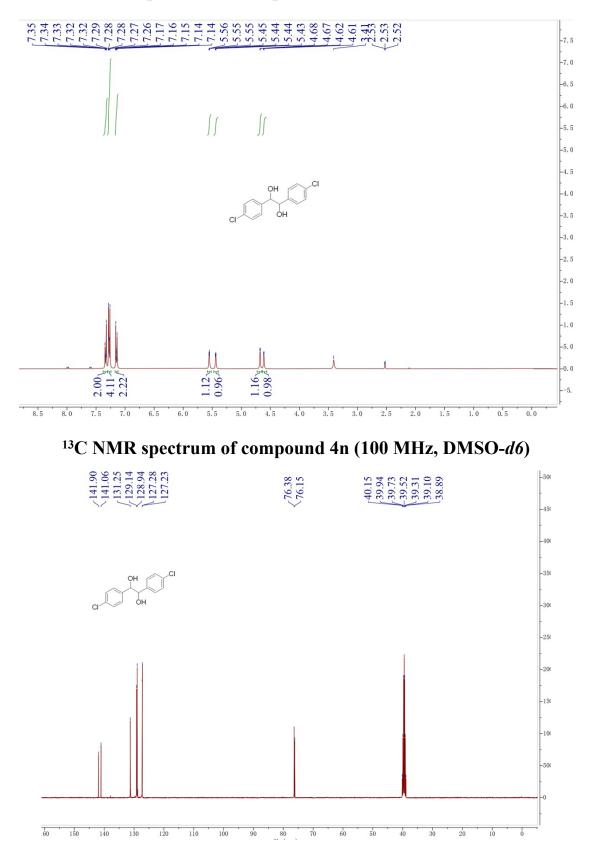


¹H NMR spectrum of compound 4l (400 MHz, DMSO-*d6*)

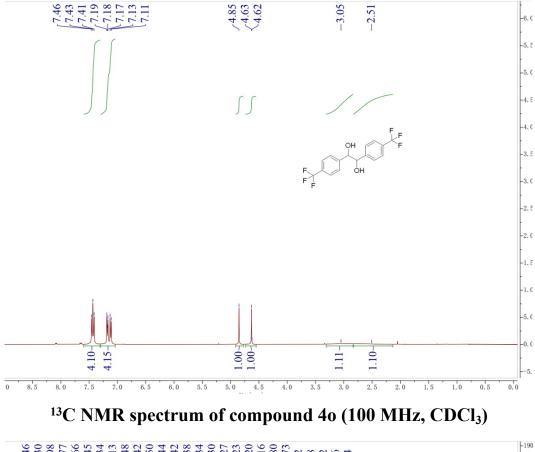
¹³C NMR spectrum of compound 4l (100 MHz, CDCl₃)





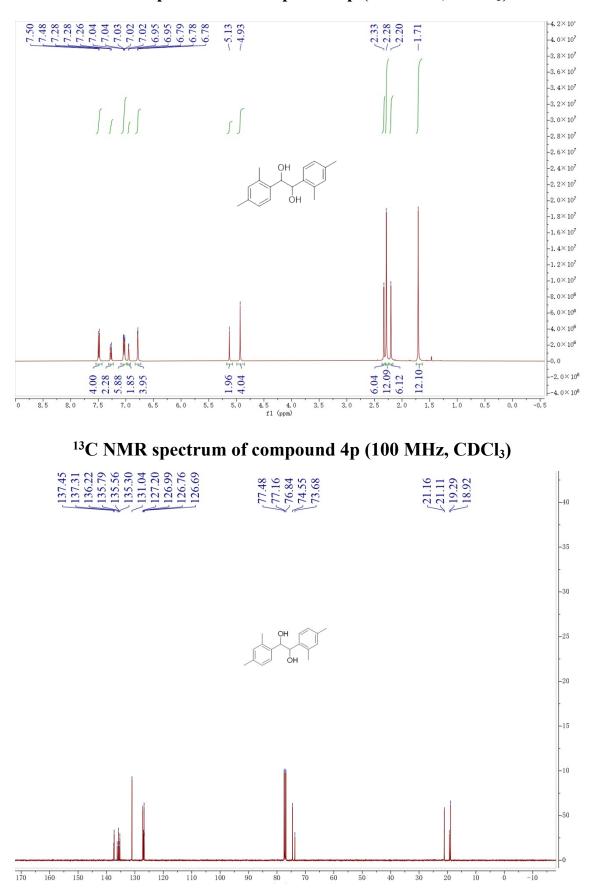


¹H NMR spectrum of compound 4n (400 MHz, DMSO-*d6*)

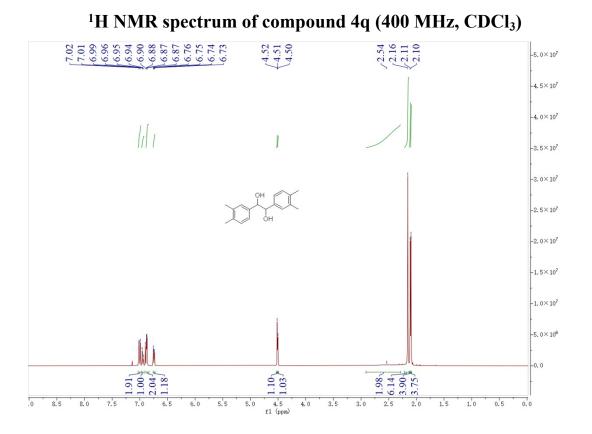


¹H NMR spectrum of compound 40 (400 MHz, CDCl₃)

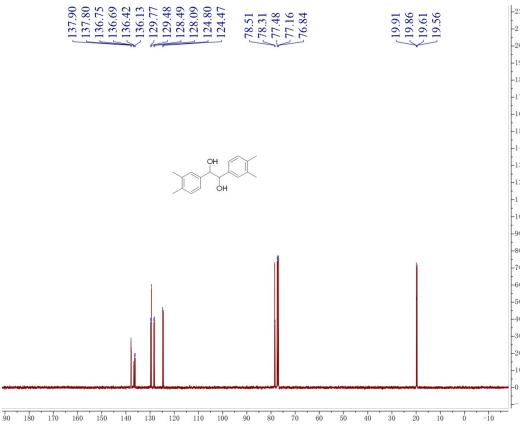
 $\begin{array}{c} 143.46\\ 143.30\\ 130.56\\ 130.56\\ 130.56\\ 130.56\\ 130.56\\ 130.54\\ 120.48\\ 120.48\\ 120.48\\ 120.48\\ 120.53\\$ -180 -170 -160 -150 -140 -130 OH F F F -120 -110 -100 -900 -800 -700 -600 -500 -400 -300 -200 -100 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

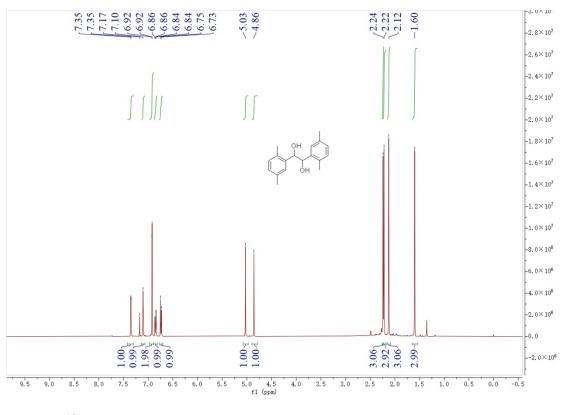


¹H NMR spectrum of compound 4p (400 MHz, CDCl₃)



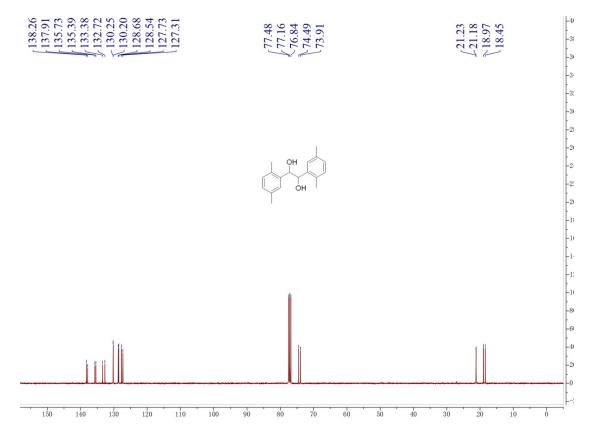
¹³C NMR spectrum of compound 4q (100 MHz, CDCl₃)

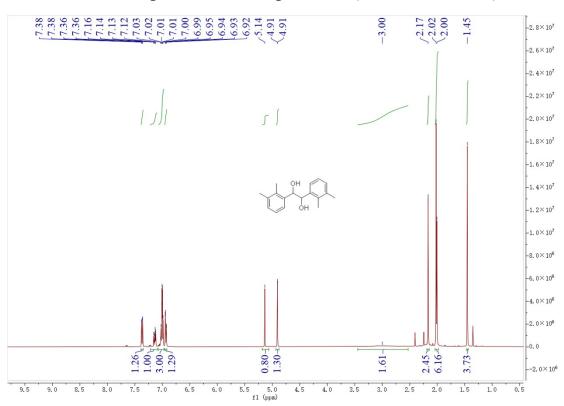




¹H NMR spectrum of compound 4r (400 MHz, CDCl₃)

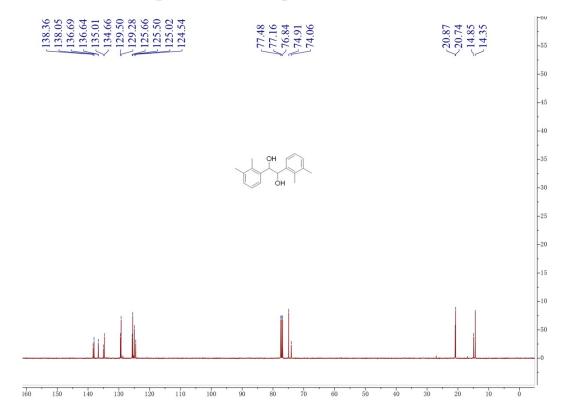
¹³C NMR spectrum of compound 4r (100 MHz, CDCl₃)

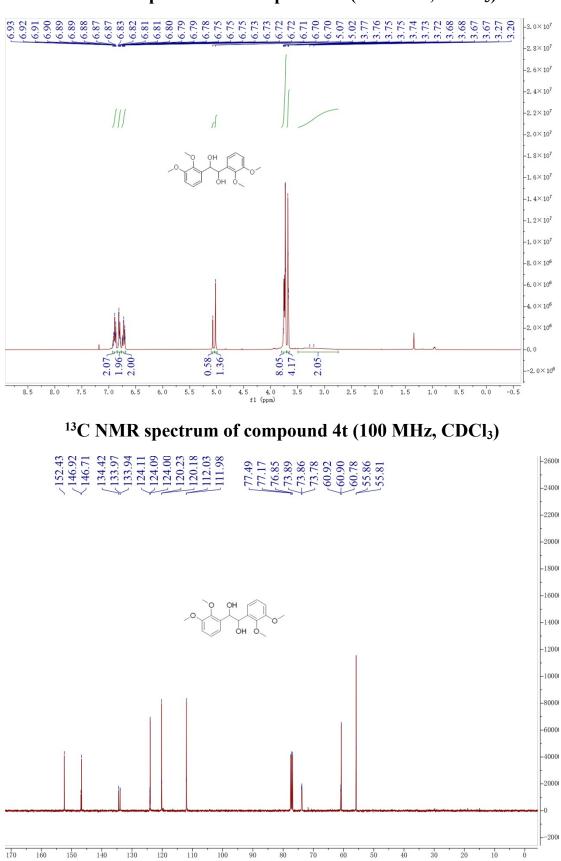




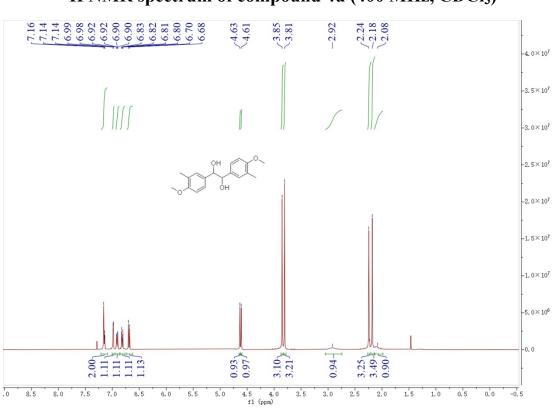
¹H NMR spectrum of compound 4s (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 4s(100 MHz, CDCl₃)



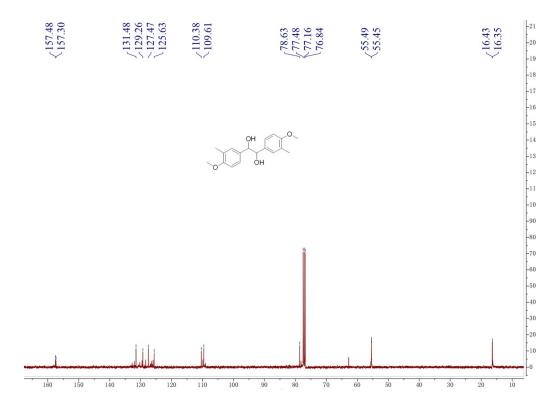


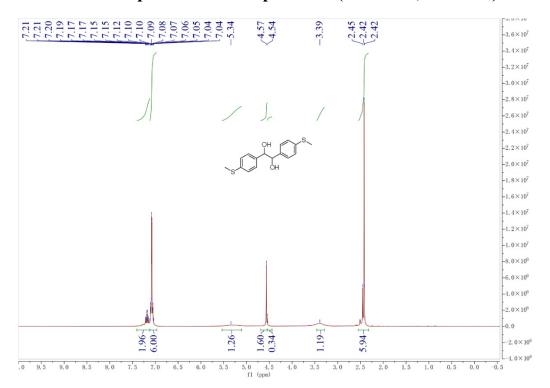
¹H NMR spectrum of compound 4t (400 MHz, CDCl₃)



¹H NMR spectrum of compound 4u (400 MHz, CDCl₃)

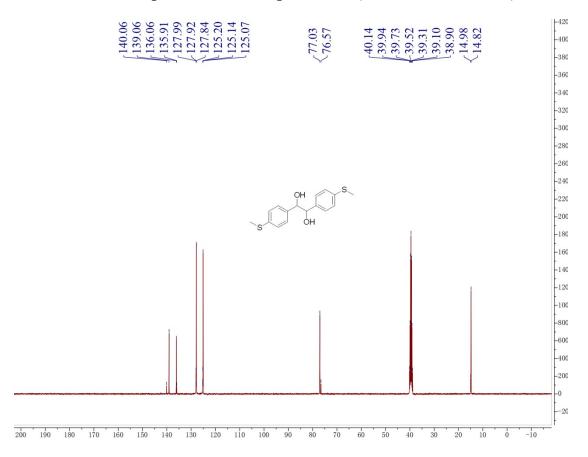
¹³C NMR spectrum of compound 4u (100 MHz, CDCl₃)

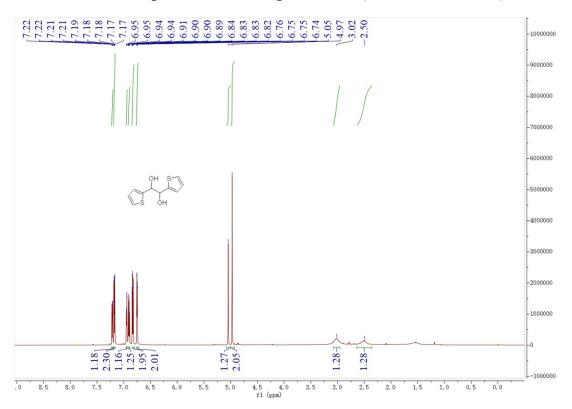




¹H NMR spectrum of compound 4v(400 MHz, DMSO-*d*₆)

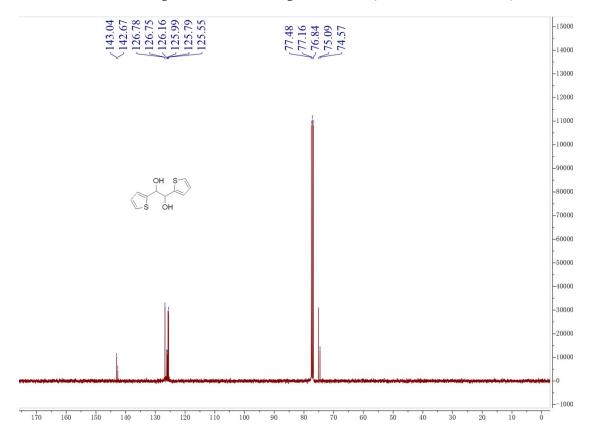
¹³C NMR spectrum of compound 4v (100 MHz, DMSO-*d*₆)

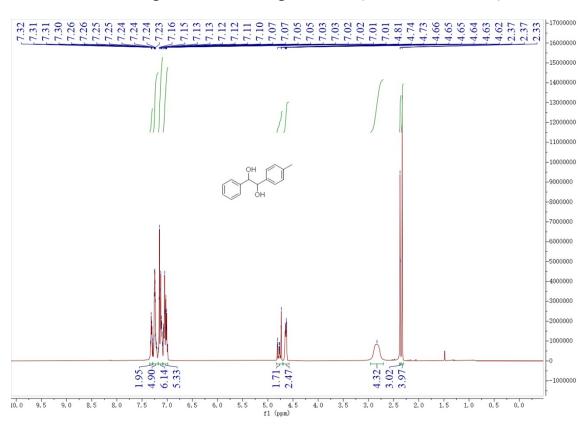




¹H NMR spectrum of compound 4w (400 MHz, CDCl₃)

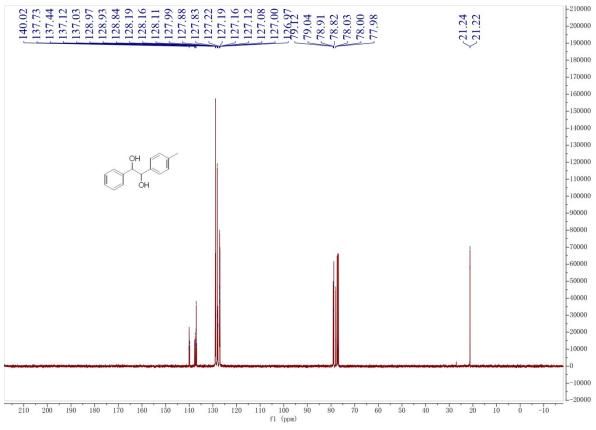
¹³C NMR spectrum of compound 4w (100 MHz, CDCl₃)

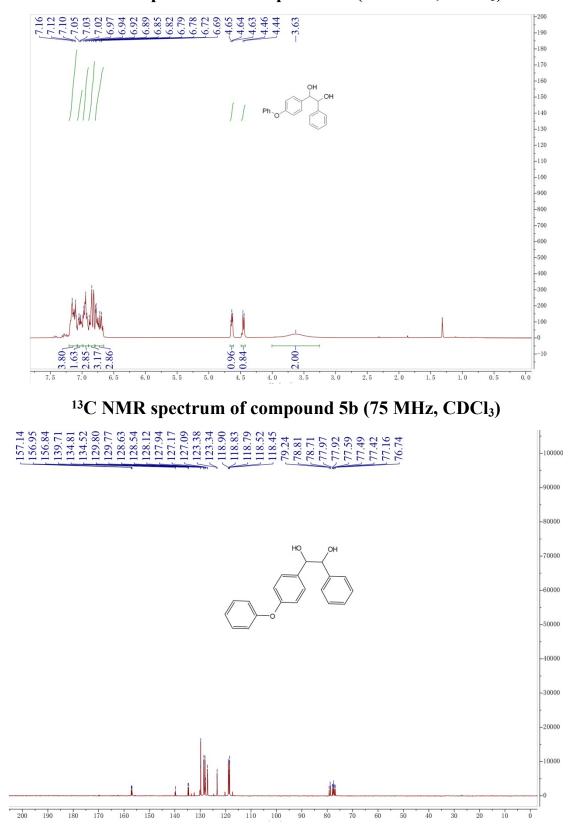




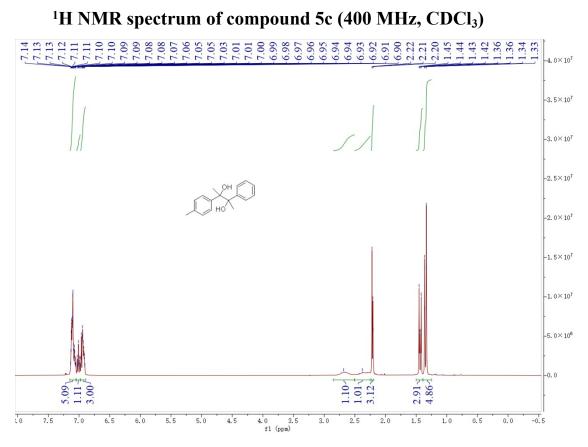
¹H NMR spectrum of compound 5a (400 MHz, CDCl₃)

¹³C NMR spectrum of compound 5a (100 MHz, CDCl₃)

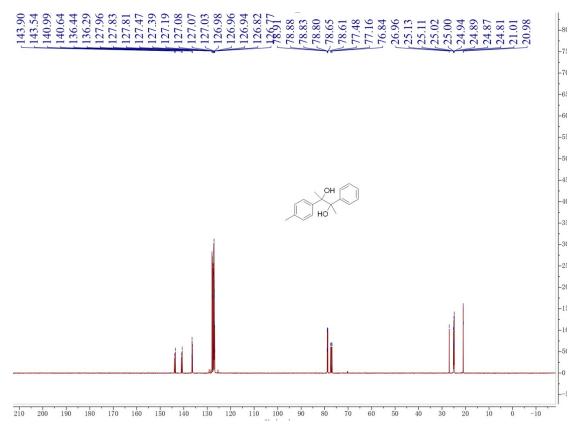


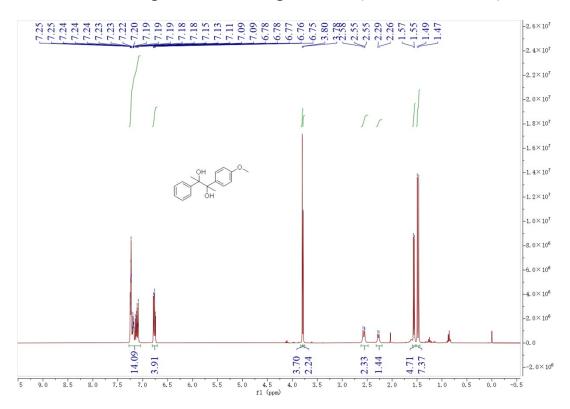


¹H NMR spectrum of compound 5b (300 MHz, CDCl₃)



¹³C NMR spectrum of compound 5c (100 MHz, CDCl₃)

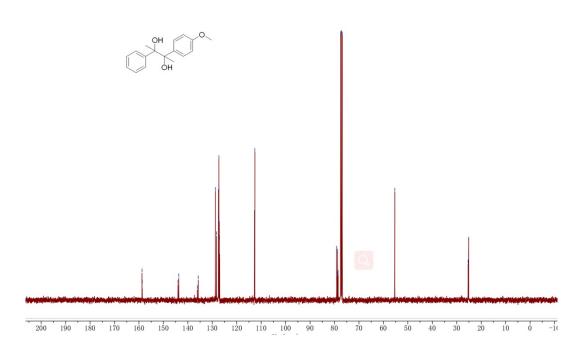


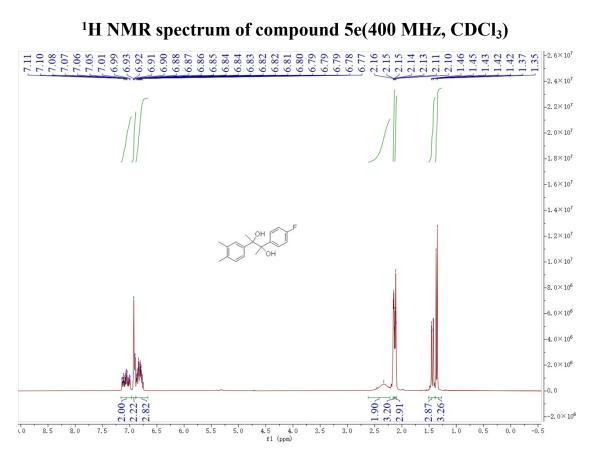


¹H NMR spectrum of compound 5d (400 MHz, CDCl₃)

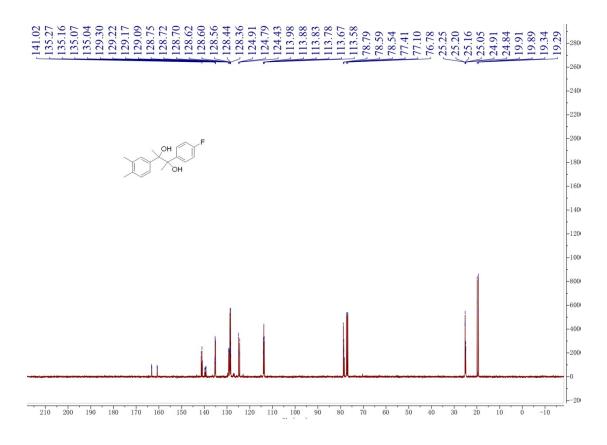
¹³C NMR spectrum of compound 5d (100 MHz, CDCl₃)

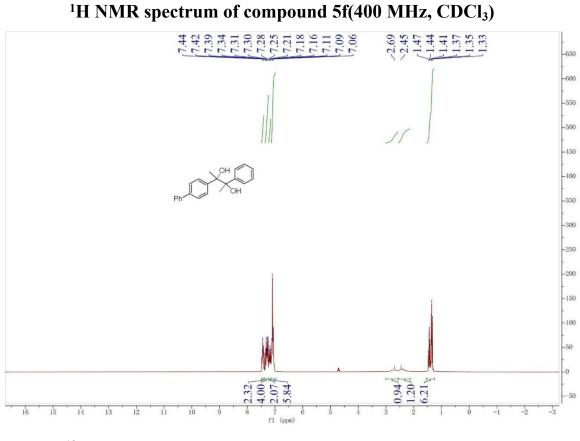
58.74 58.64 44.05 35.74 35.74 35.74 35.74 227.12 27.12	9.13 8.83 8.72 8.53 7.48 7.16 5.34 5.34	5.36 5.23 5.15 5.10
	-5, -2, -2, -2, -2, -2, -2, -2, -2, -2, -2	5555



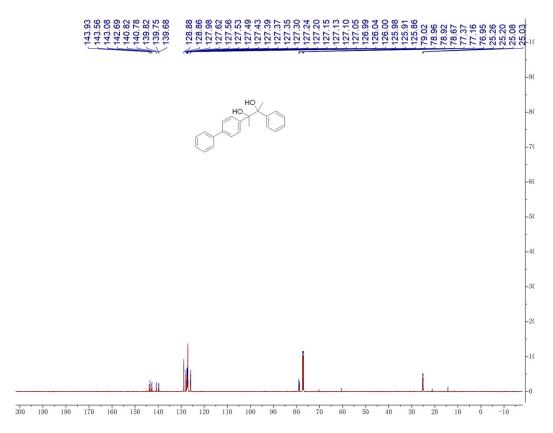


¹³C NMR spectrum of compound 5e (100 MHz, CDCl₃)

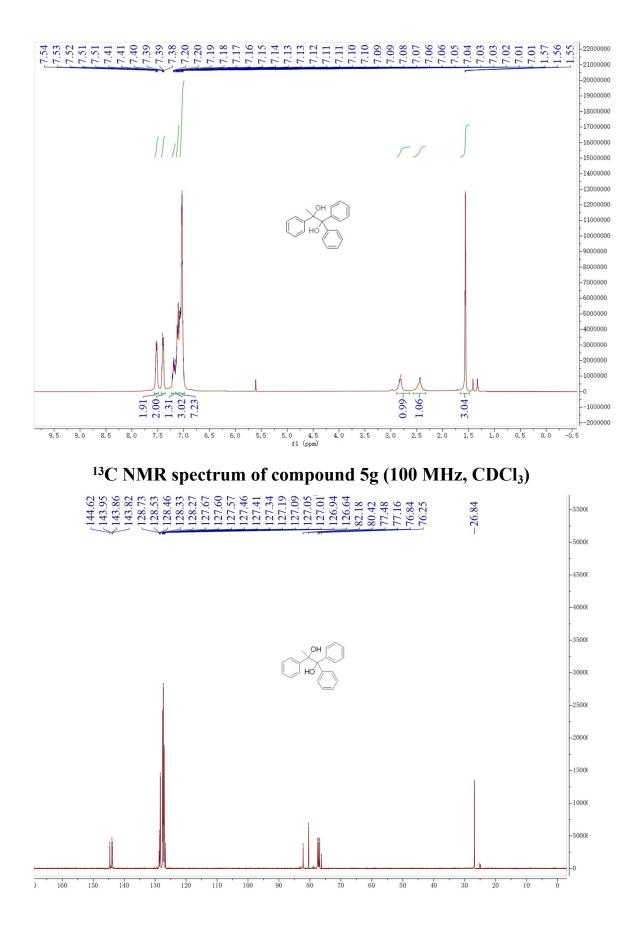




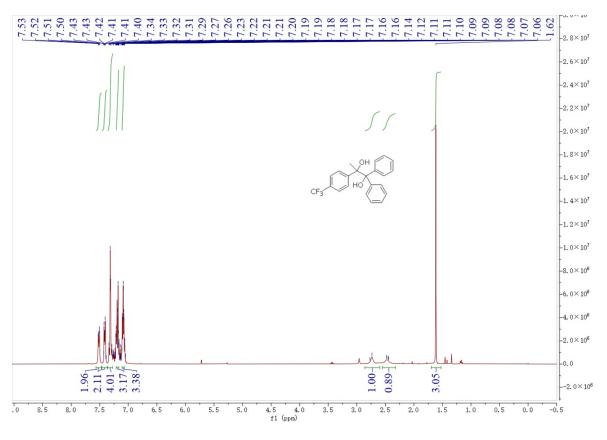
¹³C NMR spectrum of compound 5f (150 MHz, CDCl₃)



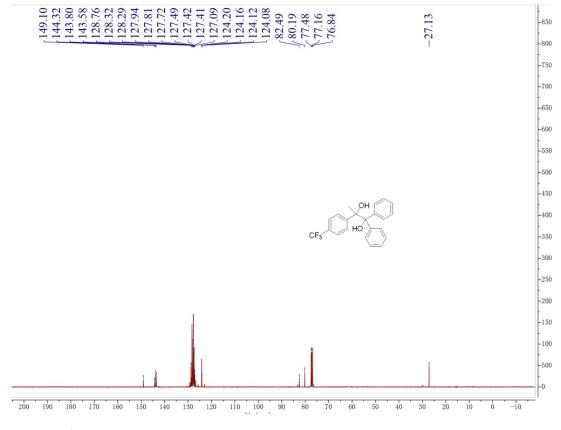
¹H NMR spectrum of compound 5g (400 MHz, CDCl₃)



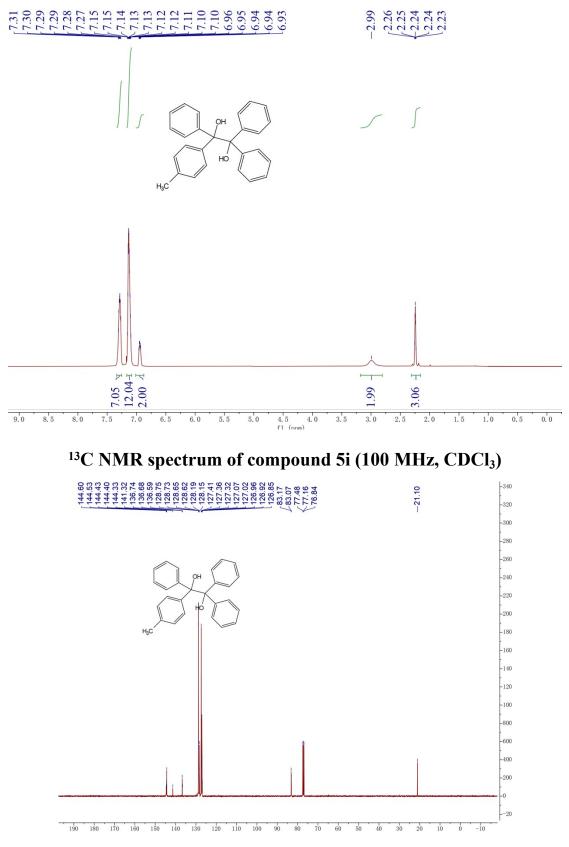
¹H NMR spectrum of compound 5h (400 MHz, CDCl₃)



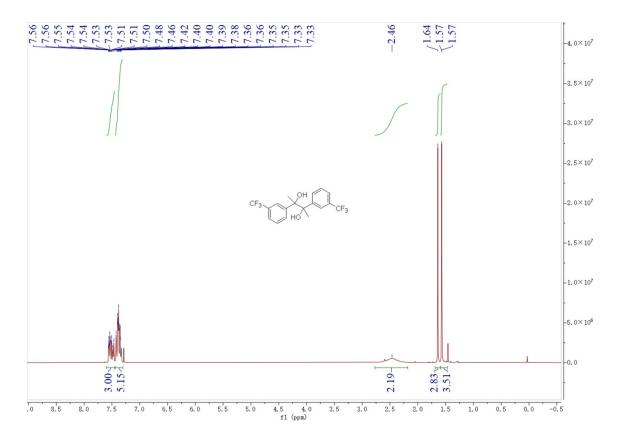
¹³C NMR spectrum of compound 5h (100 MHz, CDCl₃)



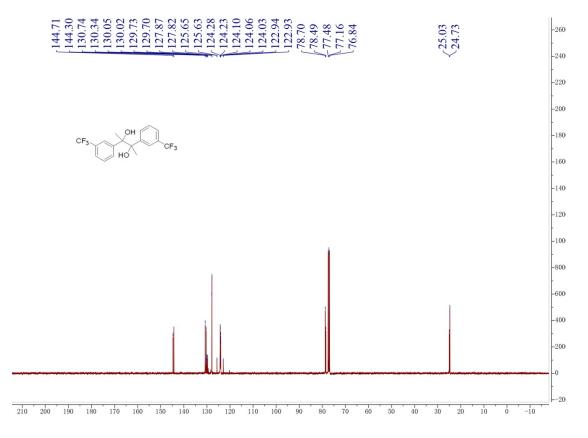
¹H NMR spectrum of compound 5i (400 MHz, CDCl₃)



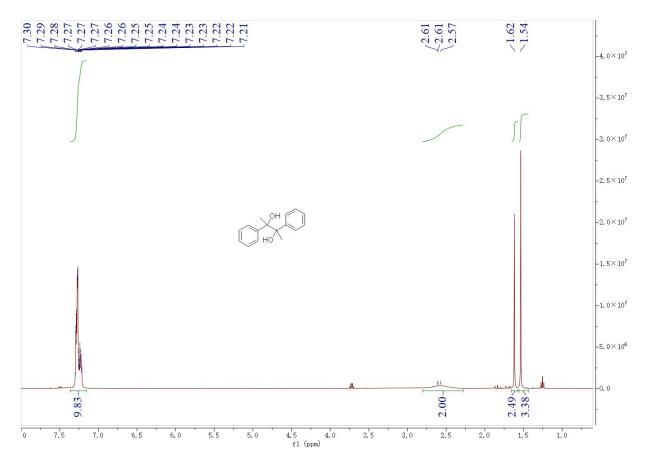
¹H NMR spectrum of compound 6a (400 MHz, CDCl₃)



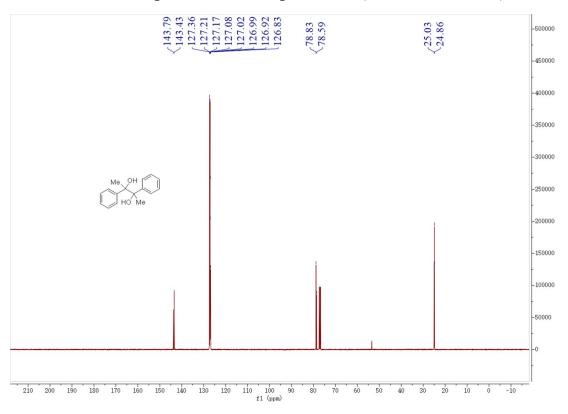
¹³C NMR spectrum of compound 6a (100 MHz, CDCl₃)



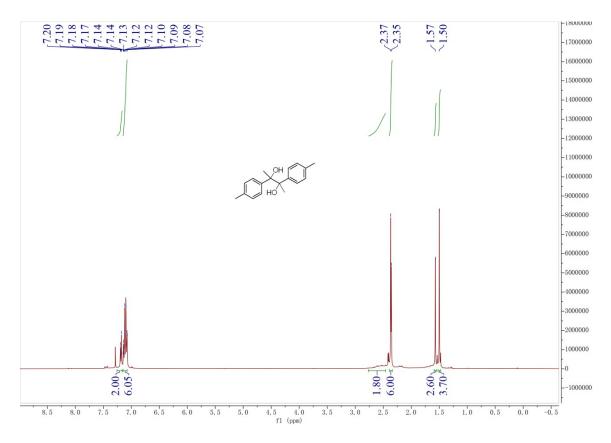
¹H NMR spectrum of compound 6b (400 MHz, CDCl₃)



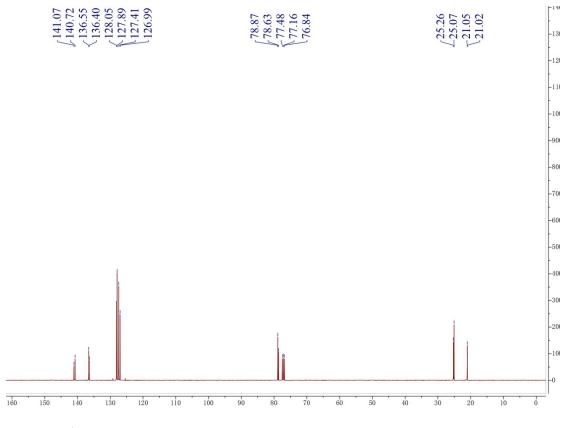
¹³C NMR spectrum of compound 6b (100 MHz, CDCl₃)



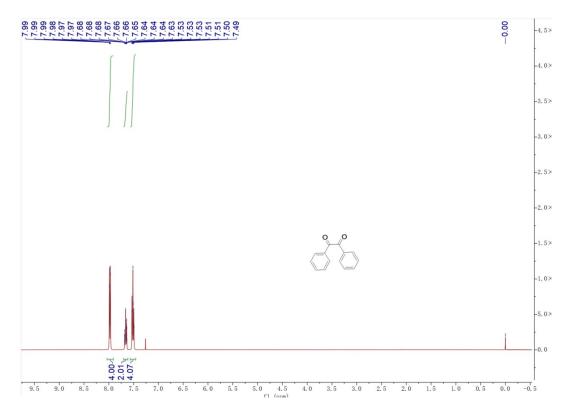
¹H NMR spectrum of compound 6c (400 MHz, CDCl₃)



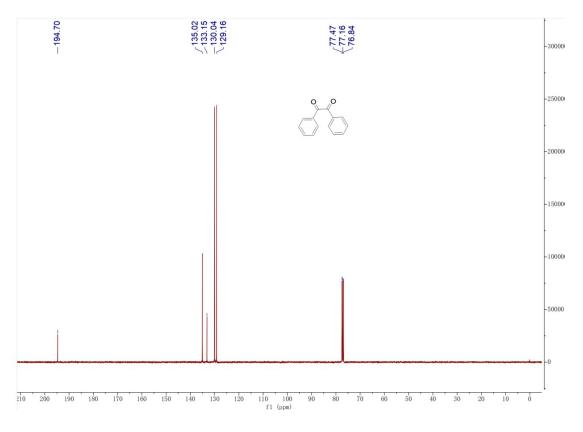
¹³C NMR spectrum of compound 6c (100 MHz, CDCl₃)



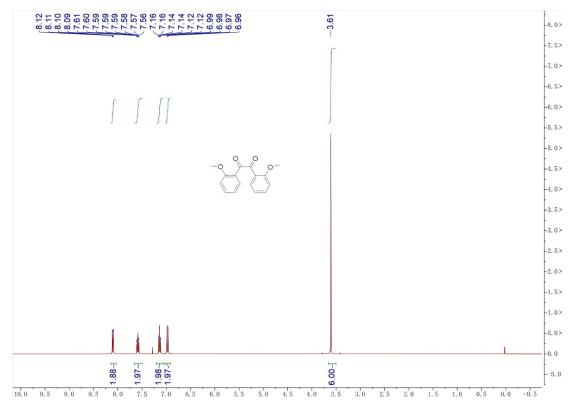
¹H NMR spectrum of compound7a (400 MHz, CDCl₃)



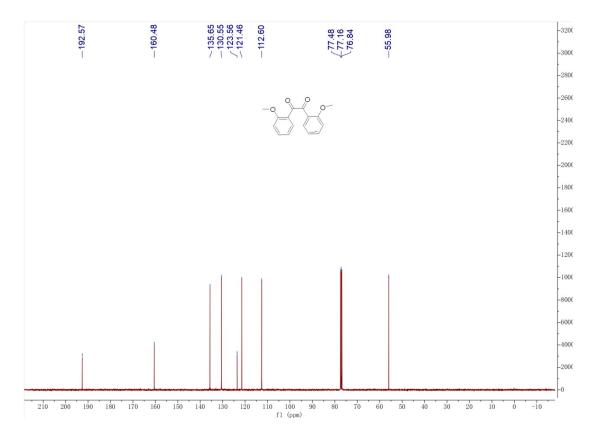
¹³C NMR spectrum of compound 7a (100 MHz, CDCl₃)



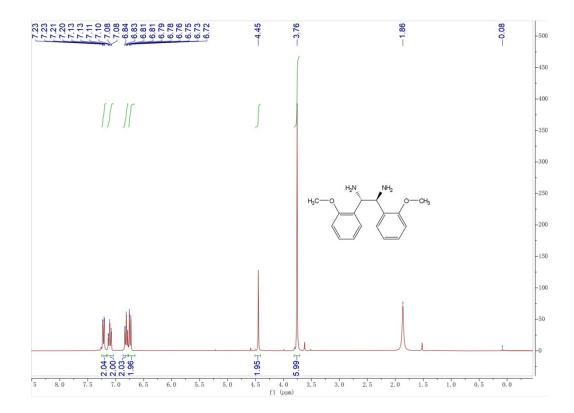
¹H NMR spectrum of compound 7b (400 MHz, CDCl₃)



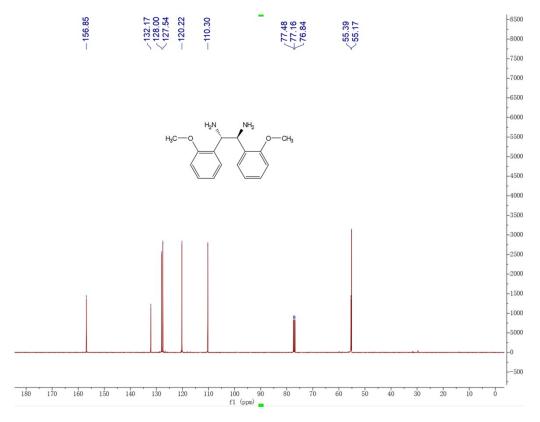
¹³C NMR spectrum of compound 7b (100 MHz, CDCl₃)



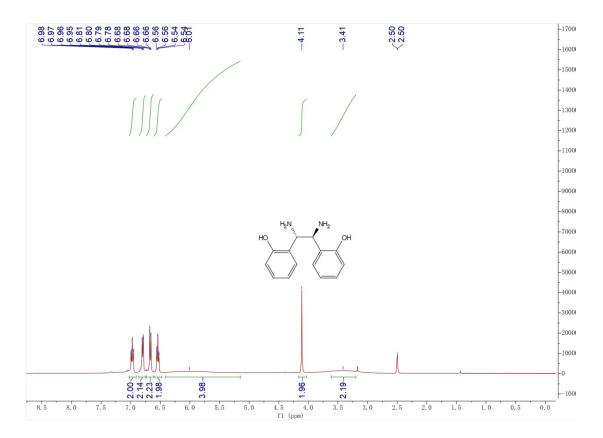
¹H NMR spectrum of compound 8 (300 MHz, CDCl₃)



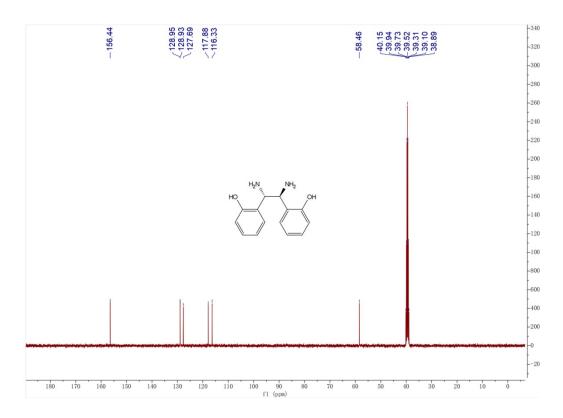
¹³C NMR spectrum of compound 8 (100 MHz, CDCl₃)



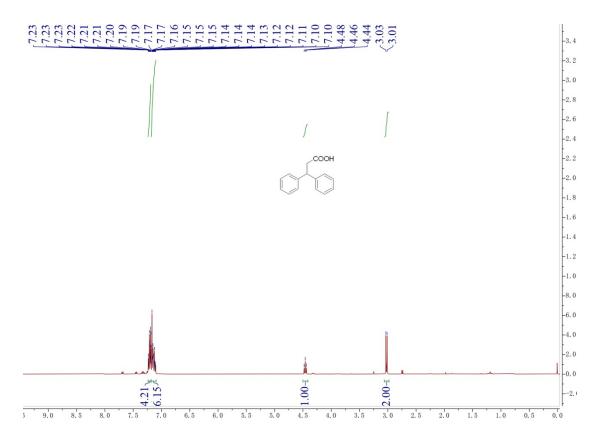
¹H NMR spectrum of compound 9 (400 MHz, CDCl₃)



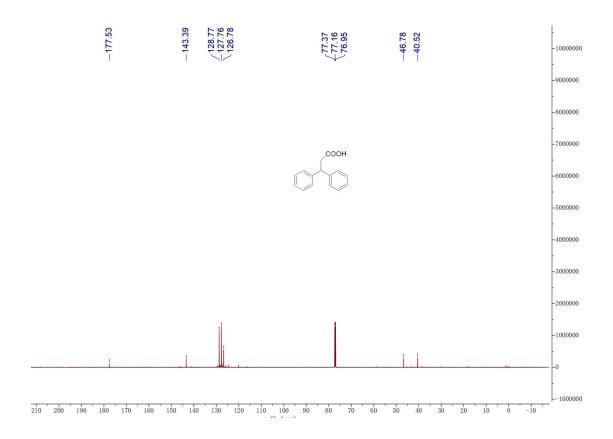
¹³C NMR spectrum of compound 9 (100 MHz, CDCl₃)



¹H NMR spectrum of compound 11 (400 MHz, CDCl₃)



¹³C NMR spectrum of compound 11 (150 MHz, CDCl₃)



18. Reference

 Nakajima, M.; Fava, E.; Loescher, S.; Jiang, Z.; Rueping, M., Photoredox-Catalyzed 1. Nakajima, M.; Fava, E.; Loescher, S.; Jiang, Z.; Rueping, M., *Angewandte Chemie International Edition* 2015, 54, 8828-8832.

2. Kronenwetter, H.; Husek, J.; Etz, B.; Jones, A.; Manchanayakage, R., *Green Chem.* 2014, *16*, 1489-1495.

3. Liu, C.; Li, R.; Zhou, W.; Liang, Y.; Shi, Y.; Li, R.-L.; Ling, Y.; Yu, Y.; Li, J.; Zhang, B., *ACS Catalysis* **2021**, *11*, 8958-8967.

4. Speckmeier, E.; Fischer, T. G.; Zeitler, K., *Journal of the American Chemical Society* **2018**, *140*, 15353-15365.

5. Ito, H.; Sudo, A., *Results in Chemistry* **2021**, *3*, 100123.

6. Caron, A.; Morin, É.; Collins, S. K., ACS Catalysis 2019, 9, 9458-9464.

7. Yoshimura, A.; Saeki, T.; Nomoto, A.; Ogawa, A., Tetrahedron 2015, 71, 5347-5355.

8. Qiu, Z.; Pham, H. D. M.; Li, J.; Li, C.-C.; Castillo-Pazos, D. J.; Khaliullin, R. Z.; Li, C.-J., *Chemical Science* **2019**, *10*, 10937-10943.

9. Liu, M.; Tan, L.; Rashid, R. T.; Cen, Y.; Cheng, S.; Botton, G.; Mi, Z.; Li, C.-J., *Chemical Science* **2020**, *11*, 7864-7870.

10. Wang, H.; Qu, J.-P.; Kang, Y.-B., Org. Lett. 2021, 23, 2900-2903.

11. Yang, Q.; Li, X.; Tang, J., Materials Today Energy 2022, 23, 100890.

12. Husain, S. M.; Stillger, T.; Dünkelmann, P.; Lödige, M.; Walter, L.; Breitling, E.; Pohl, M.; Bürchner, M.; Krossing, I.; Müller, M.; Romano, D.; Molinari, F., *Advanced Synthesis & Catalysis* **2011**, *353*, 2359-2362.

13. Nador, F.; Mascaró, E.; Castro, M.; Vitale, C.; Radivoy, G., *ARKIVOC* 2011, 2011, 312-326.

14. Yasui, M.; Hanaya, K.; Sugai, T.; Higashibayashi, S., *RSC Adv.* 2021, *11*, 24652-24655.
15. Zhang, H.; Feng, D.; Sheng, H.; Ma, X.; Wan, J.; Tang, Q., *RSC Advances* 2014, *4*, 6417-6423.