

Supporting information

Direct Metal-Free Homo-/Cross-Coupling of Carbonyls with Alcohols or Imines under Ambient Light

Tongyu Han,[†]+ Shiqian Zhang,[†]+ Jinya Li,[†]+ Quansheng Mou,[†] Jian Gao,[†] Bowen Cao,[†] Chao-Jun Li^{*‡}
and Mingxin Liu^{*†}

[†]State Key Laboratory of Applied Organic Chemistry, College of Chemistry and Chemical Engineering,
Lanzhou University, Lanzhou 730000, China

[‡]Department of Chemistry, FQRNT Centre for Green Chemistry and Catalysis, McGill University,
Montreal, Quebec H3A 0B8, Canada

+These authors contributed equally

Table of Contents

1.General information.....	S2
2.Optimization of the reaction conditions.....	S3
3.General procedure A for the synthesis of 1,2-diols 2a–2o	S5
4.General Procedure B for the synthesis of 1,2-diols 2p–2ab	S10
5.General procedure for the synthesis of 1,2-diols 4 and 5	S15
6.General Procedure for the synthesis of β -amino alcohols 7a–7s	S26
7.Radical trapping experiments.....	S33
8.Cyclic voltammetry analysis.....	S36
9.UV-vis studies.....	S36
10.Gram-scale synthesis.....	S37
11.Computational details.....	S37
12.Comparison with previous work.....	S40
13.References.....	S40
14.Copies of NMR Spectra for the products.....	S42

1. General Information

All reactions were carried out under an argon atmosphere in a flame-dried quartz tube with magnetic stirring. Petroleum ether, ethyl acetate and other solvents were dried and purified according to the procedure from “Purification of Laboratory Chemicals”.¹ The reactions were monitored by TLC analysis using silica gel GF-254 thin layer plates and compounds were visualized with a UV light at 254 nm. All products were purified by flash chromatography on silica gel. The chemical yields referred are isolated products. ¹H and ¹³C spectra were collected on a Bruker AVANCE III 400MHz and JEOL JNM-ECS 400MHz at room temperature. Chemical shifts (δ) are expressed in ppm downfield from TMS as internal standard. The letters s, d, t, q, and m are used to indicate singlet, doublet, triplet, quadruplet, and multiple, respectively. ¹⁹F NMR spectra were collected on Bruker AVANCE III 400 MHz spectrometers at room temperature. HRMS was performed on Bruker Apex II FT-ICR mass instrument (ESI) and Waters GCT Premier TOFMS (EI).

The equipment of light-reaction is a multi-channel photoreactor with 10 W purple light (395–400 nm, composed of 2 LED units in series, manufacturer: Shanghai Yukang Science and Education Instrument and Equipment company, wavelength of peak intensity: 397.7 nm). 10 W UVA LED (365–370 nm, composed of 2 LED units in series, manufacturer: Shanghai Yukang Science and Education Instrument and Equipment company, wavelength of peak intensity: 367.2 nm).

2. Optimization of reaction conditions.

Table S1. The optimization of reaction conditions for aldehyde homo-coupling.^a

Entry	Solvent	Light	Yield (%) ^b
1	MeOH	365–370 nm	61
2	EtOH	365–370 nm	63
3	<i>i</i> -PrOH	365–370 nm	98
4	MeCN	365–370 nm	trace
5	toluene	365–370 nm	21
6	DCM	365–370 nm	trace
7	DCE	365–370 nm	trace
8	DME	365–370 nm	trace
9	<i>i</i> -PrOH	395–400 nm	98
10	<i>i</i> -PrOH	425–430 nm	N.R.
11 ^c	<i>i</i> -PrOH	395–400 nm	87
12	<i>i</i> -PrOH	dark	N.R.

^aReaction condition: Benzaldehyde (0.2 mmol), solvent (2 mL), under argon atmosphere and room temperature for 8 h. ^bIsolated yield. ^cUnder air atmosphere. Dark = no light. N.R. = no reaction.

Table S2. Optimization of reaction conditions for aldehydes and alcohols.^a

Entry	Solvent	Light	Yield (%) ^b
1	EtOAc	365–370 nm	53
2	CHCl ₃	365–370 nm	trace
3	DCE	365–370 nm	trace
4	DCM	365–370 nm	trace
5	benzotrifluoride	365–370 nm	trace
6	DMF	365–370 nm	31
7	MeCN	365–370 nm	89
8	MeCN	395–400 nm	80
9	MeCN	425–430 nm	N.D.
10	MeCN	dark	N.D.

11 ^c	MeCN	365-370 nm	69
-----------------	------	------------	----

^aReaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), solvent (2 mL), 10 W LED, argon atmosphere, room temperature, 36 h. ^bIsolated yield. ^cUnder air atmosphere. Dark= without light. N.D.= No detected.

Table S3. Optimization of reaction conditions for aldehydes and imines.^a

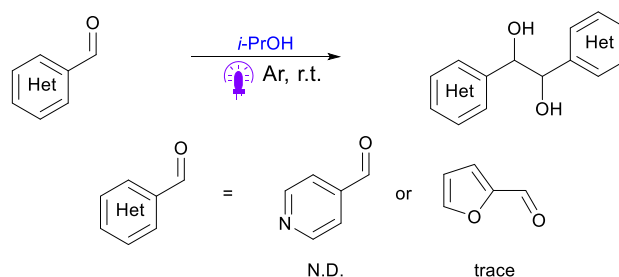
Entry	Solvent	Light	Reductant	Yield (%)
1	MeCN	365-370 nm	<i>i</i> -Pr ₂ NEt	40 ^b
2	MeCN	395-400 nm	<i>i</i> -Pr ₂ NEt	N.D.
3	MeOH	365-370 nm	<i>i</i> -Pr ₂ NEt	26 ^c
4	DME	365-370 nm	<i>i</i> -Pr ₂ NEt	39 ^c
5	1,4-dioxane	365-370 nm	<i>i</i> -Pr ₂ NEt	35 ^c
6 ^d	MeCN/H ₂ O	365-370 nm	<i>i</i> -Pr ₂ NEt	59 ^b
7 ^d	MeCN/H ₂ O	365-370 nm	Et ₃ N	36 ^b
8 ^d	MeCN/H ₂ O	365-370 nm	HCOOK	trace
9 ^d	MeCN/H ₂ O	365-370 nm	NaHSO ₃	trace
10 ^d	MeCN/H ₂ O	365-370 nm	<i>N,N</i> -Dicyclohexylmethylamine	49 ^b
11 ^e	MeCN/H ₂ O	365-370 nm	<i>i</i> -Pr ₂ NEt	60 ^b
12 ^{e,f}	MeCN/H ₂ O	365-370 nm	<i>i</i> -Pr ₂ NEt	72 ^b
13 ^{e,f,g}	MeCN/H ₂ O	365-370 nm	<i>i</i> -Pr ₂ NEt	N.D.
14 ^{e,f}	MeCN/H ₂ O	dark	<i>i</i> -Pr ₂ NEt	N.D.

^aReaction conditions: **1a** (0.3 mmol), **6a** (0.2 mmol), solvent (2 mL), 10 W LEDs, argon atmosphere, room temperature, 24 h. ^bIsolated yield. ^cThe yields were carried by ¹H NMR, Mesitylene (5 μL) as an internal standard.

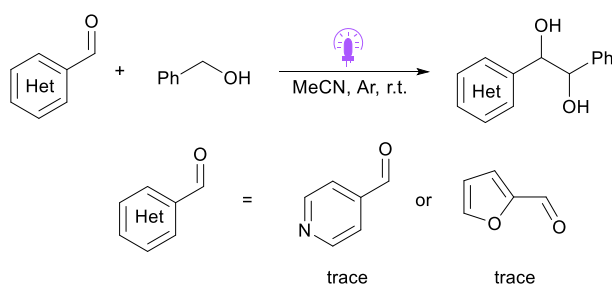
^dSolvent MeCN = 2 mL, H₂O = 0.1 mL. ^e**1a** (0.4 mmol). ^fSolvent MeCN = 0.5 mL, H₂O = 0.05 mL. ^gUnder air atmosphere. Dark= without light. N.D.= No detected.

Table S4. Other substrates

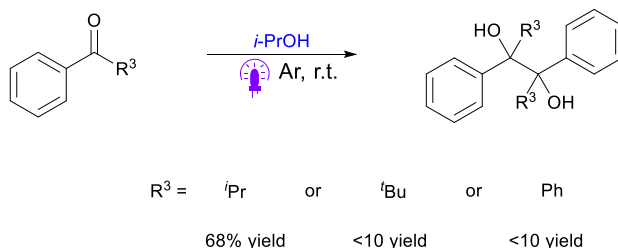
When heteroaromatic aldehydes or ketones are used as substrates, the corresponding products are detectable only in trace amounts.



Reaction condition: aldehydes (0.2 mmol), ⁱPrOH (2 mL), under argon atmosphere and room temperature for 8 h. N.D. = no detected.

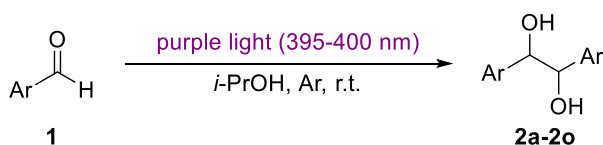


Reaction conditions: aldehydes (0.2 mmol), Benzyl alcohol (0.3 mmol), MeCN (2 mL), 10 W LED (365-370 nm), argon atmosphere, room temperature, 36 h. N.D.= No detected.



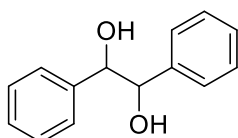
Reaction condition: ketones (0.2 mmol), *i*-PrOH (2 mL), under argon atmosphere and room temperature for 8 h.

3. General Procedure A for the synthesis of 1,2-diols 2a–2o.



In an argon-filled glovebox, a 10 mL flame-dried quartz tube with magnetic stirring was charged sequentially with *i*-PrOH (2 mL) and substrates **1** (0.2 mmol) were sequentially added into the quartz tube. Then, the quartz tube was removed from glovebox. The mixture was stirred at room temperature under 10 W purple light (395–400 nm) until the reaction was completed, as monitored by TLC analysis. The reaction mixture was then concentrated in vacuum. The crude product was purified by flash column chromatography (silica gel, PE/EA) to afford the desired products. The corresponding HRMS data of the known product are available in the literature: (**2a**, **2b**, **2e**, **2f**, **2g**, **2h**, **2j**, **2k**, **2m**)²; (**2c**, **2i**)¹⁹; (**2d**)²⁰, (**2l**)³.

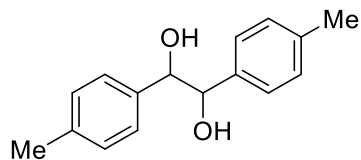
1,2-Biphenylethane-1,2-diol (**2a**)



The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 98% (21 mg) isolated yield, white solid, *dl* : *meso* = 1.02 : 1. ¹H NMR

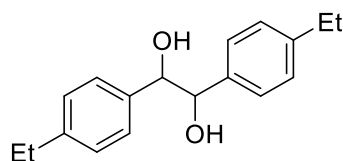
(400 MHz, CDCl₃) δ 7.31 – 7.22 (m, 8H), 7.12 – 7.10 (m, 2H), 4.81 (s, 1H), 4.68 (s, 1H), 2.95 (s, 1H), 2.29 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 139.78, 139.69, 128.20, 128.09, 127.96, 127.89, 127.05, 126.91, 79.06, 78.04.

1,2-Di-*p*-tolylethane-1,2-diol (2b)



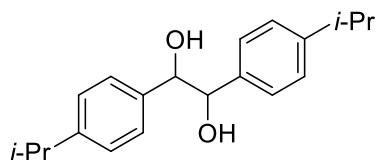
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 96% (23 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.14 (d, *J* = 7.6 Hz, 4H), 7.02 (d, *J* = 4.4 Hz, 4H), 4.71 (s, 1H), 4.62 (s, 1H), 2.92 (s, 1H), 2.34 (s, 3H), 2.29 (s, 3H), 2.19 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 137.77, 137.42, 136.91, 128.95, 128.76, 126.99, 126.81, 78.73, 77.97, 21.16, 21.13.

1,2-Bis(4-ethylphenyl)ethane-1,2-diol (2c)



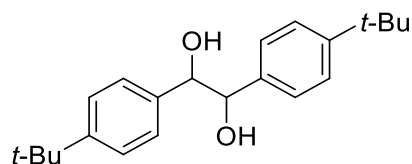
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 96% (26 mg) isolated yield, white solid, *dl* : *meso* = 1.4 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.29 – 7.15 (m, 4H), 7.08 – 7.04 (m, 4H), 4.71 (s, 1H), 4.66 (s, 1H), 2.92 (s, 1H), 2.67 – 2.57 (m, 4H), 2.22 (s, 1H), 1.25 – 1.17 (m, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 144.21, 143.81, 137.27, 137.24, 127.82, 127.59, 127.10, 126.84, 78.61, 78.06, 28.55, 28.49, 15.54, 15.51.

1,2-Bis(4-isopropylphenyl)ethane-1,2-diol (2d)



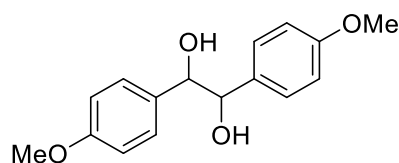
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 97% (29 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1.15. ¹H NMR (400 MHz, CDCl₃) δ 7.26 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.12 – 7.07 (m, 4H), 4.68 (d, *J* = 4.0 Hz, 2H), 2.94 – 2.81 (m, 3H), 2.10 (s, 1H), 1.25 – 1.20 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) δ 148.87, 148.41, 137.55, 137.51, 127.12, 126.74, 126.44, 126.15, 78.39, 78.07, 33.82, 33.72, 23.95, 23.92.

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



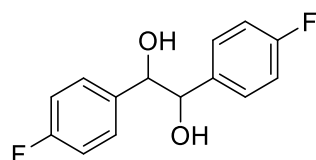
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 98% (32 mg) isolated yield, white solid, *dl* : *meso* = 1.07 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.37 (d, *J* = 8.4 Hz, 2H), 7.28 (dd, *J* = 8.0, 6.0 Hz, 4H), 7.11 (d, *J* = 8.4 Hz, 2H), 4.69 (d, *J* = 6.0 Hz, 2H), 2.91 (s, 1H), 2.26 (s, 1H), 1.32 (s, 9H), 1.28 (s, 9H). **¹³C NMR** (100 MHz, CDCl₃) δ 151.13, 150.64, 137.24, 137.22, 126.86, 126.41, 125.32, 125.01, 78.13, 78.00, 34.53, 34.45, 31.30, 31.29.

1,2-Bis(4-methoxyphenyl)ethane-1,2-diol (2f)



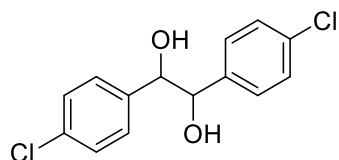
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 80% (22 mg) isolated yield, white solid, *dl* : *meso* = 2 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.17 (d, *J* = 8.4 Hz, 4H, *meso*), 7.01 (d, *J* = 8.4 Hz, 4H, *dl*), 6.84 (d, *J* = 8.4 Hz, 4H, *meso*), 6.74 (d, *J* = 8.4 Hz, 4H, *dl*), 4.71 (s, 2H, *meso*), 4.59 (s, 2H, *dl*), 3.79 (s, 6H, *meso*), 3.75 (s, 6H, *dl*), 3.02 (s, 2H, *dl*), 2.29 (s, 2H, *meso*). **¹³C NMR** (100 MHz, CDCl₃) δ 159.33, 159.07, 132.04, 131.95, 128.30, 128.14, 113.61, 113.42, 78.72, 77.70, 55.22, 55.14.

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



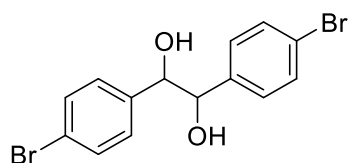
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 82% (20 mg) isolated yield, white solid, *dl* : *meso* = 1.19 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.15 – 7.12 (m, 2H), 7.05 – 6.89 (m, 6H), 4.81 (s, 1H), 4.60 (s, 1H), 3.05 (s, 1H), 2.43 (s, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 163.63 (d, *J*_{C-F} = 8.1 Hz), 161.18 (d, *J*_{C-F} = 8.1 Hz), 135.25 (d, *J*_{C-F} = 2.9 Hz), 135.10 (d, *J*_{C-F} = 3.2 Hz), 128.66 (d, *J*_{C-F} = 6.0 Hz), 128.58 (d, *J*_{C-F} = 6.0 Hz), 78.69, 77.21. **¹⁹F NMR** (376 MHz, CDCl₃) δ -113.95, -114.03.

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



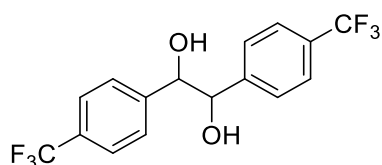
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 82% (23 mg) isolated yield, white solid, *dl* : *meso* = 1.11 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.22 (dd, *J* = 18.0, 8.4 Hz, 4H), 7.07 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 4.80 (s, 1H), 4.58 (s, 1H), 3.08 (s, 1H), 2.52 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 137.88, 133.82, 128.35, 128.33, 128.29, 128.28, 78.47, 77.09.

1,2-Bis(4-bromophenyl)ethane-1,2-diol (2i)



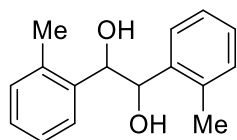
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 75% (28 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1.28. ¹H NMR (400 MHz, CDCl₃) δ 7.38 (dd, *J* = 18.4, 8.4 Hz, 4H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.94 (d, *J* = 8.0 Hz, 2H), 4.79 (s, 1H), 4.57 (s, 1H), 2.99 (s, 1H), 2.42 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 138.34, 138.20, 131.31, 131.24, 128.66, 128.63, 122.03, 78.46, 77.10.

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



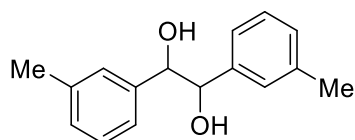
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 94% (33 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1.10. ¹H NMR (400 MHz, CDCl₃) δ 7.54 – 7.49 (m, 4H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 4.93 (s, 1H), 4.71 (s, 1H), 3.13 (s, 1H), 2.62 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 143.19, 143.04, 130.39 (q, *J*_{C-F} = 32.2 Hz), 130.27 (q, *J*_{C-F} = 32.3 Hz), 127.28, 127.23, 125.18 (q, *J*_{C-F} = 3.8 Hz), 125.02 (q, *J*_{C-F} = 3.8 Hz), 123.96 (q, *J*_{C-F} = 270.4 Hz), 123.90 (q, *J*_{C-F} = 270.4 Hz), 78.33, 77.10. ¹⁹F NMR (376 MHz, CDCl₃) δ -62.54, -62.57.

1,2-Di-o-tolyethane-1,2-diol (2k)



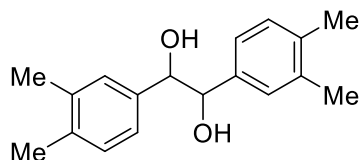
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 91% (22 mg) isolated yield, colorless oil, *dl* : *meso* = 1.25 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.60 (d, *J* = 7.6 Hz, 1H), 7.32 – 7.06 (m, 6H), 6.91 (d, *J* = 7.6 Hz, 1H), 5.17 (s, 1H), 4.94 (s, 1H), 3.05 (s, 1H), 2.30 (s, 1H), 2.15 (s, 3H), 1.63 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 138.02, 137.89, 136.08, 135.84, 130.11, 129.99, 127.70, 127.66, 127.12, 126.61, 126.02, 125.89, 74.53, 73.23, 19.13, 18.71.

1,2-Di-*m*-tolylethane-1,2-diol (2l)



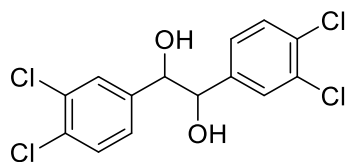
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 95% (23 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1.03. ¹H NMR (400 MHz, CDCl₃) δ 7.22 (m, 1H), 7.13 – 6.98 (m, 6H), 6.89 (d, *J* = 7.6 Hz, 1H), 4.70 (s, 1H), 4.64 (s, 1H), 2.91 (s, 1H), 2.33 (s, 3H), 2.28 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 139.8, 137.95, 137.69, 128.91, 128.55, 128.17, 127.93, 127.76, 127.42, 124.22, 123.95, 78.72, 78.17, 21.40, 21.35.

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



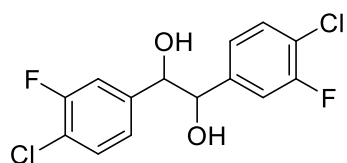
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 96% (26 mg) isolated yield, white solid, *dl* : *meso* = 1.26 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.17 – 7.09 (m, 3H), 7.02 – 7.01 (m, 2H), 6.87 (d, *J* = 7.6 Hz, 1H), 4.66 (s, 1H), 4.64 (s, 1H), 2.76 (s, 1H), 2.27 (s, 6H), 2.22 (d, *J* = 3.6 Hz, 5H), 2.01 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 137.70, 137.54, 136.67, 136.62, 136.30, 136.02, 129.63, 129.30, 128.28, 127.86, 124.63, 124.29, 78.30, 78.14, 19.81, 19.77, 19.52, 19.46.

1,2-Bis(3,4-dichlorophenyl)ethane-1,2-diol (2n)



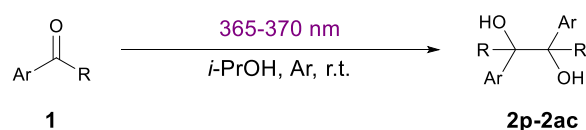
The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 71% (25 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.32 (dd, *J* = 16.8, 8.4 Hz, 4H), 6.94 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.81 (dd, *J* = 8.4, 1.2 Hz, 1H), 4.77 (s, 1H), 4.57 (s, 1H), 3.11 (br, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 139.42, 139.37, 132.60, 132.43, 132.26, 132.16, 130.17, 130.09, 128.92, 128.70, 126.35, 126.32, 77.64, 76.43.

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



The title compound was synthesized according to the general procedure A. The product was purified by flash column chromatography. 66% (21 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1.67. ¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.24 (m, 2H), 7.05 – 7.01 (m, 2H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 8.0 Hz, 1H), 4.84 (s, 1H), 4.61 (s, 1H), 2.95 (s, 1H), 2.45 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 159.08 (d, *J*_{C-F} = 6.1 Hz), 156.60 (d, *J*_{C-F} = 5.8 Hz), 140.15 (d, *J*_{C-F} = 6.2 Hz), 140.03 (d, *J*_{C-F} = 6.2 Hz), 130.36, 130.20, 123.34 (d, *J*_{C-F} = 3.7 Hz), 123.27 (d, *J*_{C-F} = 3.7 Hz), 120.70 (d, *J*_{C-F} = 17.5 Hz), 120.55 (d, *J*_{C-F} = 17.6 Hz), 115.13 (d, *J*_{C-F} = 21.8 Hz), 114.99 (d, *J*_{C-F} = 21.7 Hz), 77.83, 76.43. ¹⁹F NMR (376 MHz, CDCl₃) δ -114.53, -114.80. HRMS (ESI): *m/z* calcd for C₁₄H₉Cl₂F₂O₂ [M - H]⁻ 316.9948, found 316.9953.

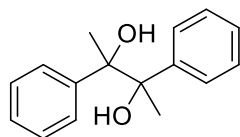
4. General Procedure B for the synthesis of pinacol products 2p–2ac.



In an argon-filled glovebox, a 10 mL flame-dried quartz tube with magnetic stirring was charged sequentially with *i*-PrOH (2 mL) and substrates **1** (0.2 mmol) were sequentially added into the quartz tube. Then, the quartz tube was removed from glovebox. The mixture was stirred at room temperature under 10 W UVA light (365–370 nm) until the reaction was completed, as monitored by TLC analysis. The reaction mixture was then concentrated in vacuum. The crude product was purified

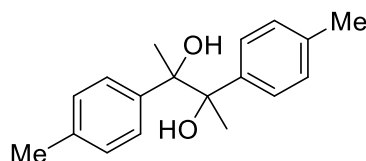
by flash column chromatography (silica gel, PE/EA) to afford the desired product. The corresponding HRMS data of the known product are available in the literature: (2p, 2q, 2s, 2t, 2u, 2y, 2ab)³; (2r, 2v)²¹, 2w²², 2aa²³.

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



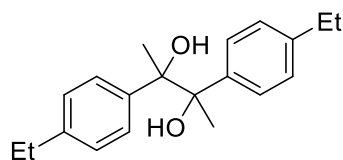
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 99% (24 mg) isolated yield, white solid, *dl* : *meso* = 1.09 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.25 – 7.17 (m, 10H), 2.61 (s, 1H), 2.30 (s, 1H), 1.58 (s, 3H), 1.49 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 143.70, 143.33, 127.33, 127.24, 127.11, 127.02, 126.88, 126.86, 78.81, 78.55, 25.05, 24.89.

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



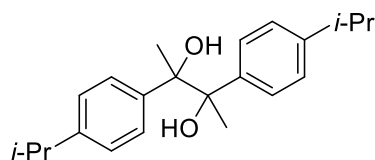
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 99% (27 mg) isolated yield, colorless oil, *dl* : *meso* = 1 : 1.01. ¹H NMR (400 MHz, CDCl₃) δ 7.17 – 7.06 (m, 10H), 2.60 (s, 1H), 2.35 (s, 3H), 2.34 (s, 3H), 2.24 (s, 1H), 1.55 (s, 3H), 1.48 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 140.84, 140.48, 136.52, 136.37, 127.98, 127.81, 127.26, 126.81, 78.72, 78.47, 25.17, 24.99, 20.96, 20.93.

2,3-Bis(4-ethylphenyl)butane-2,3-diol (2r)



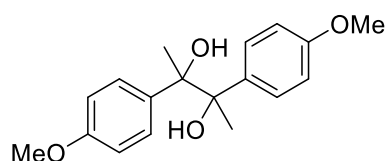
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 94% (28 mg) isolated yield, colorless oil, *dl* : *meso* = 1.24 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.22 – 7.08 (m, 8H), 2.69 – 2.60 (m, 5H), 2.21 (s, 1H), 1.55 (s, 3H), 1.48 (s, 3H), 1.25 (td, *J* = 7.6, 5.2 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 142.87, 142.77, 141.07, 140.71, 127.29, 126.86, 126.77, 126.61, 78.73, 78.46, 28.33, 25.17, 25.04.

2,3-Bis(4-isopropylphenyl)butane-2,3-diol (2s)



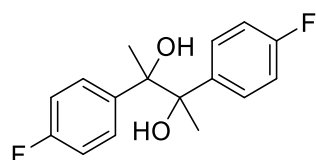
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 99% (33 mg) isolated yield, white solid, *dl* : *meso* = 1.15 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.23 (d, *J* = 8.0 Hz, 2H), 7.18 – 7.10 (m, 6H), 2.96 – 2.84 (m, 2H), 2.58 (s, 1H), 2.17 (s, 1H), 1.54 (s, 3H), 1.48 (s, 3H), 1.27 – 1.24 (m, 12H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.52, 147.41, 141.23, 140.88, 127.23, 126.81, 125.31, 125.20, 78.70, 78.41, 33.60, 25.16, 25.09, 24.03, 24.00, 23.94.

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



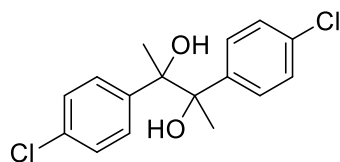
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 83% (25 mg) isolated yield, white solid, *dl* : *meso* = 1.43 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.11 (dd, *J* = 13.2, 8.8 Hz, 4H), 6.77 (dd, *J* = 9.2, 5.6 Hz, 4H), 3.80 (s, 3H), 3.79 (s, 3H), 2.56 (s, 1H), 2.28 (s, 1H), 1.55 (s, 3H), 1.46 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 158.53, 158.43, 136.03, 135.69, 128.57, 128.13, 112.56, 112.41, 78.73, 78.51, 55.22, 25.18, 25.02.

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



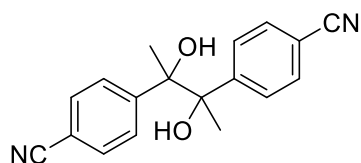
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 83% (23 mg) isolated yield, white solid, *dl* : *meso* = 1.09 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.15 (ddd, *J* = 23.2, 8.8, 5.6 Hz, 4H), 6.91 (dd, *J* = 15.6, 8.8 Hz, 4H), 2.53 (s, 1H), 2.30 (s, 1H), 1.57 (s, 3H), 1.49 (s, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 163.16 (d, *J*_{C-F} = 9.8 Hz), 160.71 (d, *J*_{C-F} = 9.5 Hz), 139.42 (d, *J*_{C-F} = 3.2 Hz), 139.03 (d, *J*_{C-F} = 3.2 Hz), 129.04 (d, *J*_{C-F} = 7.9 Hz), 128.65 (d, *J*_{C-F} = 7.9 Hz), 114.03 (d, *J*_{C-F} = 7.2 Hz), 113.82 (d, *J*_{C-F} = 7.1 Hz), 78.56, 78.30, 25.14, 24.88. **¹⁹F NMR** (376 MHz, CDCl₃) δ -115.87, -116.16.

2,3-Bis(4-chlorophenyl)butane-2,3-diol (2v)



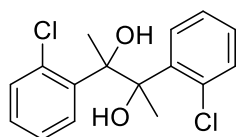
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 77% (24 mg) isolated yield, white solid, *dl* : *meso* = 1.06 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.22 – 7.15 (m, 6H), 7.09 (d, *J* = 8.4 Hz, 2H), 2.57 (s, 1H), 2.27 (s, 1H), 1.54 (s, 3H), 1.47 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 142.12, 141.66, 133.14, 132.96, 128.80, 128.44, 127.36, 127.29, 78.48, 78.20, 25.03, 24.71.

4,4'-(2,3-Dihydroxybutane-2,3-diyl)dibenzonitrile (2w)



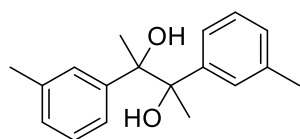
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 99% (29 mg) isolated yield, white solid, *dl* : *meso* = 1 : 1.05. ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.71 (dd, *J* = 24.8, 8.5 Hz, 4H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 5.57 (s, 1H), 5.39 (s, 1H), 3.38 (s, 2H), 1.61 (s, 3H), 1.23 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 152.44, 152.11, 130.63, 130.18, 128.71, 127.98, 119.26, 119.23, 108.95, 108.53, 77.03, 76.77, 24.65, 24.31.

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



The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 74% (23 mg) isolated yield, colorless oil, *dl* : *meso* = 1 : 1.30. ¹H NMR (400 MHz, CDCl₃) δ 7.49 (d, *J* = 8.0 Hz, 1H), 7.23 – 7.07 (m, 6H), 4.01 (s, 1H), 3.81 (s, 1H), 1.83 (s, 3H), 1.80 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 139.74, 139.70, 133.00, 132.53, 131.78, 131.74, 131.67, 131.27, 128.76, 128.69, 126.69, 126.03, 82.44, 81.92, 25.99, 25.54.

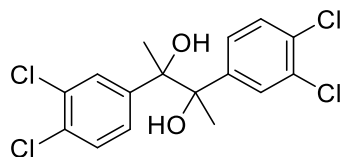
2,3-Di-m-tolylbutane-2,3-diol (2y)



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

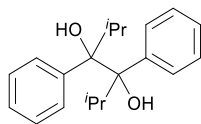
by flash column chromatography. 99% (27 mg) isolated yield, colorless oil, *dl* : *meso* = 1.18 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.16 (t, J = 7.2 Hz, 2H), 7.06 (dd, J = 22.0, 8.0 Hz, 6H), 2.60 (s, 1H), 2.32 (s, 3H), 2.30 (s, 3H), 1.57 (s, 3H), 1.49 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.63, 143.28, 136.68, 136.47, 128.17, 127.76, 127.68, 127.55, 127.12, 126.95, 124.45, 123.95, 78.80, 78.55, 25.07, 24.98, 21.54.

2,3-Di-(3,4-dichlorophenyl)butane-2,3-diol (2z)



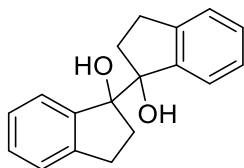
The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 68% (26 mg) isolated yield, colorless oil, *dl* : *meso* = 1. : 1.04. ^1H NMR (400 MHz, CDCl_3) δ 7.47 (d, J = 2.0 Hz, 1H), 7.35 – 7.30 (m, 3H), 7.13 (dd, J = 8.4, 2.0 Hz, 1H), 6.91 (dd, J = 8.4, 1.6 Hz, 1H), 2.52 (s, 1H), 2.13 (s, 1H), 1.50 (s, 3H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.02, 143.37, 131.66, 131.64, 131.49, 131.28, 129.48, 129.31, 129.22, 129.09, 126.76, 126.57, 78.14, 77.79, 25.14, 24.65.

2,5-dimethyl-3,4-diphenylhexane-3,4-diol (2aa)



The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 68% (41 mg) isolated yield, colorless oil, *dl* : *meso* = 1. : 1.05. ^1H NMR (400 MHz, CDCl_3) δ 7.47 (d, J = 6.8 Hz, 4H), 7.33 – 7.22 (m, 6H), 2.89 (s, 1H), 2.38 – 2.28 (m, 1H), 2.26 (s, 1H), 1.83 – 1.73 (m, 1H), 1.22 (d, J = 6.4 Hz, 3H), 0.75 (d, J = 6.8 Hz, 3H), 0.67 (d, J = 6.8 Hz, 3H), 0.36 (d, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.16, 143.05, 128.14, 127.32, 127.23, 126.98, 126.70, 126.41, 84.08, 83.80, 36.02, 35.02, 19.76, 19.64, 18.35, 18.29.

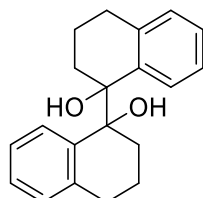
2,2',3,3'-Tetrahydro-1H,1'H-[1,1'-biindene]-1,1'-diol (2ab)



The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 83% (22 mg) isolated yield, white solid, *dl* : *meso* = 1. : 1.40. ^1H NMR

(400 MHz, CDCl₃) δ 7.80 (d, J = 7.2 Hz, 1H), 7.31 – 7.21 (m, 5H) 7.13 – 7.06 (m, 2H), 3.07 – 2.99 (m, 2H), 2.87 (s, 1H), 2.80 – 2.73 (m, 2H), 2.53 (s, 1H), 2.14 – 2.06 (m, 2H), 1.94 (t, J = 6.8 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 145.38, 144.39, 144.13, 143.76, 128.58, 128.56, 126.74, 126.20, 125.10, 125.00, 124.83, 124.54, 88.48, 88.15, 36.55, 36.43, 30.14, 29.97.

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

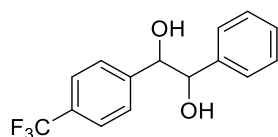


The title compound was synthesized according to the general procedure B. The product was purified by flash column chromatography. 88% (26 mg) isolated yield, white solid, *dl* : *meso* = 1.22 : 1. ¹H NMR (400 MHz, CDCl₃) δ 8.19 (d, J = 7.6 Hz, 1H), 7.24 – 6.93 (m, 7H), 3.20 (s, 1H), 2.73 – 2.50 (m, 4H), 2.24 – 2.05 (m, 2H), 1.68 – 1.52 (m, 5H), 1.35 – 1.24 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 140.47, 140.01, 139.09, 138.33, 129.01, 128.82, 128.21, 127.17, 126.35, 125.53, 78.07, 77.10, 36.39, 35.36, 31.10, 30.78, 20.36, 20.02.

5. General Procedure for the synthesis of 1,2-diols 4 and 5.

In an argon-filled glovebox, a 10 mL flame-dried quartz tube with magnetic stirring was charged sequentially with solvent (2 mL), substrates **1** (0.2 mmol), and **3** (0.3mmol) were sequentially added into the quartz tube. Then, the quartz tube was removed from glovebox. The mixture was stirred at room temperature under 10 W UVA light (365–370 nm) until the reaction was completed, as monitored by TLC analysis. The reaction mixture was then concentrated in vacuum. The crude product was purified by flash column chromatography (silica gel, PE/EA) to afford the desired products. The corresponding HRMS data of the known product are available in the literature: (**4c**, **4d**, **4h**, **4i**, **4j**, **4m**, **4n**)²⁴; (**4b**, **5m**)³, (**4e**, **4f**, **4k**)²⁵, (**4a**, **4l**)²⁶, **5c**², **5k**²⁷.

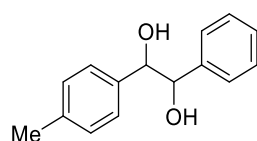
1-Phenyl-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (**4a**)



89% (50 mg) isolated yield, white solid, *dr* = 1.04 : 1. ¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.43 (m, 2H),

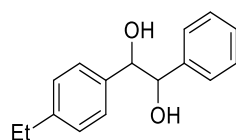
7.28 – 7.13 (m, 6H), 7.08 – 7.04 (m, 1H), 4.87 – 4.78 (m, 1H), 4.70 – 4.57 (m, 1H), 3.08 (d, $J = 162.8$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.74, 143.73, 143.41, 143.39, 143.18, 143.17, 139.71, 139.60, 139.33, 139.14, 130.51, 130.48, 130.39, 130.19, 130.16, 130.06, 129.84, 128.35, 128.32, 128.23, 128.16, 128.13, 127.99, 127.42, 127.33, 127.29, 127.10, 126.97, 126.94, 126.93, 125.48, 125.42, 125.39, 125.32, 125.22 – 124.82 (m), 122.78, 122.71, 122.68, 122.62, 120.05, 120.04, 119.98, 119.97, 79.10, 79.07, 78.48, 78.32, 78.06, 77.86, 77.28, 77.25. ^{19}F NMR (376 MHz, CDCl_3) δ -62.48, -62.51, -62.52, -62.56.

1-Phenyl-2-(*p*-tolyl)ethane-1,2-diol (4b)



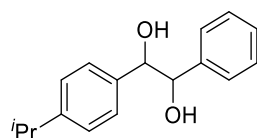
77% (35 mg) isolated yield, white solid, dr = 1.05 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.30 – 7.27 (m, 1H), 7.25 – 7.19 (m, 2H), 7.16 – 7.09 (m, 3H), 7.04 – 6.97 (m, 3H), 4.79 – 4.71 (m, 1H), 4.66 – 4.61 (m, 1H), 2.99 (s, 1H), 2.33 – 2.28 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 140.00, 139.98, 139.90, 139.77, 137.84, 137.81, 137.57, 137.49, 137.02, 137.00, 136.92, 136.80, 129.00, 128.98, 128.84, 128.24, 128.21, 128.12, 128.11, 128.07, 128.05, 127.91, 127.85, 127.14, 127.12, 127.08, 127.06, 127.02, 126.98, 126.90, 126.87, 79.11, 79.02, 78.90, 78.80, 78.09, 78.07, 78.05, 78.04, 21.21, 21.17.

1-(4-Ethylphenyl)-2-phenylethane-1,2-diol (4c)



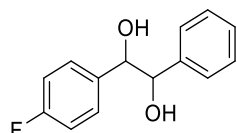
82% (40 mg) isolated yield, white solid, dr = 1.29 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.01 (m, 10H), 4.80 – 4.71 (m, 1H), 4.68 – 4.63 (m, 1H), 2.95 (s, 1H), 2.67 – 2.56 (m, 3H), 2.27 (s, 1H), 1.28 – 1.17 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.25, 143.98, 143.88, 140.04, 139.90, 139.78, 137.35, 137.34, 137.18, 137.09, 128.25, 128.21, 128.12, 128.10, 128.07, 127.91, 127.87, 127.82, 127.65, 127.17, 127.15, 127.14, 127.12, 126.98, 126.91, 79.12, 78.95, 78.88, 78.68, 78.12, 78.09, 28.60, 28.59, 28.54, 15.56, 15.53.

1-(4-Isopropylphenyl)-2-phenylethane-1,2-diol (4d)



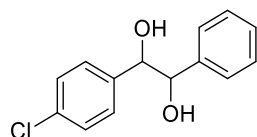
75% (38 mg) isolated yield, white solid, dr = 1.74 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.32 – 7.28 (m, 2H), 7.25 – 7.17 (m, 3H), 7.15 – 7.04 (m, 4H), 4.77 – 4.65 (m, 2H), 2.93 – 2.82 (m, 2H), 2.17 (s, 1H), 1.25 – 1.19 (m, 6H). **¹³C NMR** (100 MHz, CDCl₃) δ 148.97, 148.65, 148.52, 140.11, 140.08, 137.62, 137.58, 137.34, 137.28, 128.30, 128.12, 127.84, 127.17, 127.14, 126.95, 126.86, 126.80, 126.53, 126.46, 126.24, 78.86, 78.80, 78.47, 78.14, 33.87, 33.80, 24.00, 23.98.

1-(4-Fluorophenyl)-2-phenylethane-1,2-diol (4e)



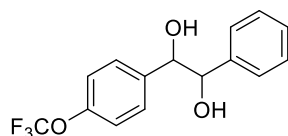
80% (37 mg) isolated yield, colorless oil, dr = 1.07 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.30 – 6.87 (m, 10H), 4.83 – 4.80 (m, 1H), 4.67 – 4.60 (m, 1H), 3.02 (s, 1H), 2.39 (s, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 163.73, 163.71, 163.65, 163.59, 161.27, 161.26, 161.20, 161.15, 139.63, 139.50, 135.52, 135.49, 135.43, 135.39, 135.38, 135.35, 135.32, 135.20, 135.17, 128.79, 128.74, 128.71, 128.67, 128.66, 128.59, 128.28, 128.23, 128.20, 128.11, 127.03, 126.96, 115.13 (dd, *J* = 8.1, 2.7 Hz), 114.92 (dd, *J* = 8.1, 2.7 Hz), 79.33, 78.72, 78.55, 78.02. **¹⁹F NMR** (376 MHz, CDCl₃) δ -114.00, -114.09, -114.30, -114.33.

1-(4-Chlorophenyl)-2-phenylethane-1,2-diol (4f)



78% (39 mg) isolated yield, white solid, dr = 1 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.30 – 7.15 (m, 6H), 7.11 – 7.04 (m, 2H), 7.00 – 6.96 (m, 1H), 4.80 – 4.77 (m, 1H), 4.67 – 4.55 (m, 1H), 3.10 (s, 1H), 2.47 (s, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 139.80, 139.70, 139.52, 139.36, 138.26, 138.16, 138.00, 137.81, 133.84, 133.83, 133.70, 133.61, 128.47, 128.38, 128.35, 128.30, 128.24, 128.17, 128.15, 128.12, 127.96, 127.10, 127.01, 126.97, 126.95, 79.18, 79.11, 78.50, 78.48, 78.09, 77.92, 77.29, 77.12. **HRMS** (ESI): *m/z* calcd for C₁₄H₁₃Cl₂O₂ [*M* + Cl]⁻ 283.0293, found 283.0298.

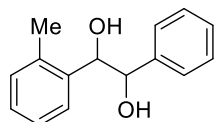
1-Phenyl-2-(4-(trifluoromethoxy)phenyl)ethane-1,2-diol (4g)



79% (47 mg) isolated yield, white solid, dr = 1.05 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.28 – 7.05 (m, 9H), 4.78 (s, 1H), 4.61 (d, *J* = 28.4 Hz, 1H), 3.27 – 3.09 (m, 1H), 2.69 – 2.44 (m, 1H). **¹³C NMR** (100 MHz,

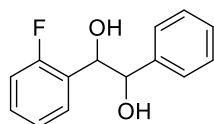
CDCl₃) δ 148.90, 148.89, 148.83, 148.82, 148.75, 148.74, 139.77, 139.65, 139.50, 139.33, 138.45, 138.36, 138.21, 138.03, 128.52, 128.42, 128.38, 128.35, 128.28, 128.24, 128.22, 128.16, 128.14, 127.98, 127.11, 127.00, 126.96, 126.92, 120.62, 120.55, 120.49, 120.42 (qt, J_{C-F} = 255.7, 2.9 Hz), 79.18, 79.10, 78.40, 78.38, 78.07, 77.91, 77.15, 76.98. **¹⁹F NMR** (376 MHz, CDCl₃) δ -57.85, -57.89, -57.90, -57.93. **HRMS** (ESI): m/z calcd for C₁₅H₁₂F₃O₃ [M - H]⁻ 297.0739, found 297.0744.

1-Phenyl-2-(*o*-tolyl)ethane-1,2-diol (4h)



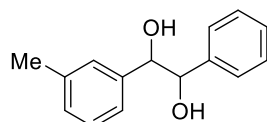
64% (29 mg) isolated yield, colorless oil, dr = 1.14 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.64 – 6.94 (m, 9H), 5.20 – 4.68 (m, 2H), 3.12 (s, 1H), 2.39 (s, 1H), 1.97 (dd, J = 202.8, 35.2 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 139.98, 139.88, 139.76, 139.70, 138.29, 138.13, 138.06, 137.99, 136.15, 136.03, 135.92, 130.18, 130.09, 130.06, 128.21, 128.17, 128.12, 128.07, 128.02, 127.91, 127.80, 127.75, 127.72, 127.25, 127.21, 127.11, 127.01, 126.97, 126.71, 126.66, 126.62, 126.07, 125.95, 79.10, 78.59, 78.07, 77.69, 74.84, 74.62, 73.86, 73.33, 19.32, 19.17, 18.83, 18.76.

1-(2-Fluorophenyl)-2-phenylethane-1,2-diol (4i)



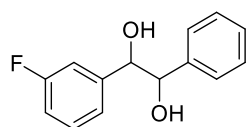
76% (35 mg) isolated yield, colorless oil, dr = 1.06 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.43 – 6.85 (m, 9H), 5.32 – 4.99 (m, 1H), 4.91 – 4.64 (m, 1H), 3.08 – 2.45 (m, 2H). **¹³C NMR** (100 MHz, CDCl₃) δ 161.31, 161.27, 161.20, 161.18, 158.86, 158.82, 158.75, 158.73, 139.83, 139.75, 139.70, 139.27, 129.60, 129.51, 129.49, 129.41, 129.33, 129.30, 129.25, 129.22, 128.50, 128.46, 128.42, 128.33, 128.29, 128.22, 128.20, 128.14, 128.10, 128.06, 127.99, 127.94, 127.12, 127.07, 126.98, 126.63, 124.16, 124.13, 124.11, 124.02, 123.99, 123.85, 123.82, 115.33, 115.29, 115.11, 115.05, 114.91, 114.83, 114.69, 79.11, 78.06, 77.76, 76.92, 72.88, 71.87, 71.46, 70.46. **¹⁹F NMR** (376 MHz, CDCl₃) δ -118.11, -118.16, -118.46, -118.61.

1-Phenyl-2-(*m*-tolyl)ethane-1,2-diol (4j)



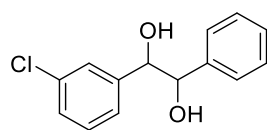
74% (34 mg) isolated yield, colorless oil, dr = 1.07 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.30 – 7.18 (m, 4H), 7.13 – 7.01 (m, 4H), 6.97 – 6.85 (m, 1H), 4.78 – 4.69 (m, 1H), 4.66 – 4.60 (m, 1H), 2.98 (s, 1H), 2.29 (dd, *J* = 21.2, 6.4 Hz, 4H). **¹³C NMR** (100 MHz, CDCl₃) δ 140.02, 139.99, 139.95, 137.99, 137.95, 137.76, 137.74, 128.95, 128.90, 128.64, 128.60, 128.23, 128.21, 128.20, 128.17, 128.11, 128.09, 128.06, 128.00, 127.99, 127.90, 127.86, 127.83, 127.79, 127.54, 127.51, 127.16, 127.11, 126.98, 126.96, 124.29, 124.23, 124.06, 124.02, 79.09, 78.98, 78.93, 78.80, 78.22, 78.18, 78.10, 78.06, 21.45, 21.41, 21.39.

1-(3-Fluorophenyl)-2-phenylethane-1,2-diol (4k)



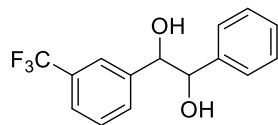
82% (38 mg) isolated yield, colorless oil, dr = 1.08 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.28 – 7.06 (m, 6H), 6.93 – 6.75 (m, 3H), 4.77 – 4.75 (m, 1H), 4.64 – 4.56 (m, 1H), 3.18 (d, *J* = 56.8 Hz, 1H), 2.70 – 2.45 (m, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 163.88, 163.83, 161.44, 161.39, 142.50, 142.42, 142.35, 142.27, 142.20, 142.07, 142.00, 139.79, 139.66, 139.55, 139.33, 129.72, 129.64, 129.62, 129.58, 129.55, 129.54, 129.50, 129.48, 128.26, 128.23, 128.18, 128.15, 128.11, 127.96, 127.11, 127.02, 126.96, 126.92, 122.76, 122.73, 122.68, 122.65, 122.62, 115.09, 115.04, 114.91, 114.88, 114.86, 114.83, 114.70, 114.65, 114.15, 114.07, 113.98, 113.93, 113.85, 113.76, 113.71, 79.09, 79.02, 78.47 (d, *J*_{C-F} = 1.7 Hz), 78.37 (d, *J*_{C-F} = 1.6 Hz), 78.06, 77.88, 77.29 (d, *J*_{C-F} = 1.7 Hz), 77.12, 77.10. **¹⁹F NMR** (376 MHz, CDCl₃) δ -112.85, -112.91, -113.08, -113.12.

1-(3-Chlorophenyl)-2-phenylethane-1,2-diol (4l)



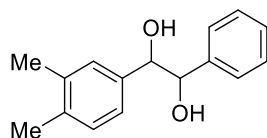
66% (33 mg) isolated yield, colorless oil, dr = 1.05 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.29 – 7.07 (m, 8H), 7.03 – 6.85 (m, 1H), 4.79 – 4.73 (m, 1H), 4.65 – 4.55 (m, 1H), 3.10 (d, *J* = 55.2 Hz, 1H), 2.59 – 2.38 (m, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 141.91, 141.85, 141.65, 141.50, 139.78, 139.68, 139.49, 139.33, 134.23, 134.18, 134.08, 134.06, 129.42, 129.37, 129.30, 129.29, 128.31, 128.29, 128.27, 128.25, 128.22, 128.16, 128.13, 128.09, 128.03, 127.97, 127.26, 127.19, 127.11, 127.03, 126.97, 126.95, 126.92, 125.34, 125.26, 125.25, 125.22, 79.09, 78.98, 78.42, 78.25, 78.08, 77.89, 77.32, 77.14.

1-Phenyl-2-(3-(trifluoromethyl)phenyl)ethane-1,2-diol (4m)



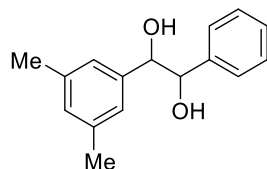
90% (51 mg) isolated yield, colorless oil, dr = 1.12 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.51 – 7.44 (m, 1H), 7.36 – 7.15 (m, 7H), 7.11 – 7.01 (m, 1H), 4.84 – 4.76 (m, 1H), 4.68 – 4.53 (m, 1H), 3.43 – 2.50 (m, 2H). **¹³C NMR** (100 MHz, CDCl₃) δ 140.72, 140.61, 140.36, 140.08, 139.69, 139.58, 139.26, 139.08, 130.71, 130.55, 130.50, 130.44, 130.31, 130.23, 130.12, 128.64, 128.46, 128.42, 128.35, 128.33, 128.30, 128.25, 128.17, 128.01, 127.11, 126.95, 126.91, 124.97, 124.93, 124.90, 124.86, 124.81, 124.78, 124.74, 124.63 (qd, J_{C-F} = 3.7, 1.7 Hz), 123.97 (q, J_{C-F} = 3.7 Hz), 123.77 (q, J_{C-F} = 3.9 Hz), 124.00 (qdd, J_{C-F} = 270.8, 10.9, 7.4 Hz), 79.18, 79.10, 78.53, 78.06, 77.81, 77.25, 76.98. **¹⁹F NMR** (376 MHz, CDCl₃) δ -62.58, -62.65, -62.71, -62.76.

1-(3,4-Dimethylphenyl)-2-phenylethane-1,2-diol (4n)



64% (31 mg) isolated yield, white solid, dr = 1.44 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.34 – 6.80 (m, 8H), 4.80 – 4.61 (m, 2H), 2.82 (s, 1H), 2.25 – 2.18 (m, 7H). **¹³C NMR** (100 MHz, CDCl₃) δ 140.20, 140.13, 139.90, 139.77, 137.76, 137.63, 137.38, 137.34, 136.69, 136.64, 136.62, 136.60, 136.37, 136.35, 136.19, 136.07, 129.69, 129.61, 129.38, 128.38, 128.33, 128.28, 128.21, 128.12, 128.10, 128.01, 127.97, 127.91, 127.80, 127.18, 127.10, 126.97, 124.70, 124.61, 124.36, 79.11, 78.82, 78.77, 78.41, 78.22, 78.18, 78.14, 78.08, 19.83, 19.81, 19.78, 19.75, 19.53, 19.49.

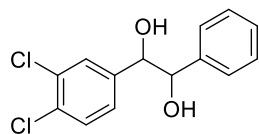
1-(3,5-Dimethylphenyl)-2-phenylethane-1,2-diol (4o)



60% (28 mg) isolated yield, colorless oil, dr = 1.15 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.34 – 7.21 (m, 5H), 7.16 – 7.10 (m, 1H), 6.94 – 6.74 (m, 2H), 4.81 – 4.61 (m, 2H), 2.94 – 2.68 (m, 1H), 2.29 – 2.08 (m, 6H). **¹³C NMR** (100 MHz, CDCl₃) δ 140.14, 140.10, 139.86, 139.84, 139.76, 137.99, 137.68, 129.94, 129.53, 129.45, 128.31, 128.24, 128.19, 128.14, 128.11, 128.10, 127.93, 127.84, 127.20, 127.10, 126.96,

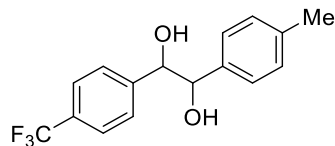
126.90, 124.93, 124.61, 124.49, 79.13, 78.89, 78.77, 78.41, 78.35, 78.17, 78.10, 21.38, 21.31, 21.27.

1-(3,4-Dichlorophenyl)-2-phenylethane-1,2-diol (4p)



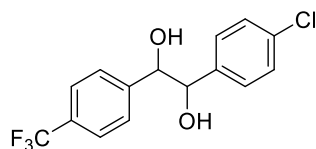
46% (26 mg) isolated yield, colorless oil, dr = 1.08 : 1. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.33 – 7.15 (m, 6H), 7.10 – 7.07 (m, 1H), 6.97 – 6.76 (m, 1H), 4.79 – 4.50 (m, 2H), 3.20 – 2.33 (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 140.34, 140.00, 139.72, 139.64, 139.63, 139.53, 139.22, 139.11, 132.59, 132.42, 132.22, 132.17, 132.12, 131.77, 131.74, 130.16, 130.08, 129.92, 129.89, 129.12, 128.98, 128.87, 128.79, 128.43, 128.41, 128.27, 128.18, 128.01, 127.10, 126.94, 126.52, 126.41, 126.38, 79.11, 79.02, 78.09, 77.90, 77.76, 77.61, 76.41.

1-(*p*-Tolyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5a)



71% (42 mg) isolated yield, colorless oil, dr = 1.09 : 1. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.54 – 7.45 (m, 2H), 7.32 – 6.96 (m, 6H), 4.89 – 4.56 (m, 2H), 3.22 (s, 1H), 2.88 (s, 1H), 2.33 – 2.29 (m, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.92, 143.85, 143.41, 143.21, 138.19, 138.09, 137.91, 137.58, 136.89, 136.37, 136.22, 130.52, 130.41, 130.20, 130.08, 129.88, 129.76, 129.10, 129.07, 129.05, 128.87, 127.44, 127.33, 127.30, 127.06, 126.92, 126.86, 126.84, 125.18 (q, $J_{\text{C-F}}$ = 3.7 Hz), 124.94 (q, $J_{\text{C-F}}$ = 3.7 Hz), 124.04 (qd, $J_{\text{C-F}}$ = 257.5, 13.1 Hz), 78.90, 78.81, 78.44, 78.36, 78.07, 77.89, 21.19, 21.16. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -62.45, -62.48, -62.52, -62.55. **HRMS** (ESI): m/z calcd for $\text{C}_{16}\text{H}_{14}\text{F}_3\text{O}_2$ [$\text{M} - \text{H}$] $^-$ 295.0946, found 295.0956.

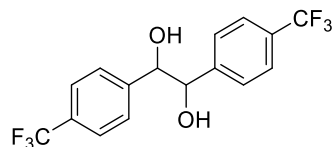
1-(4-Chlorophenyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5b)



90% (57 mg) isolated yield, white solid, dr = 1.06 : 1. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.52 – 7.46 (m, 2H), 7.23 – 7.13 (m, 4H), 7.03 (d, J = 8.4 Hz, 1H), 6.96 (dd, J = 8.4, 6.4 Hz, 1H), 4.88 – 4.75 (m, 1H), 4.65 – 4.52 (m, 1H), 3.23 (d, J = 33.2 Hz, 1H), 2.77 – 2.63 (m, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.38, 143.29, 143.28, 143.26, 143.112, 143.11, 137.84, 137.73, 137.67, 137.54, 134.06, 134.00, 133.91, 133.89,

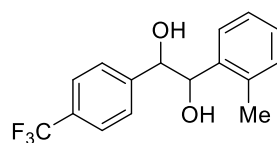
130.89, 130.57, 130.44, 130.43, 130.33, 130.25, 130.12, 130.01, 129.92, 129.80, 129.68, 128.50, 128.42, 128.37, 128.34, 128.32, 128.29, 127.34, 127.33, 127.30, 127.27, 125.23, 125.19, 125.15, 125.10, 125.06, 125.02, 124.98, 124.94, 124.00 (qdd, $J_{C-F} = 270.6, 6.5, 3.3$ Hz), 78.49, 78.48, 78.38, 78.34. ^{19}F NMR (376 MHz, CDCl_3) δ -62.48, -62.51, -62.53, -62.55.

1,2-Bis(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5c)



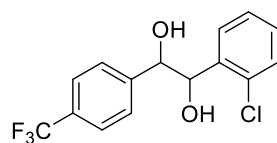
67% (47 mg) isolated yield, white solid, *dl/meso* = 1 : 1.11. ^1H NMR (400 MHz, CDCl_3) δ 7.53 – 7.49 (m, 4H), 7.25 (d, $J = 8.0$ Hz, 2H), 7.19 (d, $J = 8.4$ Hz, 2H), 4.92 (s, 1H), 4.70 (s, 1H), 3.11 (s, 1H), 2.60 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.26, 143.12, 130.45 (q, $J_{C-F} = 32.4$ Hz), 130.34 (q, $J_{C-F} = 32.3$ Hz), 127.33, 127.27, 125.23 (q, $J_{C-F} = 3.8$ Hz), 125.08 (q, $J_{C-F} = 3.6$ Hz), 124.01 (q, $J_{C-F} = 270.3$ Hz), 78.38, 77.16. ^{19}F NMR (376 MHz, CDCl_3) δ -62.56, -62.60.

1-(*o*-Tolyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5d)



52% (31 mg) isolated yield, colorless oil, *dr* = 1.08 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.61 – 7.42 (m, 2H), 7.32 – 6.90 (m, 6H), 5.16 – 4.74 (m, 2H), 3.33 – 2.31 (m, 2H), 2.21 – 1.64 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.94, 143.93, 143.66, 143.65, 138.08, 137.92, 137.78, 137.62, 136.17, 135.92, 135.75, 130.33, 130.24, 130.12, 130.21, 130.09, 129.92, 129.80, 128.09, 128.02, 127.79, 127.76, 127.63, 127.17, 127.06, 126.67, 126.56, 126.53, 126.29, 126.23, 126.09, 125.96, 124.84 (q, $J_{C-F} = 3.7$ Hz), 124.11 (qd, $J_{C-F} = 264.1, 6.3$ Hz). ^{19}F NMR (376 MHz, CDCl_3) δ -62.48, -62.52.

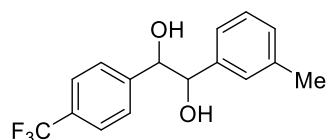
1-(2-Chlorophenyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5e)



80% (51 mg) isolated yield, colorless oil, *dr* = 1.10 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.61 – 7.46 (m, 2H), 7.39 (dd, $J = 24.8, 8.0$ Hz, 1H), 7.31 – 7.00 (m, 5H), 5.55 – 4.66 (m, 2H), 3.29 – 2.78 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 144.09, 143.30, 143.10, 142.73, 137.54, 137.12, 136.61, 136.29, 133.37, 132.63, 132.40, 132.22, 130.54, 130.42, 130.22, 130.18, 130.13, 130.10, 129.86, 129.81, 129.56, 129.49,

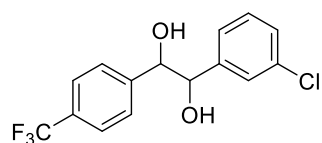
129.30, 129.23, 129.14, 129.11, 129.08, 128.94, 128.85, 128.74, 128.41, 128.23, 127.65, 127.32, 127.27, 127.07, 126.85, 126.79, 126.73, 126.46, 125.19 (q, J_{C-F} = 3.8 Hz), 125.03 (q, J_{C-F} = 3.7 Hz), 124.56 (q, J_{C-F} = 3.7 Hz), 124.04, (qt, J_{C-F} = 270.6, 6.4 Hz), 78.36, 77.11, 76.00, 75.24, 74.40, 73.45, 73.04, 72.08, ^{19}F NMR (376 MHz, CDCl_3) δ -62.47, -62.52, -62.55. HRMS (ESI): m/z calcd for $\text{C}_{15}\text{H}_{11}\text{ClF}_3\text{O}_2$ $[\text{M} - \text{H}]^-$ 315.0400, found 315.0405.

1-(*m*-Tolyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5f)



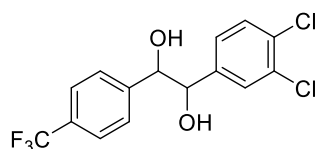
51% (30 mg) isolated yield, colorless oil, dr = 1.07 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.54 – 7.30 (m, 2H), 7.25 – 7.03 (m, 5H), 6.98 – 6.85 (m, 2H), 4.88 – 4.55 (m, 2H), 3.26 – 2.89 (m, 1H), 2.32 – 2.18 (m, 4H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.91, 143.45, 143.23, 139.89, 139.36, 139.24, 138.11, 138.04, 137.79, 130.19, 130.12, 129.86, 129.74, 129.12, 129.03, 129.00, 128.65, 128.29, 128.24, 128.02, 127.82, 127.64, 127.49, 127.47, 127.33, 127.29, 125.16 (q, J_{C-F} = 3.7 Hz), 125.00 (q, J_{C-F} = 3.7 Hz), 124.92, 124.90, 124.89, 124.87, 124.83, 124.27, 124.06, 123.98, 124.07 (qdd, J_{C-F} = 270.5, 11.0, 5.0 Hz), 79.03, 78.81, 78.39, 78.34, 78.24, 78.00, 77.31, 21.43, 21.38, 21.33. ^{19}F NMR (376 MHz, CDCl_3) δ -62.50, -62.52, -62.56. HRMS (ESI): m/z calcd for $\text{C}_{16}\text{H}_{14}\text{F}_3\text{O}_2$ $[\text{M} - \text{H}]^-$ 295.0946, found 295.0956.

1-(3-Chlorophenyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5g)



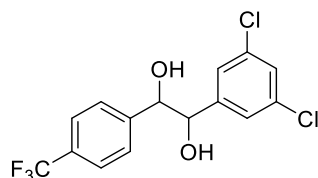
64% (40 mg) isolated yield, colorless oil, dr = 1.08 : 1. ^1H NMR (400 MHz, CDCl_3) δ 7.52 – 7.47 (m, 1H), 7.25 – 7.10 (m, 5H), 6.99 – 6.93 (m, 1H), 6.87 – 6.82 (m, 1H), 4.85 – 4.74 (m, 1H), 4.66 – 4.74 (m, 1H), 3.26 – 3.18 (m, 1H), 2.71 – 2.62 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.41, 143.27, 141.54, 141.44, 141.41, 141.27, 134.34, 134.26, 134.24, 134.21, 130.26 (qd, J_{C-F} = 32.0, 7.0 Hz), 129.52, 129.45, 129.43, 129.40, 128.42, 128.33, 128.31, 127.35, 127.25, 127.17, 127.13, 126.93, 125.25, 125.19, 125.14, 125.10, 125.06, 125.02, 124.98, 124.94, 124.04 (qd, J_{C-F} = 270.6, 5.9 Hz), 78.32, 78.25, 77.15, 77.09. ^{19}F NMR (376 MHz, CDCl_3) δ -62.50, -62.52, -62.53, -62.55.

1-(3,4-Dichlorophenyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5h)



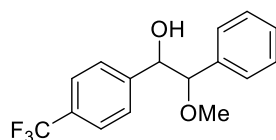
70% (49 mg) isolated yield, white solid, dr = 1.11 : 1. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.51 (dd, J = 16.0, 8.4 Hz, 2H), 7.34 – 7.17 (m, 4H), 6.93 – 6.76 (m, 1H), 4.91 – 4.53 (m, 2H), 3.18 (d, J = 8.0 Hz, 1H), 2.66 (d, J = 4.4 Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.22, 143.14, 143.05, 139.54, 139.49, 139.44, 139.40, 132.64, 132.57, 132.46, 132.39, 132.31, 132.23, 132.20, 132.12, 130.68, 130.61, 130.55, 130.49, 130.35, 130.28, 130.22, 130.17, 130.12, 130.04, 129.00, 128.96, 128.77, 128.74, 127.32, 127.30, 127.27, 126.37, 126.33, 125.34, 125.30, 125.26, 125.21, 128.18, 125.15, 125.11, 125.09, 125.05, 125.02, 123.97 (qd, $J_{\text{C-F}}$ = 270.3, 6.4 Hz), 78.37, 78.29, 77.76, 77.62, 77.14, 76.99, 76.56, 76.45. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -62.52, -62.54, -62.55, -62.57.

1-(3,5-Dichlorophenyl)-2-(4-(trifluoromethyl)phenyl)ethane-1,2-diol (5i)



60% (42 mg) isolated yield, white solid, dr = 1.12 : 1. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.57 – 7.49 (m, 2H), 7.30 – 7.19 (m, 4H), 7.00 (dd, J = 27.6, 2.0 Hz, 1H), 4.93 – 4.59 (m, 2H), 3.10 – 2.58 (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 143.22, 143.10, 143.07, 143.01, 142.81, 142.72, 134.92, 134.78, 130.78, 130.68, 130.63, 130.51, 130.45, 130.36, 130.30, 130.19, 128.34, 128.24, 127.33, 127.31, 127.27, 127.19, 125.56, 125.39, 125.34, 125.30, 125.26, 125.23, 125.20, 125.16, 125.11, 125.07, 123.97 (qd, $J_{\text{C-F}}$ = 265.3, 6.9 Hz), 78.39, 78.12, 77.77, 77.17, 76.99, 76.64. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -62.54, -62.57, -62.61, -62.63.

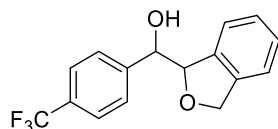
2-Methoxy-2-phenyl-1-(4-(trifluoromethyl)phenyl)ethan-1-ol (5j)



53% (31 mg) isolated yield, white solid, dr = 1.03 : 1 $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.50 (d, J = 8.0 Hz, 1H), 7.42 (d, J = 8.0 Hz, 1H), 7.30 – 7.22 (m, 4H), 7.12 (d, J = 7.2 Hz, 2H), 6.99 (dd, J = 7.6, 2.4 Hz, 1H), 4.92 – 4.70 (m, 1H), 4.33 – 4.06 (m, 1H), 3.63 – 2.57 (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 144.21, 143.25, 136.80, 136.73, 130.32, 130.15, 130.00, 129.82, 129.68, 129.50, 129.36, 129.18, 128.42, 128.34,

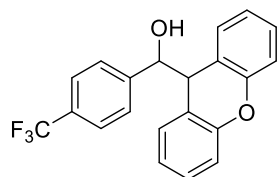
128.31, 128.27, 127.87, 127.77, 127.57, 127.37, 124.80, 124.76, 124.73, 124.69, 124.66, 124.18 (qd, $J_{C-F} = 270.2, 9.8$ Hz), 88.96, 87.31, 78.11, 76.41, 57.16, 56.91. ^{19}F NMR (376 MHz, CDCl_3) δ -62.41, -62.49.

(1,3-Dihydroisobenzofuran-1-yl)(4-(trifluoromethyl)phenyl)methanol (5k)



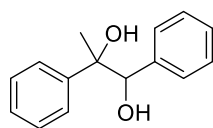
44% (26 mg) isolated yield, white solid, dr = 1.32 : 1 ^1H NMR (400 MHz, CDCl_3) δ 7.64 – 7.58 (m, 2H), 7.50 – 7.44 (m, 2H), 7.31 – 7.15 (m, 3H) 6.69 (dd, $J = 53.6, 8.0$ Hz, 1H), 5.40 (d, $J = 69.2$ Hz, 1H), 5.17 – 4.78 (m, 3H), 2.84 (d, $J = 142.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 143.67, 143.53, 140.00, 139.72, 137.09, 137.04, 134.04, 130.84, 130.52, 130.33, 130.19, 130.00, 129.87, 129.68, 129.08, 128.31, 128.25, 127.96, 127.23, 127.13, 126.96, 125.25 (q, $J_{C-F} = 3.7$ Hz), 125.07 (q, $J_{C-F} = 3.7$ Hz), 123.97 (q, $J_{C-F} = 3.7$ Hz), 124.16 (qd, $J_{C-F} = 270.3, 2.9$ Hz), 122.66, 122.45, 121.12, 121.02, 87.74, 87.47, 76.16, 75.71, 73.60, 72.94. ^{19}F NMR (376 MHz, CDCl_3) δ -62.40, -62.46.

(4-(Trifluoromethyl)phenyl)(9H-xanthen-9-yl)methanol (5l)



56% (40 mg) isolated yield, colorless oil, dr = 1 : 1 ^1H NMR (400 MHz, CDCl_3) δ 7.41 (d, $J = 8.0$ Hz, 2H), 7.28 – 7.20 (m, 2H), 7.06 – 6.92 (m, 8H), 4.78 (d, $J = 5.6$ Hz, 1H), 4.20 (d, $J = 5.6$ Hz, 1H), 2.16 (s, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 152.96, 152.94, 144.51, 129.89 (q, $J_{C-F} = 32.0$ Hz), 129.67, 129.58, 128.58, 128.43, 127.10, 124.6 (q, $J_{C-F} = 3.7$ Hz), 124.2 (q, $J_{C-F} = 270.6$ Hz), 123.01, 121.08, 120.94, 116.62, 116.31, 78.72, 48.14. ^{19}F NMR (376 MHz, CDCl_3) δ -62.34. HRMS (ESI): m/z calcd for $\text{C}_{21}\text{H}_{14}\text{F}_3\text{O}_2$ [$\text{M} - \text{H}$] $^-$ 355.0946, found 355.0942.

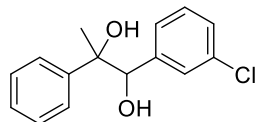
1,2-Diphenylpropane-1,2-diol (5m)



66% (30 mg) isolated yield, white solid, dr = 1.45 : 1 ^1H NMR (400 MHz, CDCl_3) δ 7.38 – 7.12 (m, 9H), 7.02 (d, $J = 6.4$ Hz, 1H), 4.79 (d, $J = 32.8$ Hz, 1H), 2.71 – 2.50 (m, 2H), 1.49 (d, $J = 100.0$ Hz, 3H). ^{13}C

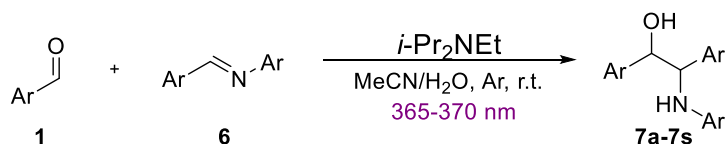
NMR (100 MHz, CDCl₃) δ 145.01, 143.64, 139.41, 139.14, 128.16, 127.88, 127.74, 127.70, 127.58, 127.31, 127.09, 126.08, 125.88, 81.04, 80.86, 77.12, 76.85, 25.58, 24.14.

1-(3-Chlorophenyl)-2-phenylpropane-1,2-diol (5n)



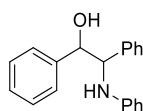
59% (28 mg) isolated yield, colorless oil, dr = 1.21 : 1 **¹H NMR** (400 MHz, CDCl₃) δ 7.36 – 7.05 (m, 8H), 6.90 (dd, *J* = 12.0, 8.0 Hz, 1H), 4.77 (d, *J* = 35.2 Hz, 1H), 2.71 – 2.41 (m, 2H), 1.51 (d, *J* = 98.0 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 144.55, 143.32, 141.57, 141.18, 133.71, 133.51, 128.85, 128.68, 128.26, 127.88, 127.81, 127.79, 127.72, 127.57, 127.33, 125.95, 125.91, 125.85, 80.38, 80.32, 77.09, 76.78, 25.55, 23.71.

6. General Procedure for the synthesis of β -amino alcohols 7a–7s.



In an argon-filled glovebox, a 10 mL flame-dried quartz tube with magnetic stirring was charged sequentially with MeCN (0.5 mL), aldehydes (0.4 mmol), imines (0.2 mmol) and *i*-Pr₂NEt (0.4 mmol) were sequentially added into the quartz tube. Then, the quartz tube was removed from glovebox, and H₂O (0.05 mL) was injected into it. The mixture was stirred at room temperature under 10 W UVA LED (365–370 nm) until the reaction was completed, as monitored by TLC analysis. The reaction mixture was then concentrated in vacuum. The crude product was purified by flash column chromatography (silica gel, PE/EA) to afford the desired products. The corresponding HRMS data of the known product are available in the literature: (**7a**, **7e**, **7g**, **7j**, **7k**)²⁸.

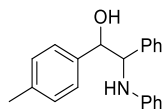
1,2-diphenyl-2-(phenylamino)ethan-1-ol (7a)



72% (42 mg) isolated yield, colorless oil, dr = 1 : 1.08. **¹H NMR** (400 MHz, CDCl₃) δ 7.28 – 7.20 (m, 8H), 7.14 – 7.11 (m, 1H), 7.09 – 7.03 (m, 3H), 6.64 (t, *J* = 7.2 Hz, 1H), 6.54 – 6.48 (m, 2H), 5.05 (d, *J* = 4.8 Hz, 0.48 × 1H), 4.85 (d, *J* = 1.2 Hz, 0.52 × 1H), 4.66 (d, *J* = 4.8 Hz, 0.48 × 1H), 4.52 (d, *J* = 6.0 Hz, 0.52 × 1H),

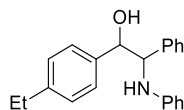
2.65 (brs, $0.48 \times 1\text{H}$), 2.37 (brs, $0.52 \times 1\text{H}$). ^{13}C NMR (100 MHz, CDCl_3) δ 147.22, 146.72, 140.52, 140.18, 139.95, 138.41, 129.07, 129.02, 128.52, 128.24, 128.21, 128.20, 127.97, 127.85, 127.57, 127.47, 127.24, 126.52, 126.49, 117.86, 117.84, 114.07, 113.88, 78.01, 77.12, 64.66, 63.63.

2-phenyl-2-(phenylamino)-1-(p-tolyl)ethan-1-ol (7b)



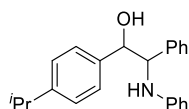
67% (41 mg) isolated yield, colorless oil, dr = 1 : 1.22. ^1H NMR (400 MHz, CDCl_3) δ 7.28 – 7.09 (m, 6H), 7.08 – 7.02 (m, 4H), 6.96 (d, J = 8.0 Hz, 1H), 6.66 – 6.59 (m, 1H), 6.54 – 6.44 (m, 2H), 4.97 (d, J = 4.8 Hz, $0.42 \times 1\text{H}$), 4.81 (d, J = 5.6 Hz, $0.58 \times 1\text{H}$), 4.62 (d, J = 4.8 Hz, $0.42 \times 1\text{H}$), 4.49 (d, J = 5.6 Hz, $0.58 \times 1\text{H}$), 2.31 (d, J = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 147.27, 146.77, 140.30, 138.69, 137.66, 137.53, 137.48, 136.84, 129.12, 129.03, 128.99, 128.90, 128.48, 128.24, 128.19, 127.85, 127.70, 127.50, 127.39, 127.26, 127.10, 126.52, 126.48, 126.43, 126.41, 117.77, 117.72, 114.02, 113.86, 77.82, 77.09, 64.53, 63.58, 21.12. HRMS (ESI): m/z calcd for $\text{C}_{21}\text{H}_{21}\text{NO}$ $[\text{M} + \text{H}]^+$ 304.1696, found 304.1693.

1-(4-ethylphenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7c)



60% (38 mg) isolated yield, colorless oil, dr = 1 : 1.17. ^1H NMR (400 MHz, CDCl_3) δ 7.25 – 7.21 (m, 4H), 7.20 – 7.17 (m, 2H), 7.12 – 7.08 (m, 2H), 7.06 – 6.99 (m, 3H), 6.65 – 6.60 (m, 1H), 6.52 – 6.47 (m, 2H), 4.98 – 4.96 (m, $0.46 \times 1\text{H}$), 4.83 (d, J = 5.2 Hz, $0.54 \times 1\text{H}$), 4.62 (d, J = 5.2 Hz, $0.46 \times 1\text{H}$), 4.51 (d, J = 5.6 Hz, $0.54 \times 1\text{H}$), 2.64 – 2.58 (m, 2H), 2.47 (brs, $0.54 \times 1\text{H}$), 2.29 (brd, J = 5.2 Hz, $0.46 \times 1\text{H}$), 1.20 (td, J = 7.6, 4.4 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 147.26, 146.78, 144.08, 143.88, 140.37, 138.76, 137.80, 137.11, 129.02, 128.98, 128.48, 128.24, 127.86, 127.73, 127.71, 127.51, 127.38, 127.24, 126.52, 126.46, 117.75, 117.66, 113.98, 113.86, 77.82, 77.15, 64.41, 63.61, 28.49, 15.52. HRMS (ESI): m/z calcd for $\text{C}_{22}\text{H}_{23}\text{NO}$ $[\text{M} + \text{H}]^+$ 318.1852, found 318.1853.

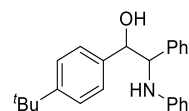
1-(4-isopropylphenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7d)



61% (40 mg) isolated yield, colorless oil, dr = 1 : 1.13. ^1H NMR (400 MHz, CDCl_3) δ 7.24 – 7.20 (m, 4H), 7.19 – 7.16 (m, 2H), 7.15 – 7.12 (m, 2H), 7.06 – 7.01 (m, 3H), 6.64 – 6.60 (m, 1H), 6.51 – 6.47 (m, 2H), 4.97 – 4.93 (m, $0.47 \times 1\text{H}$), 4.83 (d, J = 5.2 Hz, $0.53 \times 1\text{H}$), 4.61 (d, J = 5.2 Hz, $0.47 \times 1\text{H}$), 4.52 (d, J = 5.2

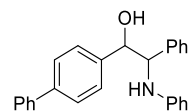
Hz, 0.53 × 1H), 2.91 – 2.83 (td, J = 12.4, 6.8 Hz, 1H), 2.45 (brs, 0.53 × 1H), 2.27 (brd, J = 5.2 Hz, 0.47 × 1H), 1.24 – 1.20 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 148.75, 148.51, 147.24, 146.78, 140.44, 138.84, 137.96, 137.26, 129.01, 128.98, 128.48, 128.25, 127.86, 127.52, 127.37, 127.22, 126.54, 126.42, 126.31, 126.28, 117.73, 117.59, 113.93, 113.86, 77.79, 77.18, 64.24, 63.62, 33.75, 23.94, 23.92. HRMS (ESI): m/z calcd for $\text{C}_{23}\text{H}_{25}\text{NO}$ [$\text{M} + \text{H}$] $^+$ 332.2009, found 332.2008.

1-(4-(tert-butyl)phenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7e)



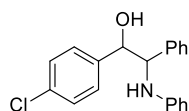
63% (43 mg) isolated yield, colorless oil, dr = 1 : 1.13. ^1H NMR (400 MHz, CDCl_3) δ 7.31 – 7.30 (m, 2H), 7.26 – 7.19 (m, 6H), 7.06 – 7.02 (m, 3H), 6.64 – 6.60 (m, 1H), 6.51 – 6.47 (m, 2H), 4.95 (d, J = 8.0 Hz, 0.47 × 1H), 4.85 (d, J = 5.2 Hz, 0.53 × 1H), 4.61 (d, J = 5.2 Hz, 0.47 × 1H), 4.53 (d, J = 5.2 Hz, 0.53 × 1H), 2.40 (brs, 0.53 × 1H), 2.25 (brs, 0.47 × 1H), 1.29 (d, J = 3.2 Hz, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 151.05, 150.77, 147.21, 146.78, 140.50, 138.90, 137.59, 136.87, 129.01, 128.98, 128.50, 128.27, 127.86, 127.54, 127.39, 127.20, 126.29, 126.13, 125.19, 125.16, 117.73, 117.56, 113.90, 113.88, 77.71, 77.16, 64.09, 63.62, 34.52, 34.48, 31.29.

1-([1,1'-biphenyl]-4-yl)-2-phenyl-2-(phenylamino)ethan-1-ol (7f)



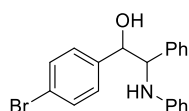
74% (54 mg) isolated yield, colorless oil, dr = 1 : 1.22. ^1H NMR (400 MHz, CDCl_3) δ 7.57 – 7.54 (m, 2H), 7.49 (d, J = 7.6 Hz, 2H), 7.43 – 7.40 (m, 2H), 7.31 (d, J = 8.4 Hz, 2H), 7.24 – 7.21 (m, 4H), 7.18 – 7.11 (m, 2H), 7.08 – 7.04 (m, 2H), 6.66 – 6.63 (m, 1H), 6.55 – 6.51 (m, 2H), 5.06 (d, J = 4.8 Hz, 0.45 × 1H), 4.87 (d, J = 5.6 Hz, 0.55 × 1H), 4.68 (d, J = 4.8 Hz, 0.45 × 1H), 4.55 (d, J = 6.0 Hz, 0.55 × 1H), 2.66 (brs, 0.55 × 1H), 2.44 (brs, 0.45 × 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 147.15, 146.67, 140.67, 140.59, 140.57, 140.47, 140.14, 139.56, 138.90, 138.39, 129.07, 129.02, 128.75, 128.72, 128.54, 128.27, 127.86, 127.58, 127.49, 127.36, 127.28, 127.24, 126.99, 126.98, 126.96, 126.86, 126.81, 117.88, 117.84, 114.05, 113.90, 77.71, 76.89, 64.50, 63.54. HRMS (ESI): m/z calcd for $\text{C}_{26}\text{H}_{23}\text{NO}$ [$\text{M} + \text{H}$] $^+$ 366.1852, found 366.1848.

1-(4-chlorophenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7g)



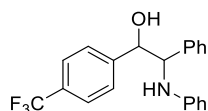
55% (36 mg) isolated yield, pale yellow oil, dr = 1: 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.26 – 7.14 (m, 7H), 7.10 – 7.04 (m, 3H), 6.98 – 6.95 (m, 1H), 6.66 (t, *J* = 7.8 Hz, 1H), 6.57 – 6.48 (m, 2H), 5.04 (t, *J* = 4.8 Hz, 0.50 × 1H), 4.80 (d, *J* = 6.0 Hz, 0.50 × 1H), 4.63 (d, *J* = 4.8 Hz, 0.50 × 1H), 4.45 (d, *J* = 6.0 Hz, 0.50 × 1H), 2.71 (brs, 0.50 × 1H), 2.42 (brd, *J* = 5.6 Hz, 0.50 × 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 146.99, 146.51, 139.74, 139.00, 138.47, 137.90, 133.61, 133.48, 129.13, 129.08, 128.63, 128.33, 128.30, 128.27, 127.91, 127.84, 127.80, 127.71, 127.65, 127.19, 118.15, 118.10, 114.17, 113.94, 77.35, 76.30, 64.76, 63.49.

1-(4-bromophenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7h)



50% (37 mg) isolated yield, pale yellow oil, dr = 1.13 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.39 – 7.36 (m, 2H), 7.25 – 7.19 (m, 4H), 7.11 – 7.05 (m, 4H), 6.91 (d, *J* = 8.4 Hz, 1H), 6.68 – 6.64 (m, 1H), 6.55 – 6.50 (m, 2H), 5.06 – 5.01 (m, 0.53 × 1H), 4.80 (d, *J* = 6.0 Hz, 0.47 × 1H), 4.64 (d, *J* = 4.8 Hz, 0.53 × 1H), 4.45 (d, *J* = 6.4 Hz, 0.47 × 1H), 2.74 (brs, 0.53 × H), 2.46 (brs, 0.47 × 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 146.98, 146.52, 139.74, 139.56, 139.02, 137.89, 131.24, 131.20, 129.13, 129.08, 128.64, 128.34, 128.26, 128.19, 127.81, 127.71, 127.66, 127.20, 121.78, 121.66, 118.14, 118.10, 114.17, 113.94, 77.39, 76.33, 64.70, 63.44. **HRMS** (ESI): *m/z* calcd for C₂₀H₁₈BrNO [M + H]⁺ 368.0645, found 368.0645.

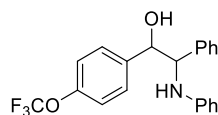
2-phenyl-2-(phenylamino)-1-(4-(trifluoromethyl)phenyl)ethan-1-ol (7i)



61% (43 mg) isolated yield, colorless oil, dr = 1 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.50 (d, *J* = 6.4 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.25 – 7.20 (m, 4H), 7.15 (d, *J* = 8.0 Hz, 1H), 7.10 – 7.04 (m, 3H), 6.69 – 6.64 (m, 1H), 6.56 – 6.52 (m, 2H), 5.13 (d, *J* = 4.4 Hz, 0.5 × 1H), 4.89 (d, *J* = 6.0 Hz, 0.5 × 1H), 4.68 (d, *J* = 4.4 Hz, 0.5 × 1H), 4.50 (d, *J* = 5.6 Hz, 0.5 × 1H), 2.82 (brs, 0.5 × 1H), 2.57 (brs, 0.5 × 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 146.86, 146.44, 144.54, 144.53, 144.08, 144.07, 139.62, 137.62, 129.95 (qd, *J*_{C-F} = 32.2, 7.6 Hz), 129.17, 129.11, 128.72, 128.37, 127.82, 127.78, 127.77, 127.15, 126.84, 126.82, 125.12 – 124.93 (m), 124.03 (qd, *J*_{C-F} = 270.3, 1.9 Hz), 118.26, 114.20, 114.01, 77.40, 76.28, 64.67, 63.51. **¹⁹F NMR** (376

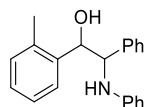
MHz, CDCl₃) δ -62.41, -62.42. **HRMS** (ESI): m/z calcd for C₂₁H₁₈F₃NO [M + H]⁺ 358.1413, found 358.1410.

2-phenyl-2-(phenylamino)-1-(4-(trifluoromethoxy)phenyl)ethan-1-ol (7j)



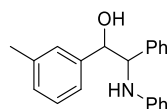
51% (38 mg) isolated yield, colorless oil, dr = 1 : 1.13. **¹H NMR** (400 MHz, CDCl₃) δ 7.26 – 7.21 (m, 4H), 7.19 – 7.16 (m, 1H), 7.12 – 7.05 (m, 6H), 6.66 (t, J = 7.2 Hz, 1H), 6.55 – 6.51 (m, 2H), 5.07 (d, J = 4.4 Hz, 0.47 \times 1H), 4.84 (d, J = 6.0 Hz, 0.53 \times 1H), 4.63 (d, J = 4.8 Hz, 0.47 \times 1H), 4.47 (d, J = 6.0 Hz, 0.53 \times 1H), 2.74 (brs, 0.53 \times 1H), 2.46 (brs, 0.47 \times 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 148.83, 148.81, 148.79, 148.78, 148.74, 148.72, 148.70, 148.69, 147.03, 146.57, 139.80, 139.28, 138.78, 137.94, 129.21, 129.16, 128.71, 128.41, 128.04, 127.98, 127.86, 127.84, 127.77, 127.23, 120.68, 120.61, 120.46 (q, J_{C-F} = 255.6) 118.26, 118.22, 114.27, 113.96, 77.27, 76.27, 64.83, 63.65. **¹⁹F NMR** (376 MHz, CDCl₃) δ -57.82, -57.83.

2-phenyl-2-(phenylamino)-1-(o-tolyl)ethan-1-ol (7k)



53% (32 mg) isolated yield, colorless oil, dr = 1 : 1.08. **¹H NMR** (400 MHz, CDCl₃) δ 7.62 (d, J = 6.4 Hz, 0.47 \times 1H), 7.24 – 7.17 (m, 6H), 7.09 – 7.03 (m, 4H), 6.93 (d, J = 7.6 Hz, 1H), 6.64 (t, J = 7.2 Hz, 1H), 6.53 – 6.49 (m, 2H), 5.29 – 5.26 (m, 0.48 \times 1H), 5.05 (d, J = 6.4 Hz, 0.52 \times 1H), 4.62 (d, J = 4.4 Hz, 0.48 \times 1H), 4.50 (d, J = 6.0 Hz, 0.52 \times 1H), 2.43 (brd, J = 3.2 Hz, 0.52 \times 1H), 2.26 (s, 1.40 \times 1H), 2.20 (brd, J = 5.2 Hz, 0.48 \times 1H), 1.96 (s, 1.59 \times 1H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.33, 146.71, 140.18, 139.01, 138.31, 138.14, 135.43, 135.07, 130.36, 130.22, 129.06, 128.99, 128.45, 128.11, 128.08, 127.71, 127.67, 127.55, 127.43, 127.13, 126.31, 126.27, 126.14, 125.84, 117.75, 117.73, 114.09, 113.84, 74.05, 73.52, 63.78, 62.12, 19.16, 18.95.

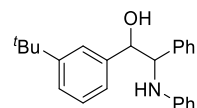
2-phenyl-2-(phenylamino)-1-(m-tolyl)ethan-1-ol (7l)



53% (32 mg) isolated yield, colorless oil, dr = 1 : 1.17. **¹H NMR** (400 MHz, CDCl₃) δ 7.25 – 7.22 (m, 4H), 7.17 – 7.14 (m, 2H), 7.09 – 7.03 (m, 4H), 6.90 – 6.87 (m, 1H), 6.63 (t, J = 7.4 Hz, 1H), 6.53 – 6.48 (m, 2H), 4.97 (d, J = 5.2 Hz, 0.46 \times 1H), 4.83 (d, J = 5.6 Hz, 0.54 \times 1H), 4.63 (d, J = 5.2 Hz, 0.46 \times 1H), 4.51 (d,

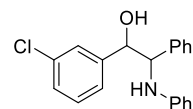
$J = 5.6$ Hz, $0.54 \times 1\text{H}$), 2.29 (d, $J = 6.8$ Hz, 3H). **^{13}C NMR** (100 MHz, CDCl_3) δ 147.24, 146.75, 140.44, 140.28, 139.84, 138.63, 137.90, 137.84, 129.04, 128.99, 128.75, 128.59, 128.50, 128.23, 128.09, 127.87, 127.57, 127.44, 127.23, 127.11, 123.60, 123.53, 117.80, 117.75, 114.05, 113.89, 77.97, 77.26, 64.56, 63.64, 21.41, 21.39. **HRMS** (ESI): m/z calcd for $\text{C}_{21}\text{H}_{21}\text{NO}$ $[\text{M} + \text{H}]^+$ 304.1696, found 304.1693.

1-(3-(tert-butyl)phenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7m)



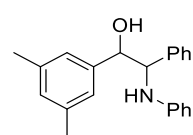
63% (43 mg) isolated yield, colorless oil, dr = 1 : 1.08. **^1H NMR** (400 MHz, CDCl_3) δ 7.28 – 7.14 (m, 8H), 7.09 – 7.04 (m, 3H), 6.64 (t, $J = 7.2$ Hz, 1H), 6.54 – 6.49 (m, 2H), 5.10 – 5.06 (m, $0.48 \times 1\text{H}$), 4.82 (d, $J = 5.2$ Hz, $0.52 \times 1\text{H}$), 4.68 – 4.63 (m, $0.48 \times 1\text{H}$), 4.46 (d, $J = 6.4$ Hz, $0.52 \times 1\text{H}$), 2.57 (brs, $0.52 \times 1\text{H}$), 2.40 (brd, $J = 5.6$ Hz, $0.48 \times 1\text{H}$), 1.19 (d, $J = 9.2$ Hz, 9H). **^{13}C NMR** (100 MHz, CDCl_3) δ 150.84, 150.81, 147.34, 146.75, 140.15, 140.00, 139.29, 138.43, 129.06, 129.00, 128.40, 128.20, 127.97, 127.91, 127.83, 127.44, 127.38, 127.31, 124.87, 124.77, 123.98, 123.74, 123.38, 123.35, 117.82, 117.78, 114.11, 113.84, 78.42, 77.35, 64.99, 63.51, 34.54, 31.21, 31.16. **HRMS** (ESI): m/z calcd for $\text{C}_{24}\text{H}_{27}\text{NO}$ $[\text{M} + \text{H}]^+$ 346.2165, found 346.2165.

1-(3-chlorophenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7n)



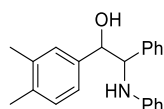
66% (43 mg) isolated yield, colorless oil, dr = 1 : 1.56. **^1H NMR** (400 MHz, CDCl_3) δ 7.30 – 7.16 (m, 7H), 7.13 – 7.01 (m, 4H), 6.68 – 6.64 (m, 1H), 6.55 – 6.51 (m, 2H), 5.04 (d, $J = 8.0$ Hz, $0.39 \times 1\text{H}$), 4.83 (d, $J = 5.6$ Hz, $0.39 \times 1\text{H}$), 4.65 (d, $J = 4.8$ Hz, $0.39 \times 1\text{H}$), 4.48 (d, $J = 6.0$ Hz, $0.61 \times 1\text{H}$), 2.69 (brs, $0.33 \times 1\text{H}$), 2.40 (brs, $0.37 \times 1\text{H}$). **^{13}C NMR** (100 MHz, CDCl_3) δ 146.93, 146.47, 142.62, 142.16, 139.73, 137.79, 134.12, 129.37, 129.34, 129.12, 129.06, 128.65, 128.32, 128.00, 127.92, 127.78, 127.70, 127.15, 126.69, 126.64, 124.67, 124.62, 118.10, 114.16, 113.97, 77.20, 76.29, 64.62, 63.48. **HRMS** (ESI): m/z calcd for $\text{C}_{20}\text{H}_{18}\text{ClNO}$ $[\text{M} + \text{H}]^+$ 324.1150, found 324.1149.

1-(3,5-dimethylphenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7o)



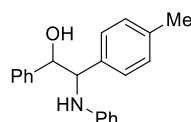
58% (36 mg) isolated yield, colorless oil, dr = 1 : 1.04. **¹H NMR** (400 MHz, CDCl₃) δ 7.28 – 7.16 (m, 6H), 7.06 – 7.02 (m, 2H), 6.87 (s, 1H), 6.70 (d, *J* = 1.2 Hz, 1H), 6.62 (t, *J* = 7.2 Hz, 1H), 6.52 – 6.47 (m, 2H), 4.93 – 4.86 (m, 0.49 × 1H), 4.79 (d, *J* = 2.8 Hz, 0.51 × 1H), 4.59 (d, *J* = 4.0 Hz, 0.49 × 1H), 4.50 (d, *J* = 5.2 Hz, 0.51 × 1H), 2.25 (d, *J* = 3.6 Hz, 6H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.25, 146.76, 140.38, 140.37, 139.74, 138.82, 137.77, 137.69, 129.66, 129.44, 129.00, 128.95, 128.47, 128.20, 127.87, 127.56, 127.40, 127.22, 124.37, 124.15, 117.73, 117.63, 114.01, 113.89, 77.92, 77.38, 64.41, 63.61, 21.29, 21.27. **HRMS** (ESI): *m/z* calcd for C₂₂H₂₃NO [M + H]⁺ 318.1852, found 318.1852.

1-(3,4-dimethylphenyl)-2-phenyl-2-(phenylamino)ethan-1-ol (7p)



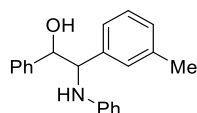
57% (36 mg) isolated yield, colorless oil, dr = 1.22 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.25 – 7.18 (m, 5H), 7.06 – 6.96 (m, 4H), 6.86 – 6.80 (m, 1H), 6.62 (t, *J* = 7.2 Hz, 1H), 6.49 (t, *J* = 7.4 Hz, 2H), 4.92 – 4.89 (m, 0.55 × 1H), 4.80 (d, *J* = 5.6 Hz, 0.45 × 1H), 4.60 (d, *J* = 5.4 Hz, 0.55 × 1H), 4.50 (d, *J* = 5.6 Hz, 0.45 × 1H), 2.21 (dd, *J* = 9.2, 6.8 Hz, 6H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.38, 146.90, 140.52, 139.04, 138.04, 137.33, 136.58, 136.49, 136.46, 136.20, 129.53, 129.51, 129.08, 129.05, 128.56, 128.32, 127.96, 127.89, 127.70, 127.60, 127.45, 127.35, 124.08, 123.93, 117.80, 117.71, 114.07, 113.98, 77.86, 77.35, 64.47, 63.71, 19.87, 19.85, 19.56, 19.54. **HRMS** (ESI): *m/z* calcd for C₂₂H₂₃NO [M + H]⁺ 318.1852, found 318.1850.

1-phenyl-2-(phenylamino)-2-(p-tolyl)ethan-1-ol (7q)



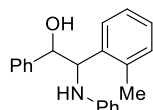
61% (36 mg) isolated yield, colorless oil, dr = 1 : 1.04. **¹H NMR** (400 MHz, CDCl₃) δ 7.28 – 7.26 (m, 3H), 7.11 – 6.99 (m, 8H), 6.63 (t, *J* = 7.2 Hz, 1H), 6.52 – 6.47 (m, 2H), 4.99 (d, *J* = 4.4 Hz, 0.49 × 1H), 4.83 (d, *J* = 5.6 Hz, 0.51 × 1H), 4.61 (d, *J* = 4.8 Hz, 0.49 × 1H), 4.48 (d, *J* = 5.6 Hz, 0.51 × 1H), 2.28 (d, *J* = 2.8 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.25, 146.76, 140.58, 140.04, 137.17, 137.05, 137.03, 135.25, 129.23, 129.03, 129.01, 128.99, 128.18, 127.90, 127.76, 127.68, 127.07, 126.51, 117.76, 117.74, 114.04, 113.85, 77.94, 77.14, 64.28, 63.37, 21.09, 21.06. **HRMS** (ESI): *m/z* calcd for C₂₁H₂₁NO [M + H]⁺ 304.1696, found 304.1696.

1-phenyl-2-(phenylamino)-2-(m-tolyl)ethan-1-ol (7r)



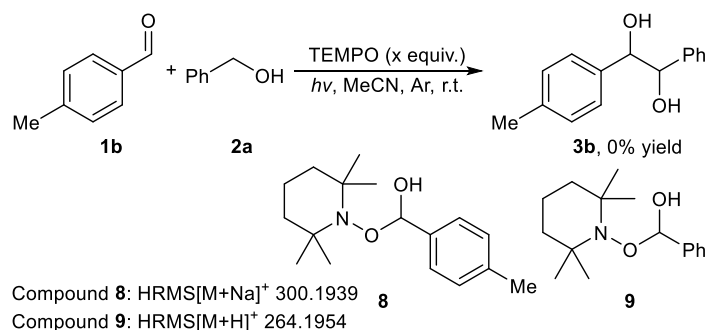
68% (41 mg) isolated yield, colorless oil, dr = 1.08 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.30 – 7.20 (m, 3H), 7.15 – 7.00 (m, 7H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.65 – 6.61 (m, 1H), 6.52 – 6.48 (m, 2H), 4.97 (d, *J* = 4.2 Hz, 0.52 × 1H), 4.84 (d, *J* = 5.6 Hz, 0.48 × 1H), 4.59 (d, *J* = 4.8 Hz, 0.52 × 1H), 4.47 (d, *J* = 5.6 Hz, 0.48 × 1H), 2.56 (brs, 0.43 × 1H), 2.34 (brs, 0.57 × 1H), 2.26 (d, *J* = 5.6 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.29, 146.80, 140.57, 140.16, 139.96, 138.42, 138.13, 137.85, 129.04, 128.99, 128.45, 128.40, 128.39, 128.26, 128.16, 127.96, 127.79, 127.76, 126.55, 126.43, 124.84, 124.23, 117.77, 117.68, 113.96, 113.83, 77.88, 77.19, 64.52, 63.69, 21.45, 21.44. **HRMS** (ESI): *m/z* calcd for C₂₁H₂₁NO [M + H]⁺ 304.1696, found 304.1694.

1-phenyl-2-(phenylamino)-2-(o-tolyl)ethan-1-ol (7s)



66% (40 mg) isolated yield, colorless oil, dr = 3 : 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.28 – 7.18 (m, 4H), 7.16 – 7.01 (m, 7H), 6.63 (t, *J* = 7.2 Hz, 1H), 6.49 – 6.42 (m, 2H), 5.02 (t, *J* = 4.0 Hz, 0.75 × 1H), 4.90 (d, *J* = 4.8 Hz, 0.75 × 1H), 4.85 (d, *J* = 5.6 Hz, 0.25 × 1H), 4.73 (d, *J* = 5.6 Hz, 0.25 × 1H), 2.69 (brs, 0.25 × 1H), 2.44 (brs, 0.75 × 1H), 2.13 (d, *J* = 10.0 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ 147.23, 146.74, 140.50, 139.91, 138.39, 136.66, 136.55, 135.76, 130.65, 130.29, 129.10, 129.06, 128.14, 128.08, 127.72, 127.31, 126.91, 126.77, 126.44, 126.22, 126.10, 117.81, 115.10, 113.80, 113.63, 76.64, 76.53, 60.41, 59.01, 19.27, 19.20. **HRMS** (ESI): *m/z* calcd for C₂₁H₂₁NO [M + H]⁺ 304.1696, found 304.1696.

7. Radical Trapping Experiments



In an argon-filled glovebox, a 10 mL flame-dried quartz tube with magnetic stirring was charged sequentially with 2,2,6,6-tetramethylpiperidinoxy (TEMPO) (0.8 mmol, 4.0 eq), MeCN (2 mL), *p*-tolualdehyde **1b** (0.2 mmol, 1.0 eq) and benzyl alcohol **2a**

(0.3 mmol, 1.5 eq) sequentially added into the quartz tube. Then, the quartz tube was removed from glovebox. After stirring the mixture at room temperature under 10 W UVA light (365–370 nm) for 36 h, the 1,2-diol **3b** not obtained. Moreover, the ketyl radical addition product **8** and **9** were detected by HRMS. HRMS (ESI) of compound **8** : m/z calcd for C₁₇H₂₇NO₂Na [M + Na]⁺ 300.1934, found 300.1939. HRMS (ESI) of compound **9**: m/z calcd for C₁₆H₂₆NO₂ [M + H]⁺ 264.1958, found 264.1954.

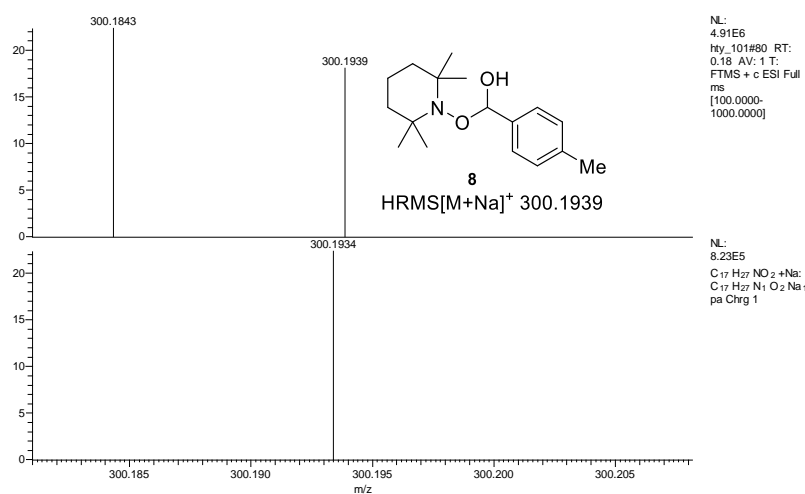


Figure S1. HRMS (ESI) of ketyl radical addition product **8**.

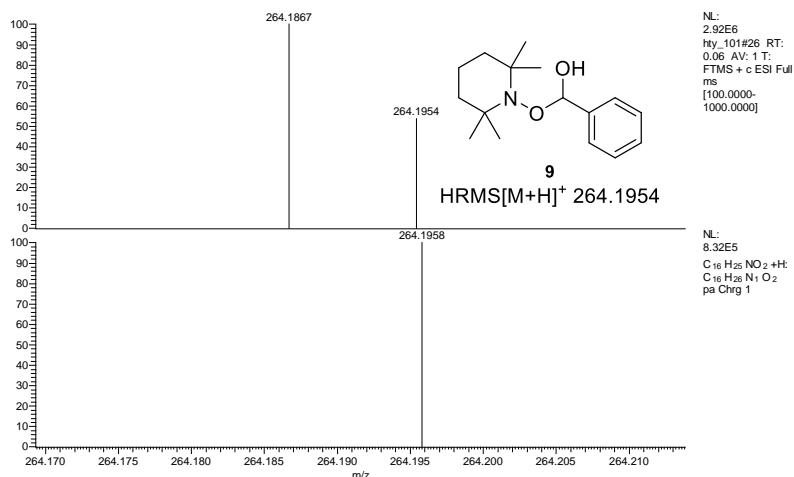
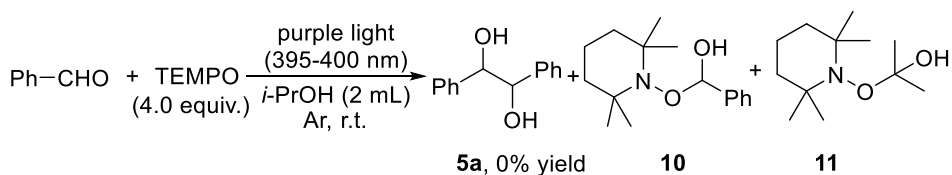


Figure S2. HRMS (ESI) of ketyl radical addition product **9**.



In an argon-filled glovebox, a 10 mL flame-dried quartz tube with magnetic stirring

was charged sequentially with 2,2,6,6-tetramethylpiperidinoxy (TEMPO) (0.8 mmol, 4.0 eq), *i*-PrOH (2 mL) and benzaldehyde (0.2 mmol, 1.0 eq) sequentially added into the quartz tube. Then, the quartz tube was removed from glovebox. The mixture was stirred at room temperature under 10 W purple light for 12 h. The pinacol coupling of benzaldehyde was inhibited completely by the addition of 4.0 equiv. of 2,2,6,6-tetramethylpiperidinoxy (TEMPO) as a radical scavenger. Moreover, the ketyl radical addition product **10** and carbon center radical addition product **11** were detected by HRMS. HRMS (ESI) of compound **10**: m/z calcd for $C_{16}H_{26}NO_2$ $[M + H]^+$ 264.1958, found 264.1951. HRMS (ESI) of compound **11**: m/z calcd for $C_{12}H_{26}NO_2$ $[M + H]^+$ 216.1958, found 216.1955.

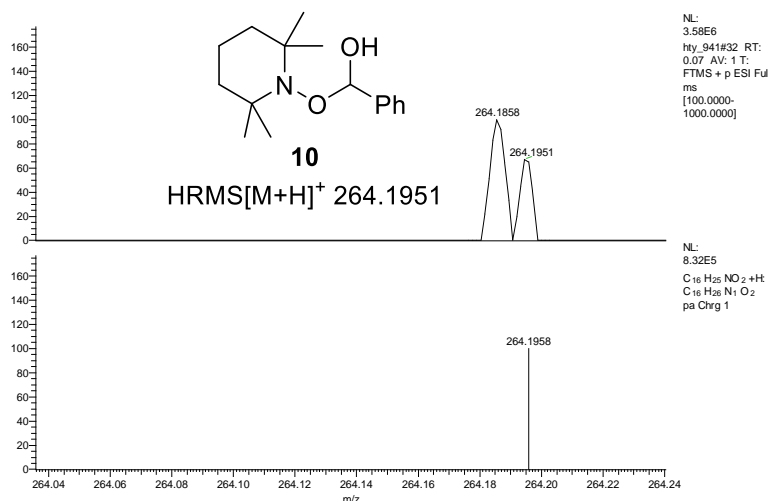


Figure S3. HRMS (ESI) of ketyl radical addition product **10**.

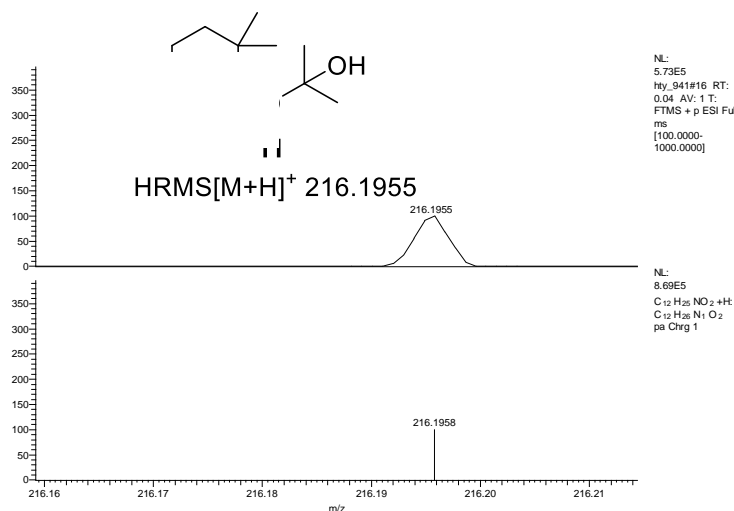


Figure S4. HRMS (ESI) of carbon center radical addition product **11**.

8. Cyclic voltammetry analysis

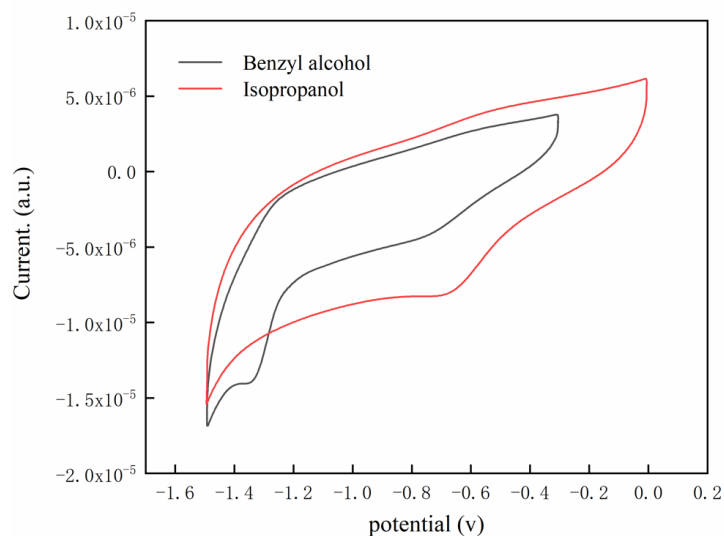


Figure S5. Cyclic voltammograms during controlled potential coulometry of 5.0 mmol benzyl alcohol or isopropanol in aqueous solution of phosphatebuffer (30 mL, pH = 7.8–8.0) and MeCN (1mL). Scan rate: 100 mV s^{-1} ; $t = 25 \pm 1 \text{ }^{\circ}\text{C}$.

9. UV-vis Studies

In a glass tube, 4-(trifluoromethyl)benzaldehyde (0.2 mmol, 28 μL), benzyl alcohol (0.2 mmol, 21 μL) or its mixture into MeCN (20 mL). The glass tube was placed in the UV-Vis and a wavelength scan from 450 nm to 335 nm.

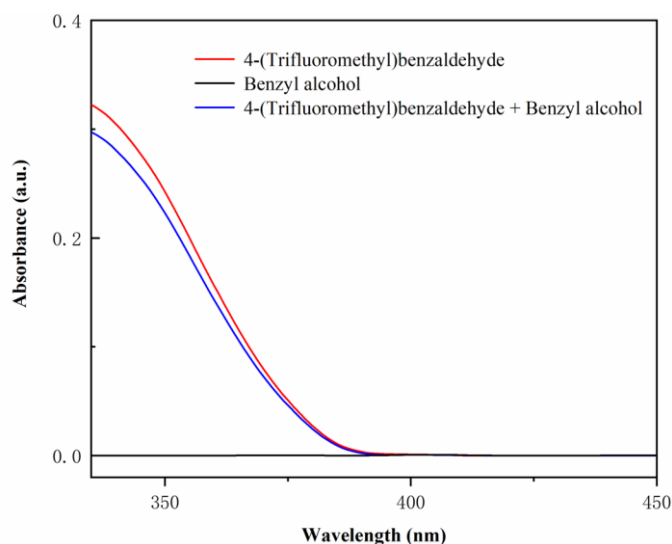


Figure S6. UV-vis absorption spectrum of 4-(trifluoromethyl)benzaldehyde, benzyl alcohol or its mixture in MeCN (10^{-2} mmol/mL).

In a glass tube, benzaldehyde (1.1 mmol, 112 μL), benzenamine (0.011 mmol, 2.0 mg), $i\text{Pr}_2\text{Net}$ (0.11 mmol, 19 μL) or its mixture into MeCN (10 mL) and H_2O (1 mL). The glass tube was placed in the UV-Vis and a wavelength scan from 390 nm to 340

nm.

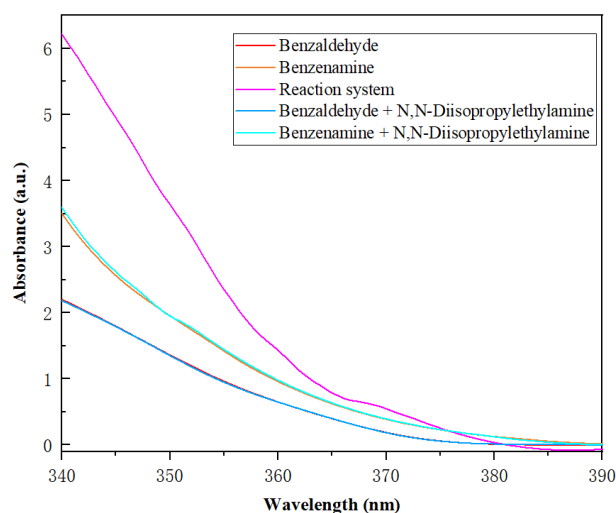
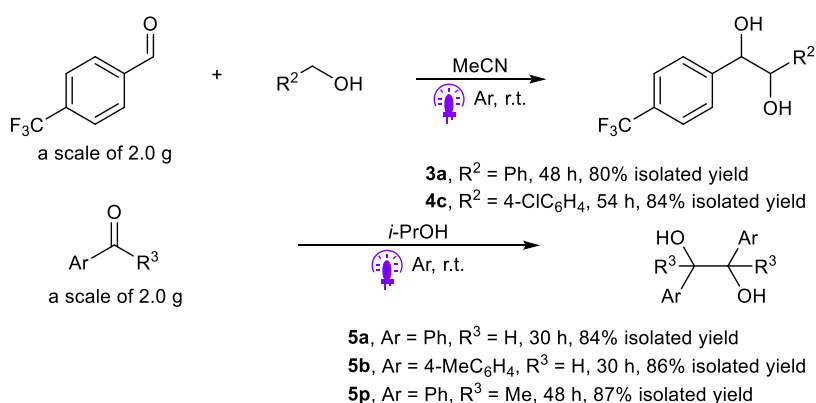


Figure S7. UV-vis absorption spectrum of benzaldehyde, benzenamine, reaction system or its mixture in MeCN and H₂O.

10. Gram-scale synthesis



The flange was then evacuated by oil pump for 30 min before the injection of 2.0 g of substrates **1** dissolved in 20 mL solvent.^[4] The mixture was stirred at room temperature under 10 W UVA LED (365–370 nm) until the reaction was completed, as monitored by TLC analysis. The reaction mixture was then concentrated in vacuum. The crude product was purified by flash column chromatography (silica gel, PE/EA) to afford the desired product.

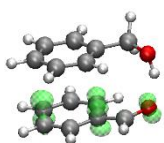
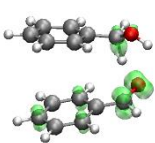
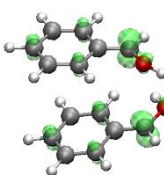
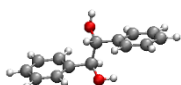
11. Computational details

All the calculations were carried out with Gaussian 16 software package.⁵ Time dependent density functional theory (TD-DFT) calculation was also performed at M06/6-311G(d,p) level of theory and the excited states were reported.⁶⁻¹⁰ Geometry

optimizations of reactants, products, intermediates, and transition states were carried out at M06/6-311G(d,p) level.¹¹ The vibration frequencies of all stationary points were also calculated at the same level to prove that the optimized structure is a stable point or a transition state. The minimum-energy path (MEP) was constructed from the transition state by using the intrinsic reaction coordinate method (IRC) to prove that the optimized transition state is the desired one.¹⁰⁻¹² Route 2 and Route 3 use the solvent effect was considered by the polarization continuous model (PCM) with 2-propanol as solvent.^{13, 14} The Minimum Energy Crossing Points (MECP) were located by KST48^{15, 16} with Gaussian 16 software at M06/6-311G(d,p)/PCM(2-propanol) level of theory. The spin population analysis was conducted using Multiwfn 3.8 (dev)¹⁷ and visualized with VMD¹⁸.

11.1 The spin density and the mulliken atomic spin populations

Table S5. The spin density and the sums of mulliken atomic spin populations, both for hydrogen atom donor and acceptor, are presented for T1, TS1-1, IM1-1 and IM1-2 (isovalue: 0.02).

Name	T1	TS1-1	IM1-1	IM1-2
Spin density				
Sum of hydrogen atom donor spin populations	0.233	0.502	1.000	-
Sum of hydrogen atom acceptor spin populations	1.813	1.522	1.004	-

The spin density in Table S5, along with the mulliken atomic spin populations analysis, indicate that under the T1 state, the unpaired electron is mainly distributed on the hydrogen atom acceptor benzaldehyde, with the spin populations of 1.813. Subsequently, as the hydrogen atom transfers towards the hydrogen atom donor via the transition state TS1-1, with the spin populations of 1.522, each of the two radicals eventually carries an unpaired electron upon the completion of hydrogen transfer (IM1-1). Therefore, this reaction process is a hydrogen atom transfer

process.

11.2 Kohn-Sham orbitals

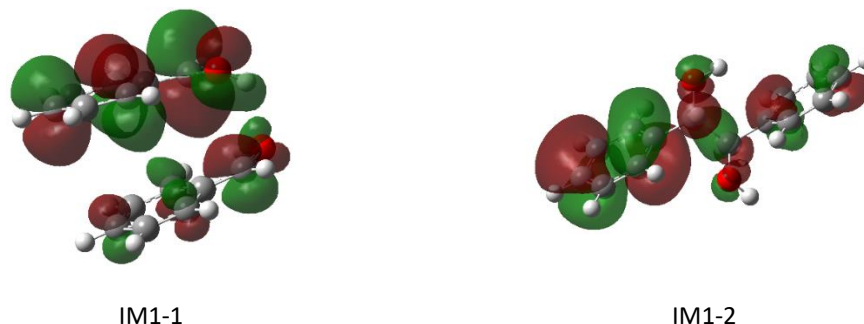


Figure S8. Kohn-Sham orbitals of IM1-1 and IM1-2.

11.3 Potential energy surface scanning

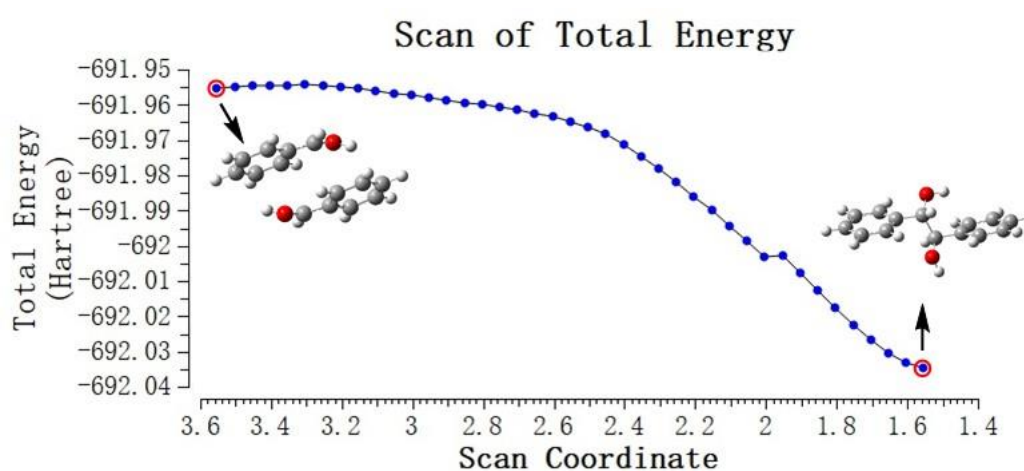
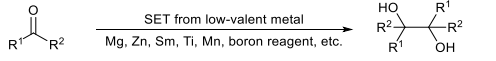
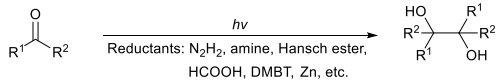
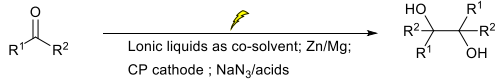
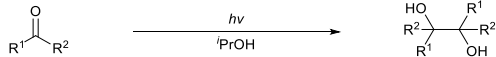


Figure S9. Potential energy surface scanning.

From IM1-1 to IM1-2 represents a radical cross-coupling process. To assist in locating the transition state for this process, we perform potential energy surface scanning, scanning the entire process from the radical to the target product (IM1-2). The scanning results are presented in Fig. S9. As the scanning progressed, no distinct peak emerged in the graph, suggesting that a transition state may not exist for the radical coupling process.

12. Comparison with previous work

	Reaction	Metal	Reductant or additives	E-factor
Previous work	Traditional homo-coupling of aldehydes: 	Excessive	Metal reductant or boron reagent. etc.	E = 23.3 (R ¹ = Ph, R ² = H, <i>J. Am. Chem. Soc.</i> 2004, 126 , 13198–13199.)
	Light-induced homo-coupling of aldehydes: 	Metal-based Photosensitizer	Dangerous and toxic reductant	E = 91.7 (R ¹ = Ph, R ² = H, <i>J. Org. Chem.</i> 2024, 89 , 11537–11541.)
	Electrochemical homo-coupling of aldehydes: 	Costly electrodes	NaN ₃ or acids	E = 77.9 (R ¹ = 4-CNPh, R ² = H, <i>J. Org. Chem.</i> 2025, 90 , 2139–2147.)
This Work	homo-coupling reactions of aldehydes/ketones: 	No	No	E = 3.9 (R ¹ = Ph, R ² = H)

Note: The E-factor (environmental factor) is defined as the ratio of the total mass of waste generated to the mass of the target product produced. In this calculation, solvents are accounted for with a 50% recovery rate. In this work, the E-factor was determined at gram scale as the ratio of total waste mass (g) to product mass (g), with solvent contributions corrected for 50% recovery.

Comparison of the cross-coupling reaction between carbonyls and alcohols/imines with previous works: As showed in the introduction section of this manuscript, the simple 1,2-addition of carbonyls using abundant non-organohalides (such as alcohols, aldehydes or imines), without the need of metal, has not been explored. Based on calculations for the gram-scale reaction involving compound **4a**, an E-factor of 3.0 was determined, accounting for a 50% solvent recovery rate.

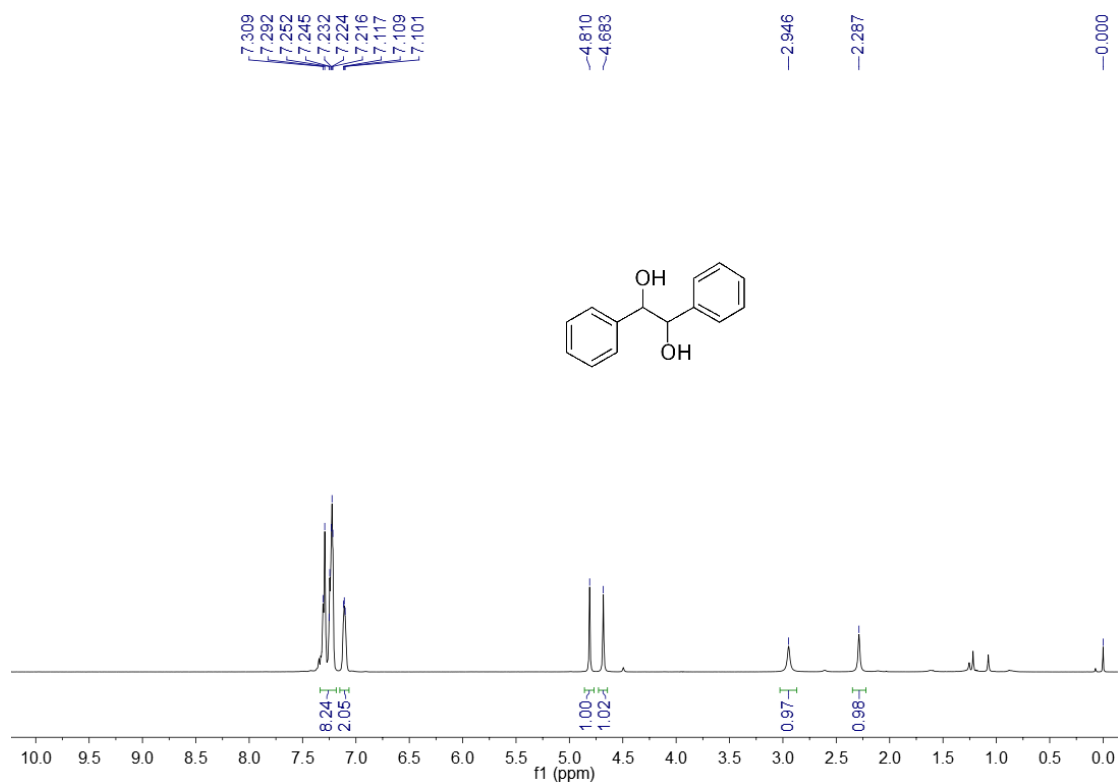
13. References

- (1) W. L. F. Armarego, C. C. L. Chai, Purification of Laboratory Chemicals. Butterworth- Heinemann, 5th ed (2003).
- (2) Y. Yang, G. Li, J. Ma, C. Wang, J. Xiao, D. Xue, *Green Chem.*, 2023, **25**, 4129–4136.
- (3) Q. Shen, K. Cao, Q. Chen, X. Li, N. Zhang, Y.-B. Miao, J. Li, *Green Chem.*, 2023, **25**, 9665–9671.
- (4) M. Liu, Y. Wang, X. Kong, R. T. Rashid, S. Chu, C.-C. Li, Z. Hearne, H. Guo, Z. Mi, C.-J. Li, *Chem.*, 2019, **5**, 858–867.

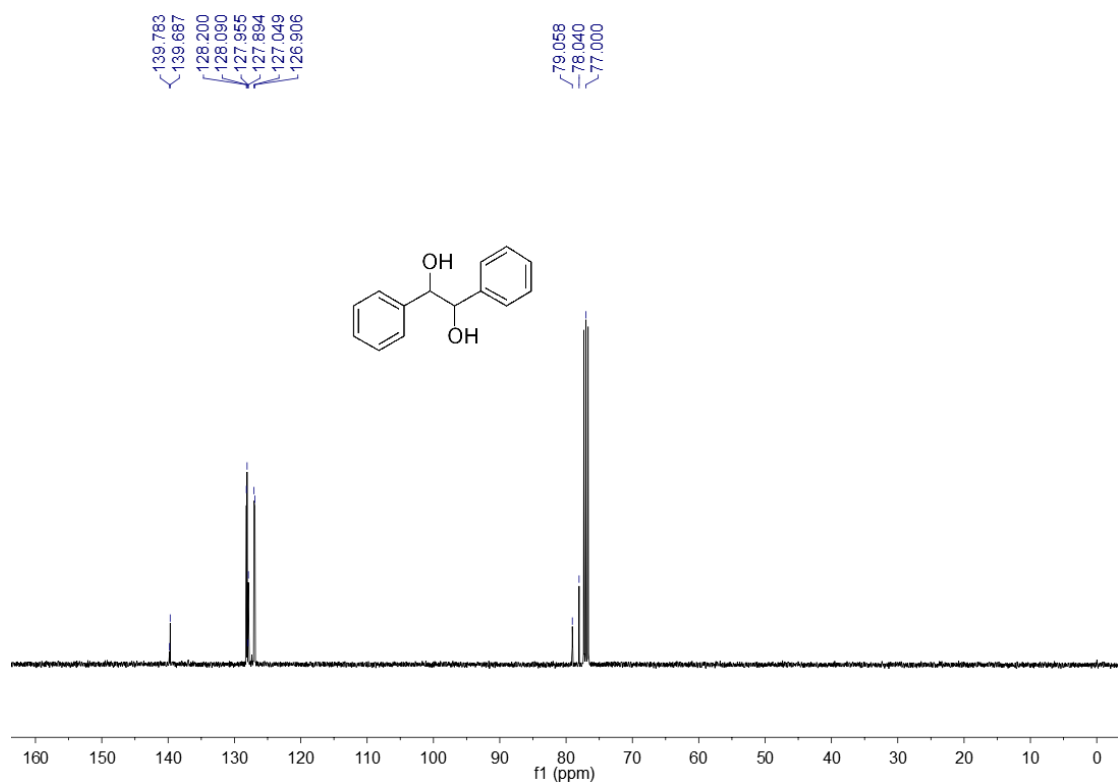
- (5) M.J. Frisch, G.W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A.V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, Jr. J. A. Montgomery, J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar, J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, D. J. Fox, Gaussian 16, Revision A.03, Gaussian, Inc., Wallingford CT, 2016.
- (6) C. Adamo, D. Jacquemin, *Chem. Soc. Rev.*, 2013, **42**, 845-856.
- (7) C. Lee, W. Yang, R. G. Parr, *Phys. Rev. B*, 1988, **37**, 785.
- (8) V. A. Rassolov, M. A. Ratner, J. A. Pople, P. C. Redfern, L. A. J. *Comp. Chem.*, 2001, **22**, 976-984.
- (9) Y. Zhao, D. G. Truhlar, *Chem. Acc.*, 2008, **120**, 215-241.
- (10) K. Fukui, *Acc. Chem. Res.*, 1981, **14**, 363-368.
- (11) C. Gonzalez, B. H. Schlegel, *J. Chem. Phys.*, 1989, **90**, 2154-2161.
- (12) K. Fukui, *J. Phys. Chem.*, 1970, **74**, 4161-4163.
- (13) S. Miertuš, E. Scrocco, J. Tomasi, *Chem. Phys.*, 1981, **55**, 117-129.
- (14) S. Miertuš, J. Tomasi, *Chem. Phys.*, 1982, **65**, 239-245.
- (15) Y. Ma, A. A. Hussein, *ChemistrySelect*, 2022, **7**, e202202354.
- (16) Y. Ma, KST48: A Powerful Tool for MECP locating. <https://github.com/RimoAccelerator/KST48>.
- (17) T. Lu, F. Chen, *J. Comput. Chem.*, 2012, **33**, 580-592.
- (18) W. Humphrey, A. Dalke, K. Schulten, *J. Molec. Graphics*, 1996, **14**, 33-38.
- (19) A. Caron, É. Morin, S. K. Collins, *ACS Catal.*, 2019, **9**, 9458-9464.
- (20) X. Huang, N. Li, Z. Geng, F. Pan, X. Wang, *Chin. J. Chem.*, 2012, **30**, 2657-2663.
- (21) S. Guo, Y. Jiang, J. Yang, G. Lu, M. Liu, W. Liu, H. Cai, *J. Org. Chem.*, 2025, **90**, 2139-2147.
- (22) J. Wang, N. B. Üner, S. E. Dubowsky, M. P. Confer, R. Bhargava, Y. Sun, Y. Zhou, R. M. Sankaran, J. S. Moore, *J. Am. Chem. Soc.*, 2023, **145**, 10470-10474.
- (23) C. Liu, R. Li, W. Zhou, Y. Liang, Y. Shi, R.-L. Li, Y. Ling, Y. Yu, J. Li, B. Zhang, *ACS Catal.*, 2021, **11**, 8958-8967.
- (24) T. Liu, W. Meng, X. Feng, H. Du, *Angew. Chem. Int. Ed.*, 2024, **63**, e202313957.
- (25) J.-Y. Li, M.-Y. Qi, Y.-J. Xu, *Chin. J. Catal.* 2022, **43**, 1084-1091.
- (26) H. Zhang, D. Feng, H. Sheng, X. Ma, J. Wan, Q. Tang, *RSC Adv.*, 2014, **4**, 6417-6423.
- (27) B. M. Kim, H. Kim, W. Kim, K. Y. Im, J. K. Park, *J. Org. Chem.*, 2004, **69**, 5104-5107.
- (28) M. Takeda, A. Mitsui, K. Nagao, H. Ohmiya, *J. Am. Chem. Soc.* 2019, **141**, 3664-3669.

14. Copies of NMR spectra for the products

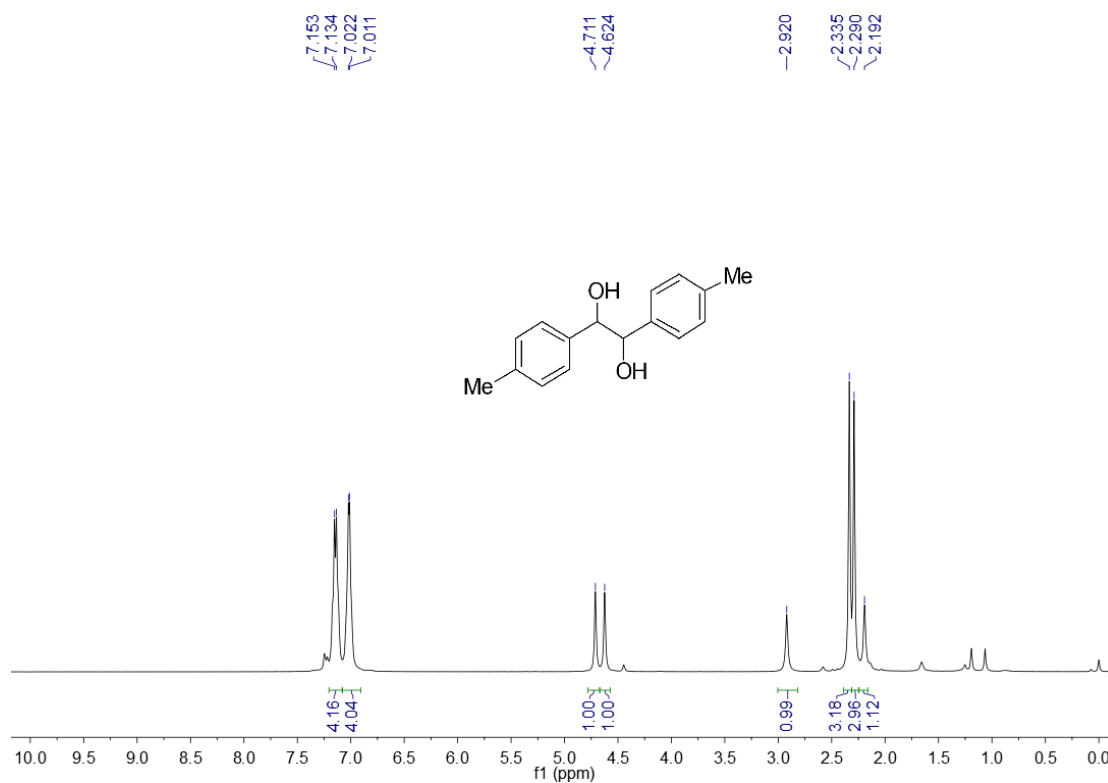
^1H NMR spectrum of compound **2a** (400 MHz) in CDCl_3



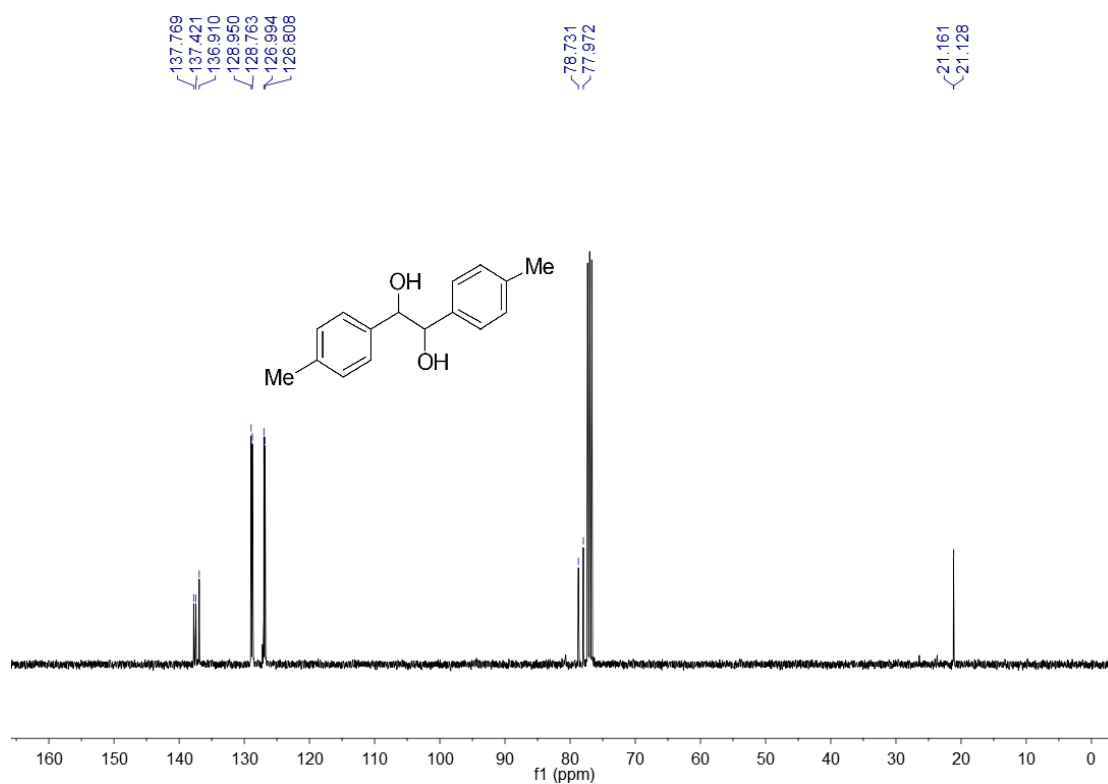
^{13}C NMR spectrum of compound **2a** (100 MHz) in CDCl_3



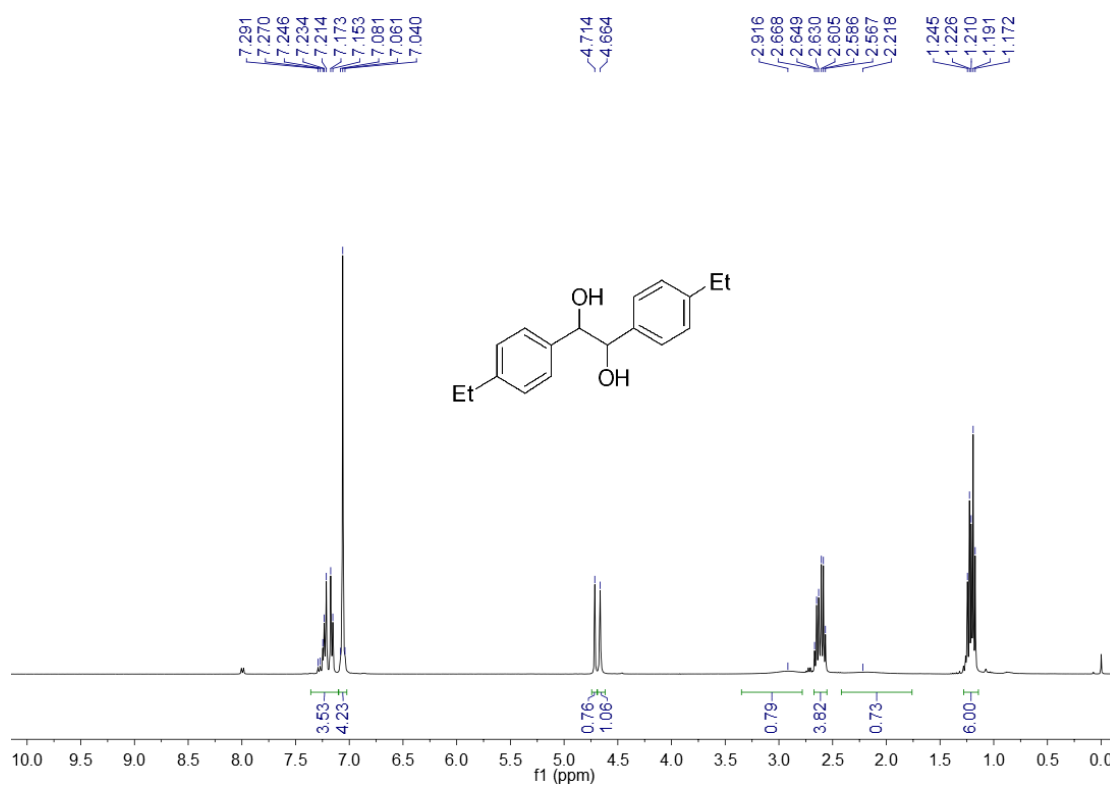
^1H NMR spectrum of compound **2b** (400 MHz) in CDCl_3



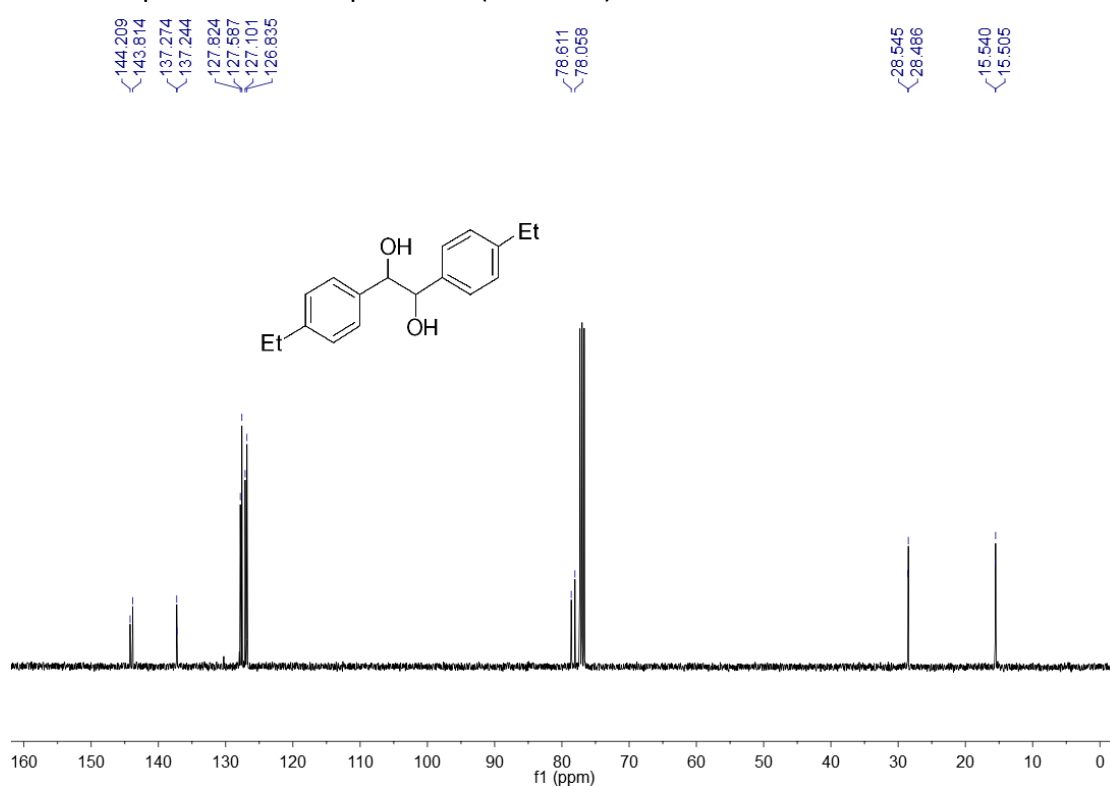
^{13}C NMR spectrum of compound **2b** (100 MHz) in CDCl_3



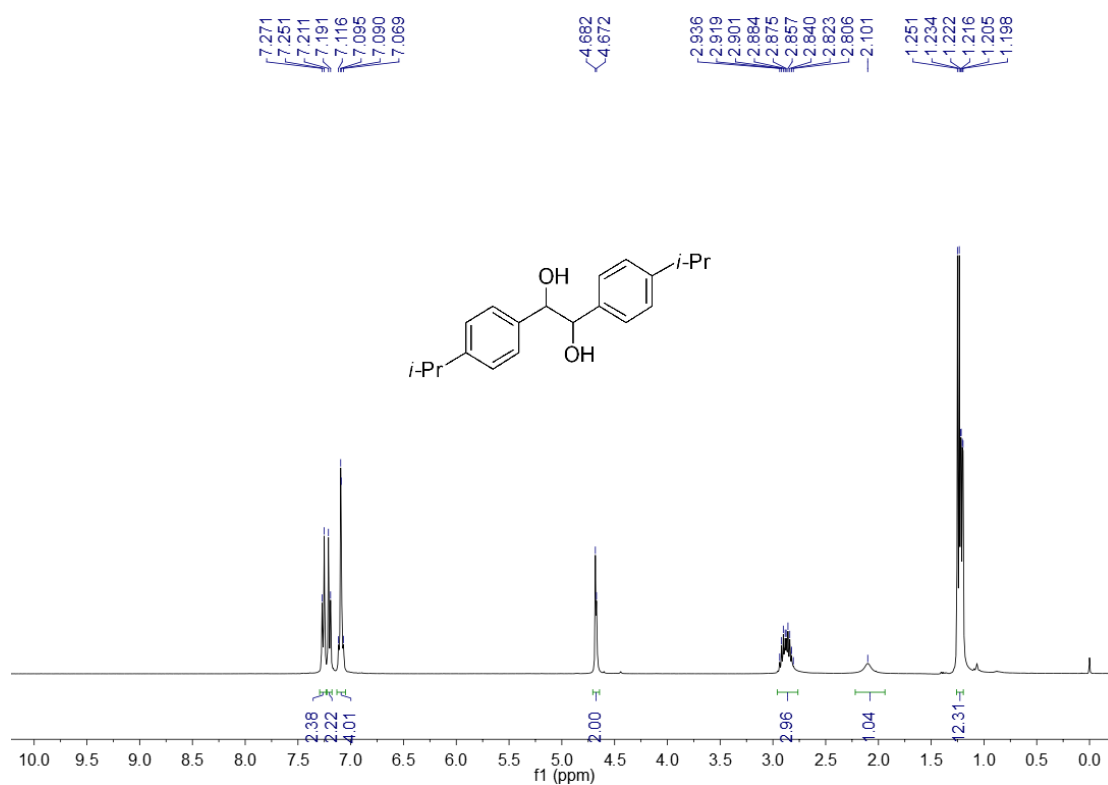
^1H NMR spectrum of compound **2c** (400 MHz) in CDCl_3



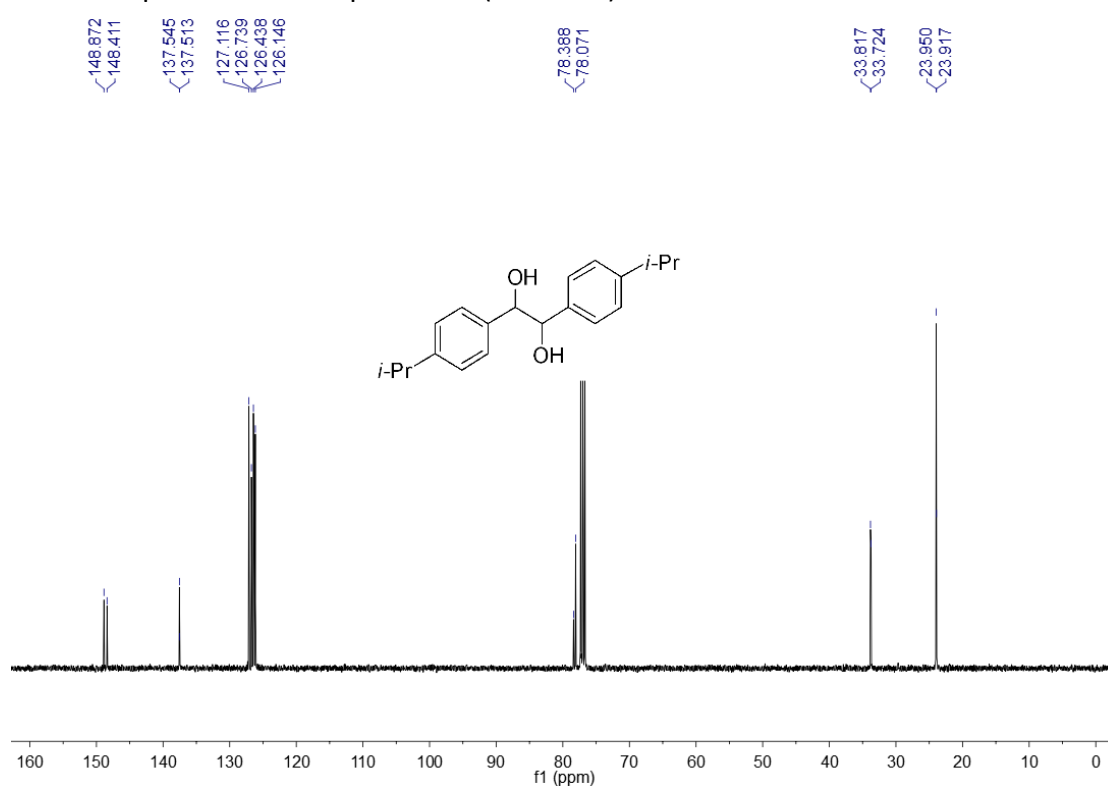
^{13}C NMR spectrum of compound **2c** (100 MHz) in CDCl_3



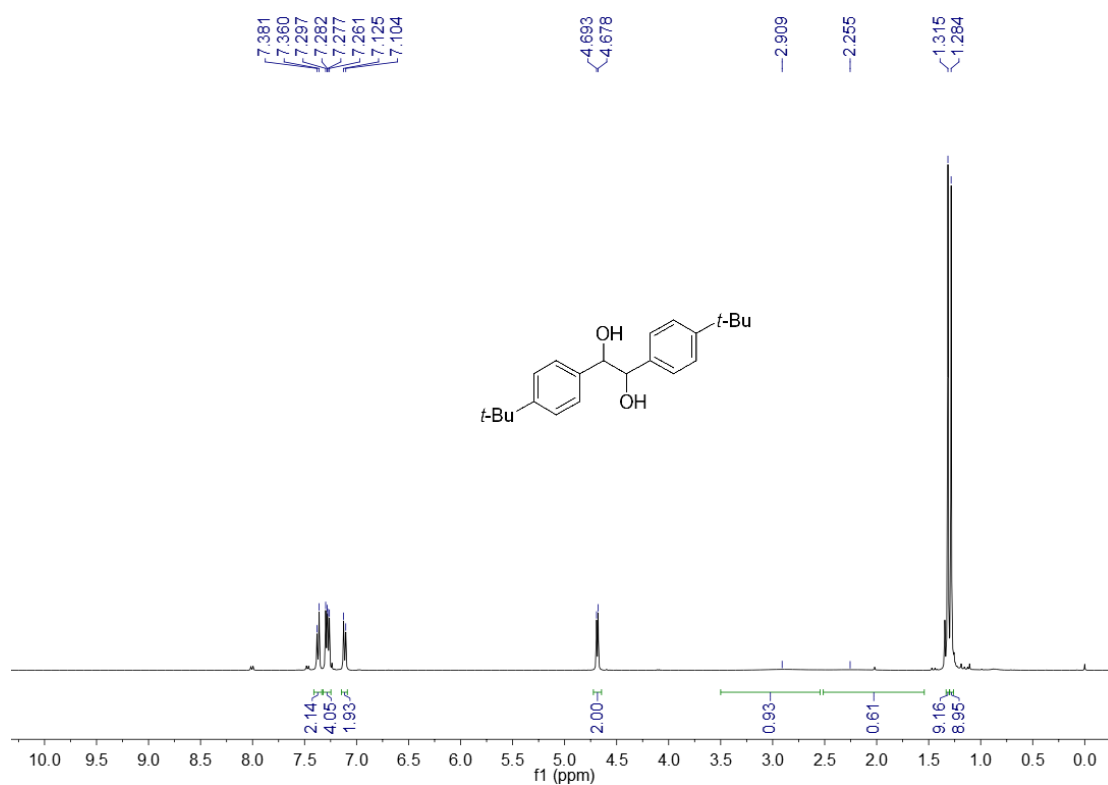
^1H NMR spectrum of compound **2d** (400 MHz) in CDCl_3



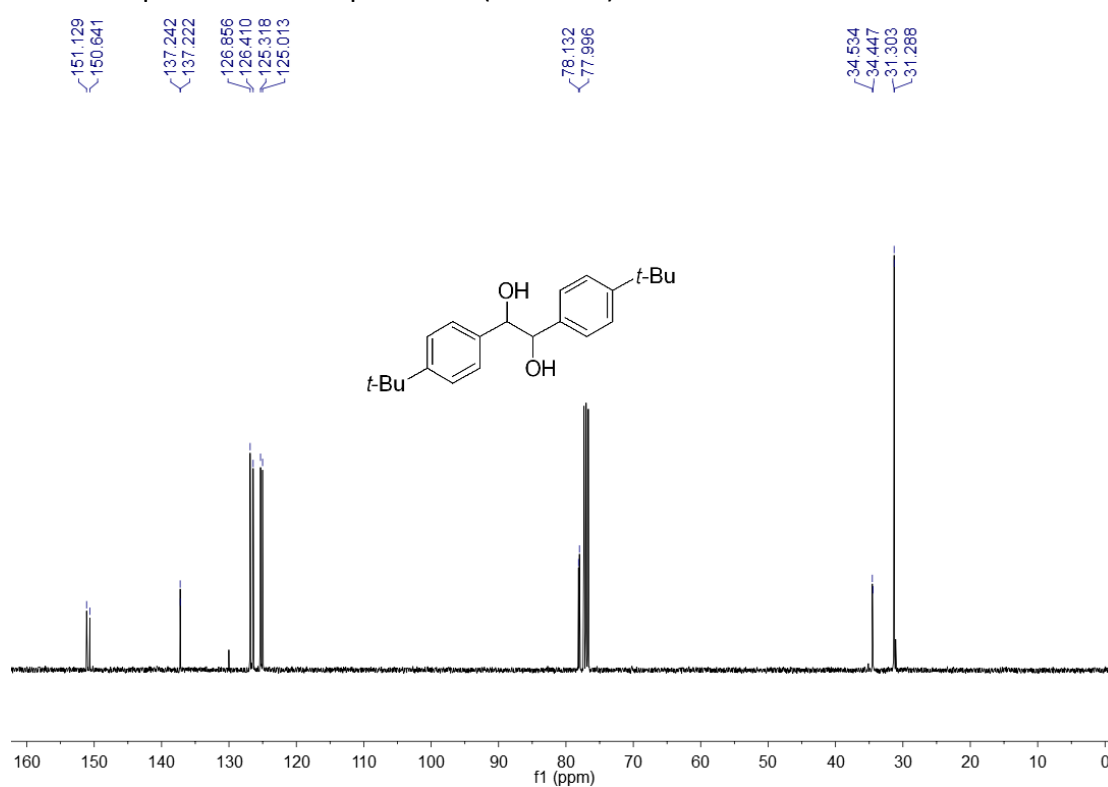
^{13}C NMR spectrum of compound **2d** (100 MHz) in CDCl_3



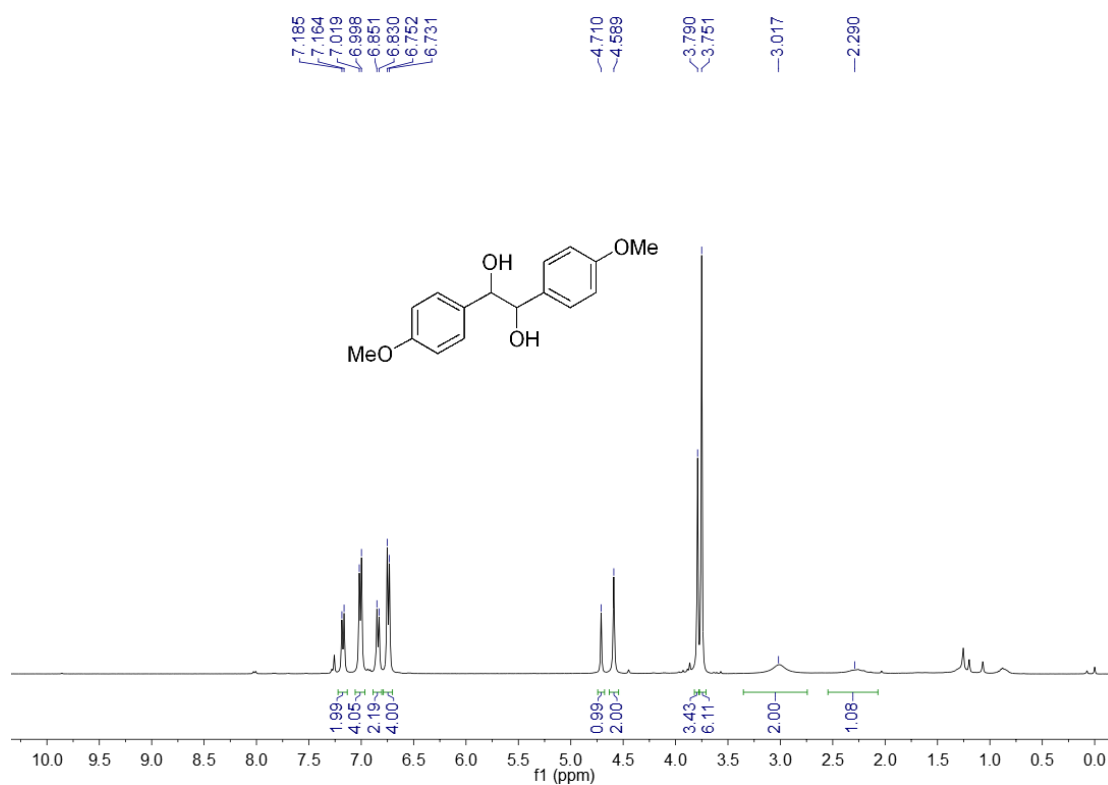
^1H NMR spectrum of compound **2e** (400 MHz) in CDCl_3



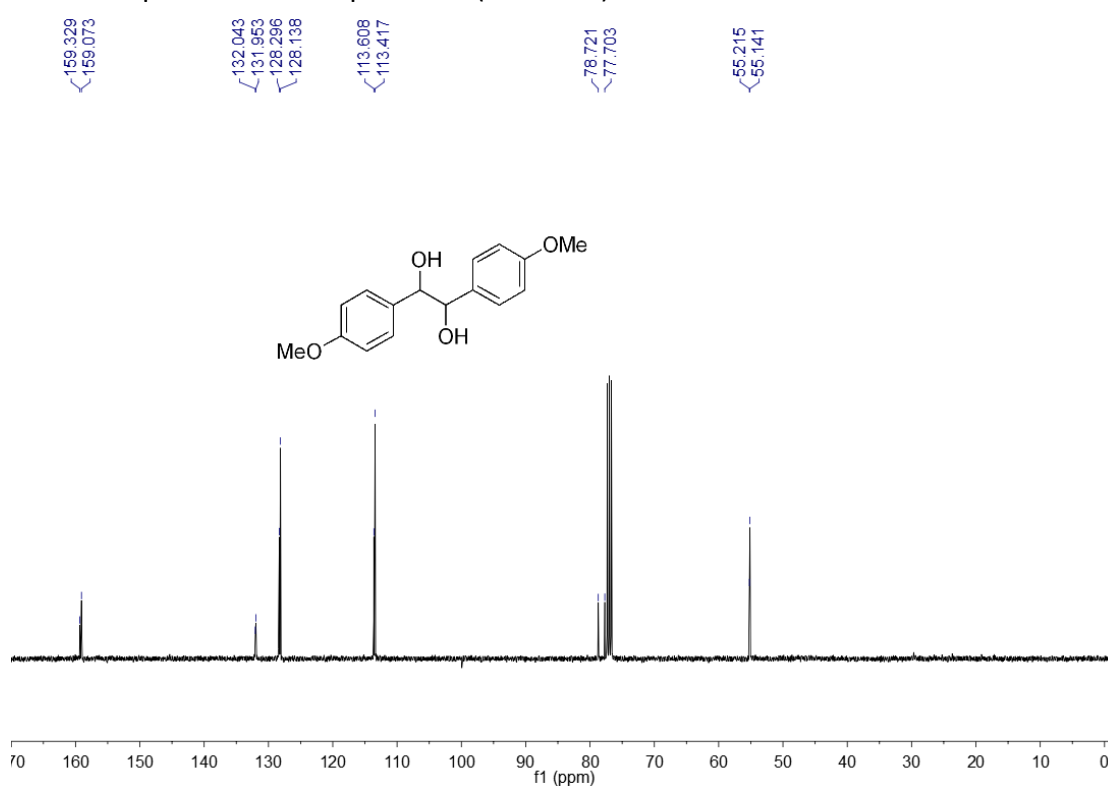
^{13}C NMR spectrum of compound **2e** (100 MHz) in CDCl_3



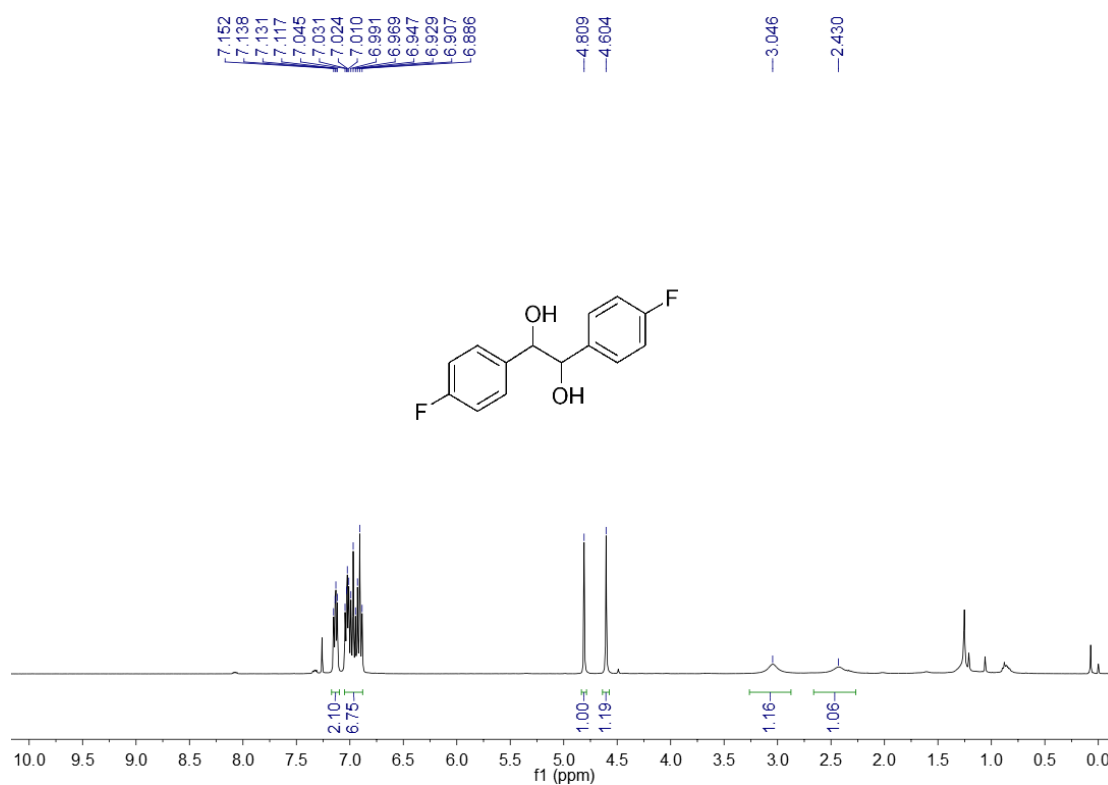
^1H NMR spectrum of compound **2f** (400 MHz) in CDCl_3



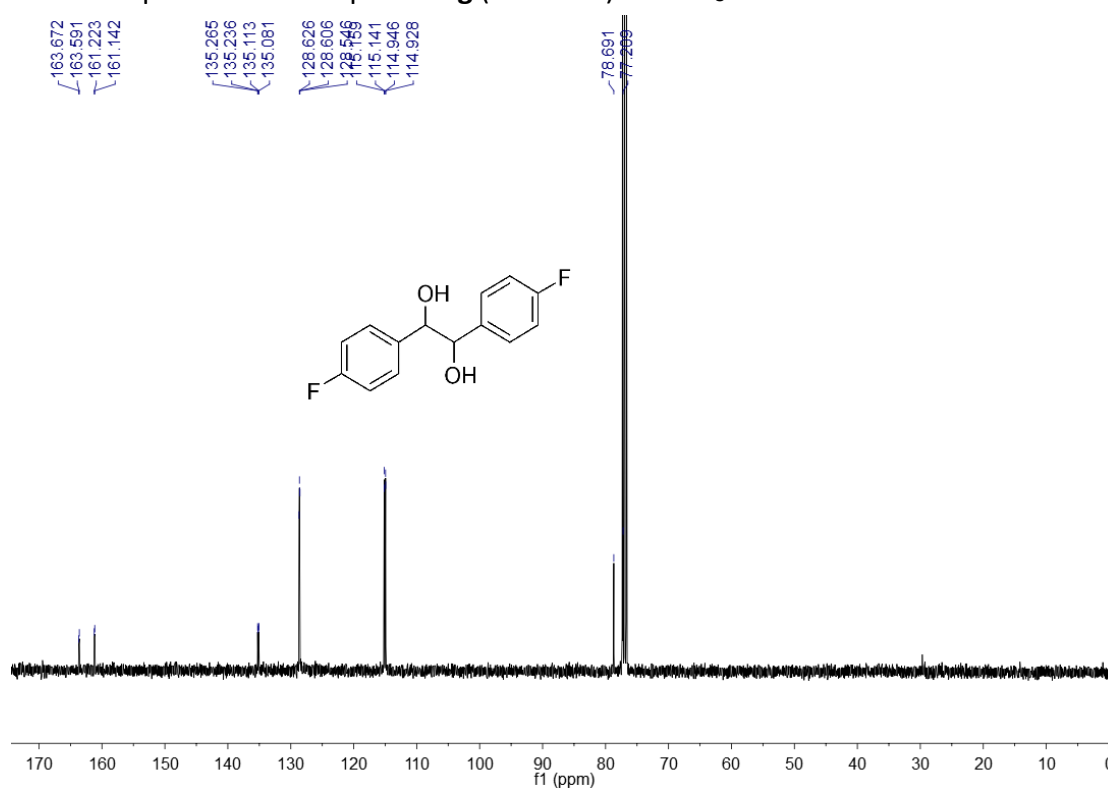
^{13}C NMR spectrum of compound **2f** (100 MHz) in CDCl_3



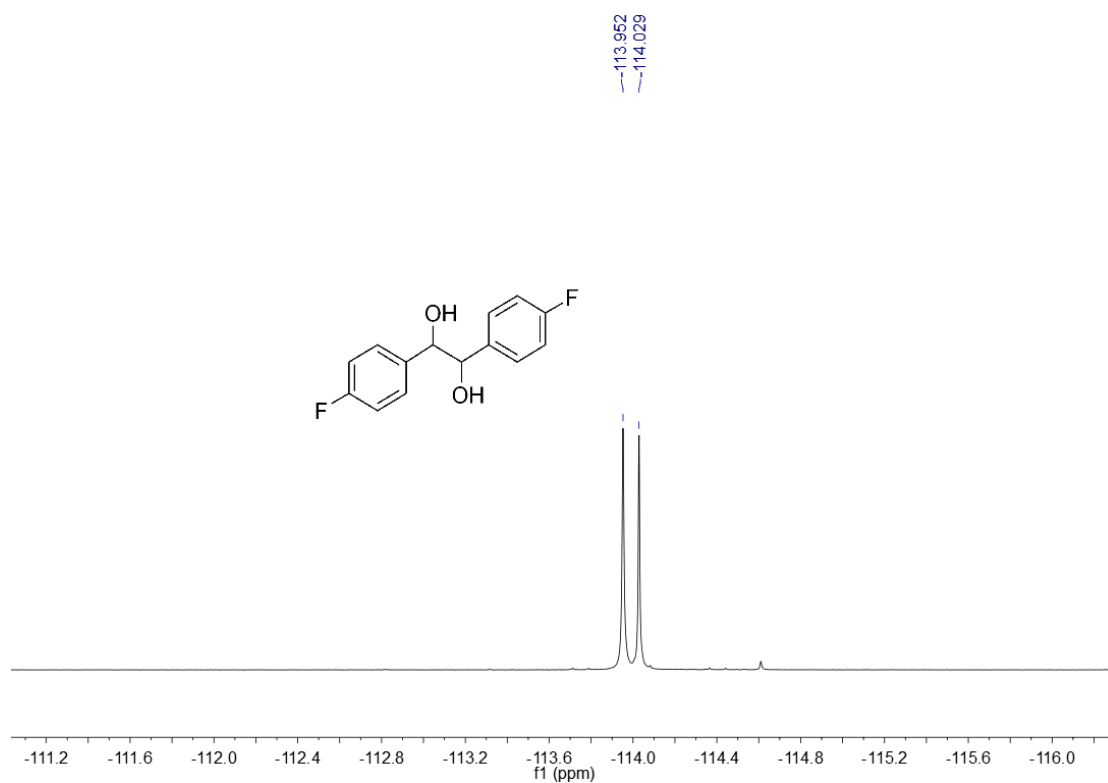
^1H NMR spectrum of compound **2g** (400 MHz) in CDCl_3



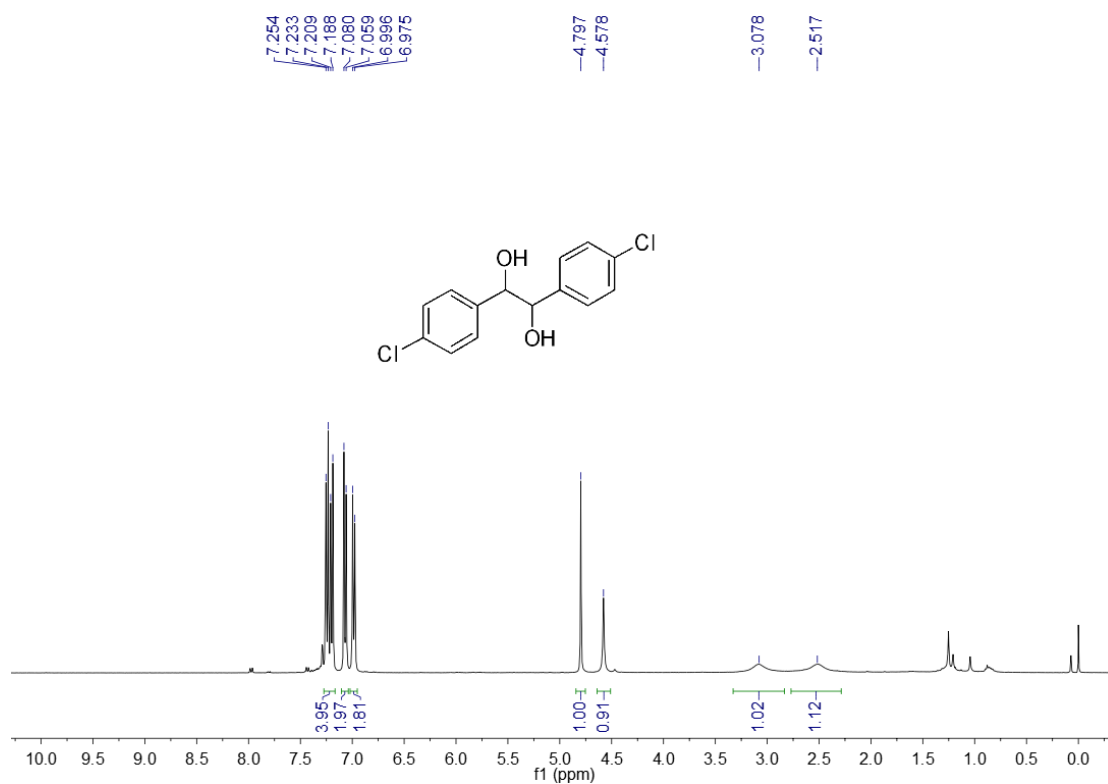
^{13}C NMR spectrum of compound **2g** (100 MHz) in CDCl_3



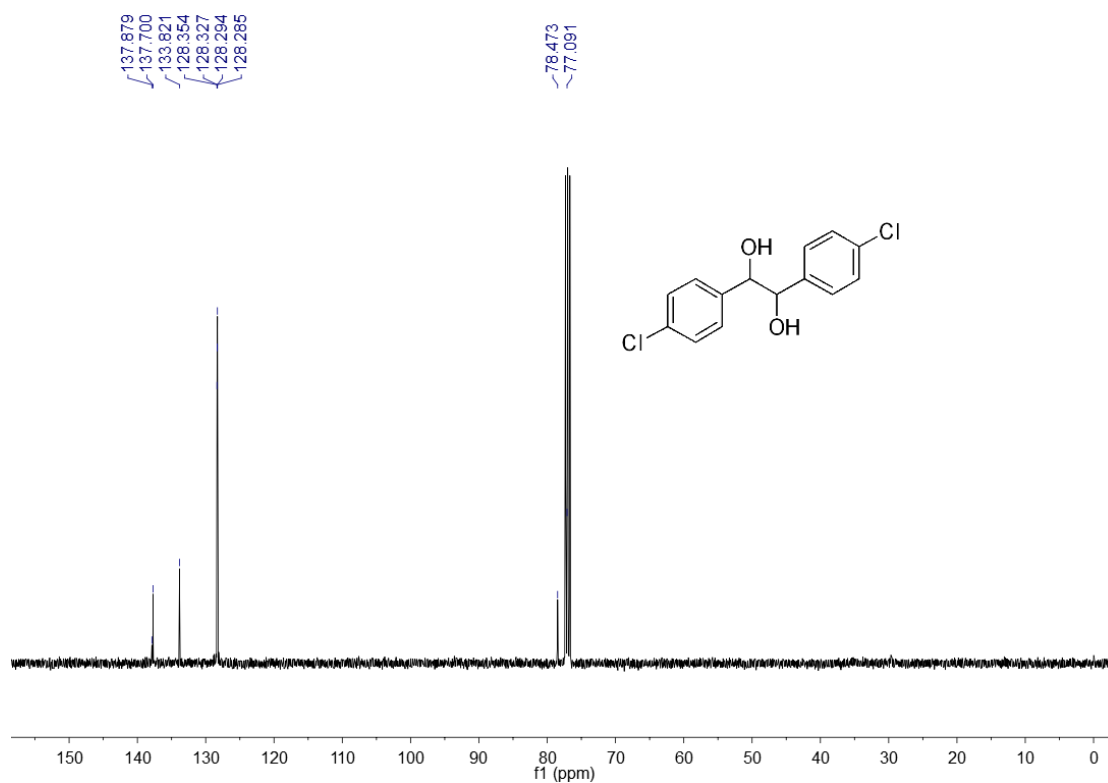
^{19}F NMR spectrum of compound **2g** (376 MHz) in CDCl_3



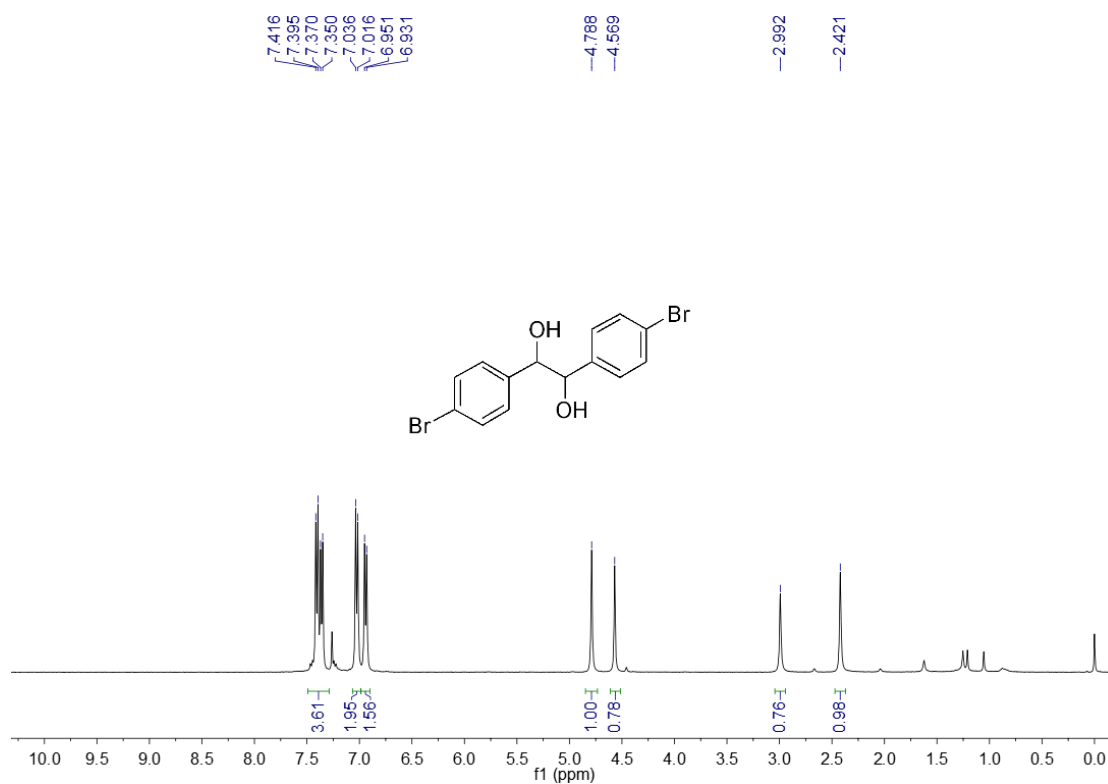
^1H NMR spectrum of compound **2h** (400 MHz) in CDCl_3



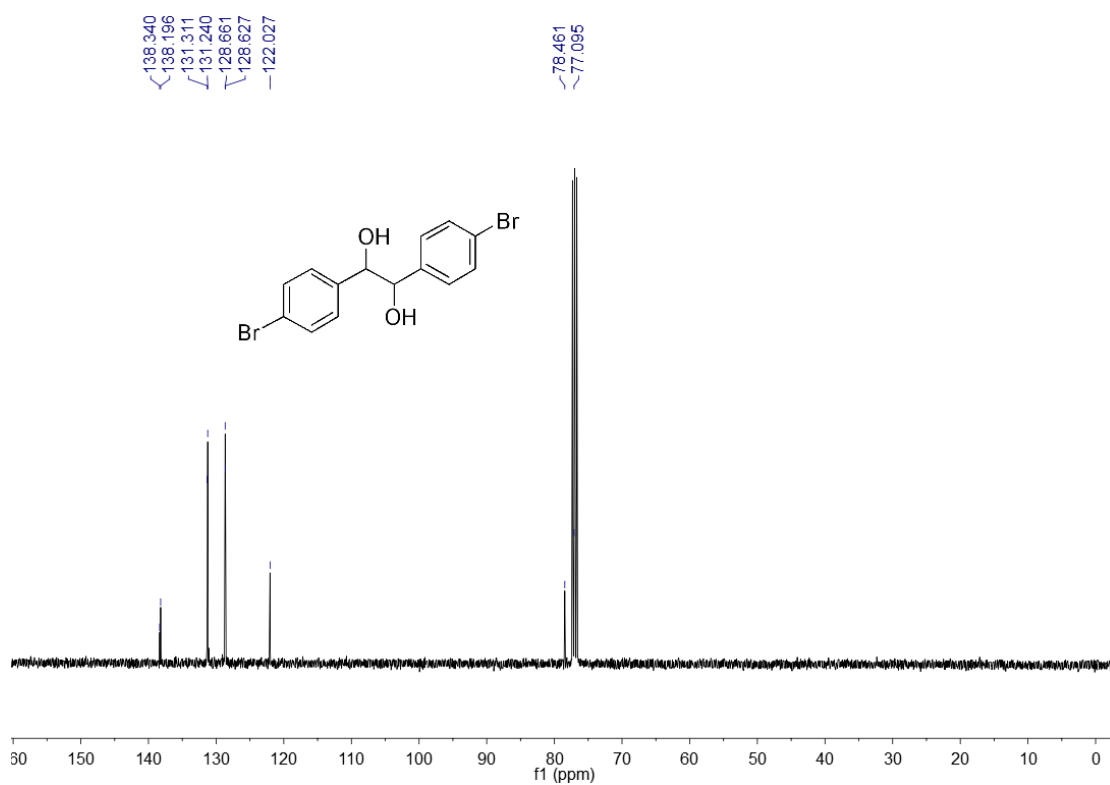
^{13}C NMR spectrum of compound **2h** (100 MHz) in CDCl_3



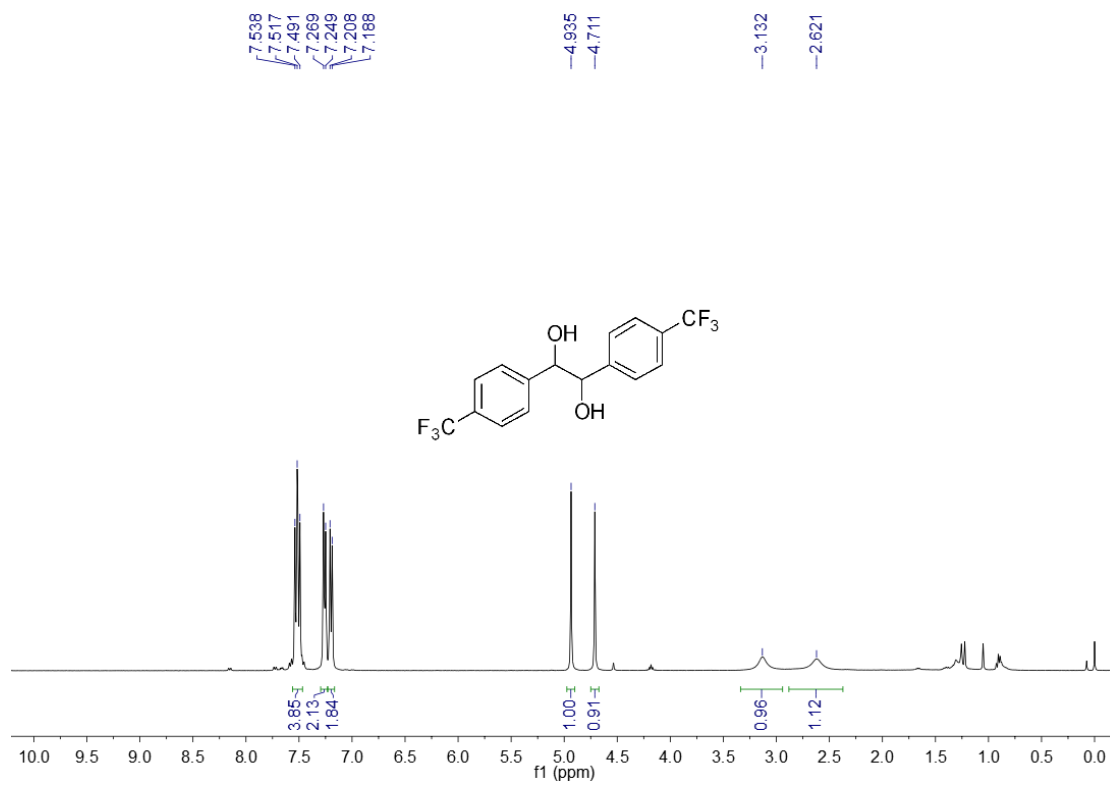
^1H NMR spectrum of compound **2i** (400 MHz) in CDCl_3



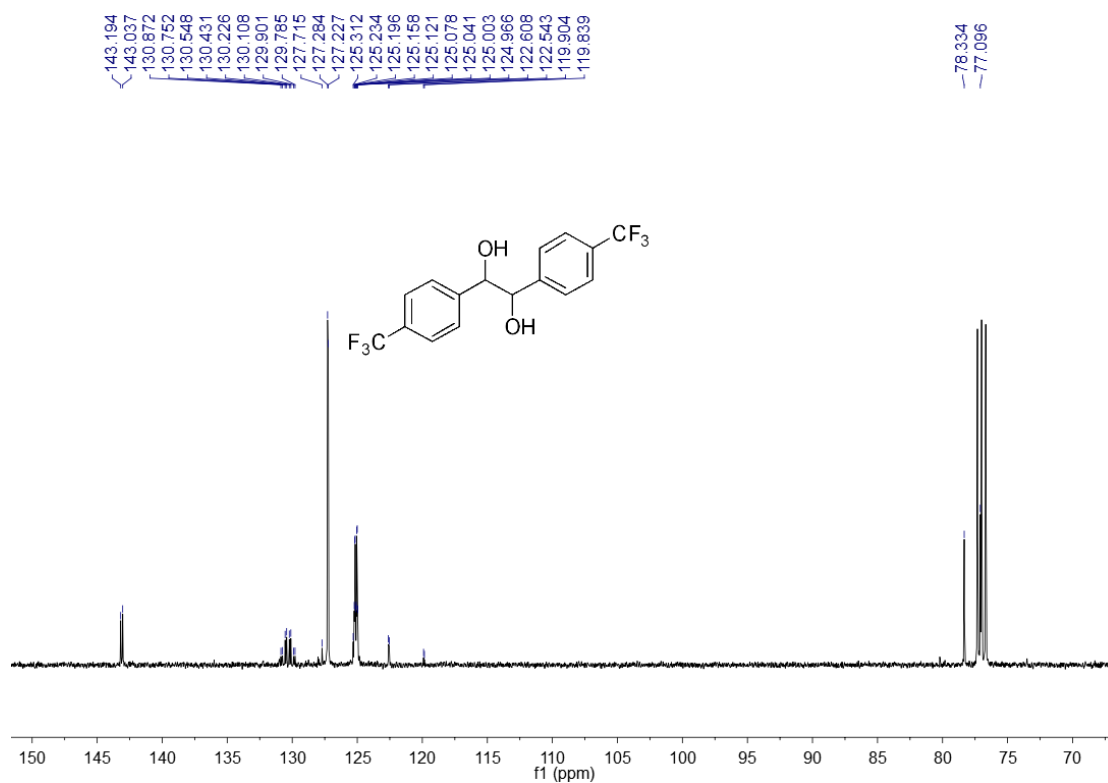
^{13}C NMR spectrum of compound **2i** (100 MHz) in CDCl_3



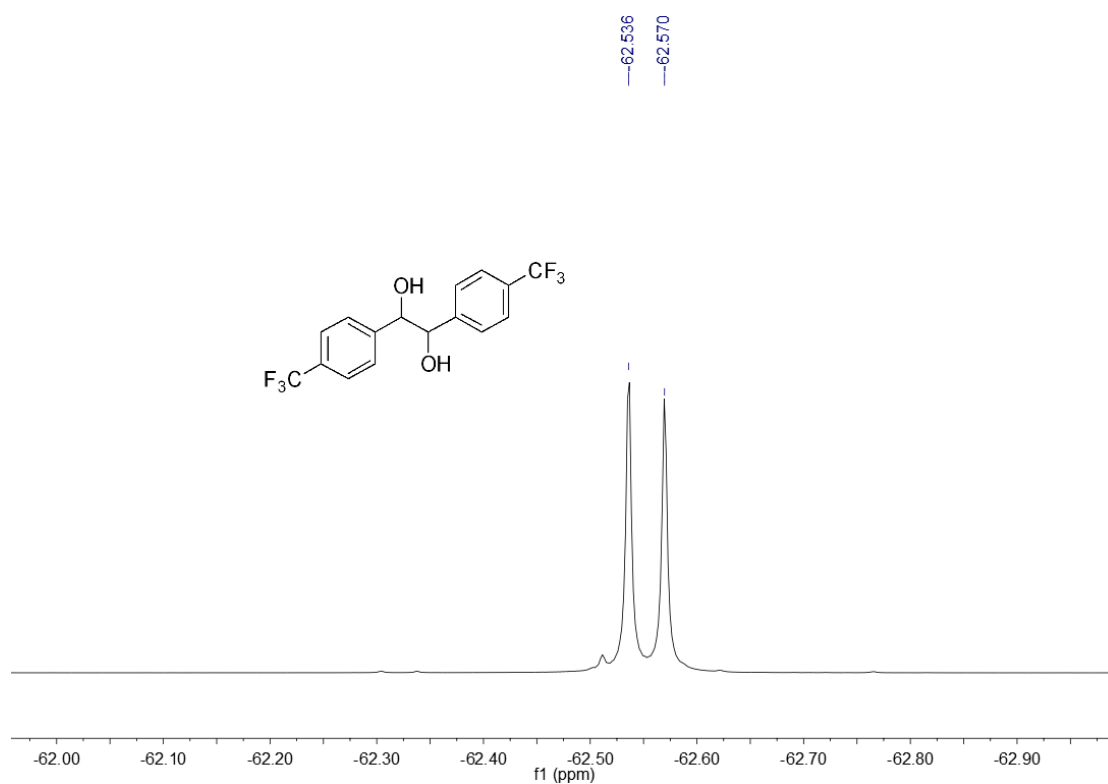
^1H NMR spectrum of compound **2j** (400 MHz) in CDCl_3



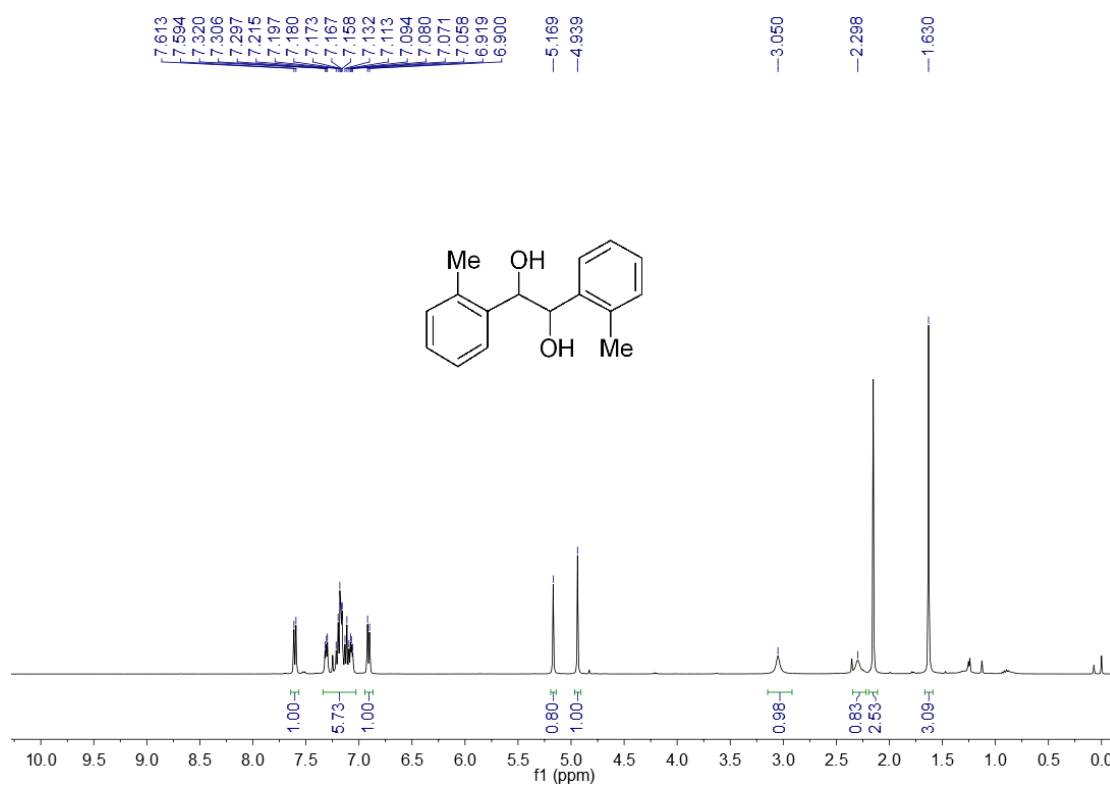
^{13}C NMR spectrum of compound **2j** (100 MHz) in CDCl_3



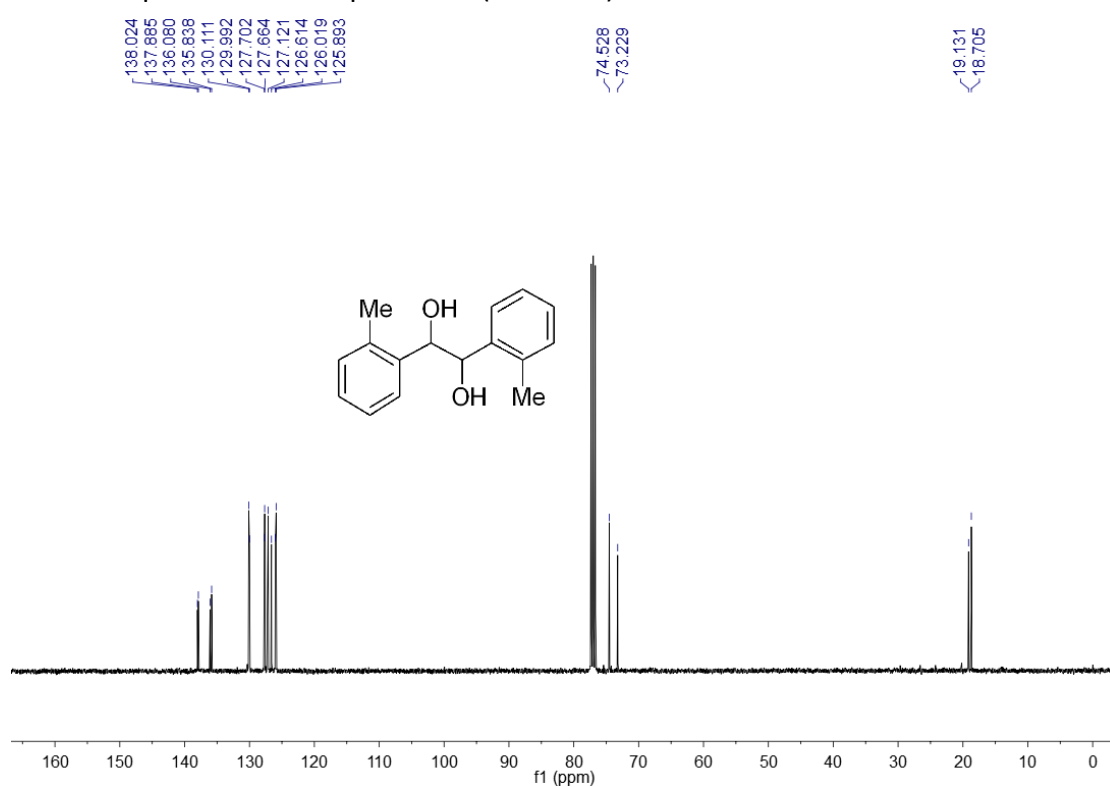
^{19}F NMR spectrum of compound **2j** (100 MHz) in CDCl_3



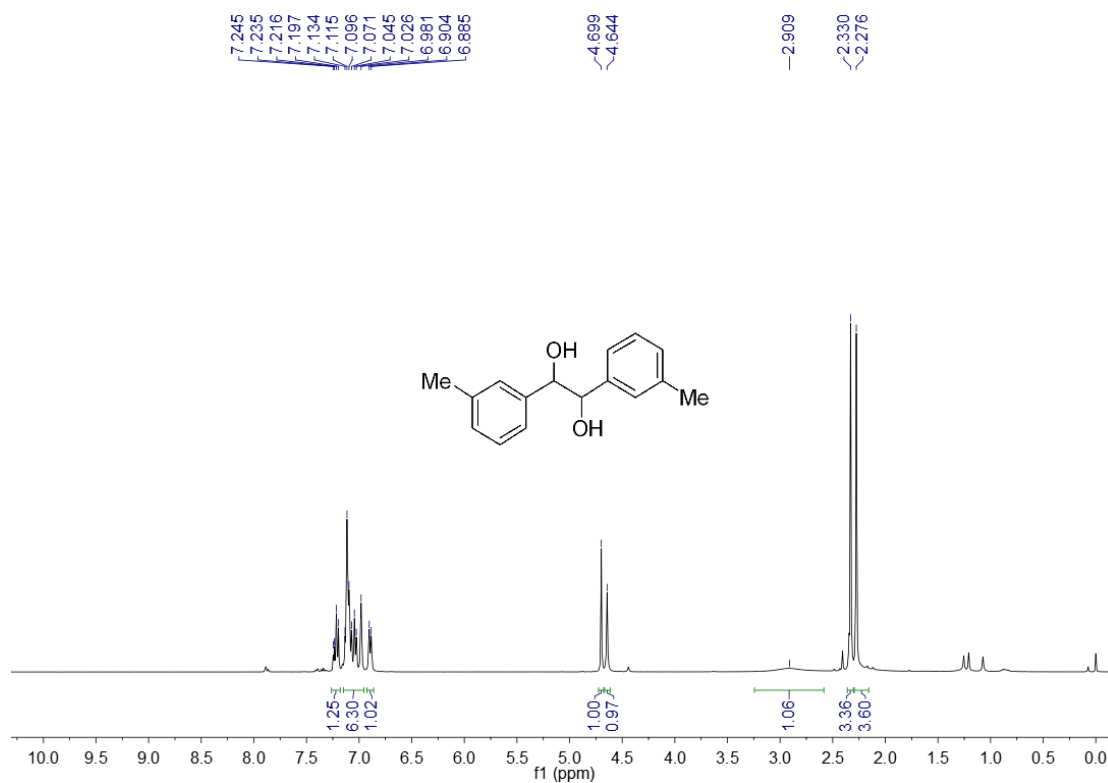
^1H NMR spectrum of compound **2k** (400 MHz) in CDCl_3



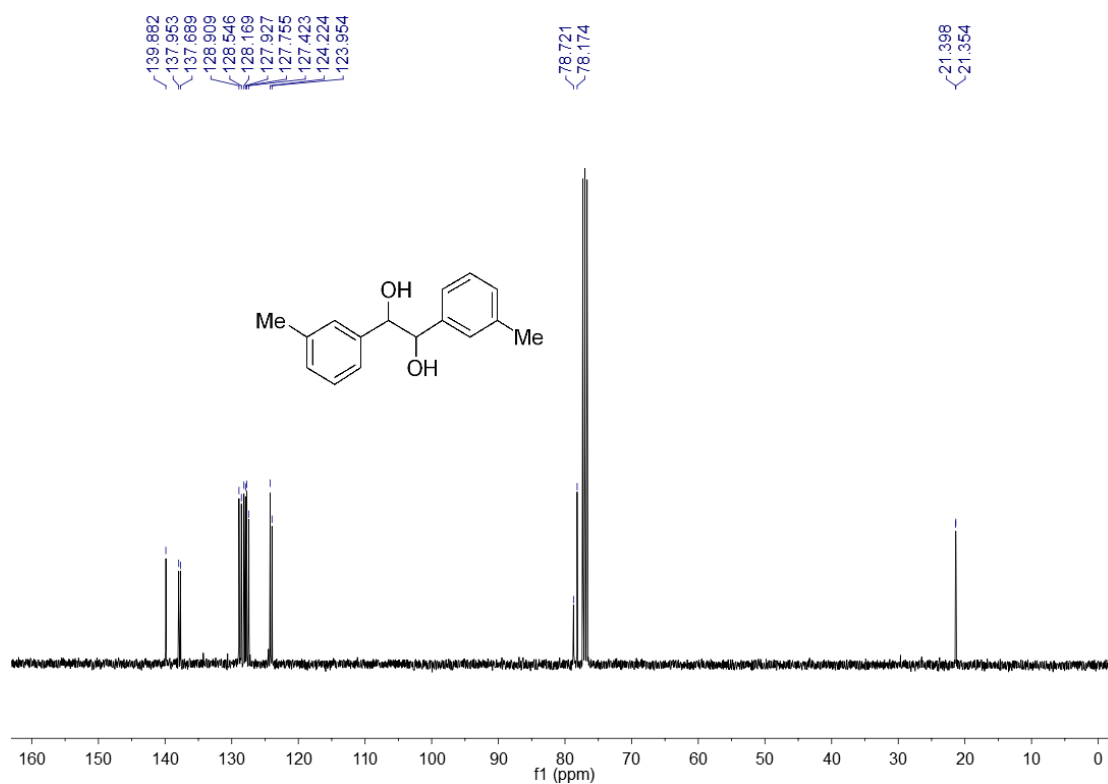
^{13}C NMR spectrum of compound **2k** (100 MHz) in CDCl_3



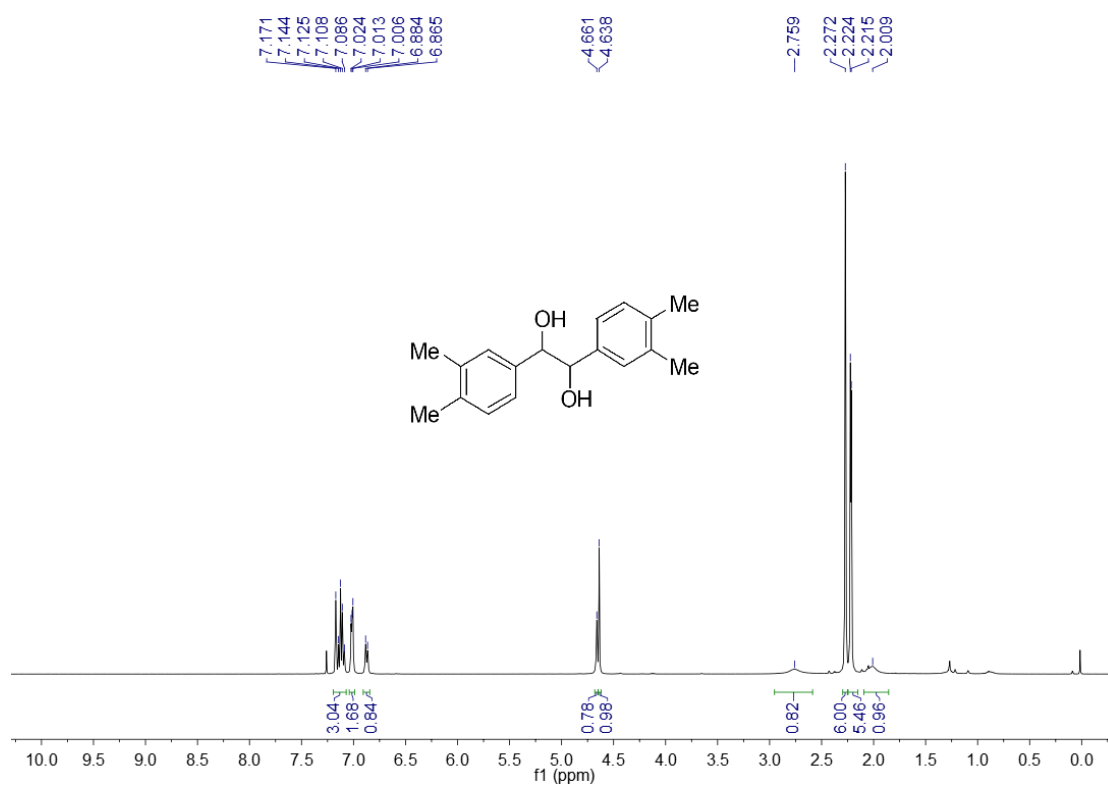
^1H NMR spectrum of compound **2l** (400 MHz) in CDCl_3



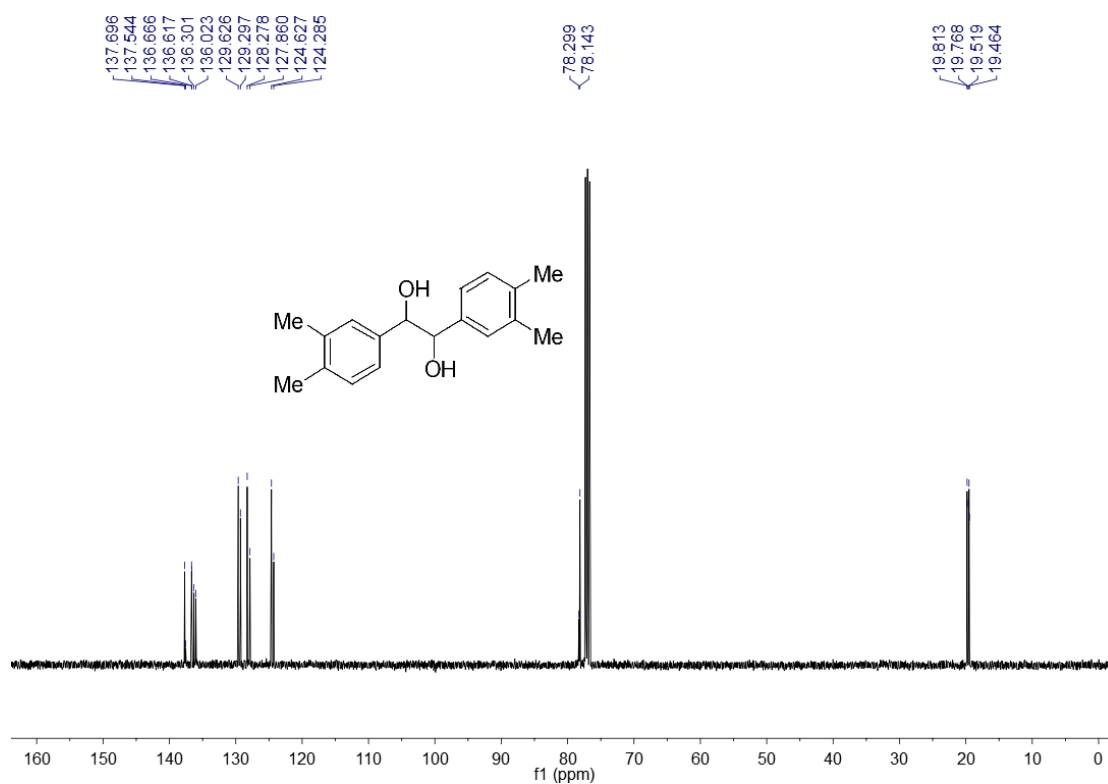
^{13}C NMR spectrum of compound **2l** (100 MHz) in CDCl_3



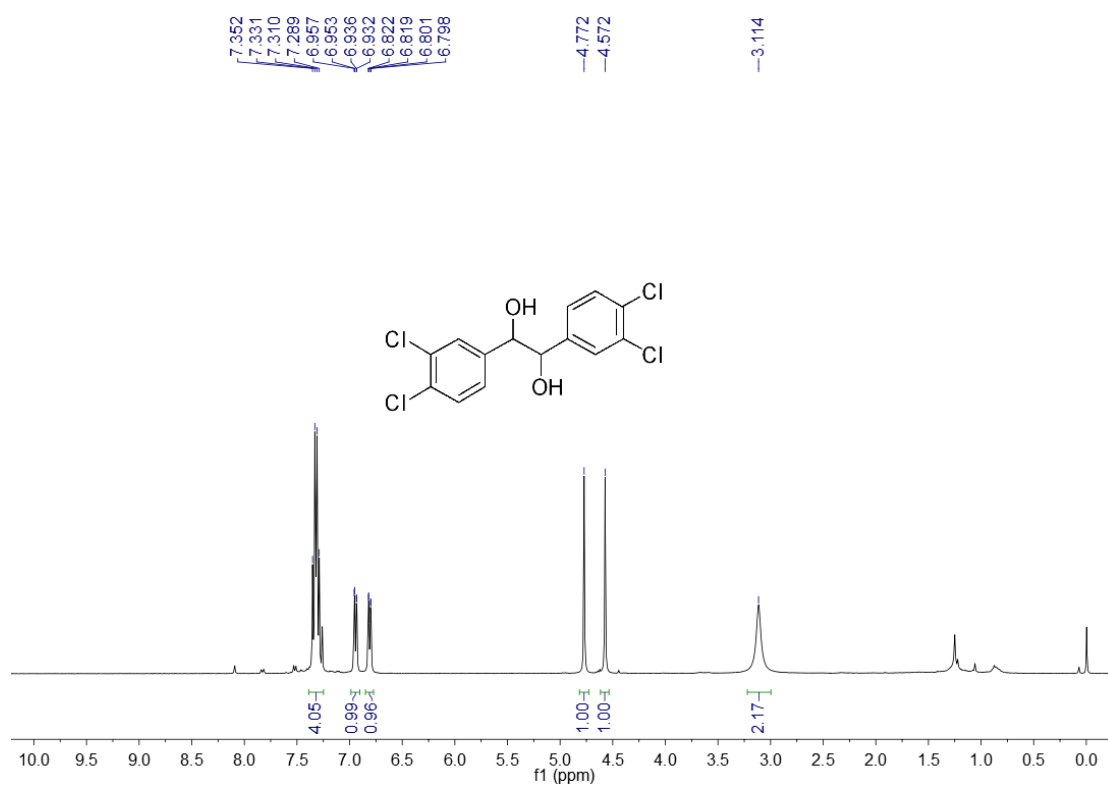
^1H NMR spectrum of compound **2m** (400 MHz) in CDCl_3



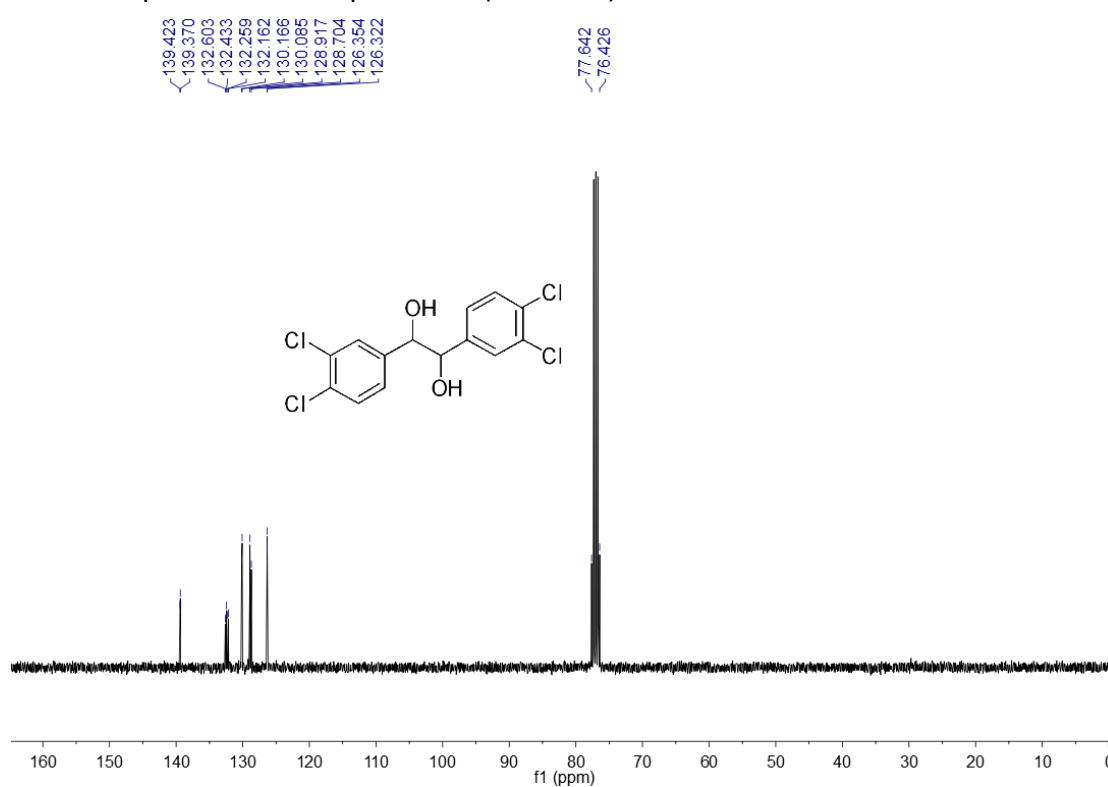
^{13}C NMR spectrum of compound **2m** (100 MHz) in CDCl_3



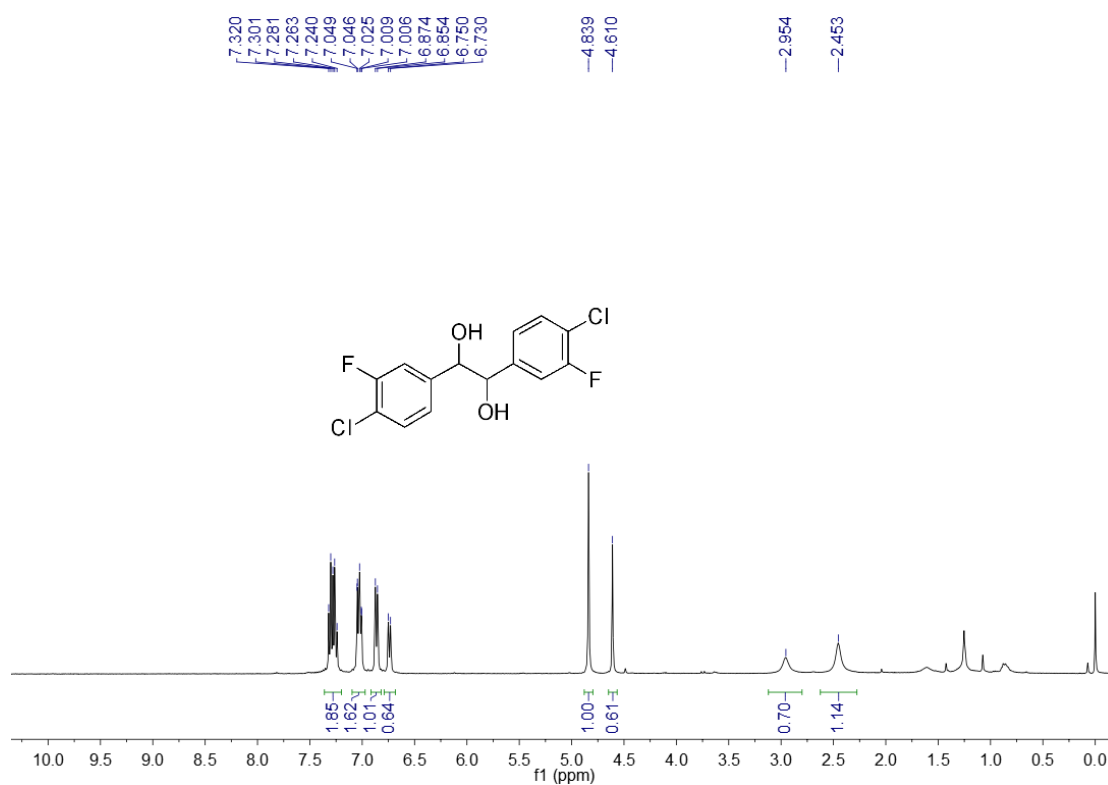
^1H NMR spectrum of compound **2n** (400 MHz) in CDCl_3



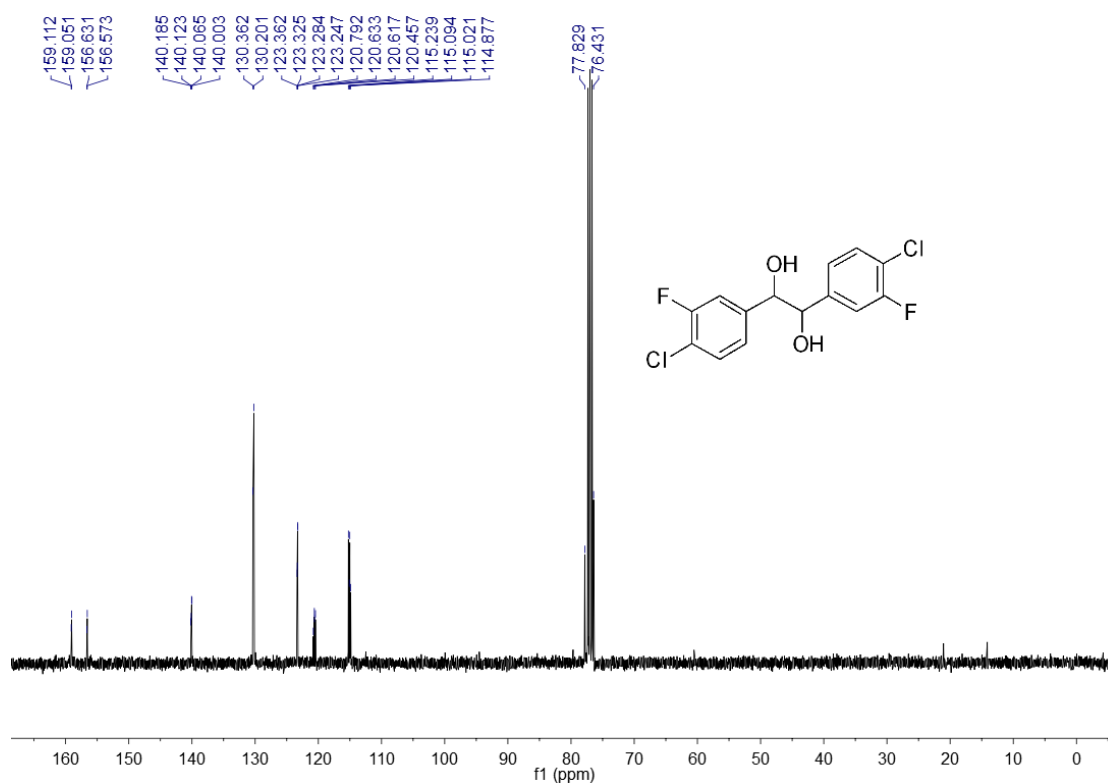
^{13}C NMR spectrum of compound **2n** (100 MHz) in CDCl_3



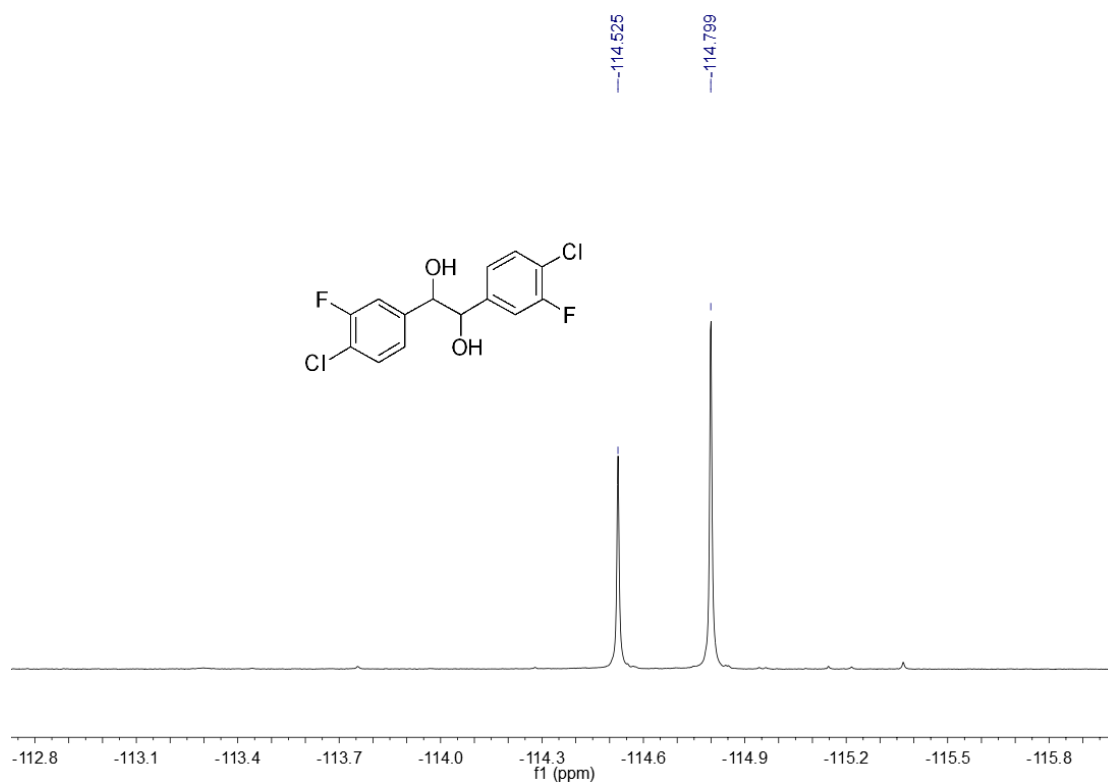
^1H NMR spectrum of compound **2o** (400 MHz) in CDCl_3



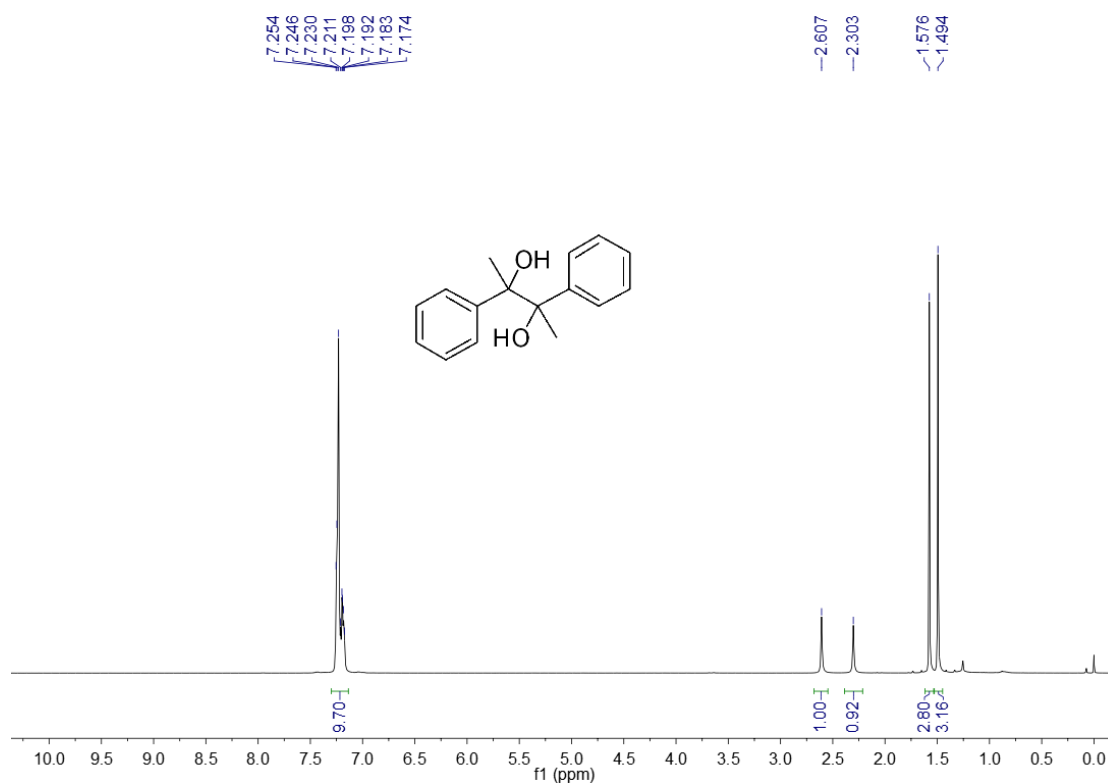
^{13}C NMR spectrum of compound **2o** (100 MHz) in CDCl_3



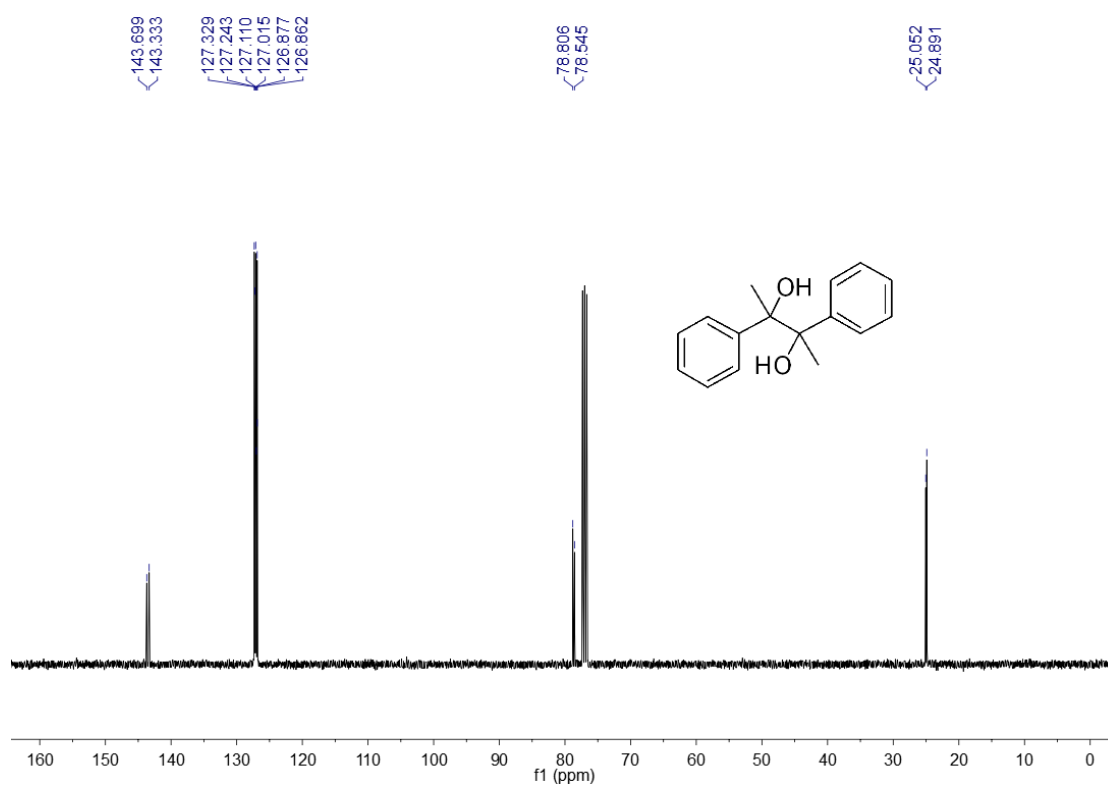
^{19}F NMR spectrum of compound **2o** (376 MHz) in CDCl_3



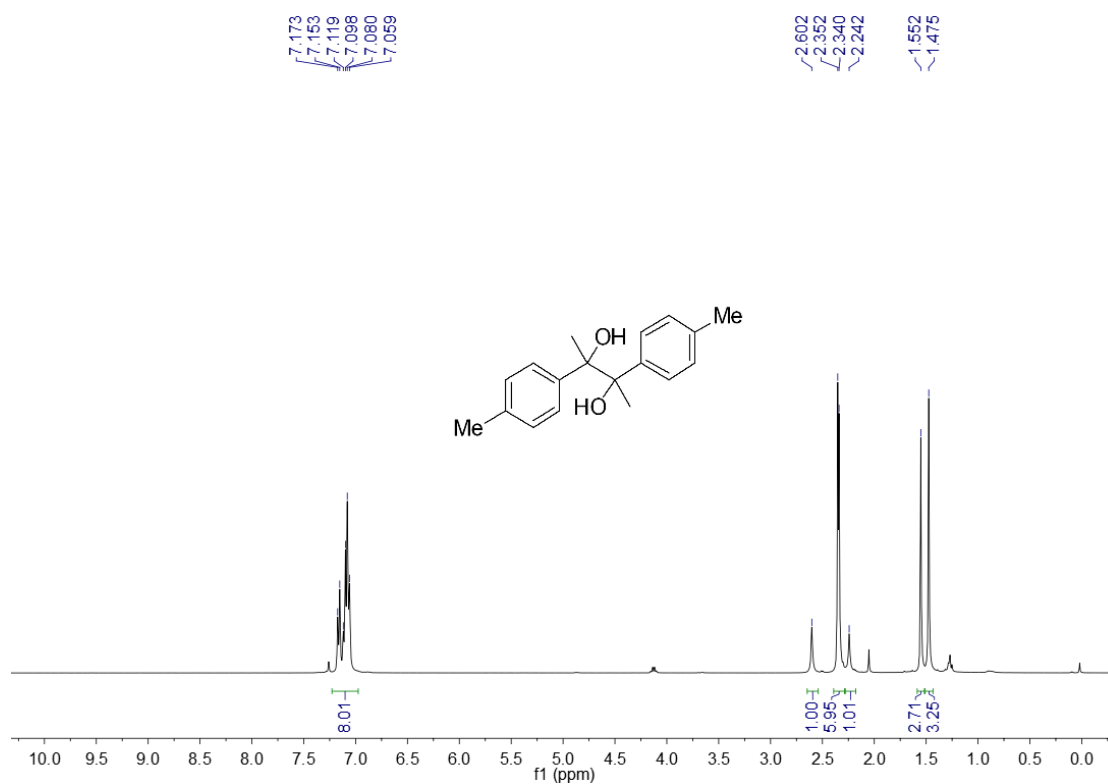
^1H NMR spectrum of compound **2p** (400 MHz) in CDCl_3



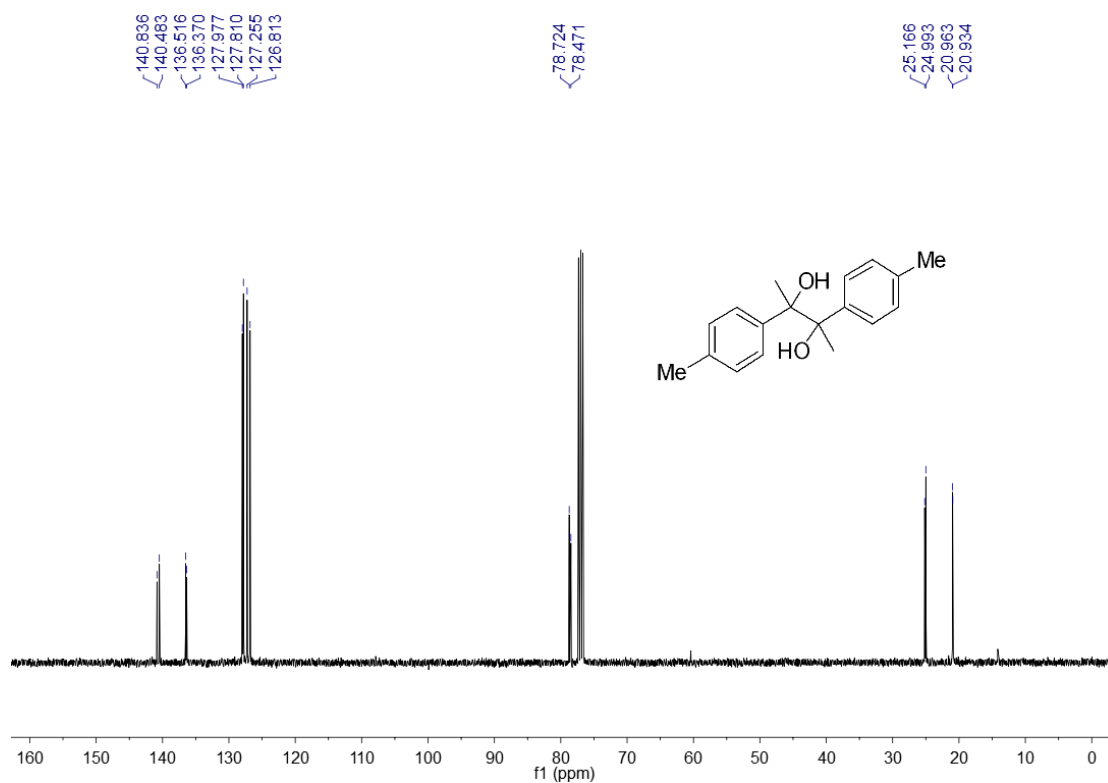
^{13}C NMR spectrum of compound **2p** (100 MHz) in CDCl_3



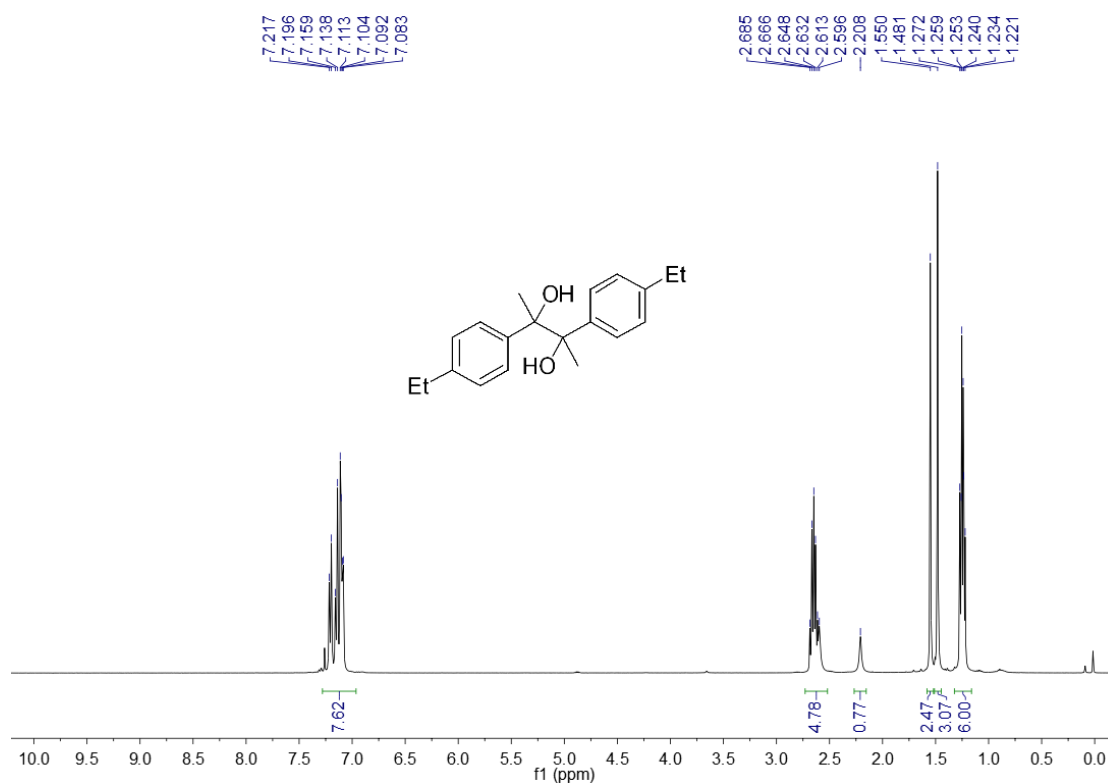
^1H NMR spectrum of compound **2q** (400 MHz) in CDCl_3



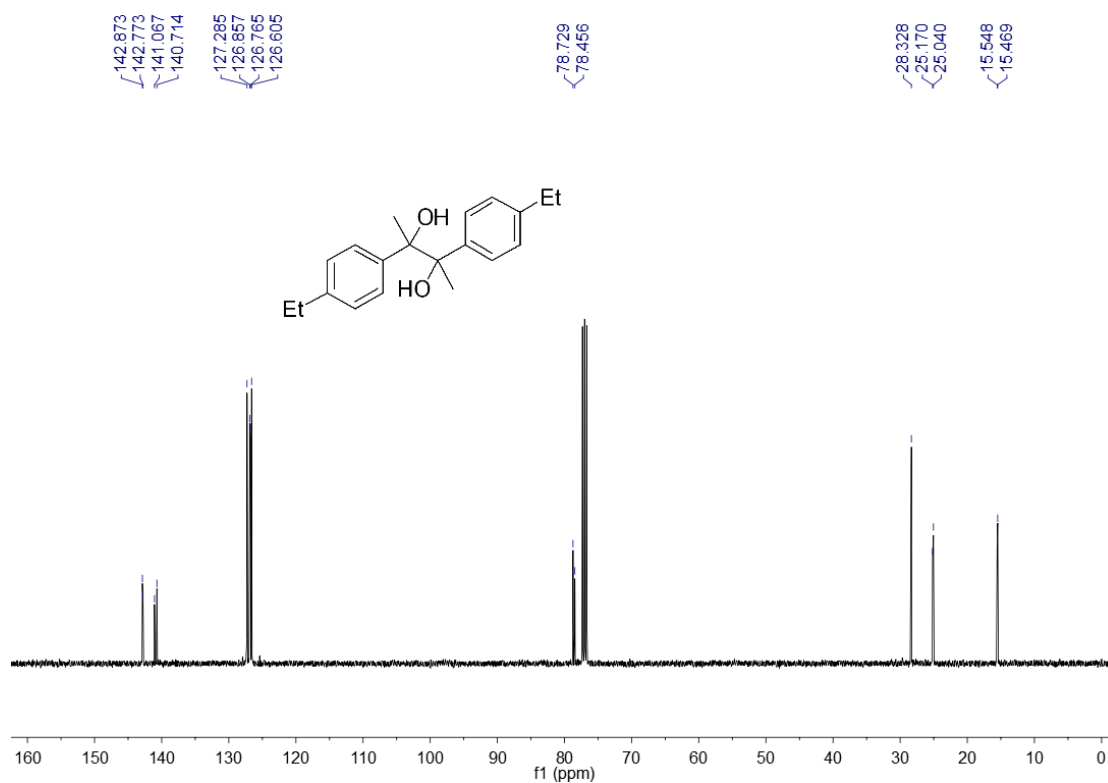
^{13}C NMR spectrum of compound **2q** (100 MHz) in CDCl_3



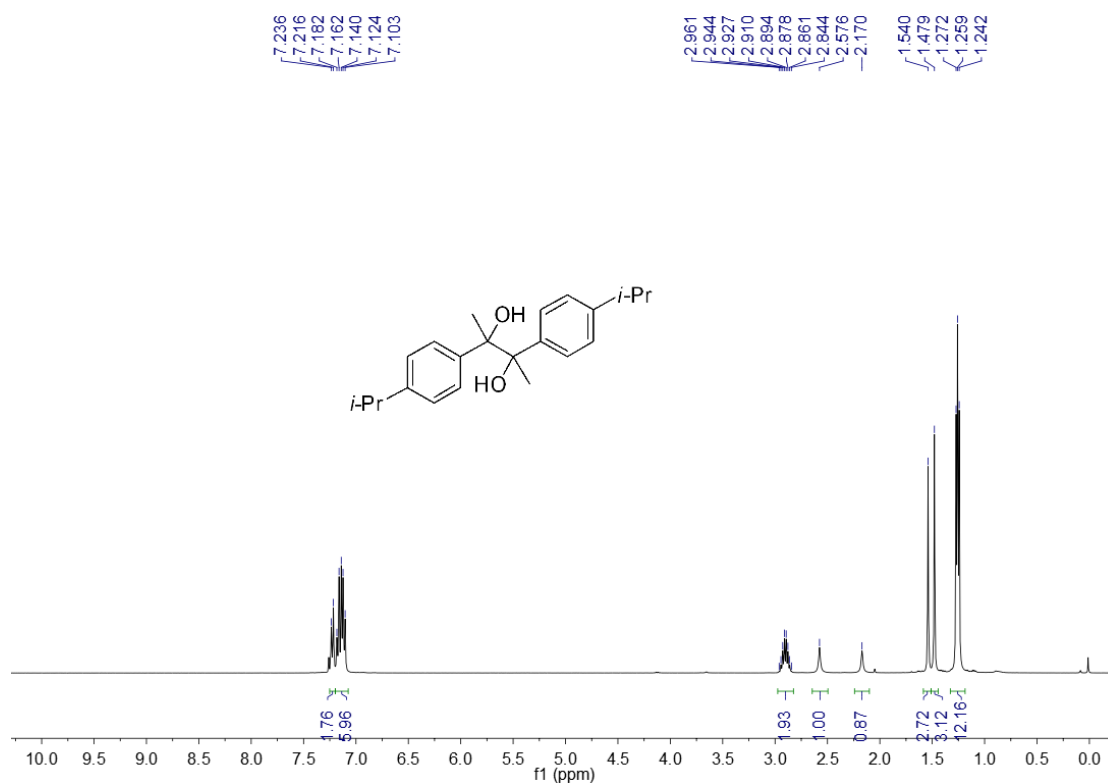
^1H NMR spectrum of compound **2r** (400 MHz) in CDCl_3



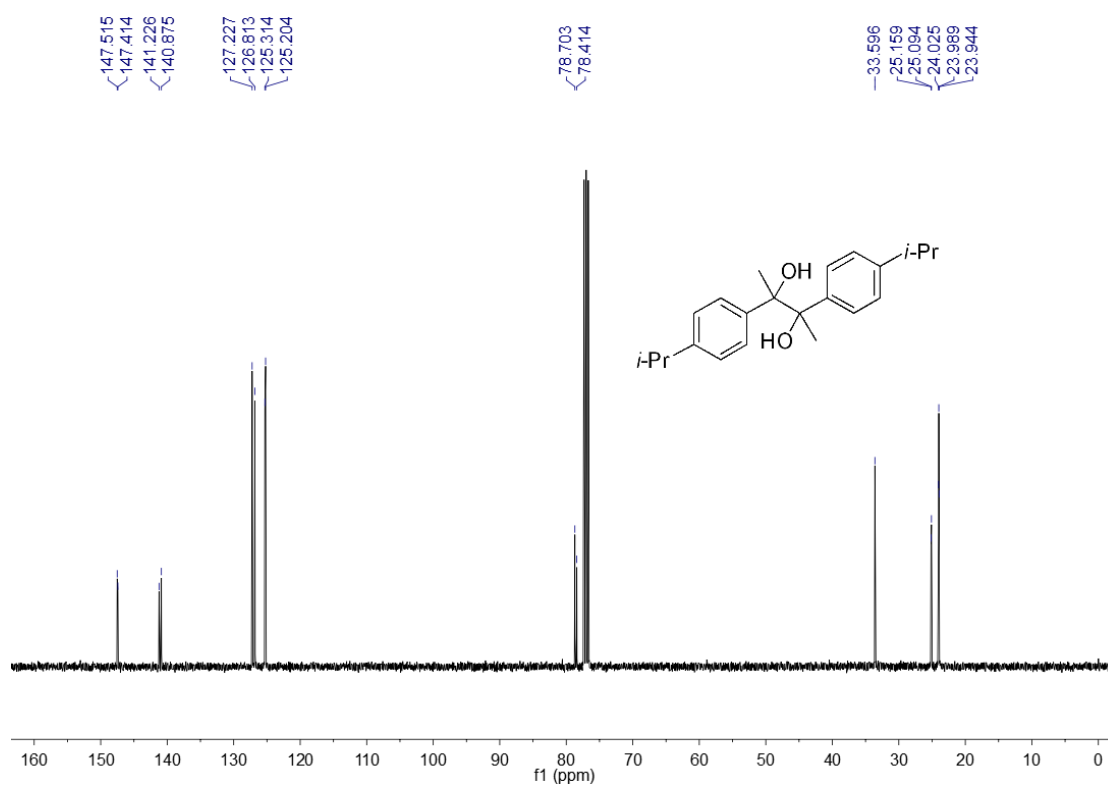
^{13}C NMR spectrum of compound **2r** (100 MHz) in CDCl_3



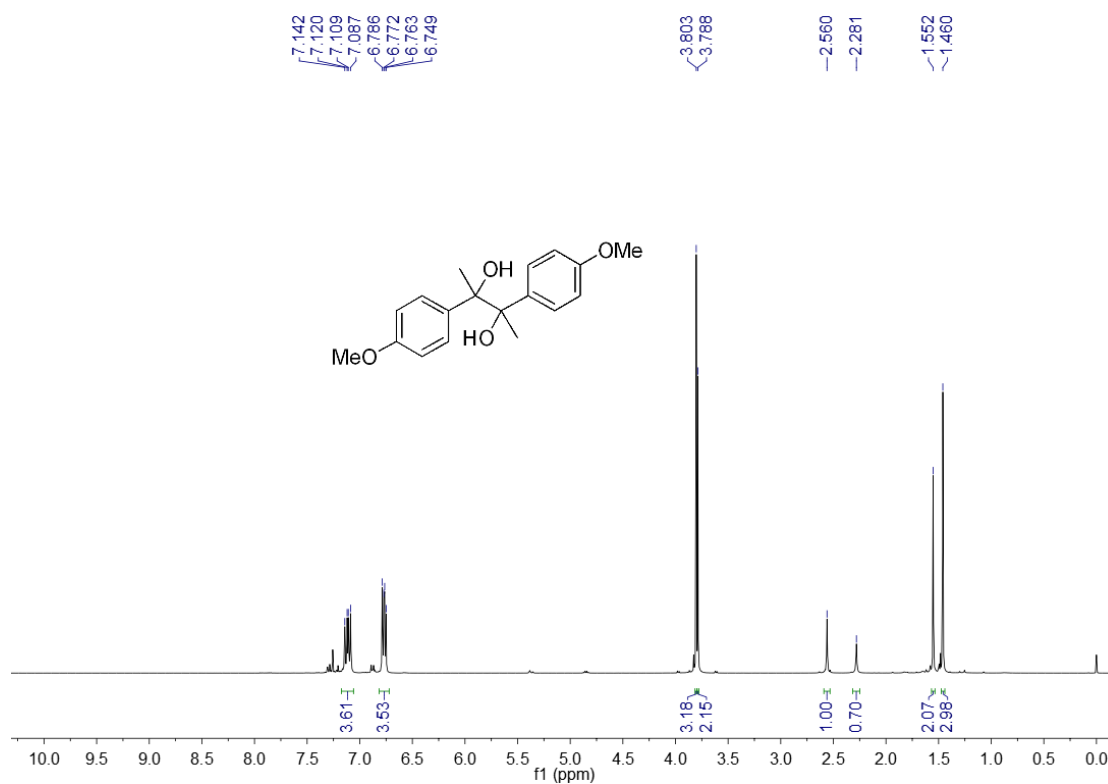
^1H NMR spectrum of compound **2s** (400 MHz) in CDCl_3



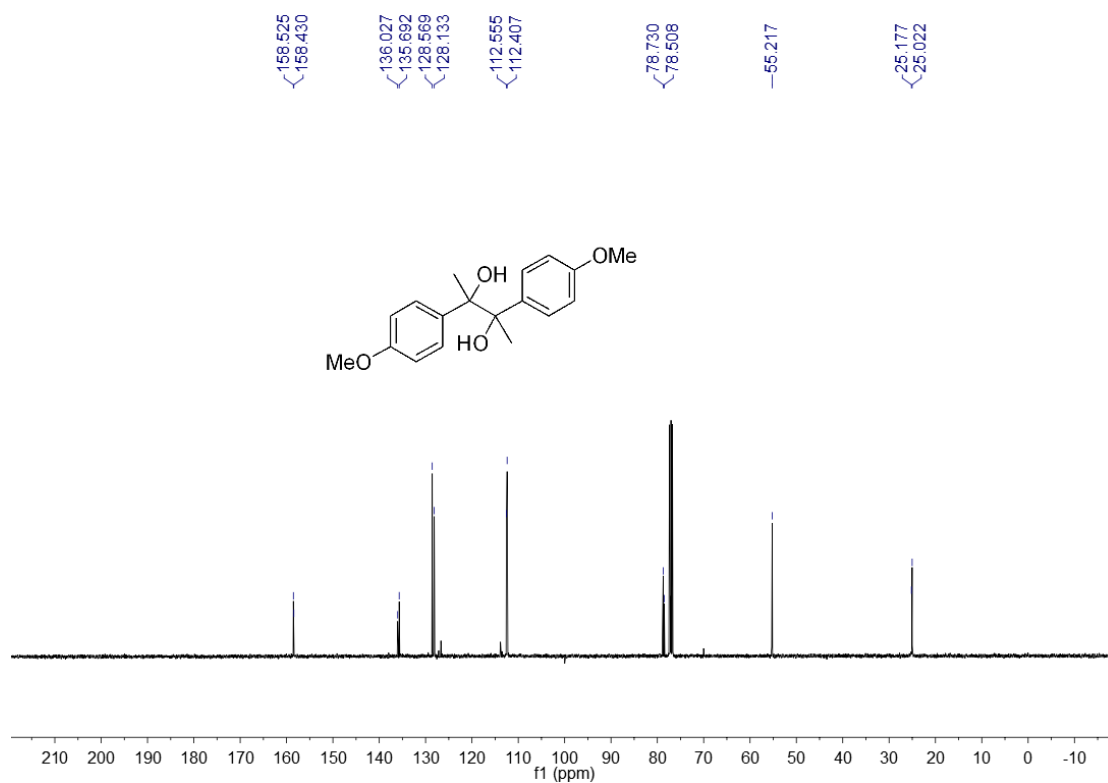
^{13}C NMR spectrum of compound **2s** (100 MHz) in CDCl_3



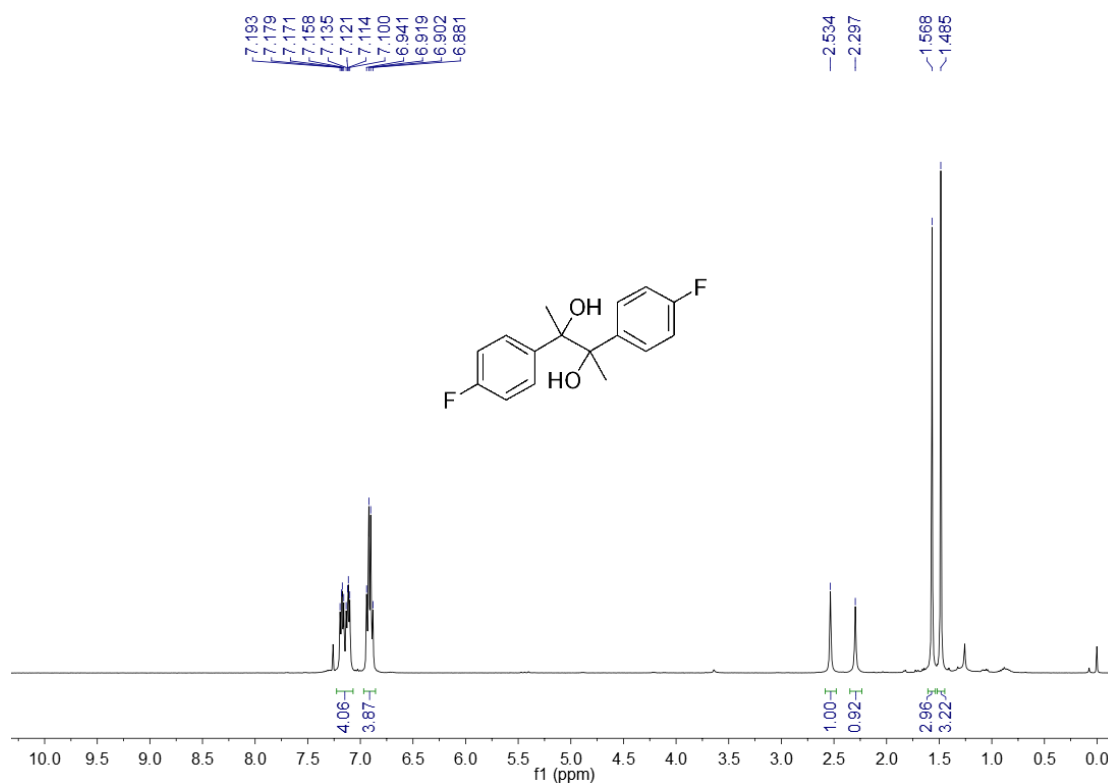
^1H NMR spectrum of compound **2t** (400 MHz) in CDCl_3



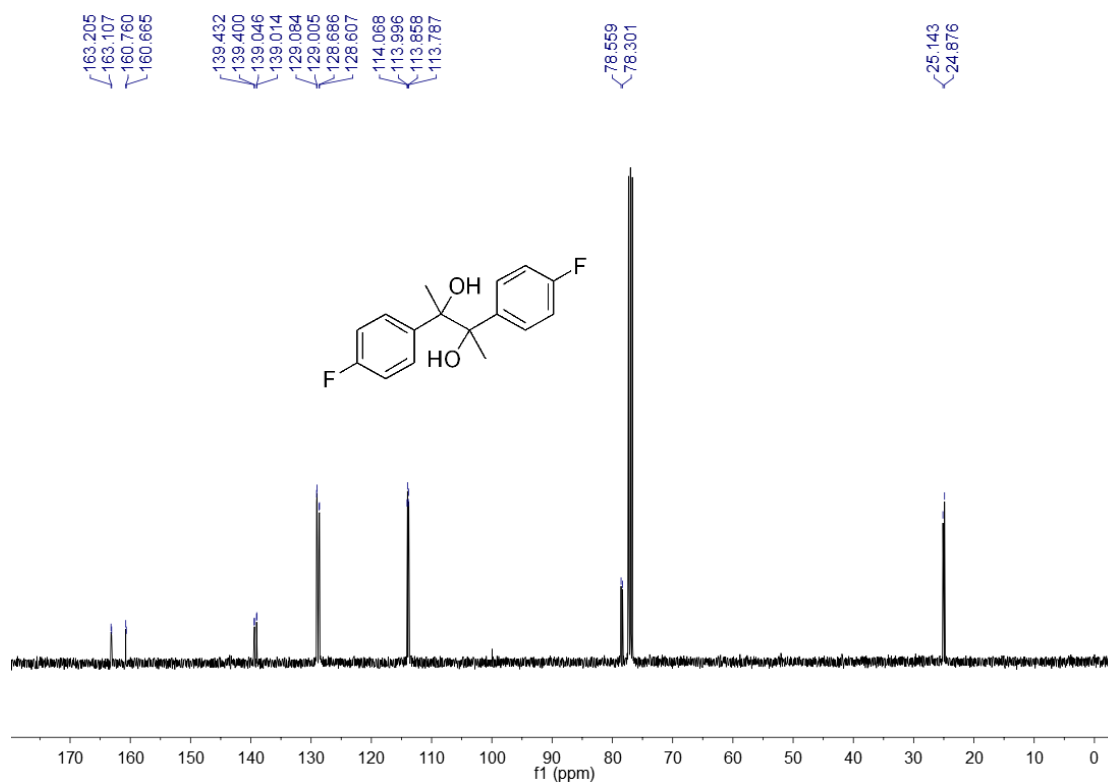
^{13}C NMR spectrum of compound **2t** (100 MHz) in CDCl_3



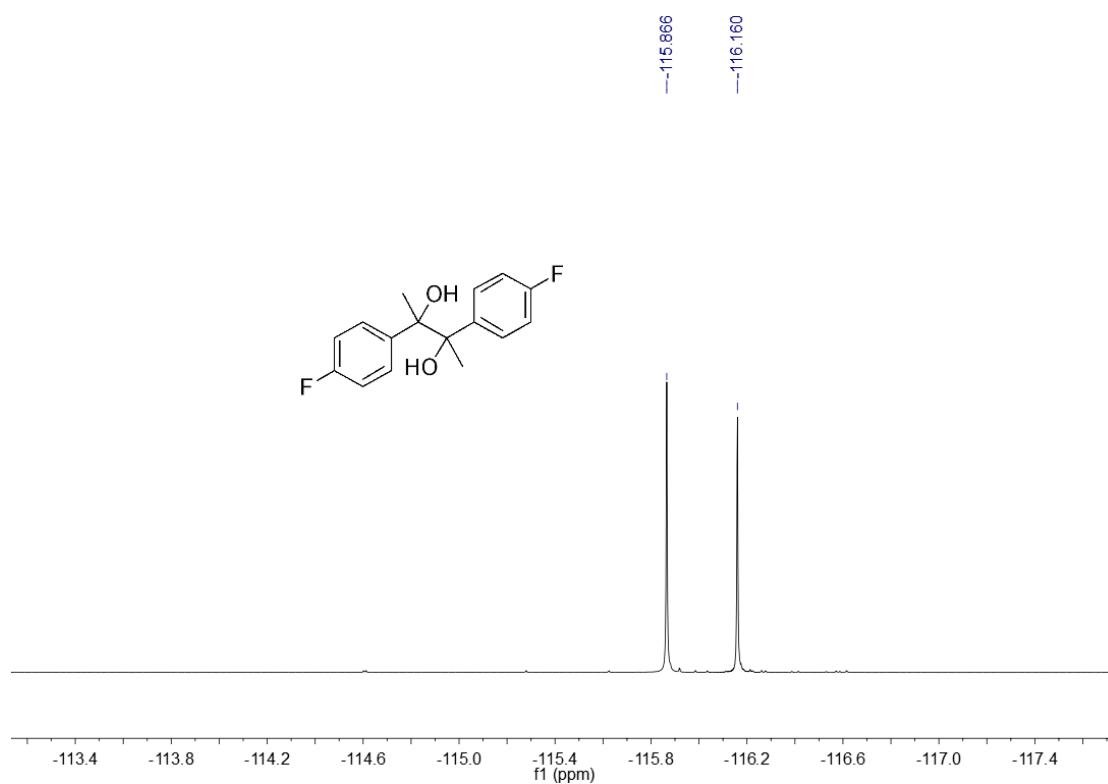
^1H NMR spectrum of compound **2u** (400 MHz) in CDCl_3



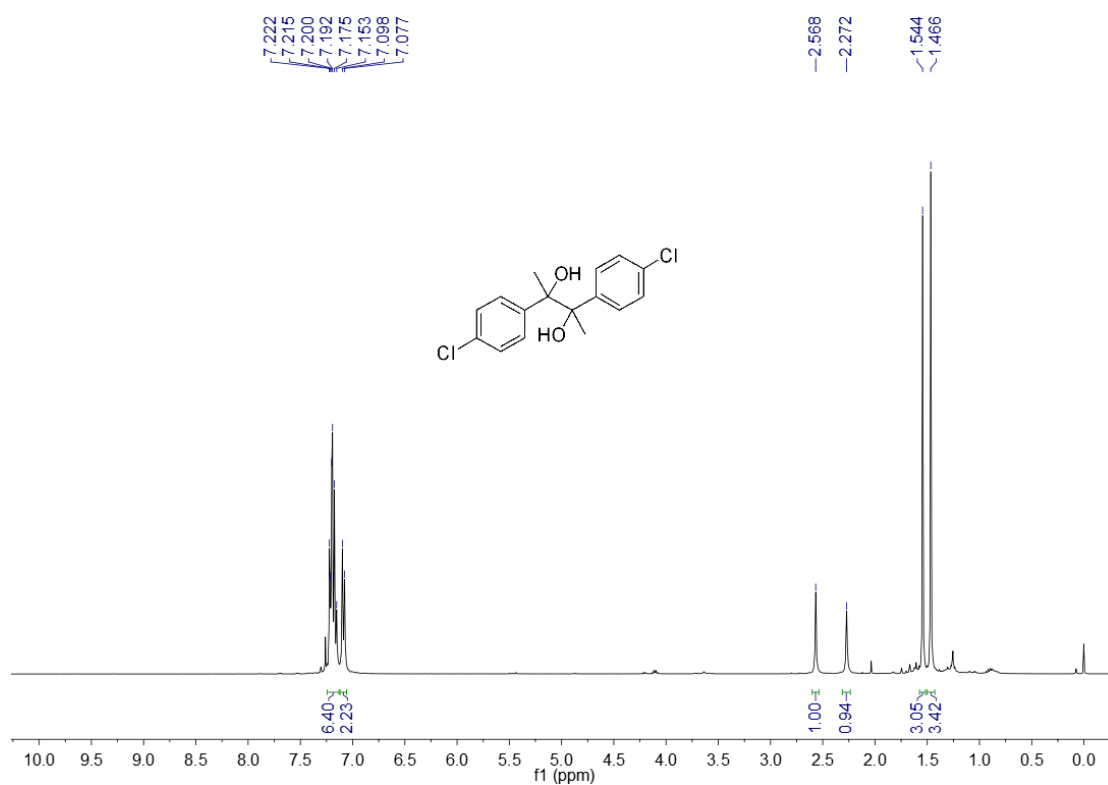
^{13}C NMR spectrum of compound **2u** (100 MHz) in CDCl_3



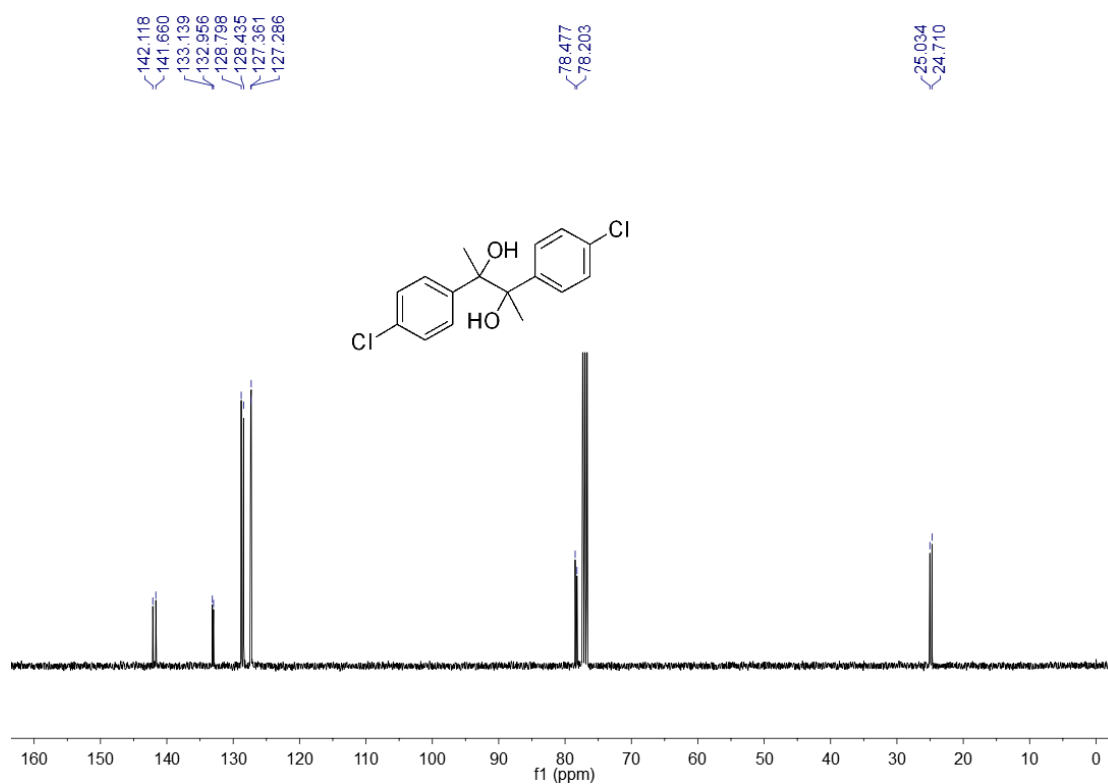
^{19}F NMR spectrum of compound **2u** (376 MHz) in CDCl_3



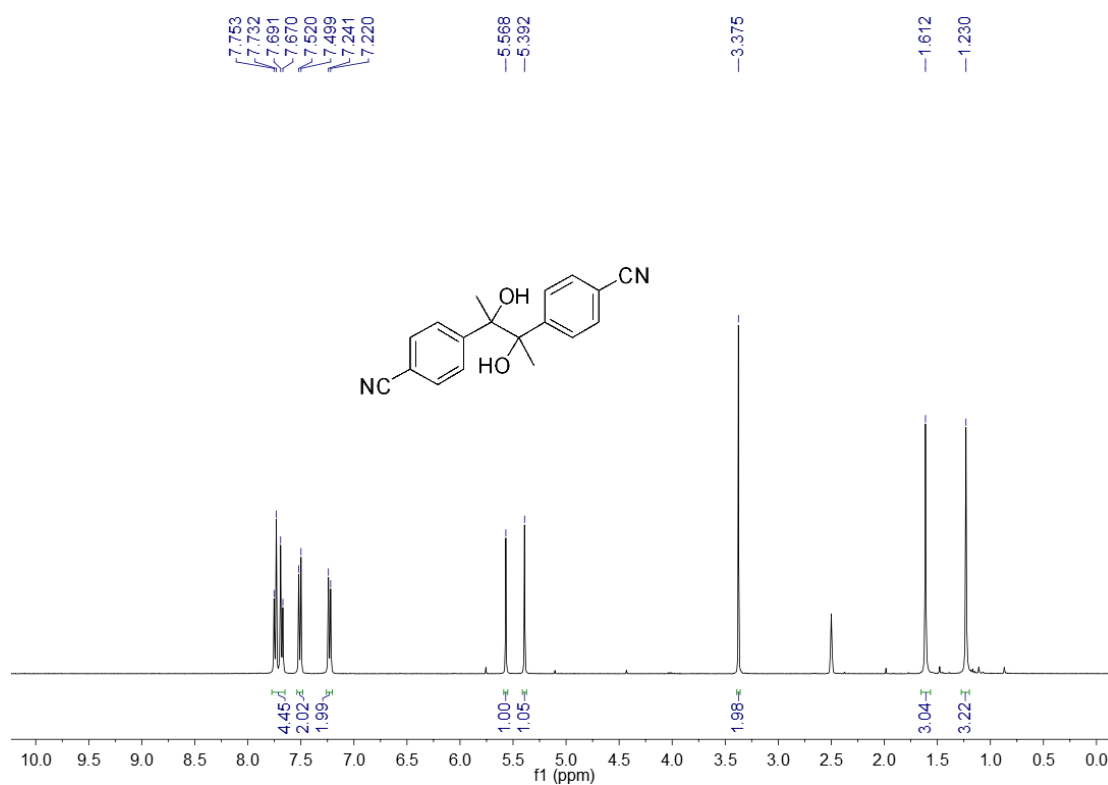
^1H NMR spectrum of compound **2v** (400 MHz) in CDCl_3



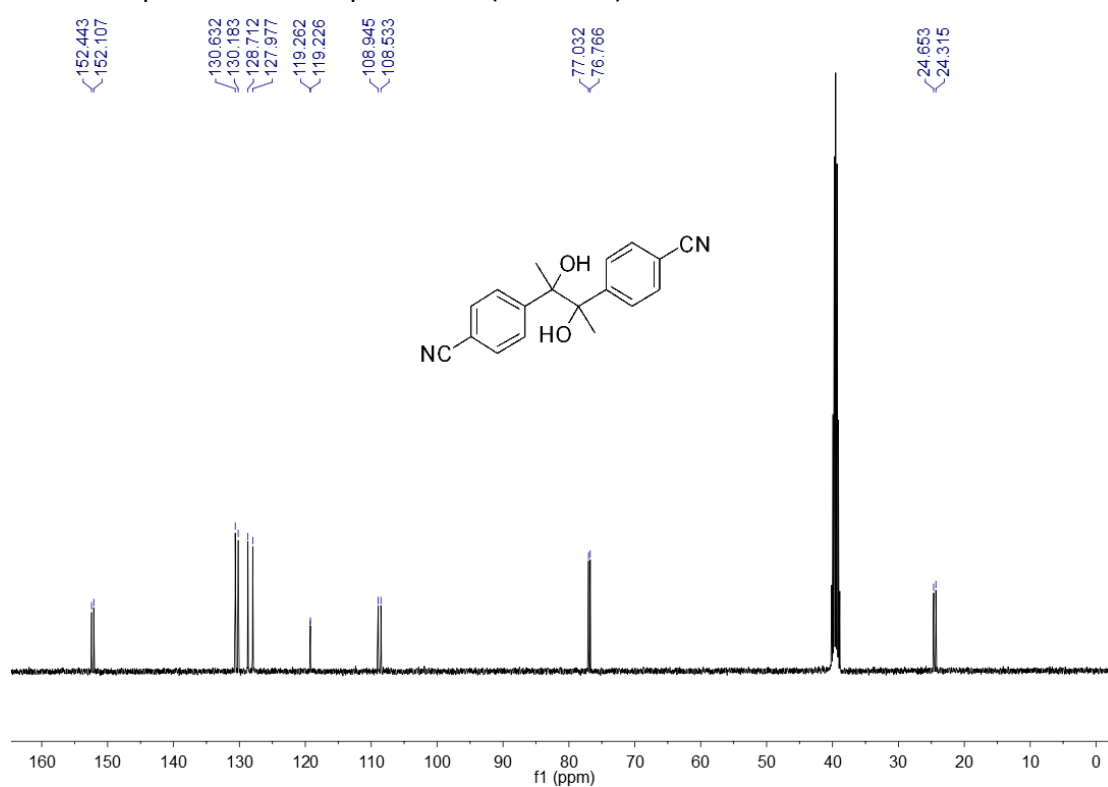
^{13}C NMR spectrum of compound **2v** (100 MHz) in CDCl_3



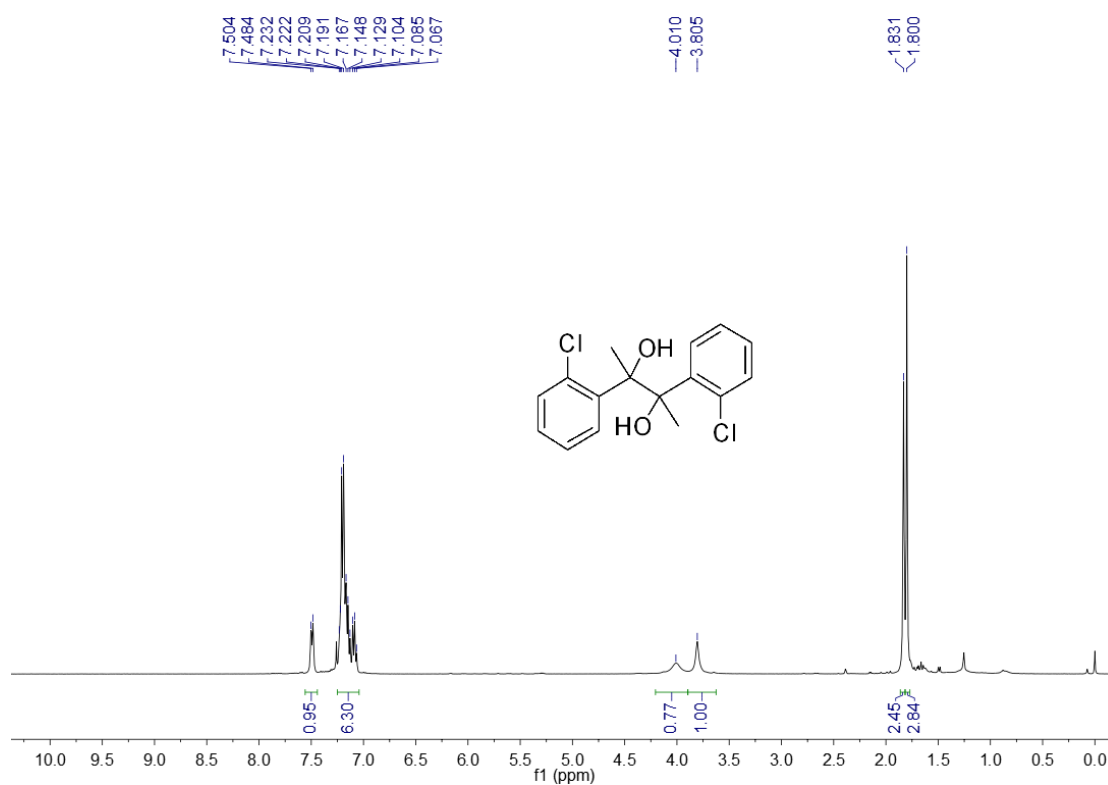
^1H NMR spectrum of compound **2w** (400 MHz) in $\text{DMSO-}d_6$



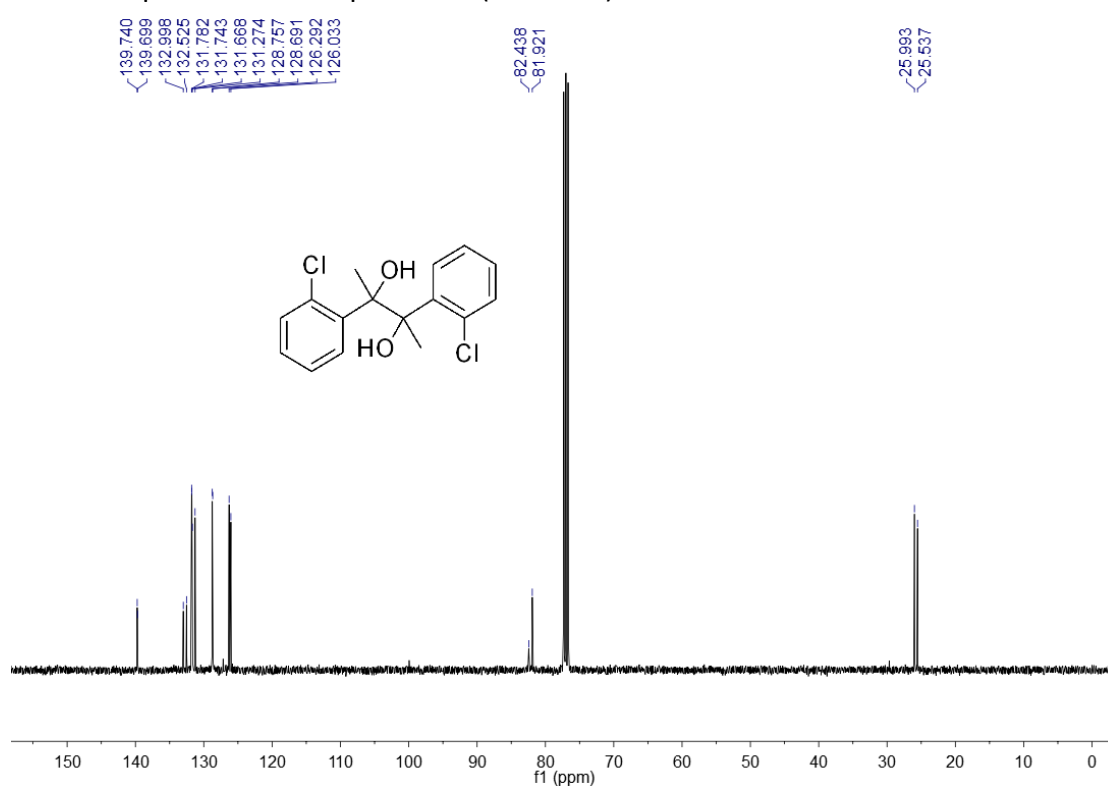
^{13}C NMR spectrum of compound **2w** (100 MHz) in $\text{DMSO-}d_6$



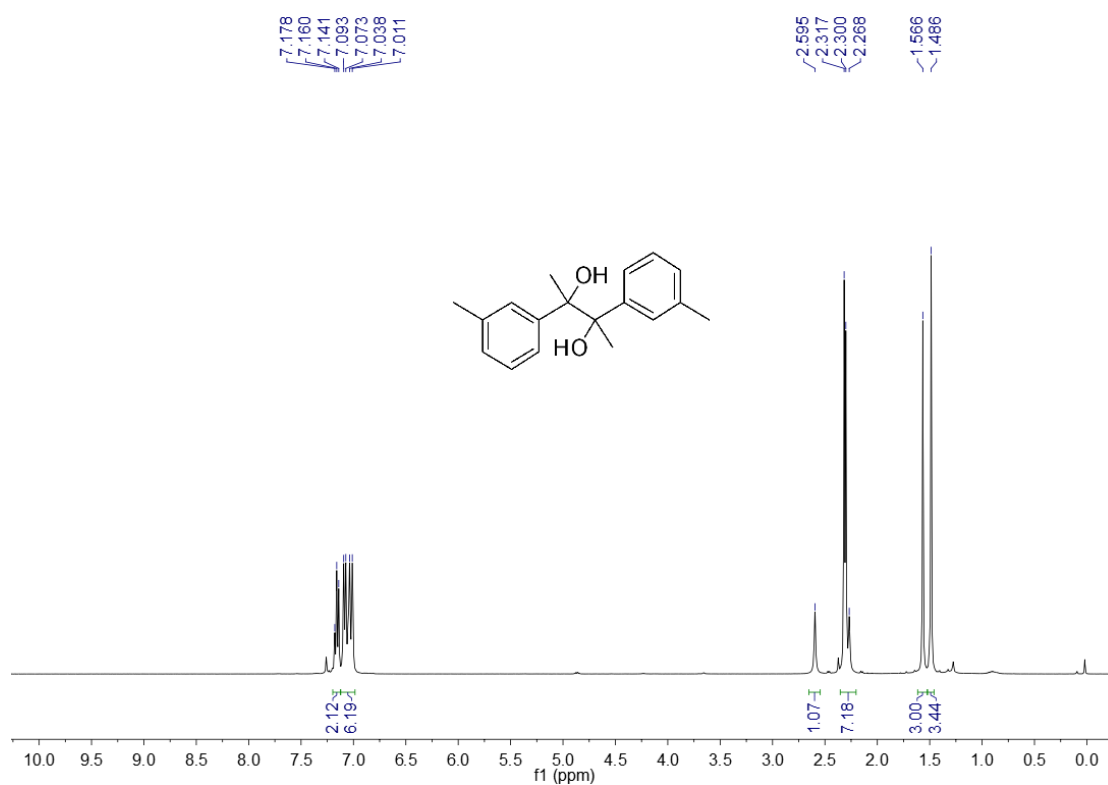
^1H NMR spectrum of compound **2x** (400 MHz) in CDCl_3



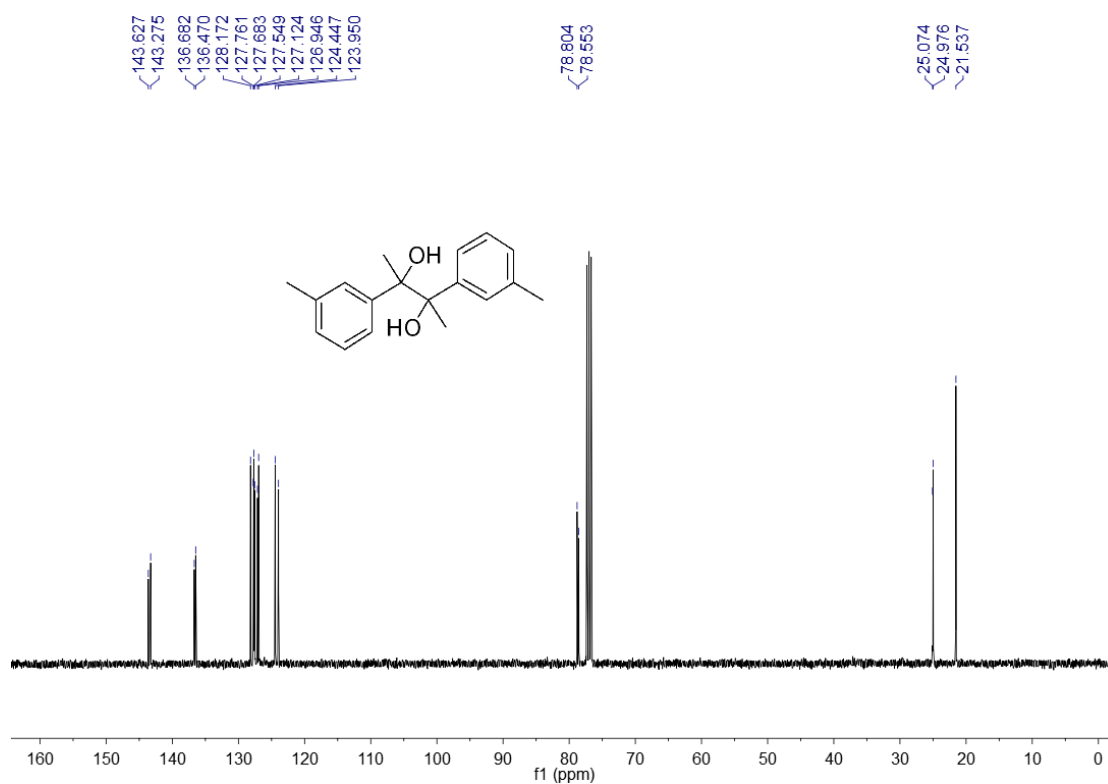
^{13}C NMR spectrum of compound **2x** (100 MHz) in CDCl_3



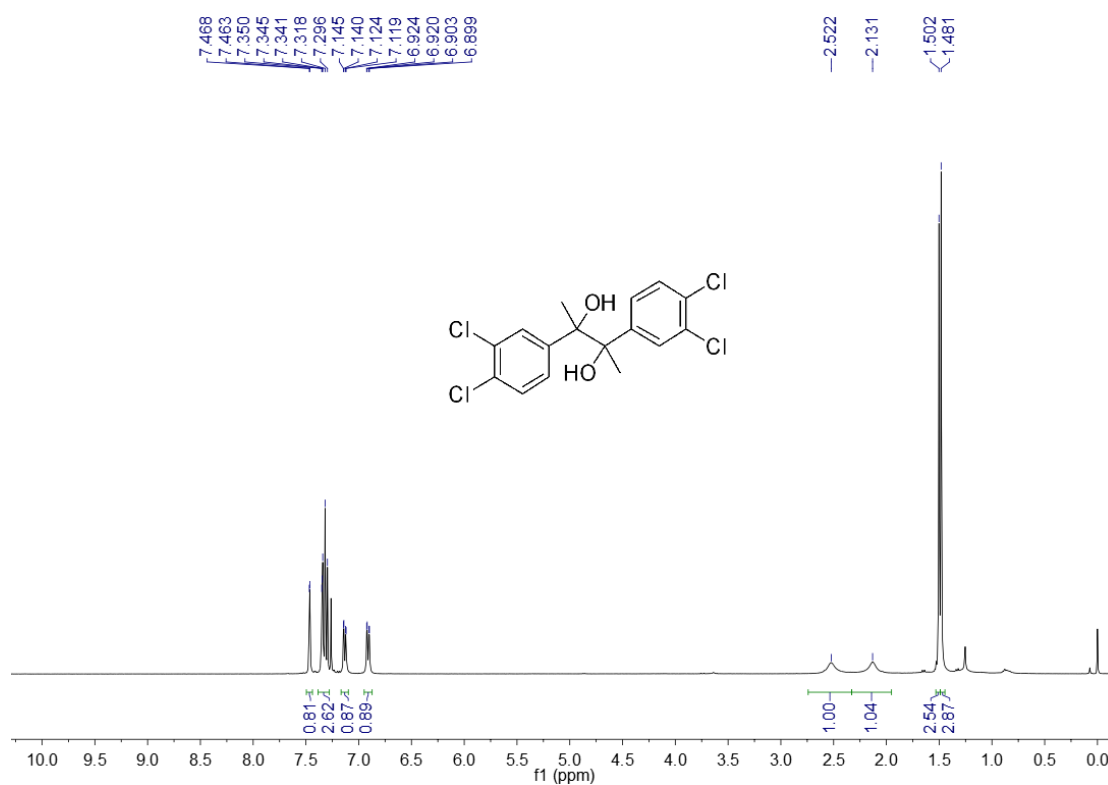
^1H NMR spectrum of compound **2y** (400 MHz) in CDCl_3



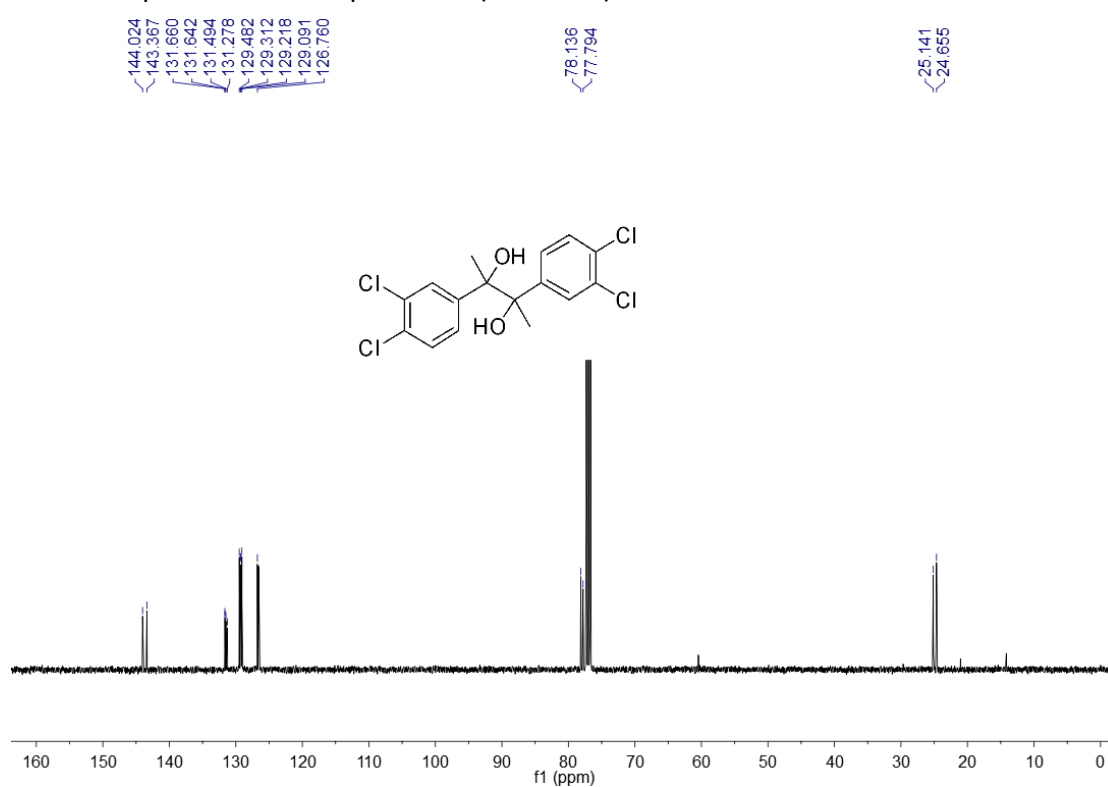
^{13}C NMR spectrum of compound **2y** (100 MHz) in CDCl_3



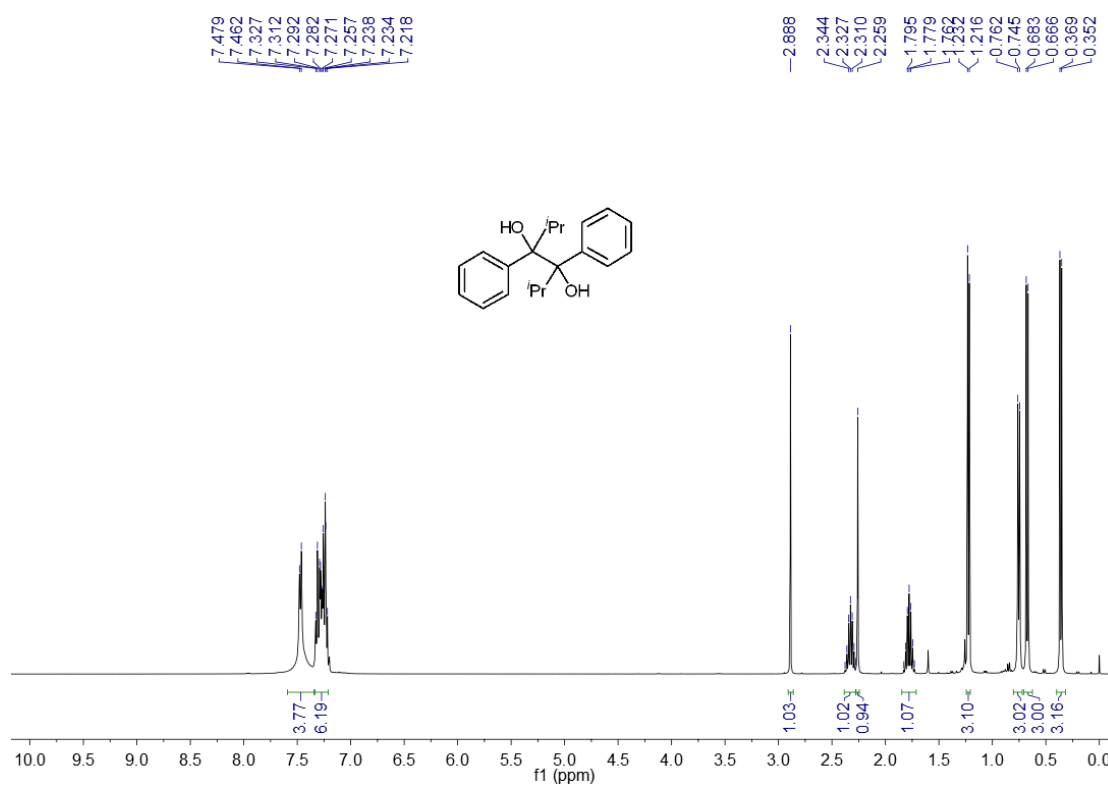
^1H NMR spectrum of compound **2z** (400 MHz) in CDCl_3



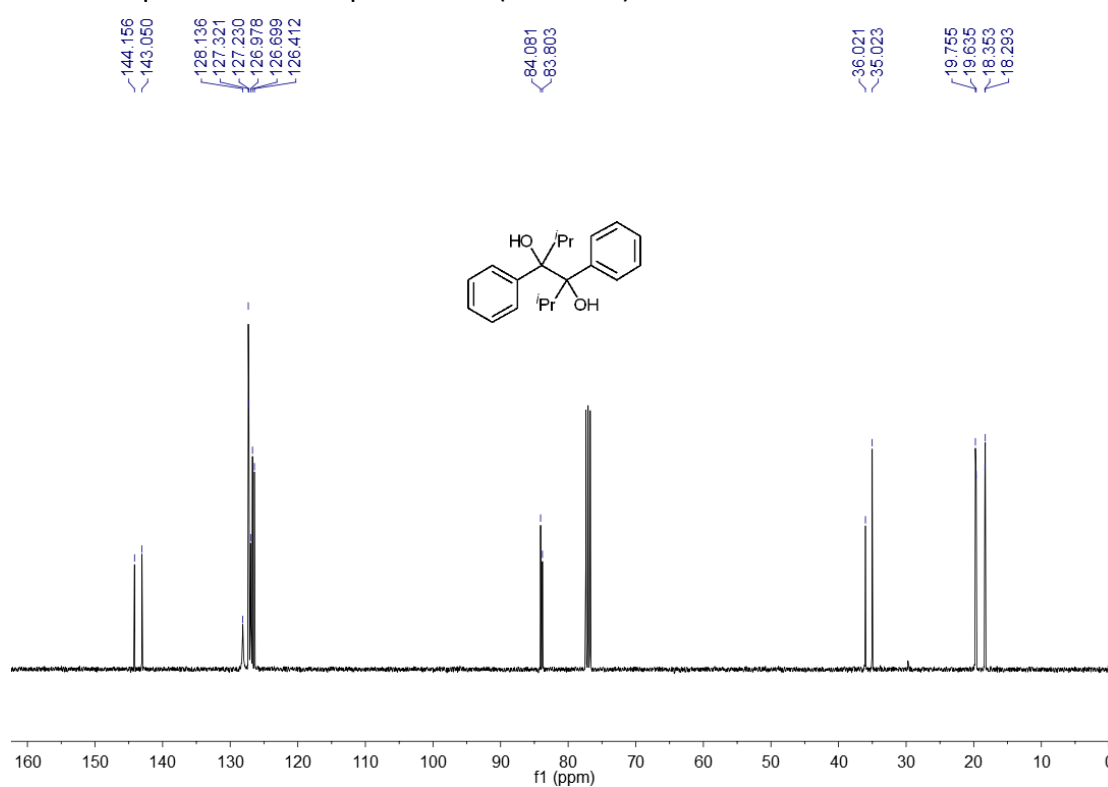
^{13}C NMR spectrum of compound **2z** (100 MHz) in CDCl_3



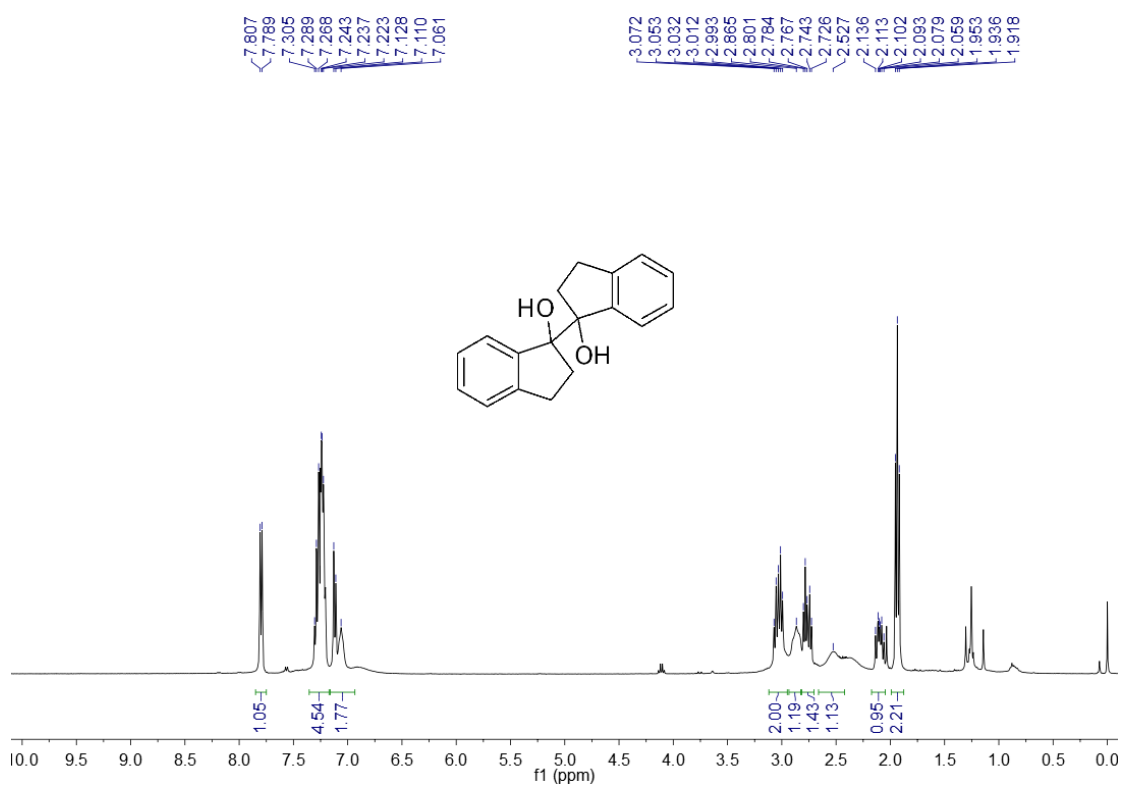
^1H NMR spectrum of compound **2aa** (400 MHz) in CDCl_3



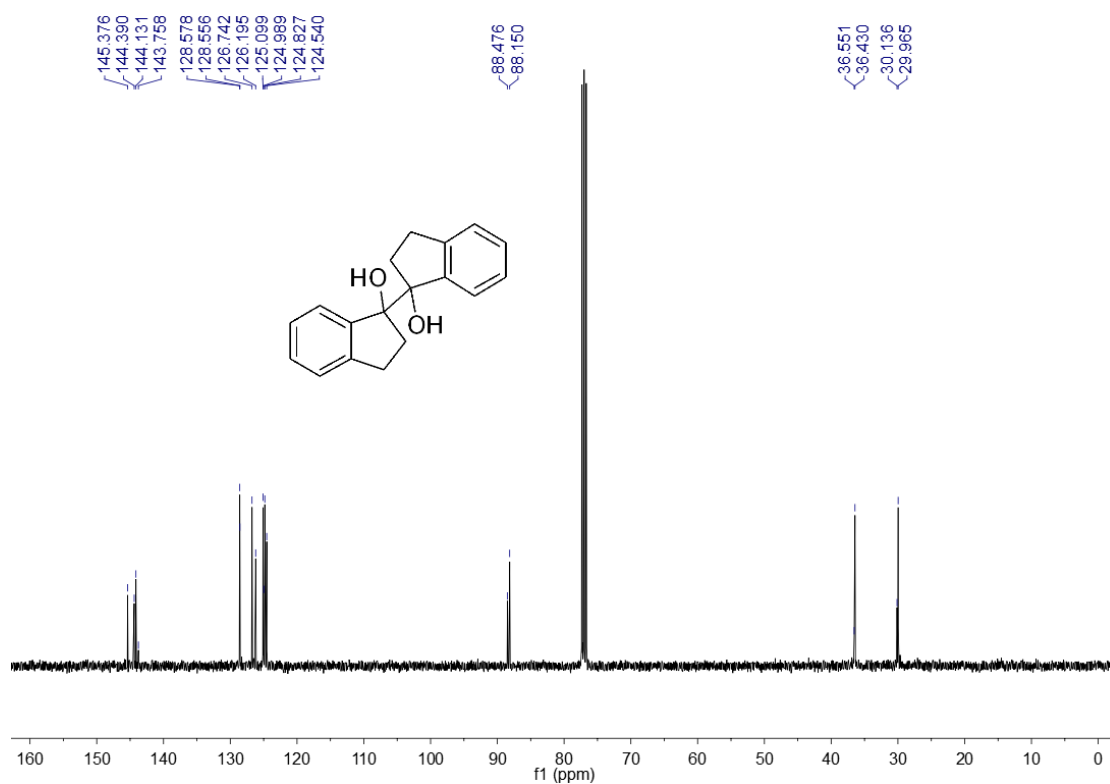
^{13}C NMR spectrum of compound **2aa** (100 MHz) in CDCl_3



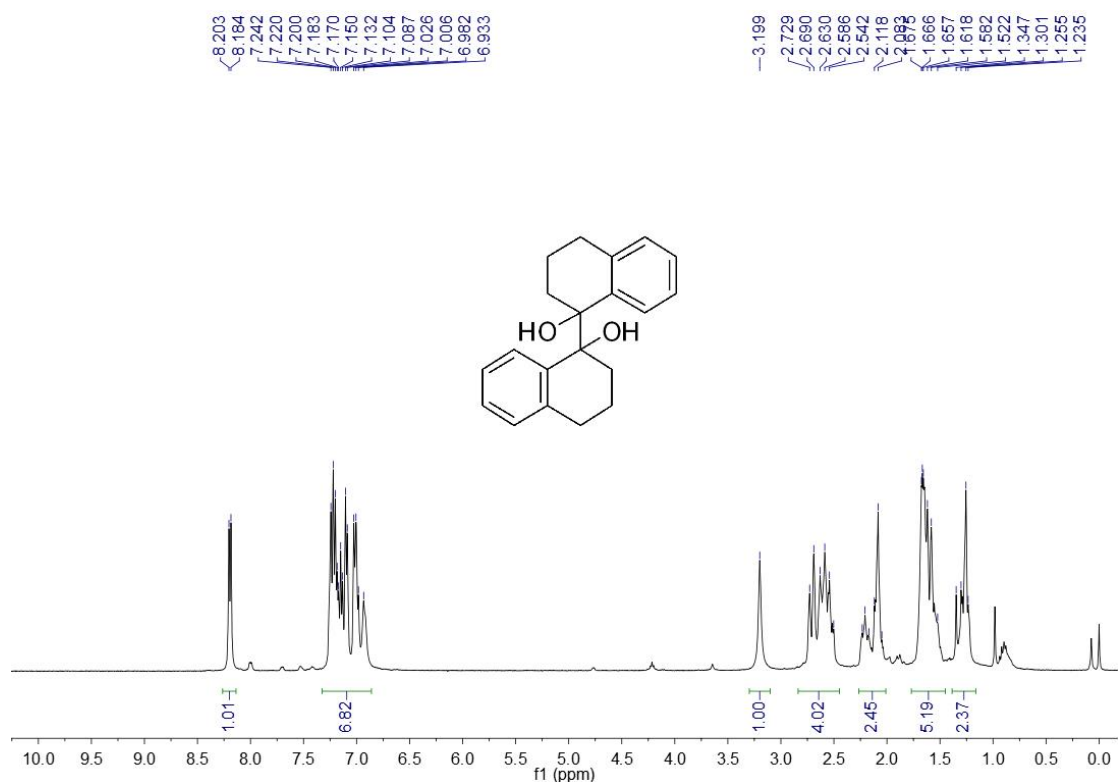
^1H NMR spectrum of compound **2ab** (400 MHz) in CDCl_3



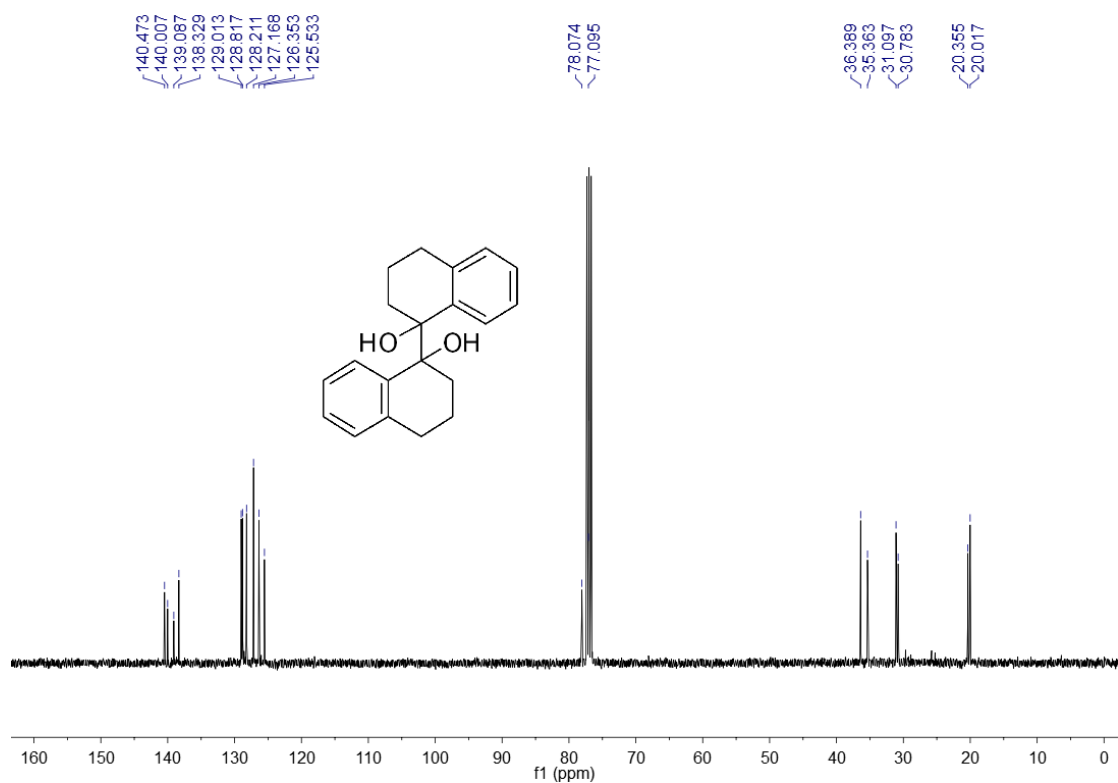
^{13}C NMR spectrum of compound **2ab** (100 MHz) in CDCl_3



^1H NMR spectrum of compound **2ac** (400 MHz) in CDCl_3



^{13}C NMR spectrum of compound **2ac** (100 MHz) in CDCl_3



Chemical structure: OC(C(O)c1ccccc1)c2ccc(C(F)(F)F)cc2

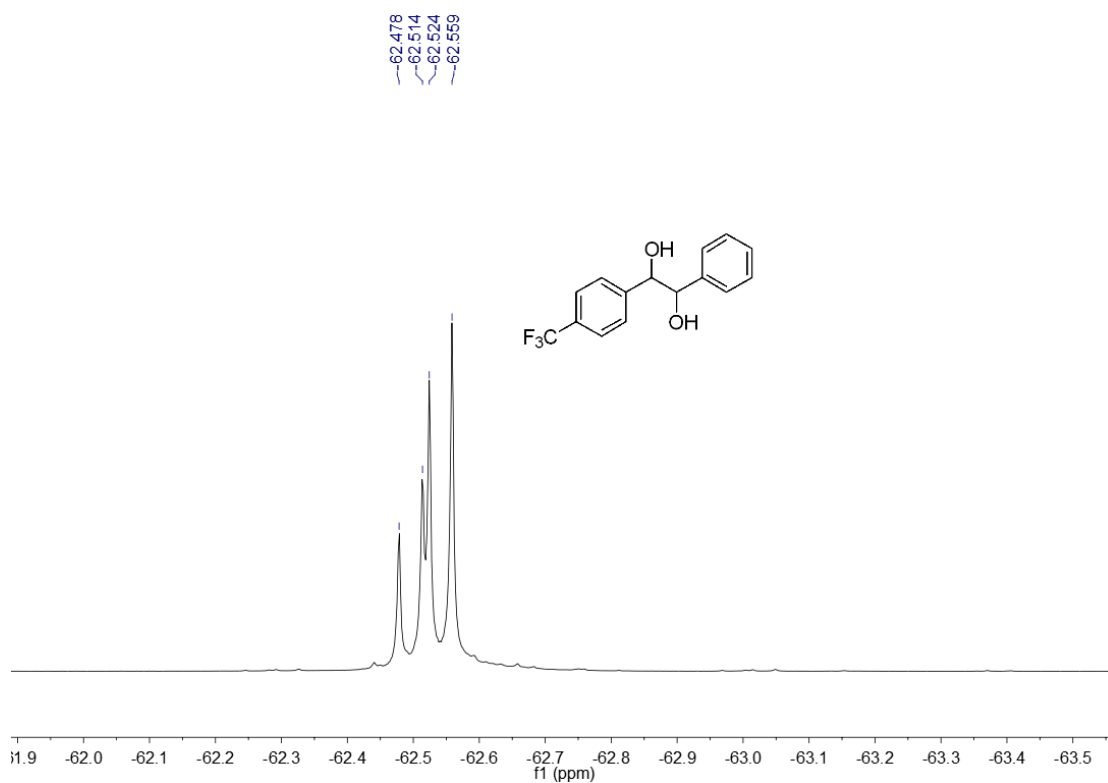
¹H NMR spectrum (ppm):

- 7.503, 7.483, 7.462, 7.448, 7.428, 7.276, 7.271, 7.265, 7.255, 7.233, 7.211, 7.206, 7.194, 7.159, 7.139, 7.134, 7.082, 7.075, 7.070, 7.061, 7.051, 7.042, 4.868, 4.849, 4.806, 4.793, 4.777, 4.703, 4.684, 4.638, 4.585, 4.566
- Integration: 1.96, 6.44, 1.35, 1.00, 1.04, 2.19
- Peak labels: -3.281, -2.874, -0.000

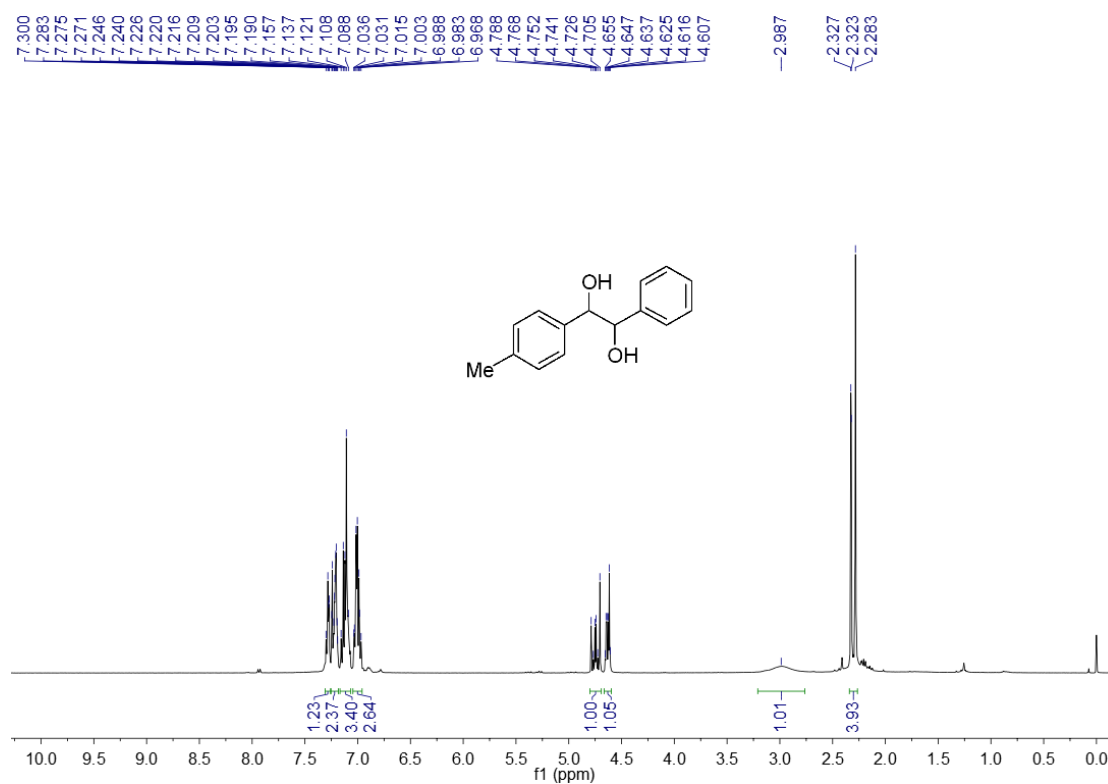
Figure 1 displays the ^{13}C NMR spectra of (S)-1-(4-(trifluoromethyl)phenyl)-2-phenylethane-1,2-diol. The chemical structure of the compound is shown above the spectra.

The top spectrum shows the full ^{13}C NMR spectrum (f1 (ppm)) with peaks labeled from 143.729 to 124.898 ppm. The middle spectrum shows a zoomed-in region of the spectrum (f1 (ppm)) with peaks labeled from 79.096 to 77.253 ppm. The bottom spectrum shows the full ^{13}C NMR spectrum (f1 (ppm)) with peaks labeled from 145 to 74 ppm.

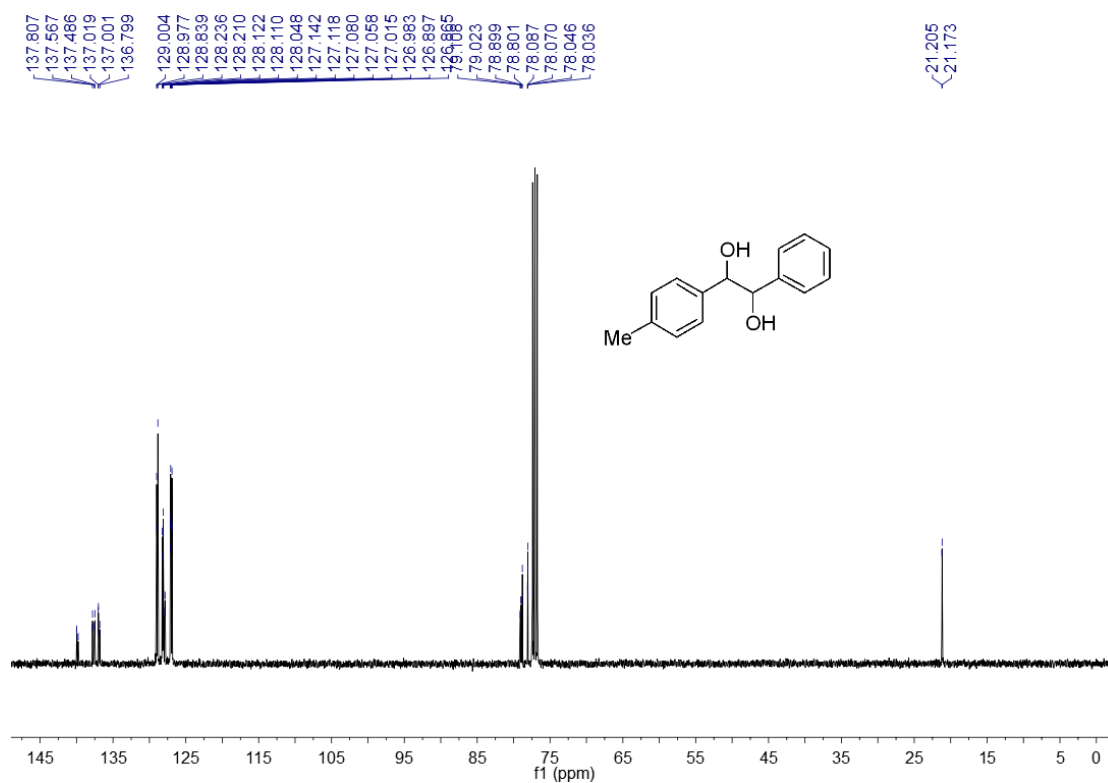
^{19}F NMR spectrum of compound **4a** (376 MHz) in CDCl_3



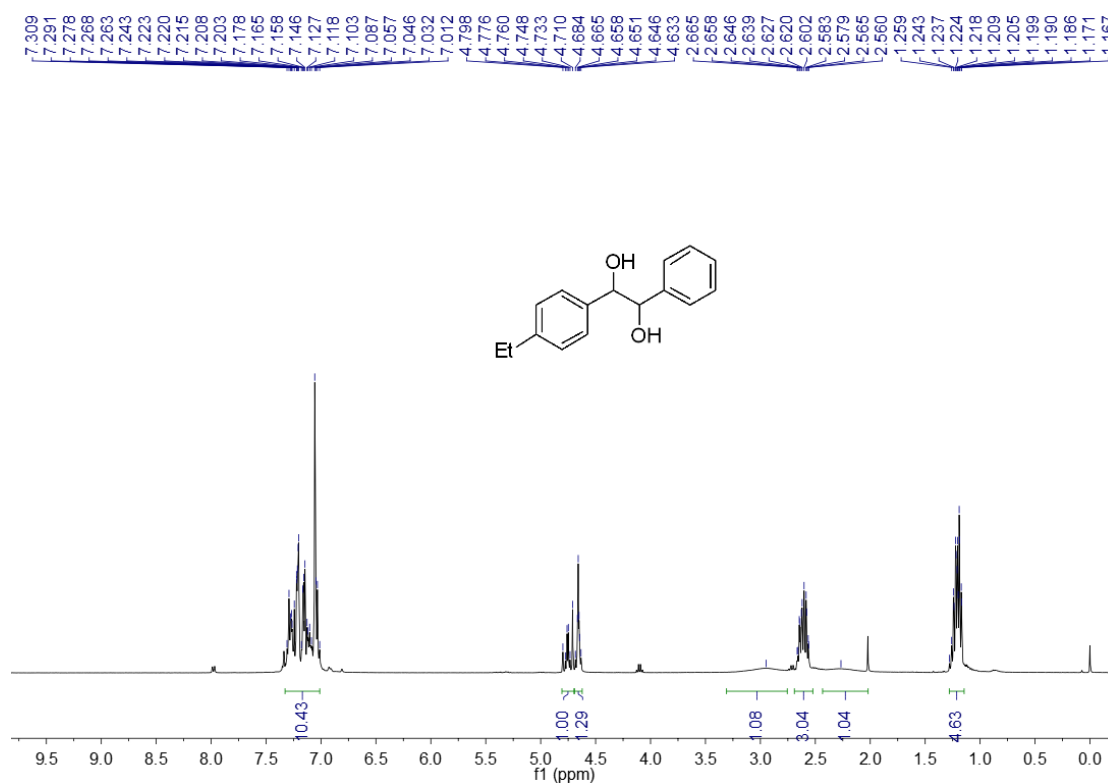
^1H NMR spectrum of compound **4b** (400 MHz) in CDCl_3



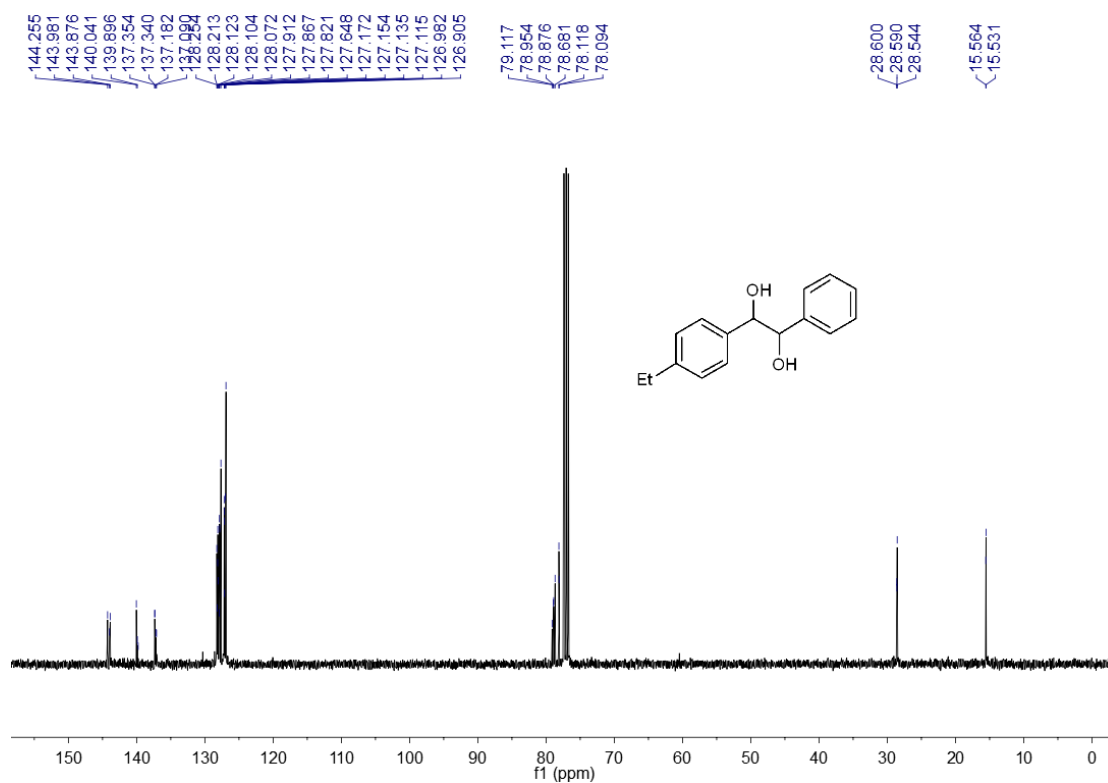
^{13}C NMR spectrum of compound **4b** (100 MHz) in CDCl_3



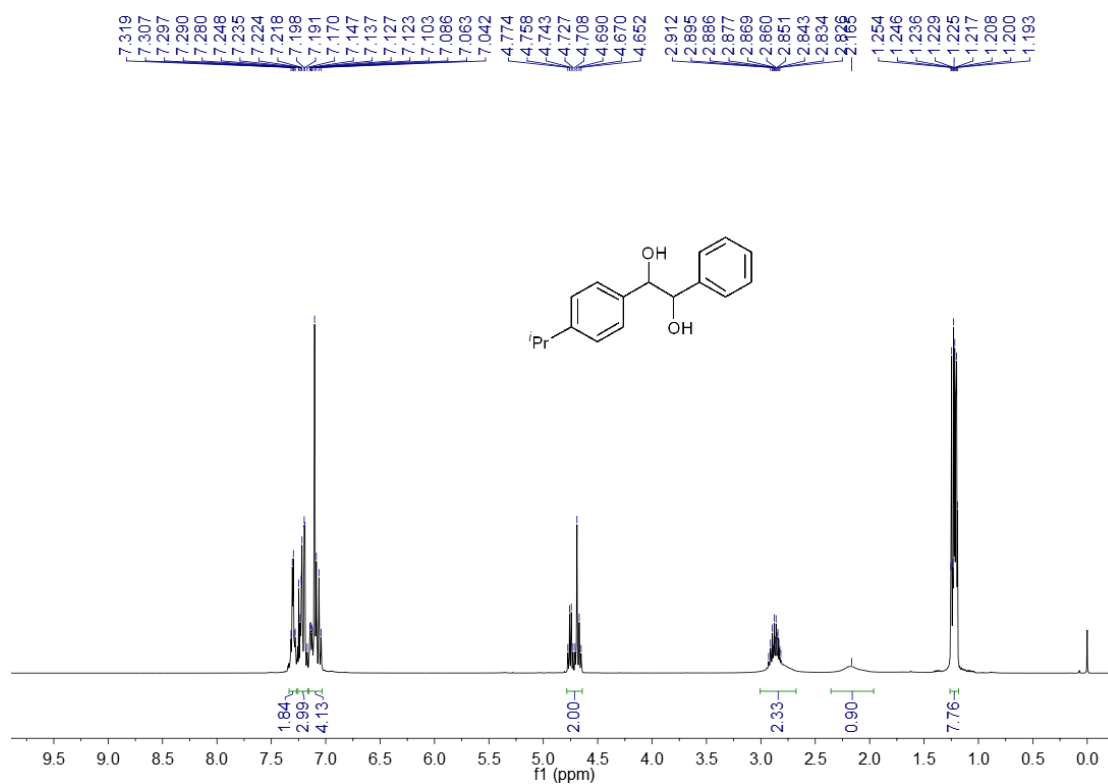
^1H NMR spectrum of compound **4c** (400 MHz) in CDCl_3



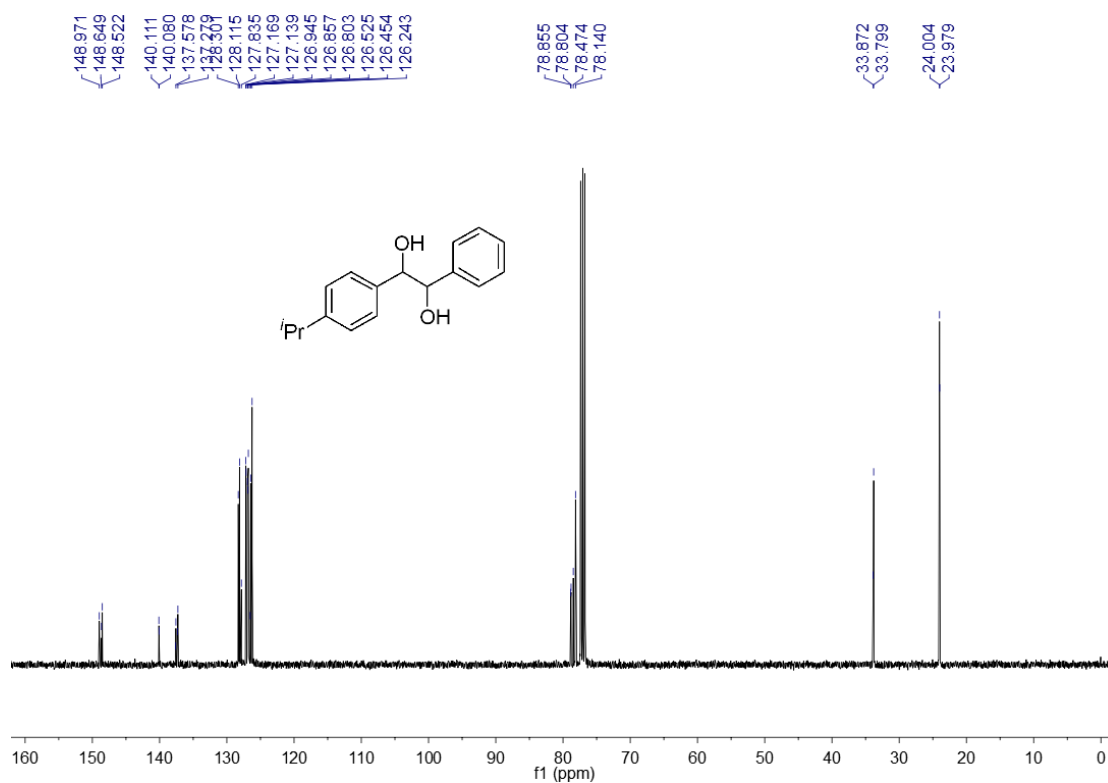
^{13}C NMR spectrum of compound **4c** (100 MHz) in CDCl_3



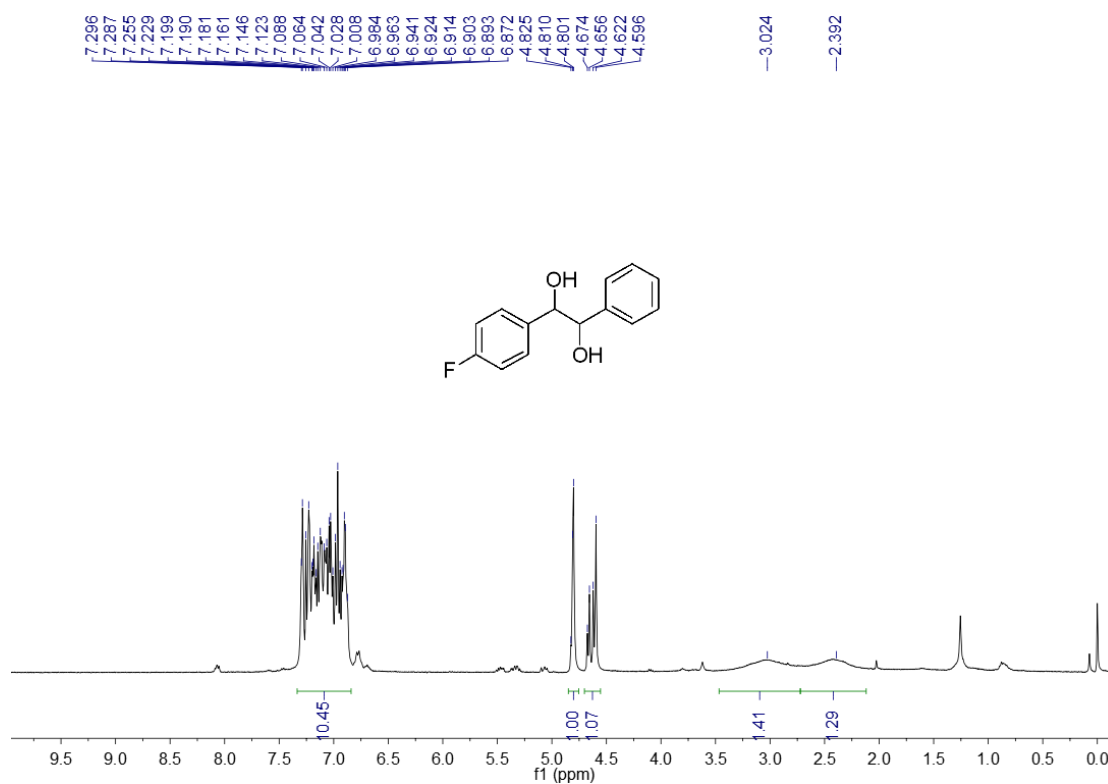
^1H NMR spectrum of compound **4d** (400 MHz) in CDCl_3



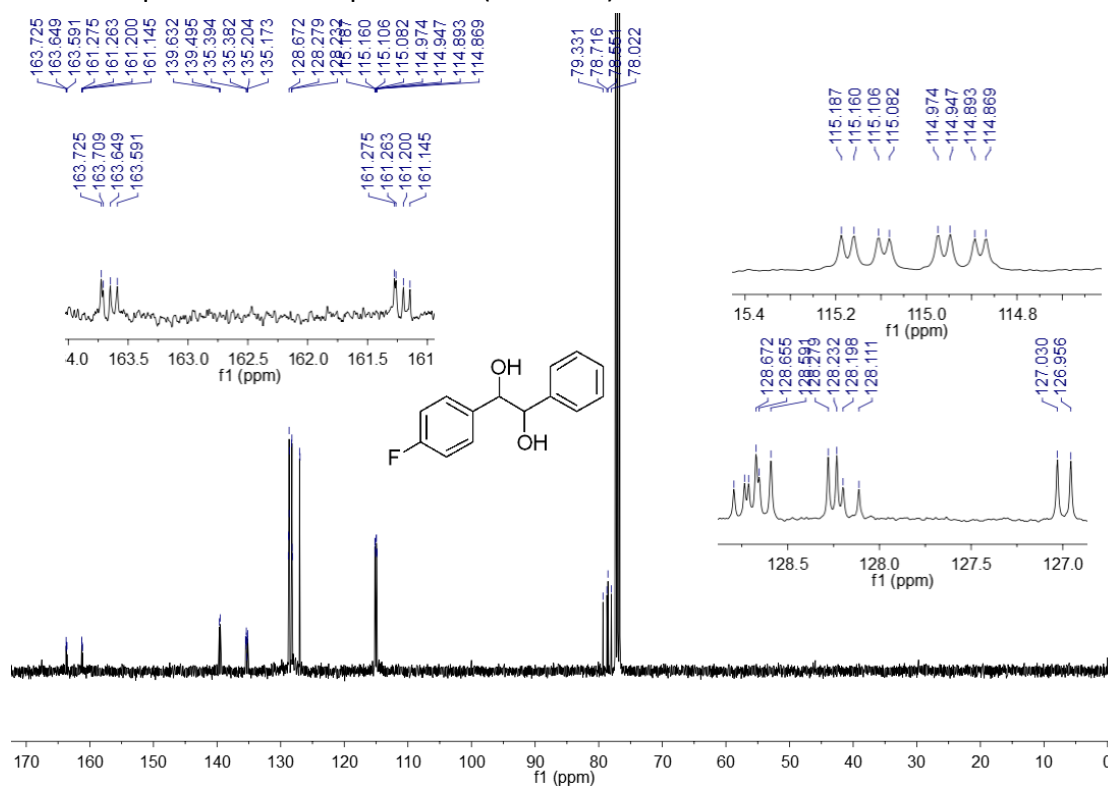
^{13}C NMR spectrum of compound **4d** (100 MHz) in CDCl_3



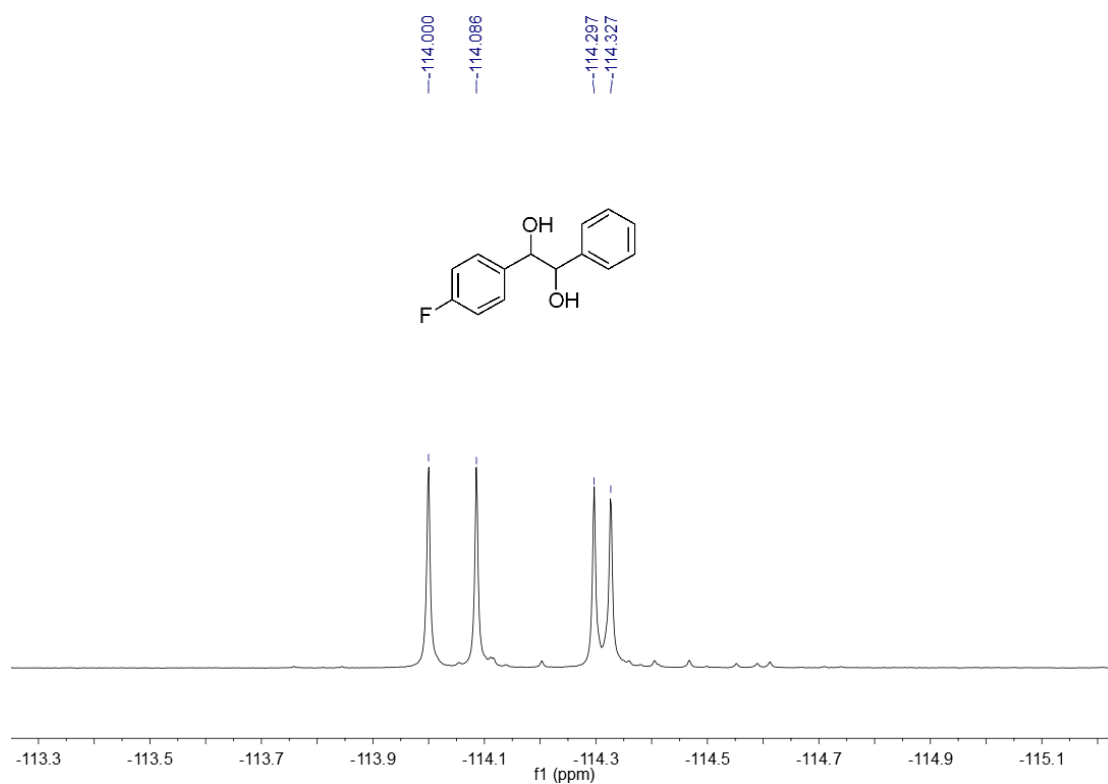
^1H NMR spectrum of compound **4e** (400 MHz) in CDCl_3



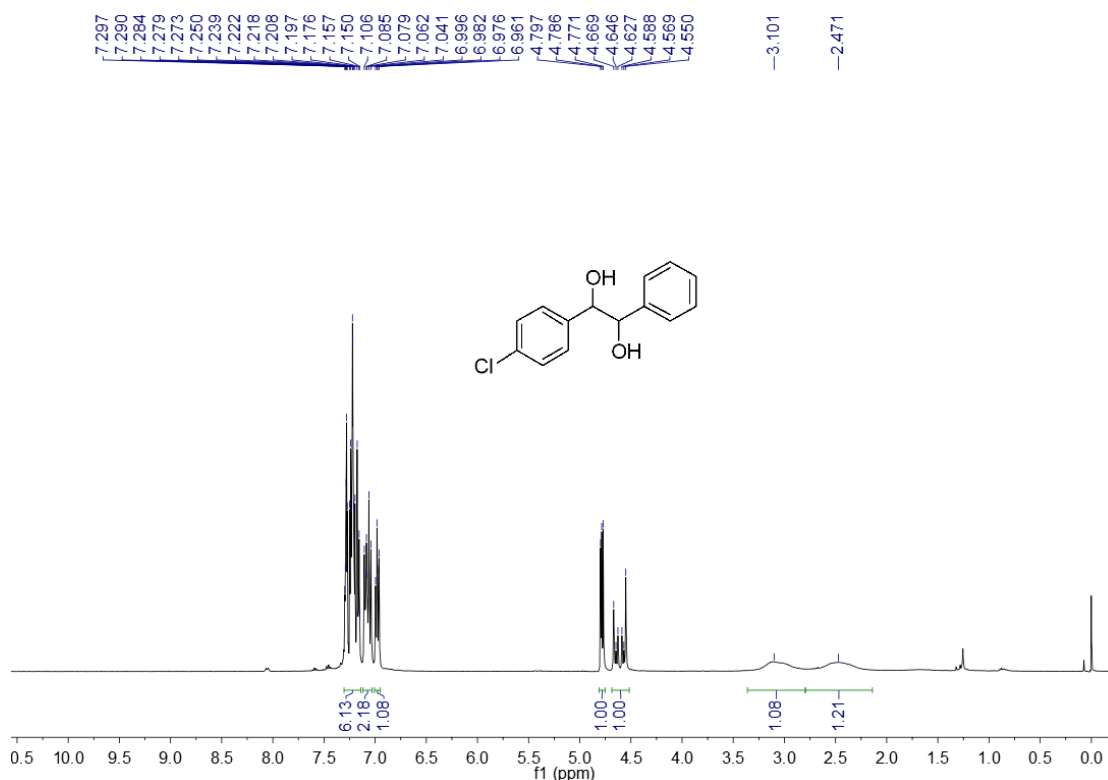
^{13}C NMR spectrum of compound **4e** (100 MHz) in CDCl_3



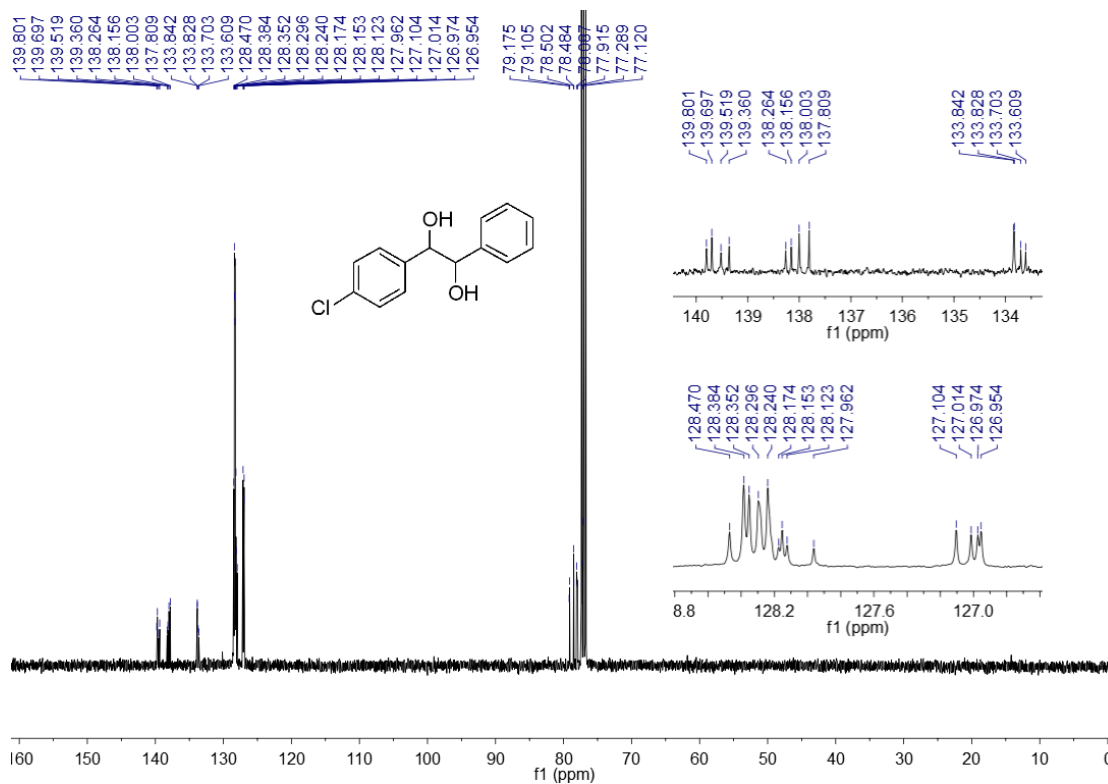
^{19}F NMR spectrum of compound **4e** (376 MHz) in CDCl_3



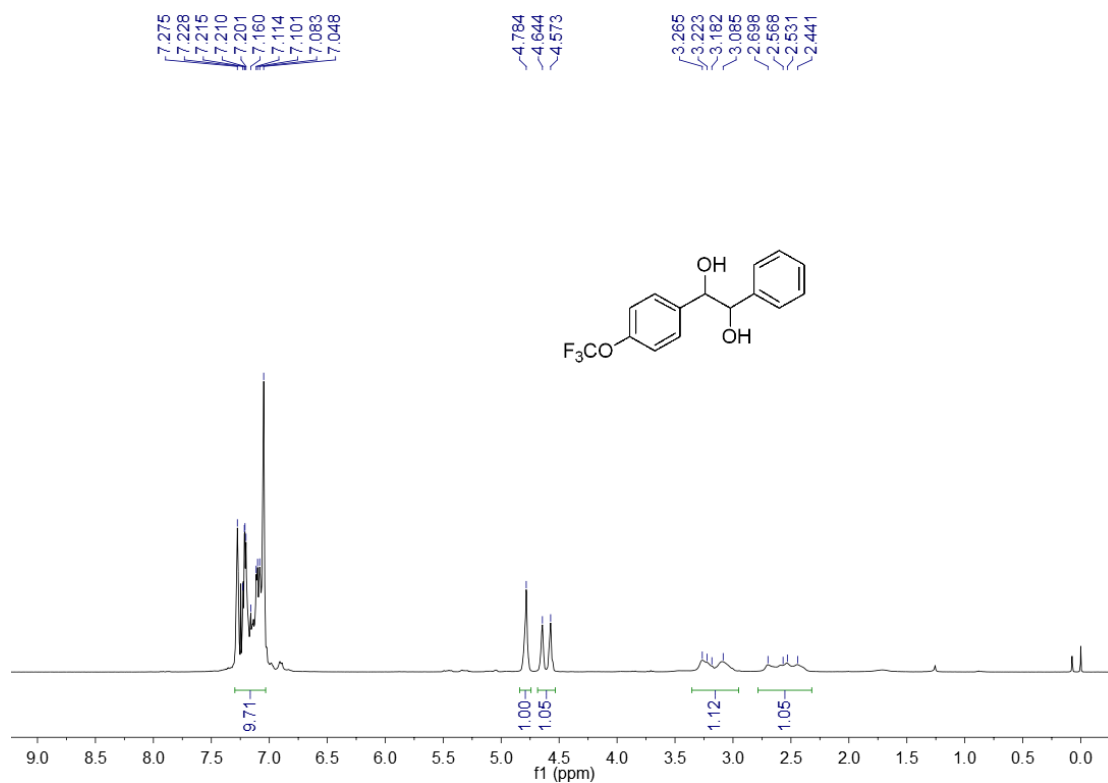
^1H NMR spectrum of compound **4f** (400 MHz) in CDCl_3



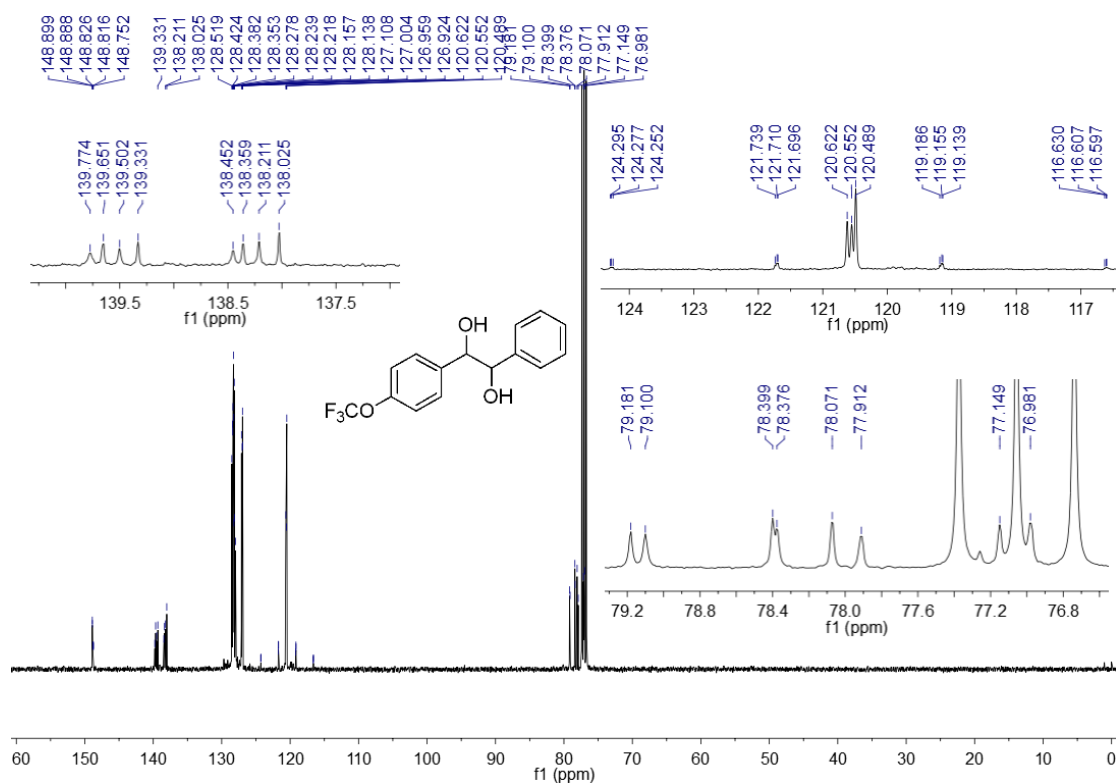
^{13}C NMR spectrum of compound **4f** (100 MHz) in CDCl_3



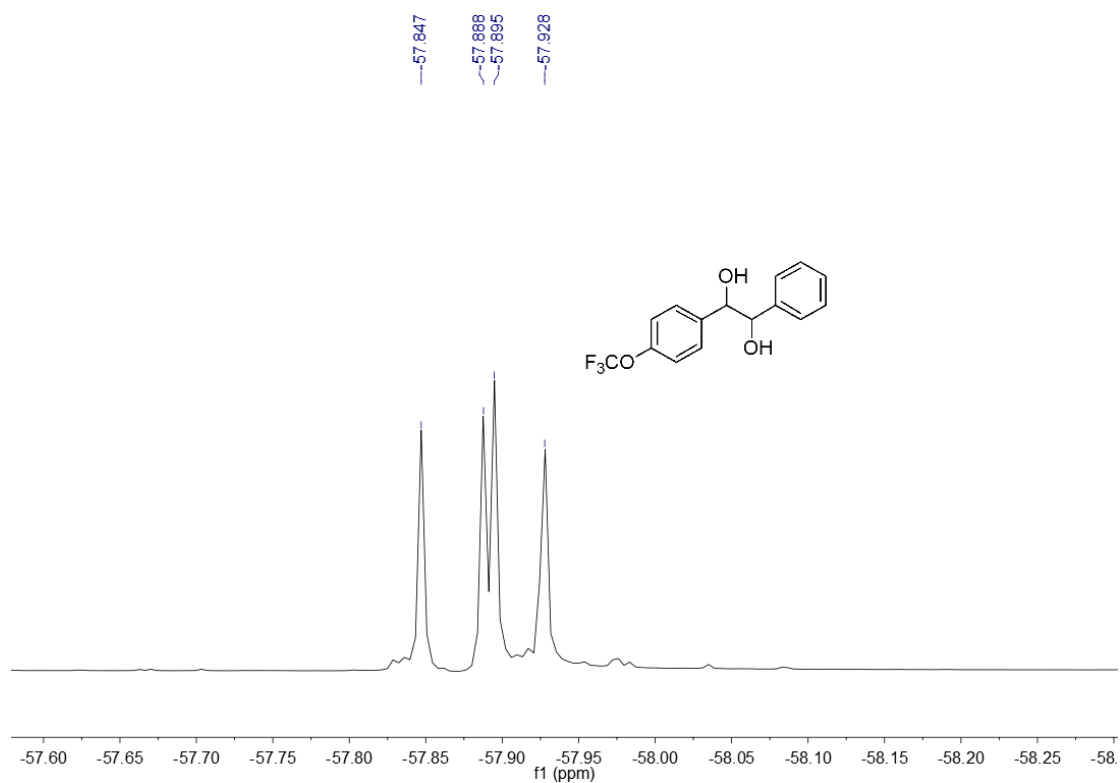
^1H NMR spectrum of compound **4g** (400 MHz) in CDCl_3



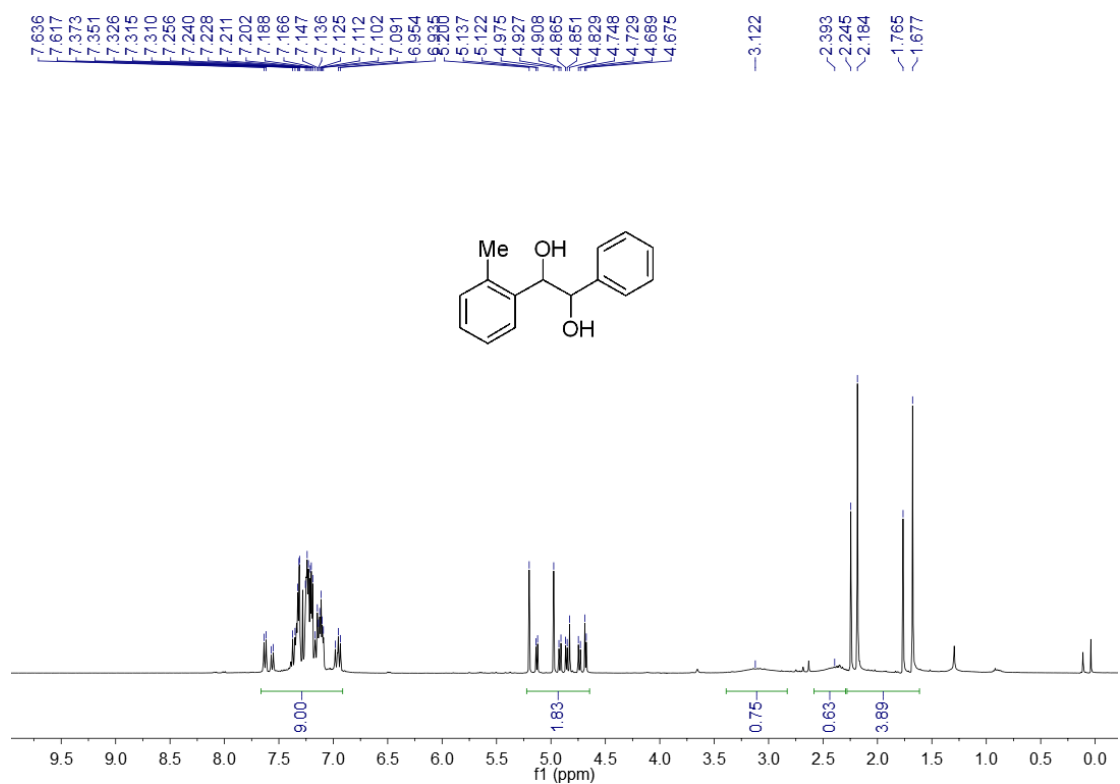
^{13}C NMR spectrum of compound **4g** (100 MHz) in CDCl_3



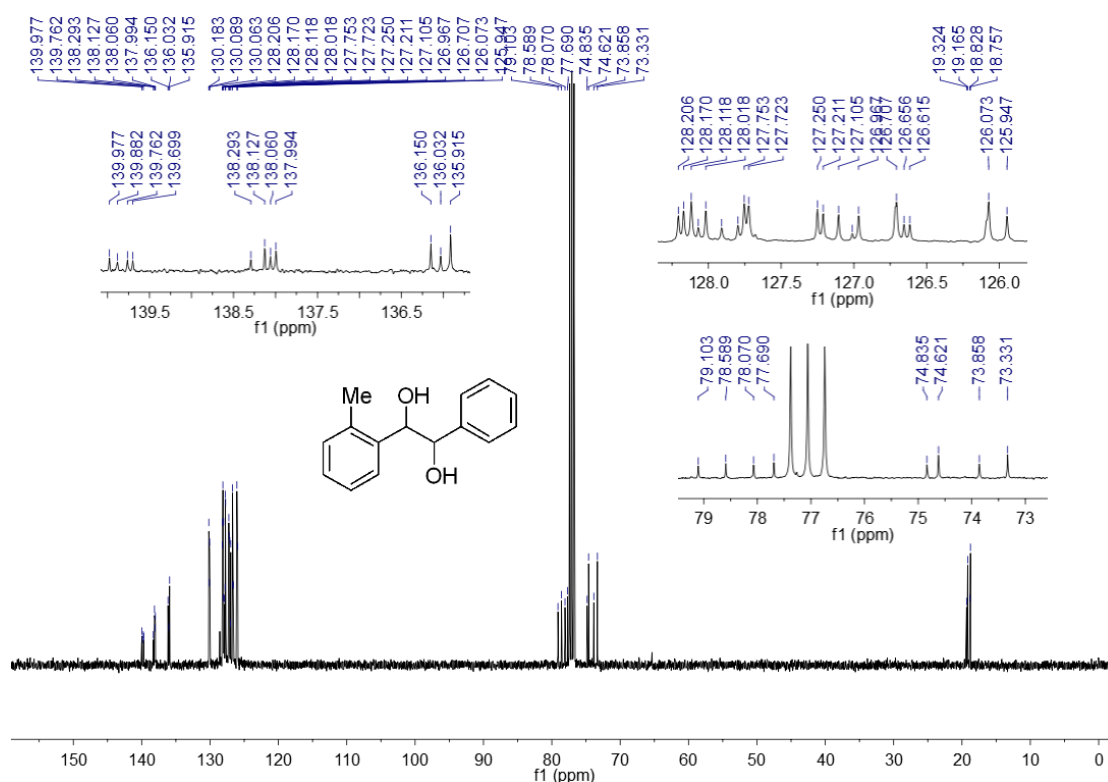
^{19}F NMR spectrum of compound **4g** (376 MHz) in CDCl_3



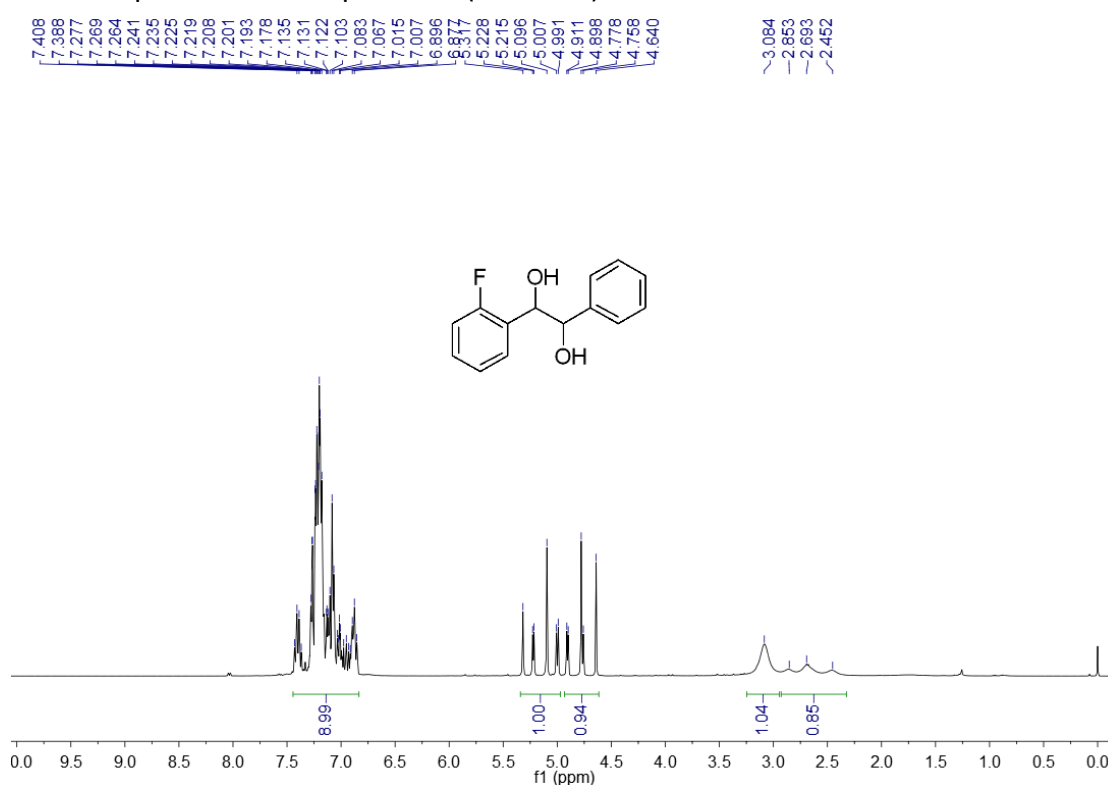
^1H NMR spectrum of compound **4h** (400 MHz) in CDCl_3



^{13}C NMR spectrum of compound **4h** (100 MHz) in CDCl_3



^1H NMR spectrum of compound **4i** (400 MHz) in CDCl_3



Chemical structure: O[C@H](c1ccccc1)[C@H](O)c2ccccc2F

¹³C NMR spectrum (ppm):

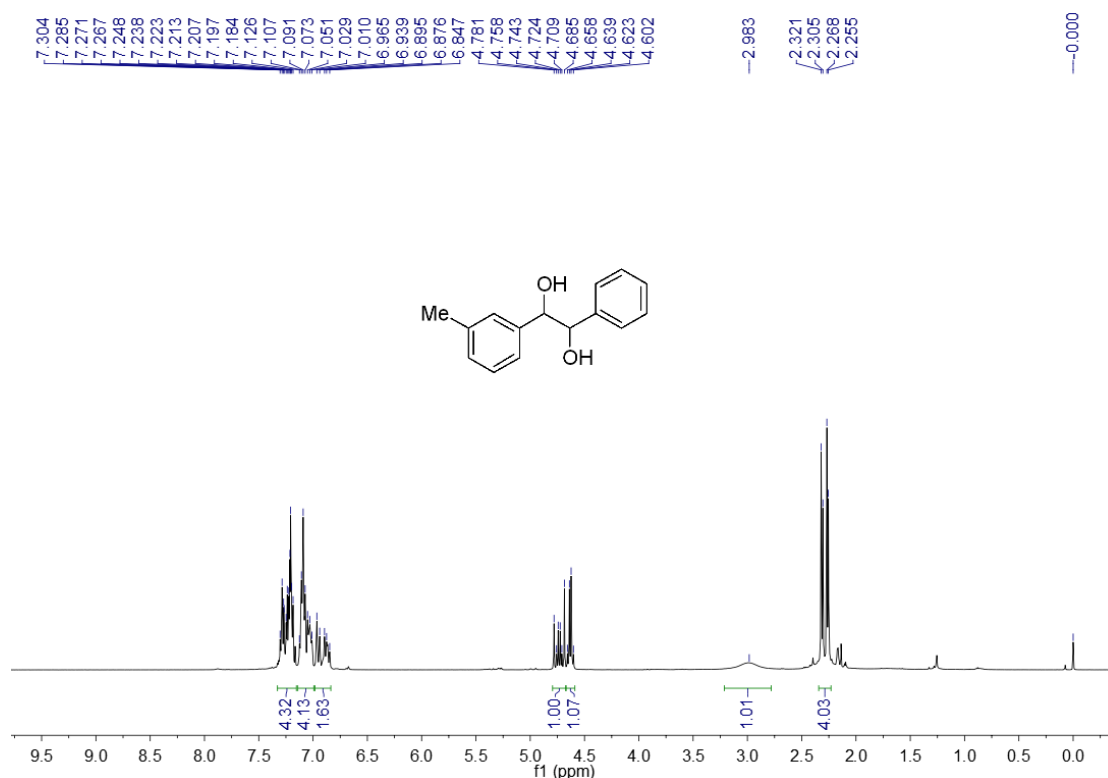
- 161.305, 161.267, 161.200, 161.176, 158.863, 158.824, 158.750, 158.728, 139.828, 139.752, 139.699, 139.272, 128.222, 128.204, 128.137, 128.096, 128.057, 128.057, 128.057, 127.991, 127.935, 127.920, 127.121, 127.073, 126.980, 126.626, 124.162, 124.126, 124.108, 124.022, 123.990, 115.325, 115.281, 115.107, 115.054, 79.110, 78.063, 77.762, 76.916, 72.879, 71.870, 71.463, 70.455

Chemical structure: O[C@H](c1ccccc1)[C@H](O)c2ccccc2F

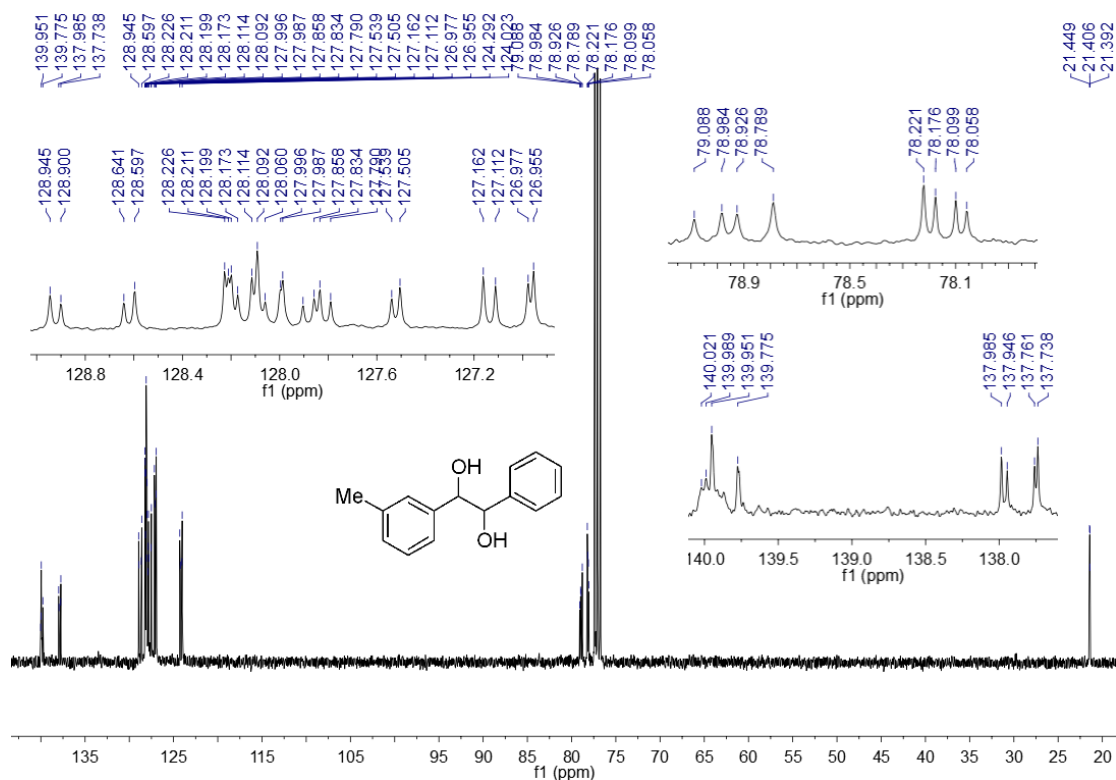
¹H NMR spectrum (400 MHz, DMSO-d₆) showing peaks at the following chemical shifts (ppm):

- 7.18105
- 7.18156
- 7.18459
- 7.18605

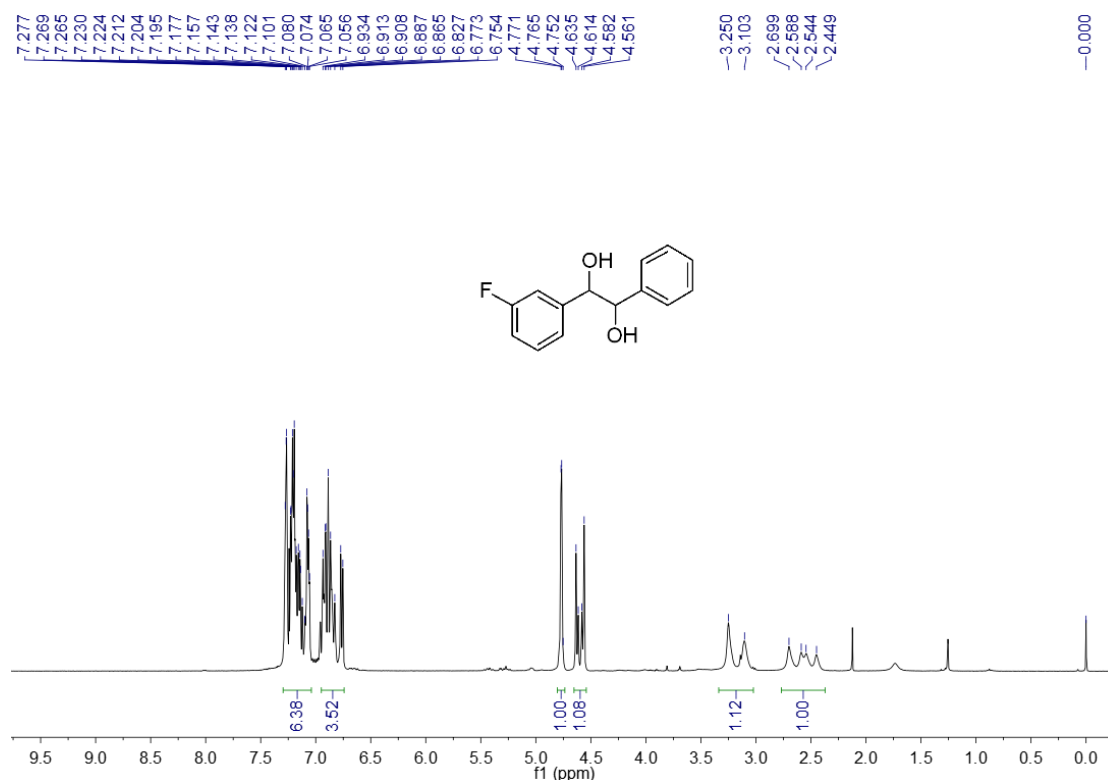
^1H NMR spectrum of compound **4j** (400 MHz) in CDCl_3



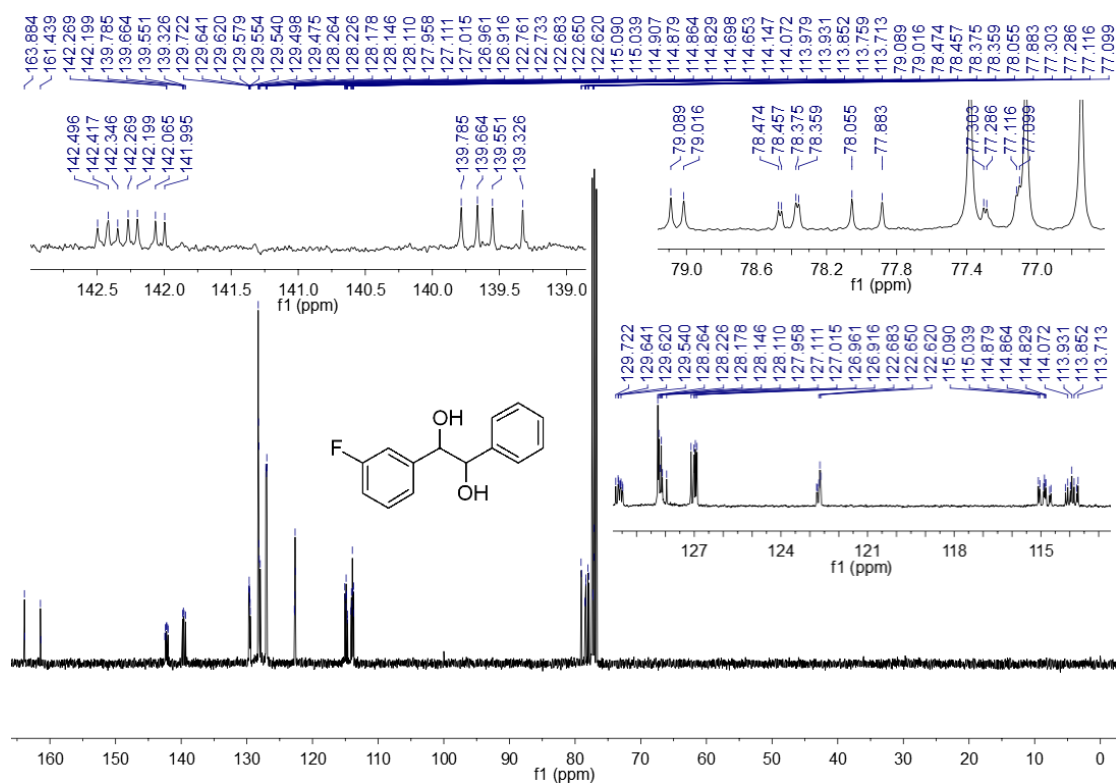
^{13}C NMR spectrum of compound **4j** (100 MHz) in CDCl_3



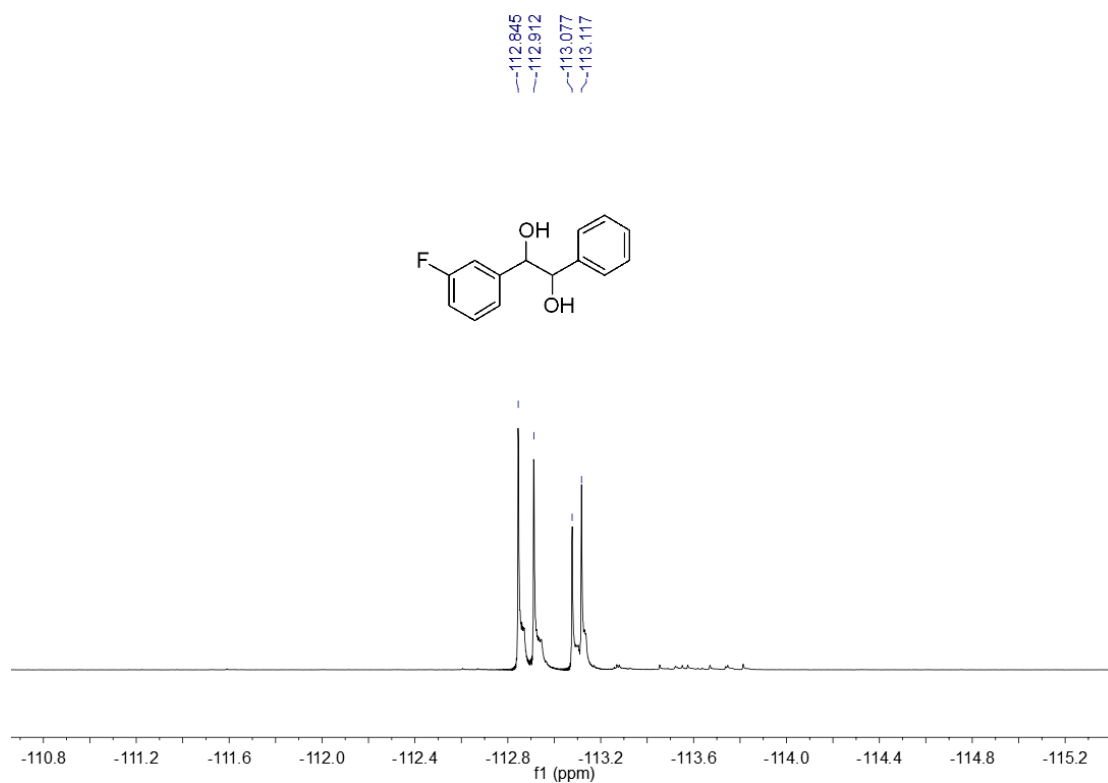
^1H NMR spectrum of compound **4k** (400 MHz) in CDCl_3



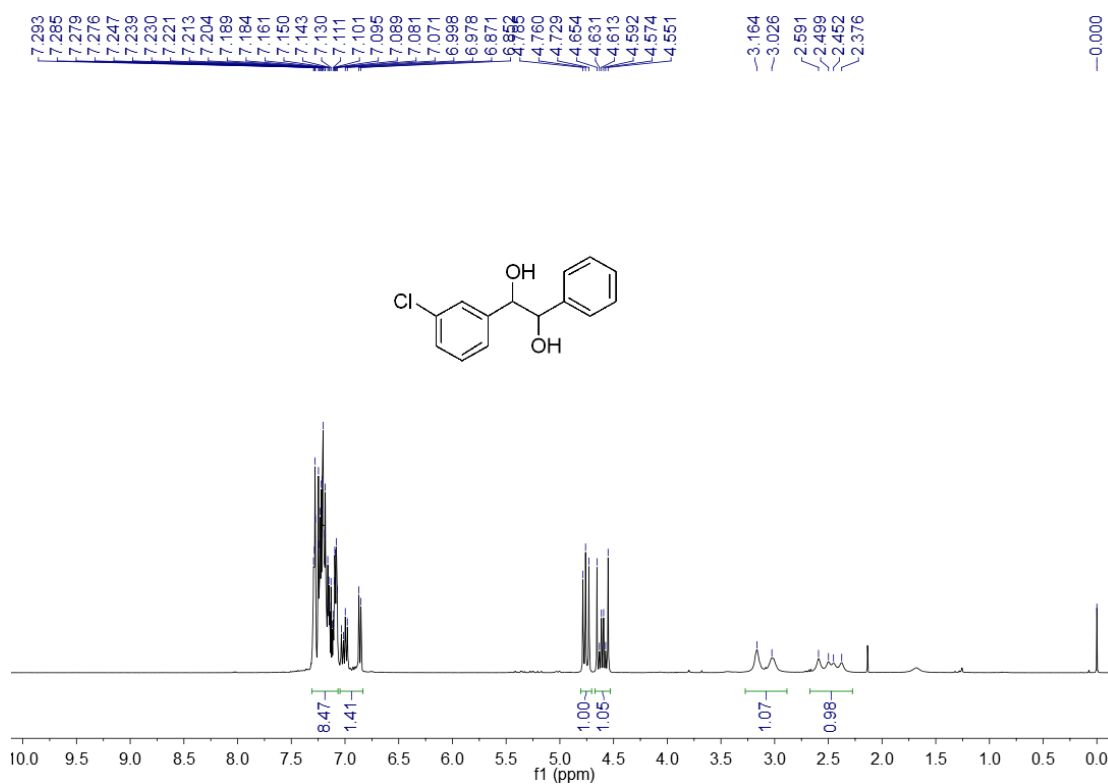
^{13}C NMR spectrum of compound **4k** (100 MHz) in CDCl_3



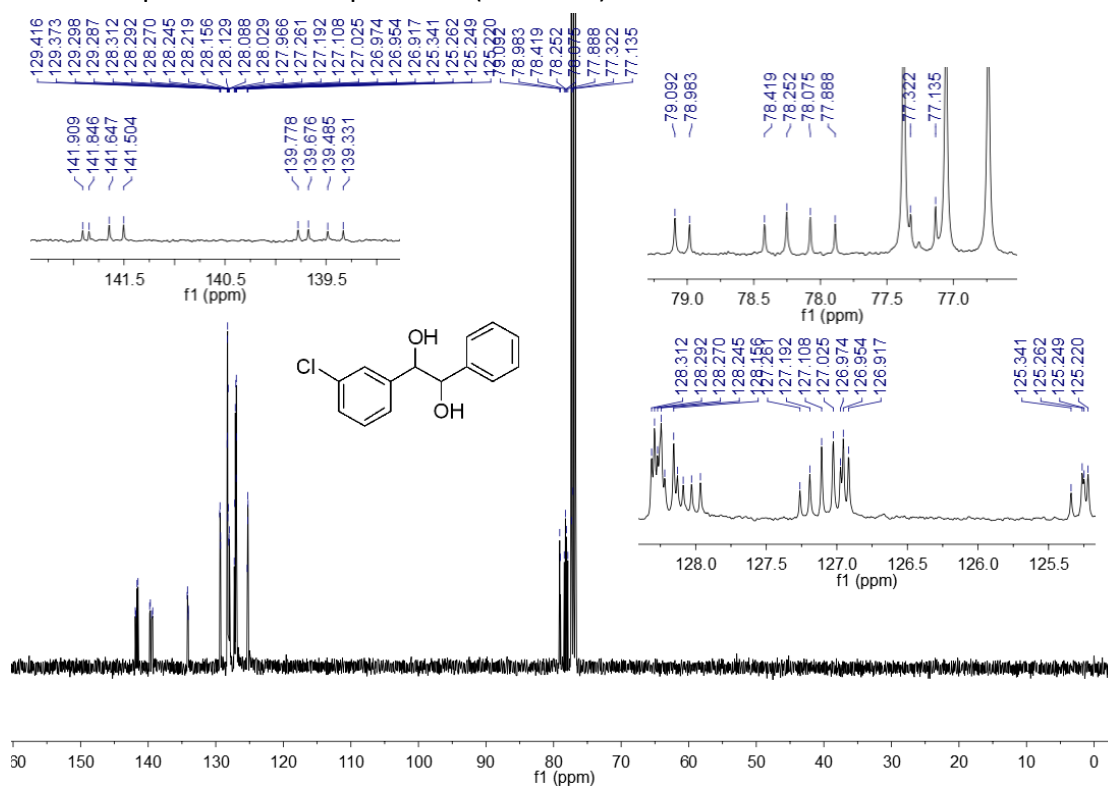
^{19}F NMR spectrum of compound **4k** (376 MHz) in CDCl_3



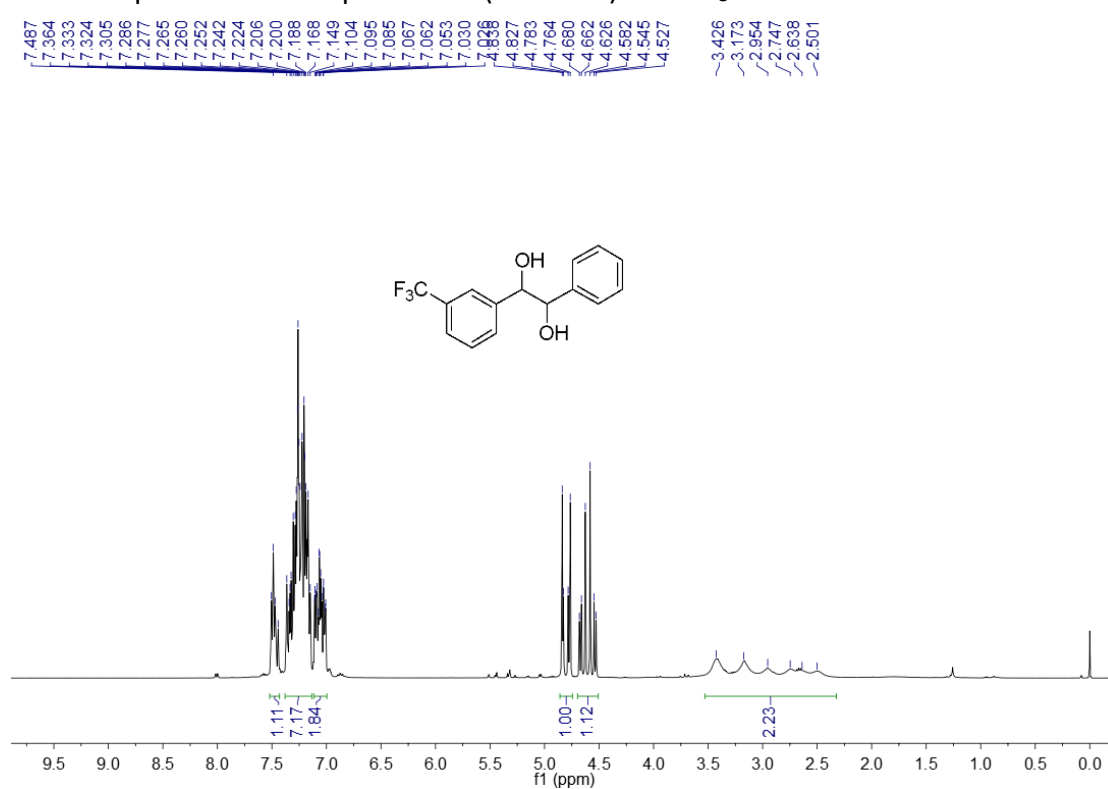
^1H NMR spectrum of compound **4l** (400 MHz) in CDCl_3



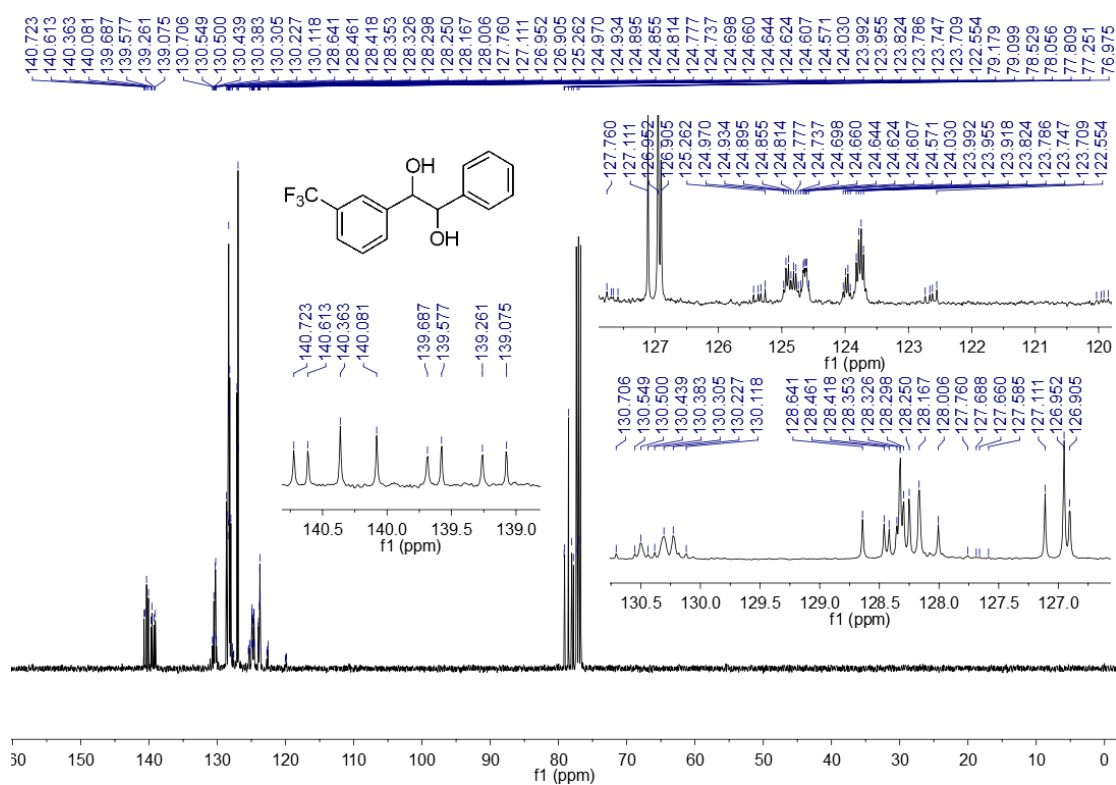
^{13}C NMR spectrum of compound **4l** (100 MHz) in CDCl_3



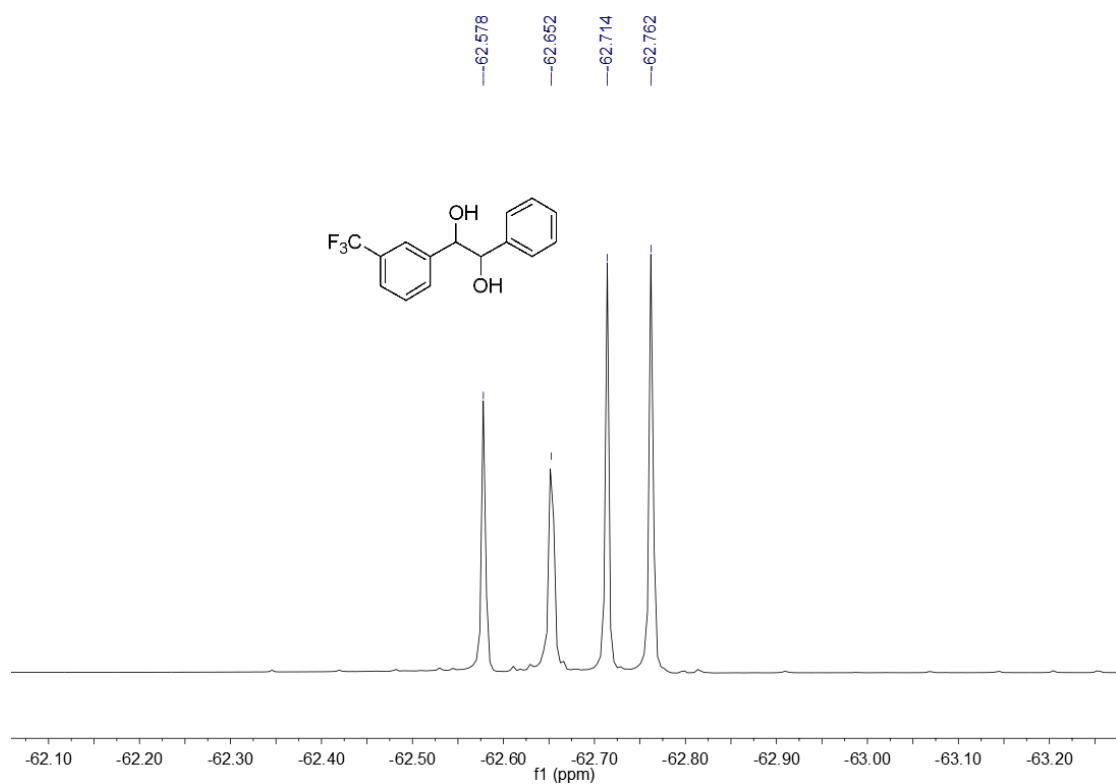
^1H NMR spectrum of compound **4m** (400 MHz) in CDCl_3



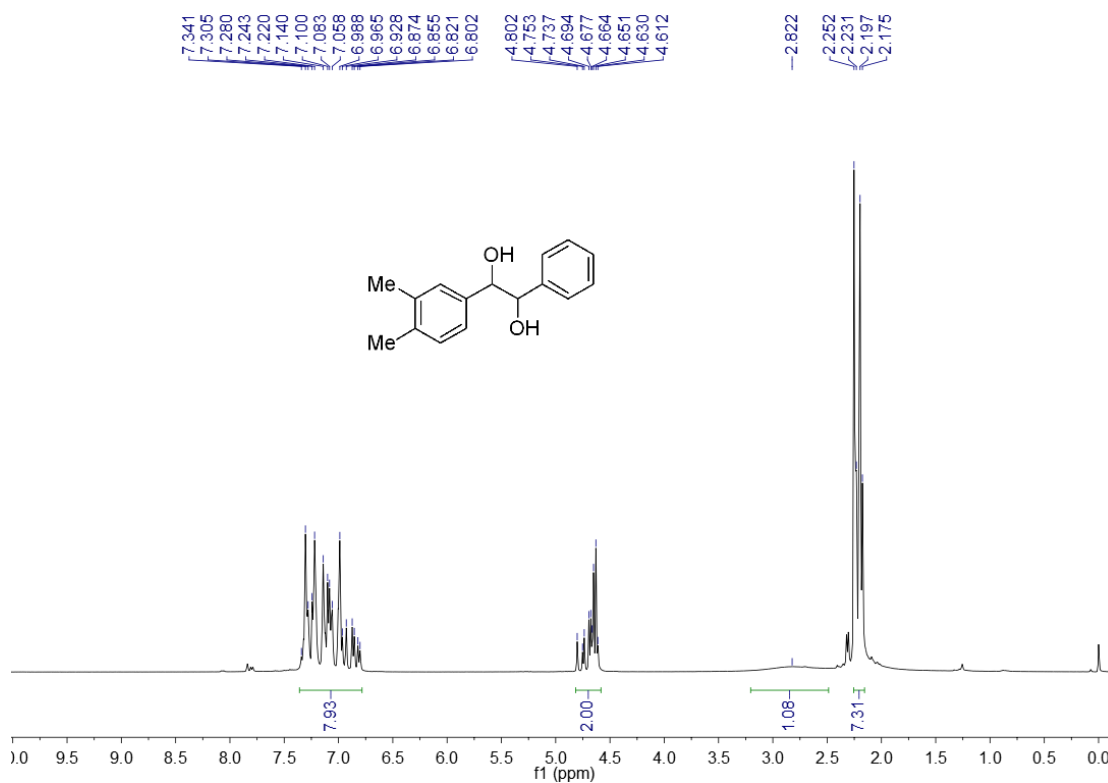
^{13}C NMR spectrum of compound **4m** (100 MHz) in CDCl_3



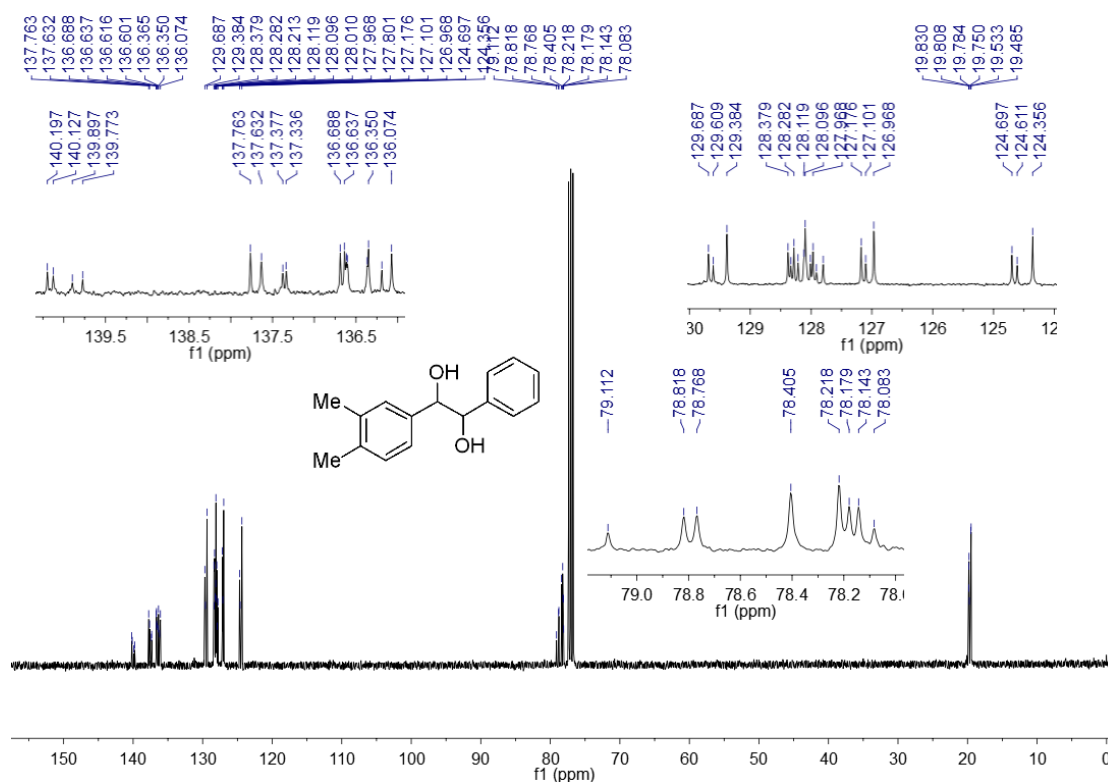
^{19}F NMR spectrum of compound **4m** (376 MHz) in CDCl_3



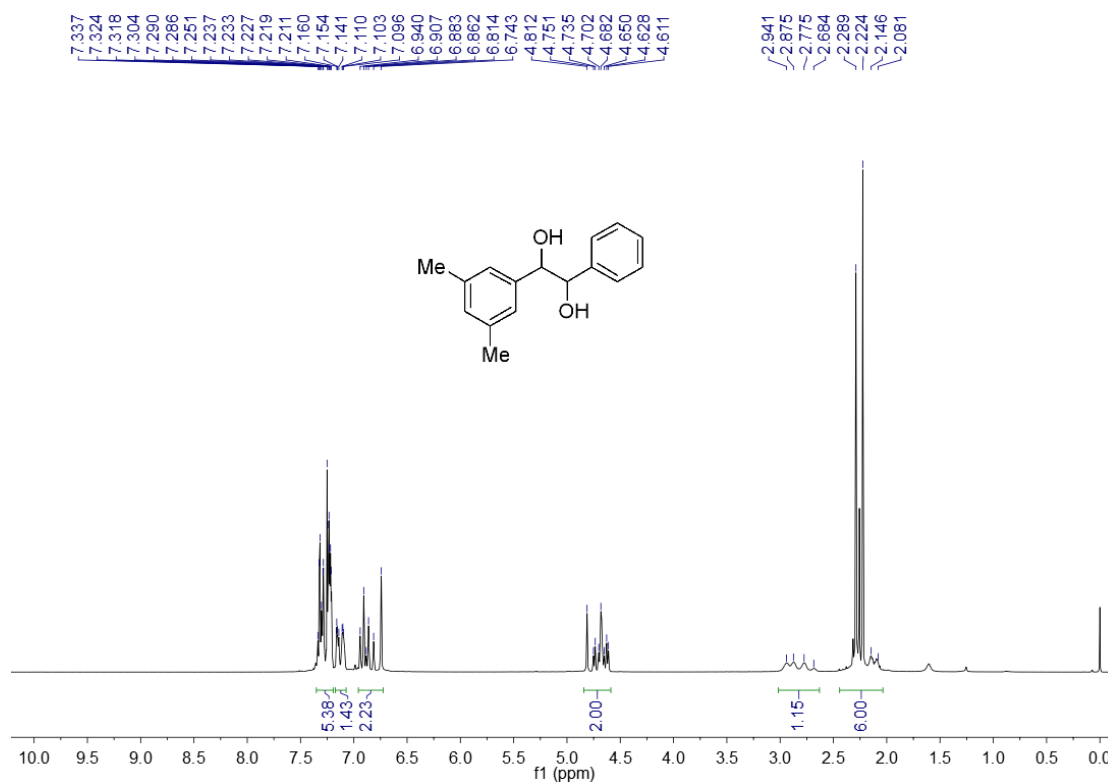
^1H NMR spectrum of compound **4n** (400 MHz) in CDCl_3



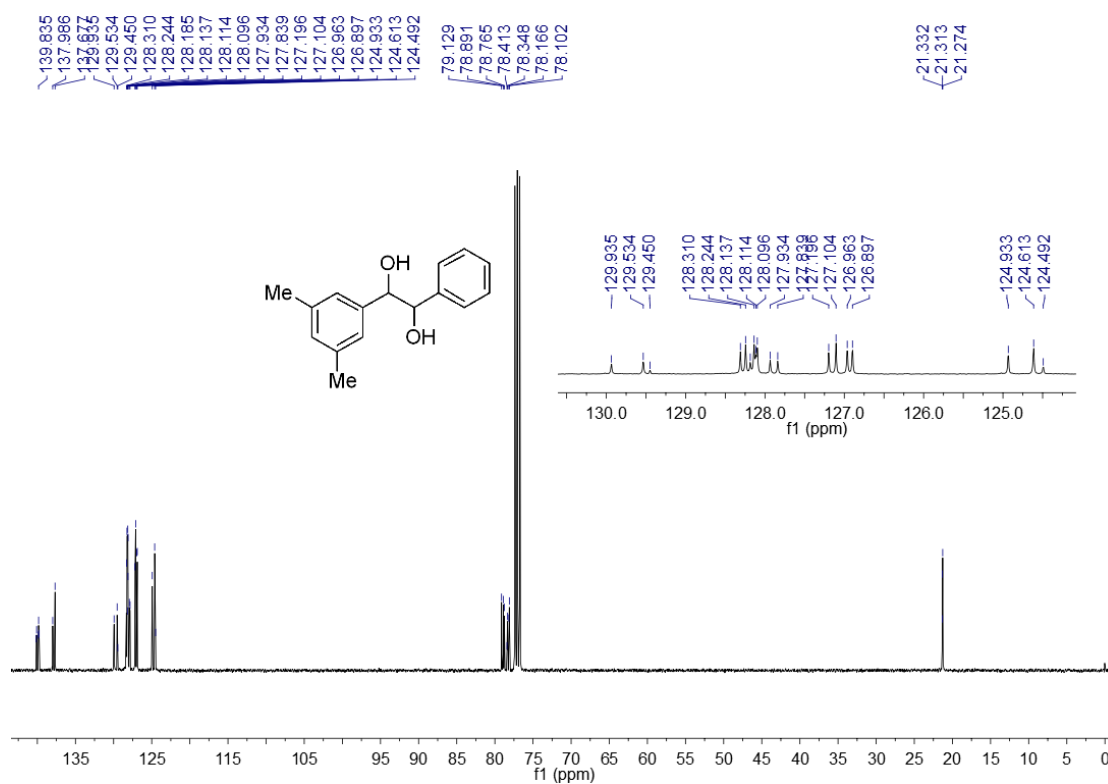
^{13}C NMR spectrum of compound **4n** (100 MHz) in CDCl_3



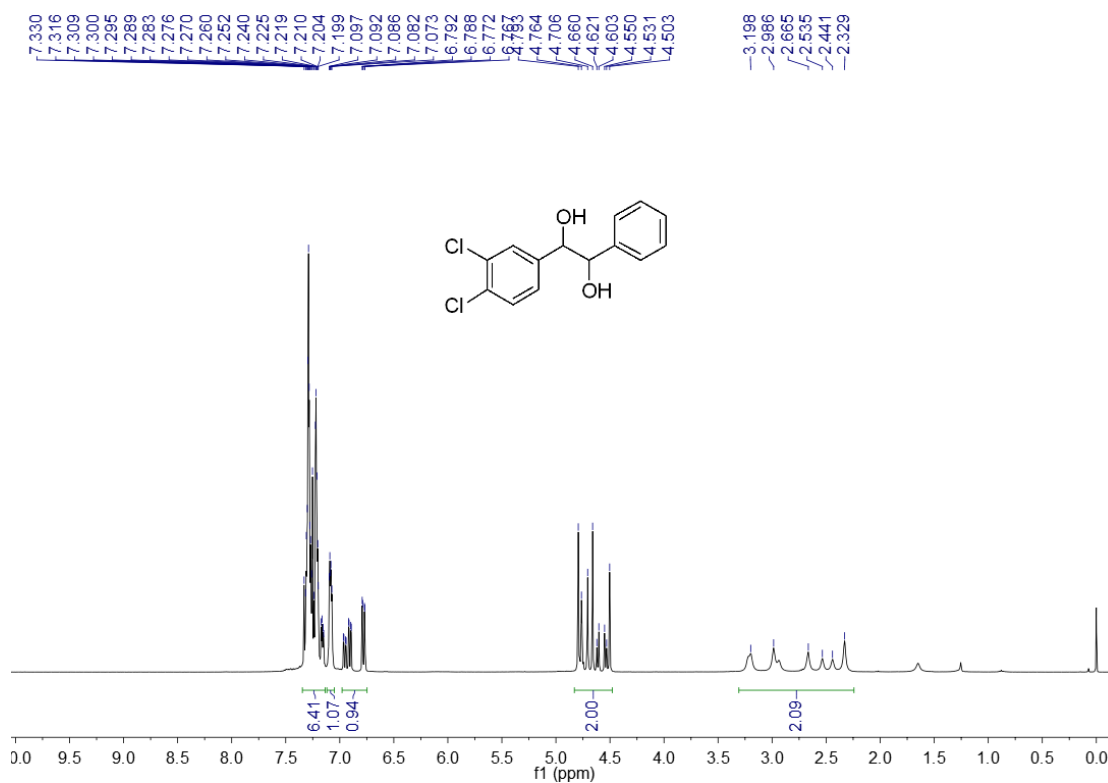
^1H NMR spectrum of compound **4o** (400 MHz) in CDCl_3



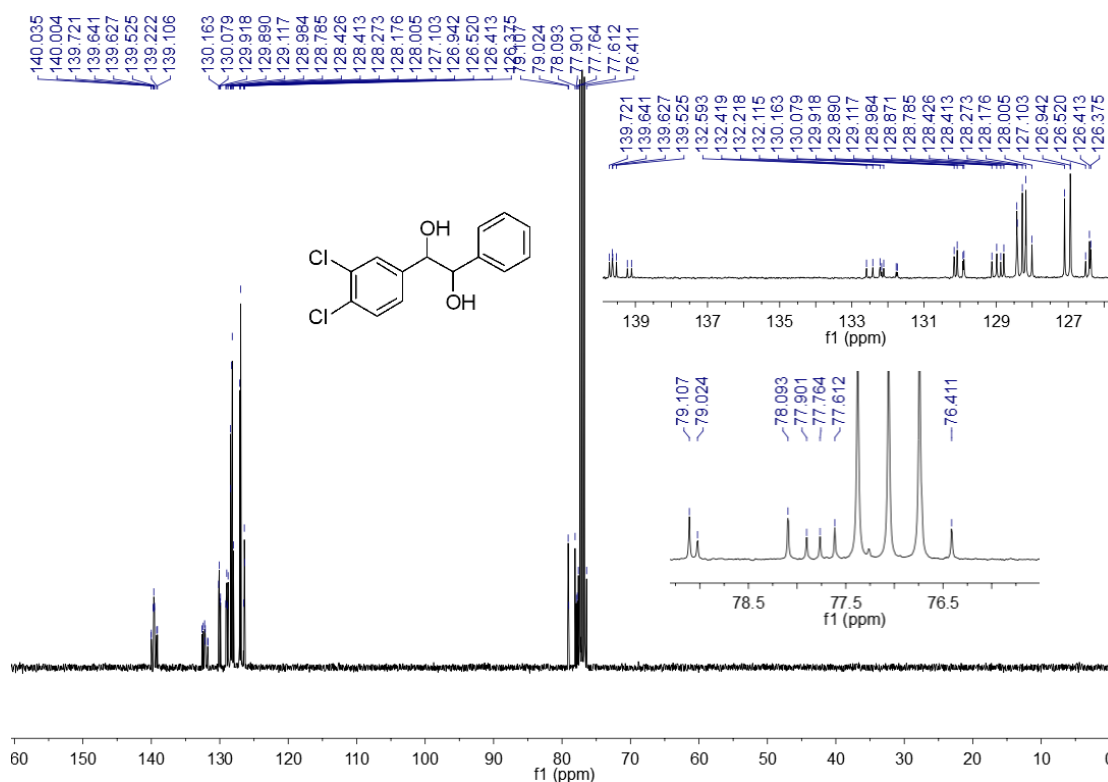
^{13}C NMR spectrum of compound **4o** (100 MHz) in CDCl_3



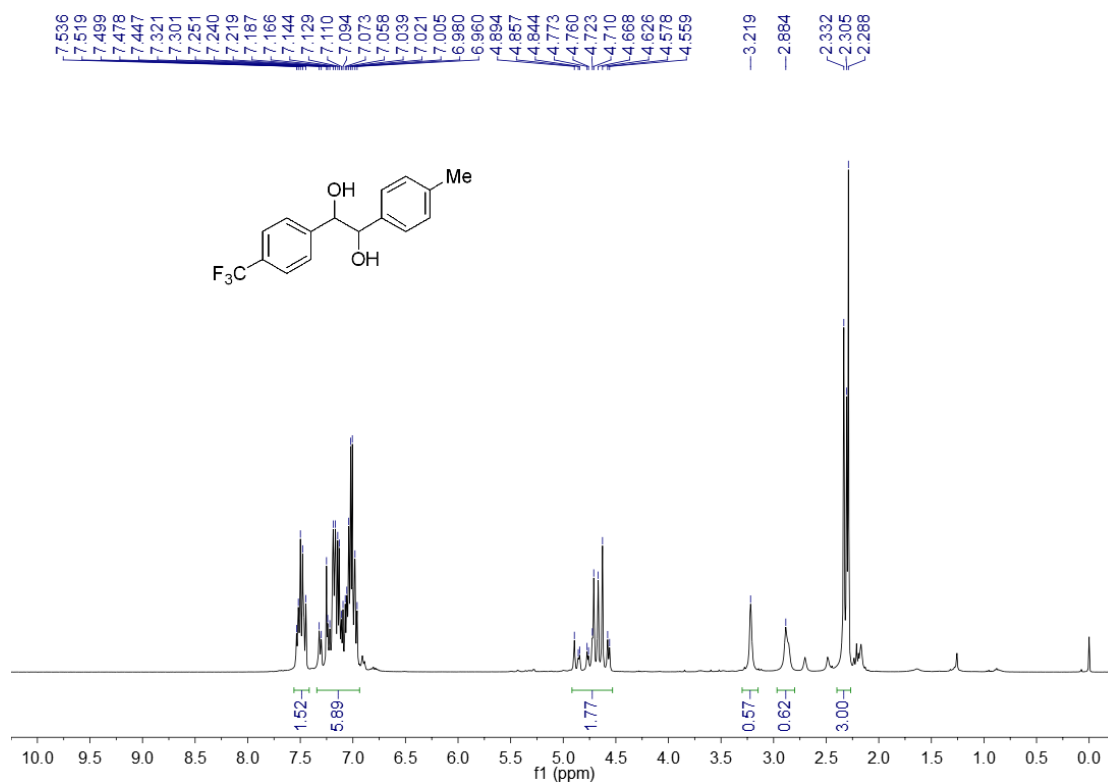
^1H NMR spectrum of compound **4p** (400 MHz) in CDCl_3



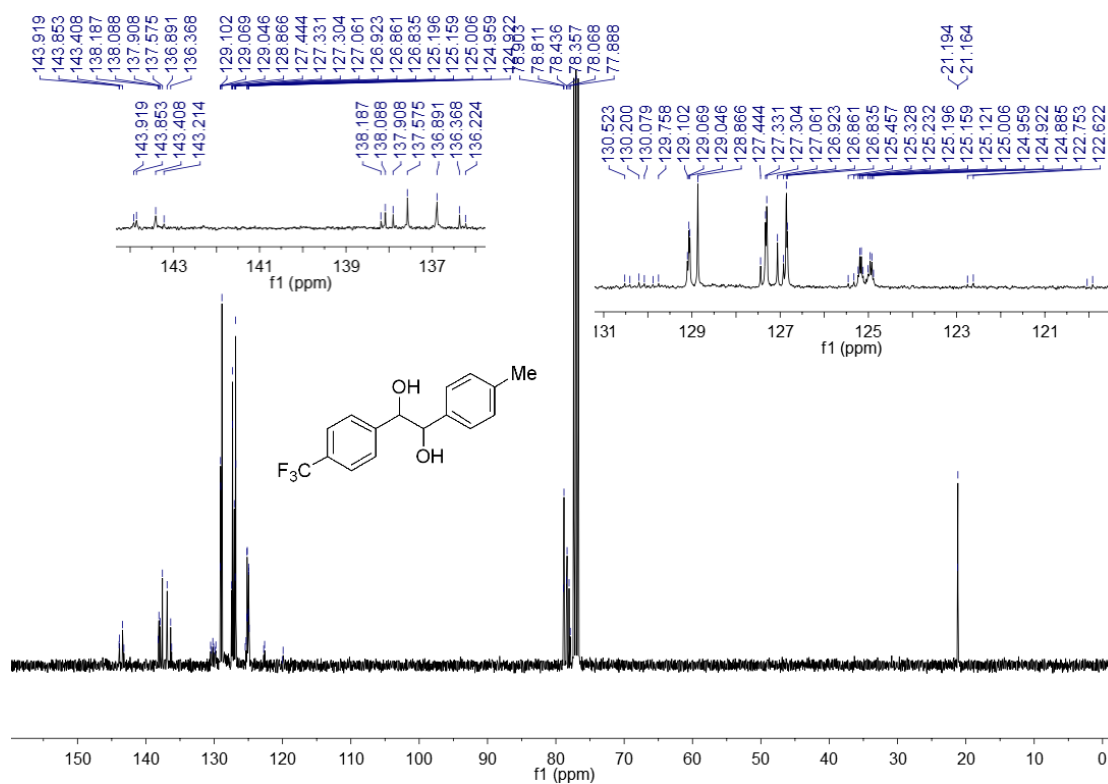
^{13}C NMR spectrum of compound **4p** (100 MHz) in CDCl_3



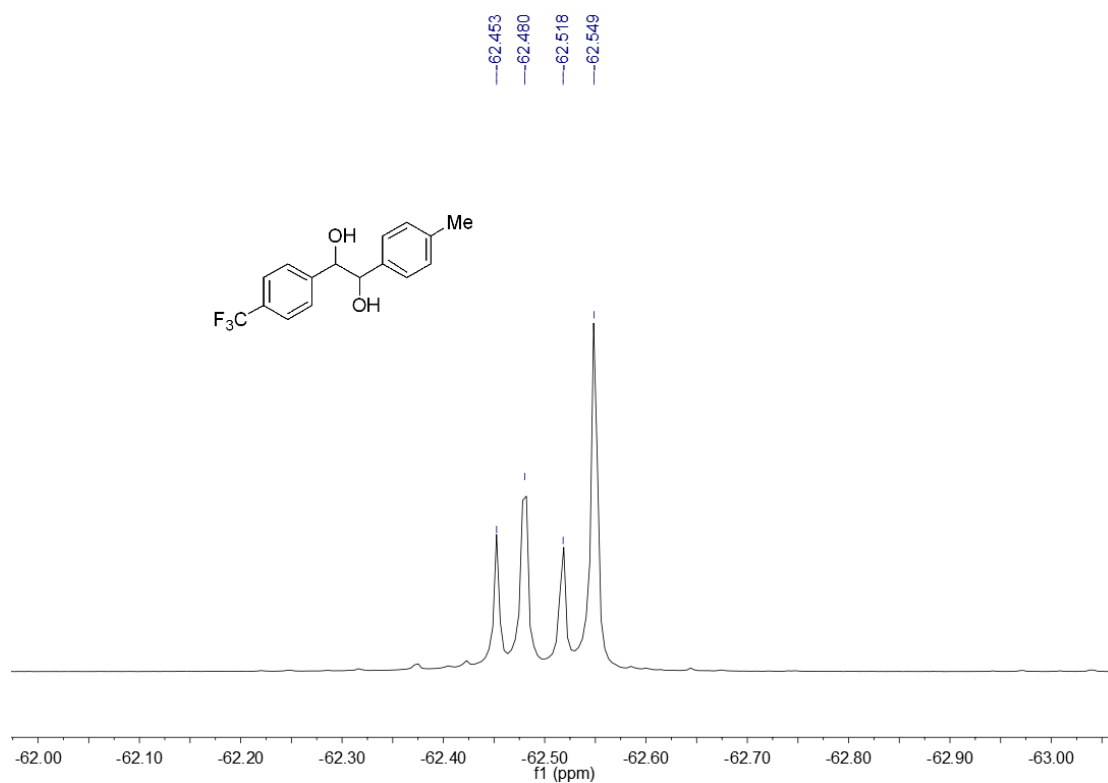
^1H NMR spectrum of compound **5a** (400 MHz) in CDCl_3



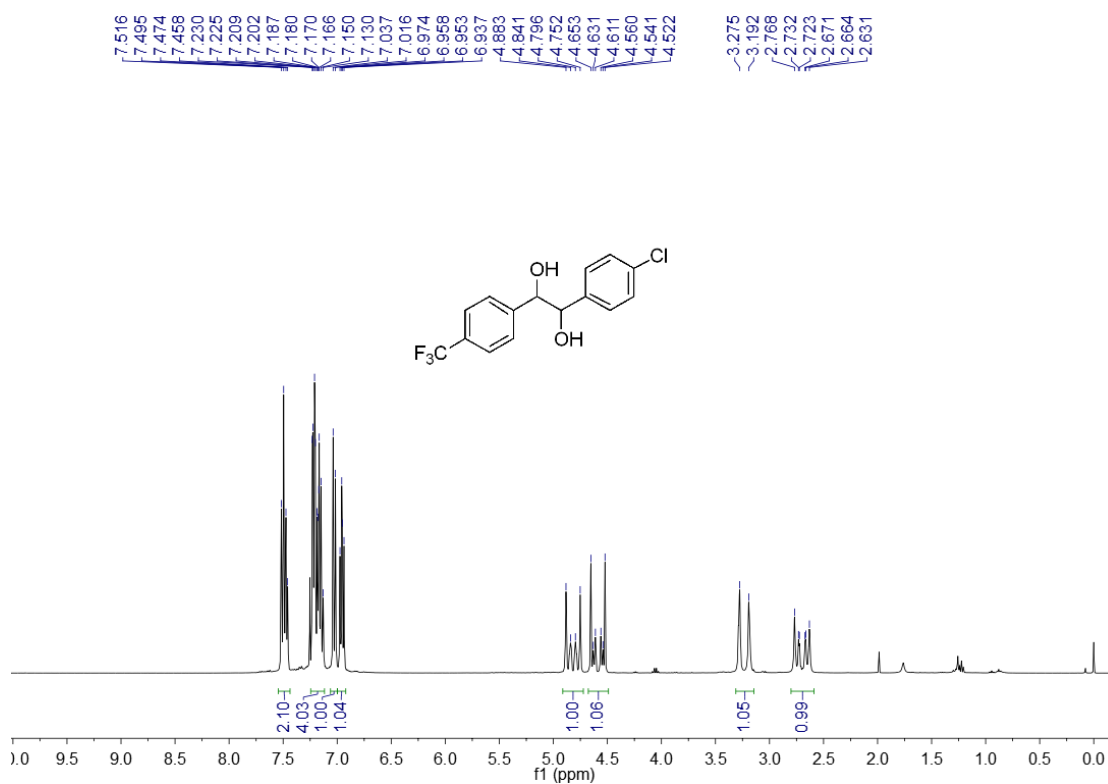
^{13}C NMR spectrum of compound **5a** (100 MHz) in CDCl_3



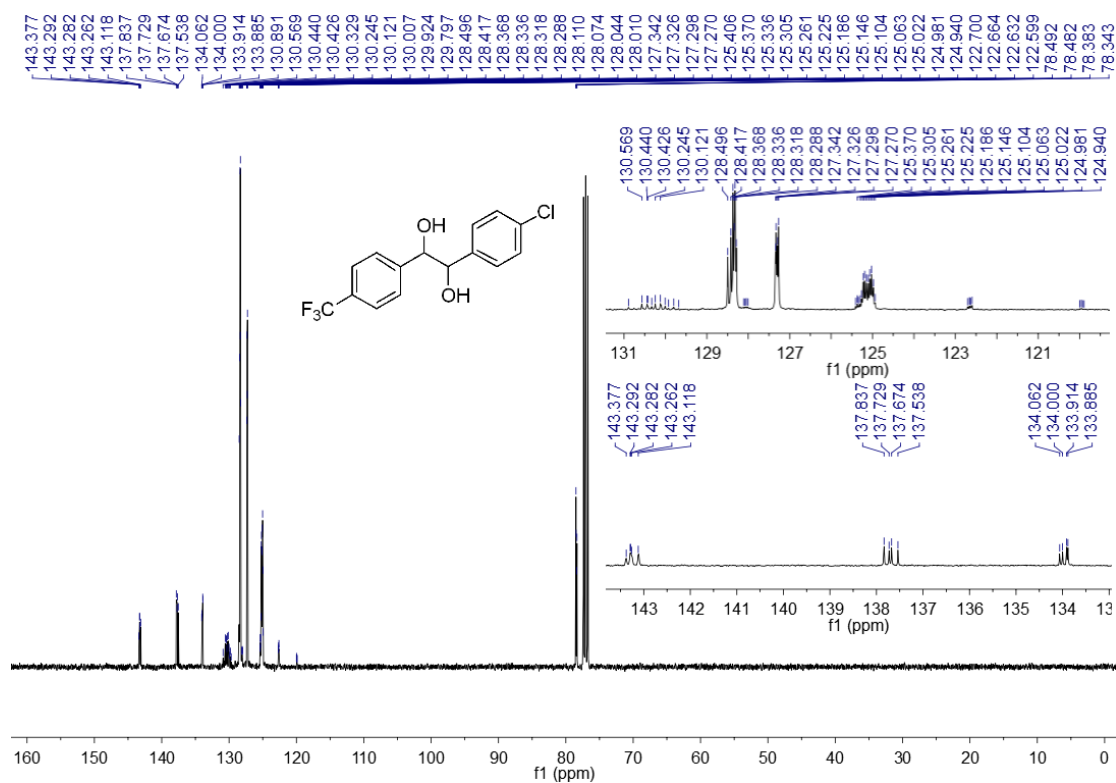
^{19}F NMR spectrum of compound **5a** (376 MHz) in CDCl_3



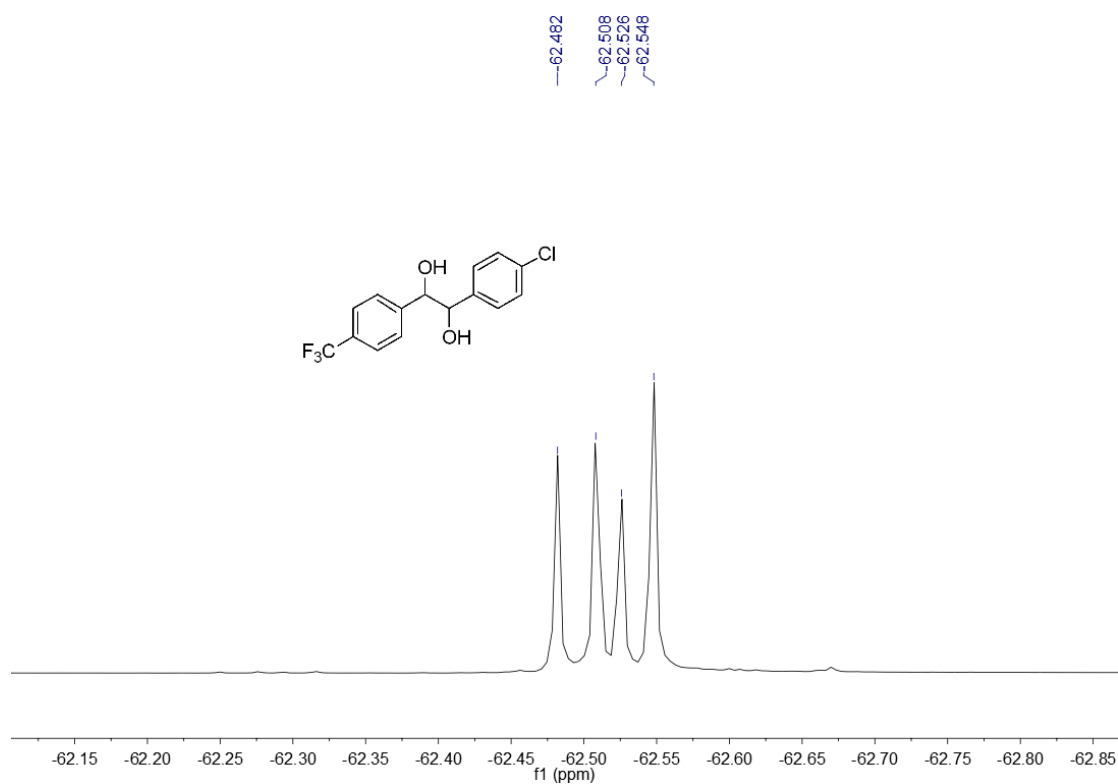
^1H NMR spectrum of compound **5b** (400 MHz) in CDCl_3



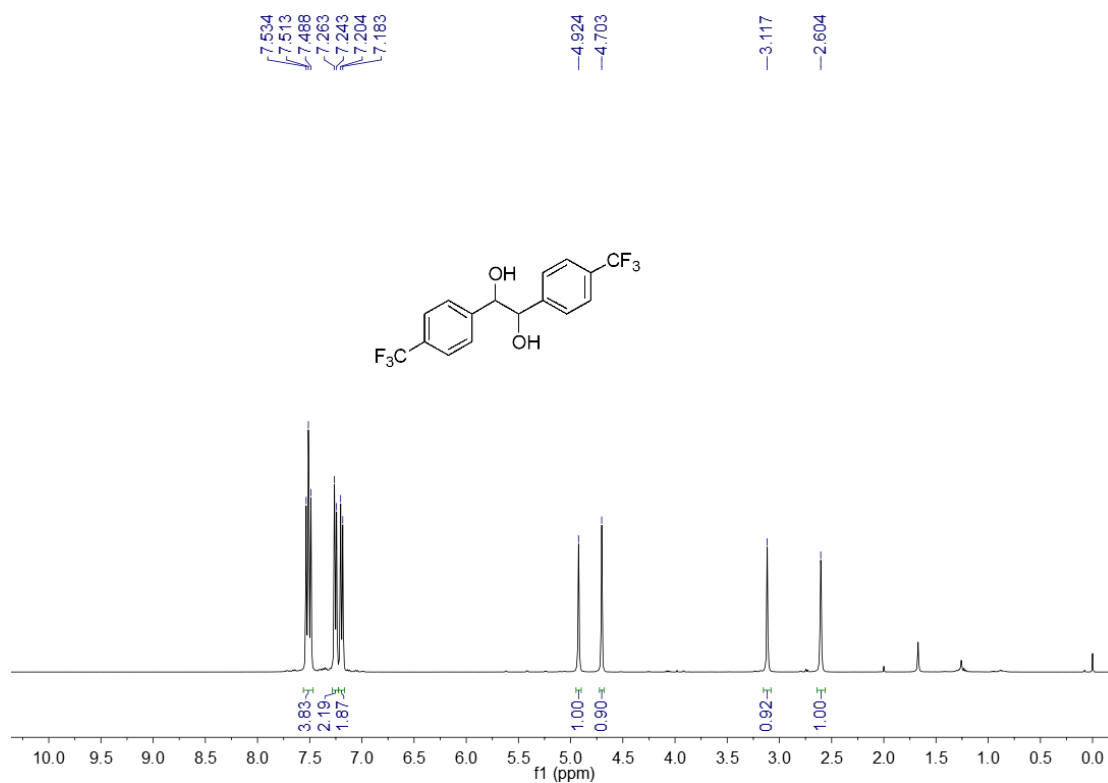
^{13}C NMR spectrum of compound **5b** (100 MHz) in CDCl_3



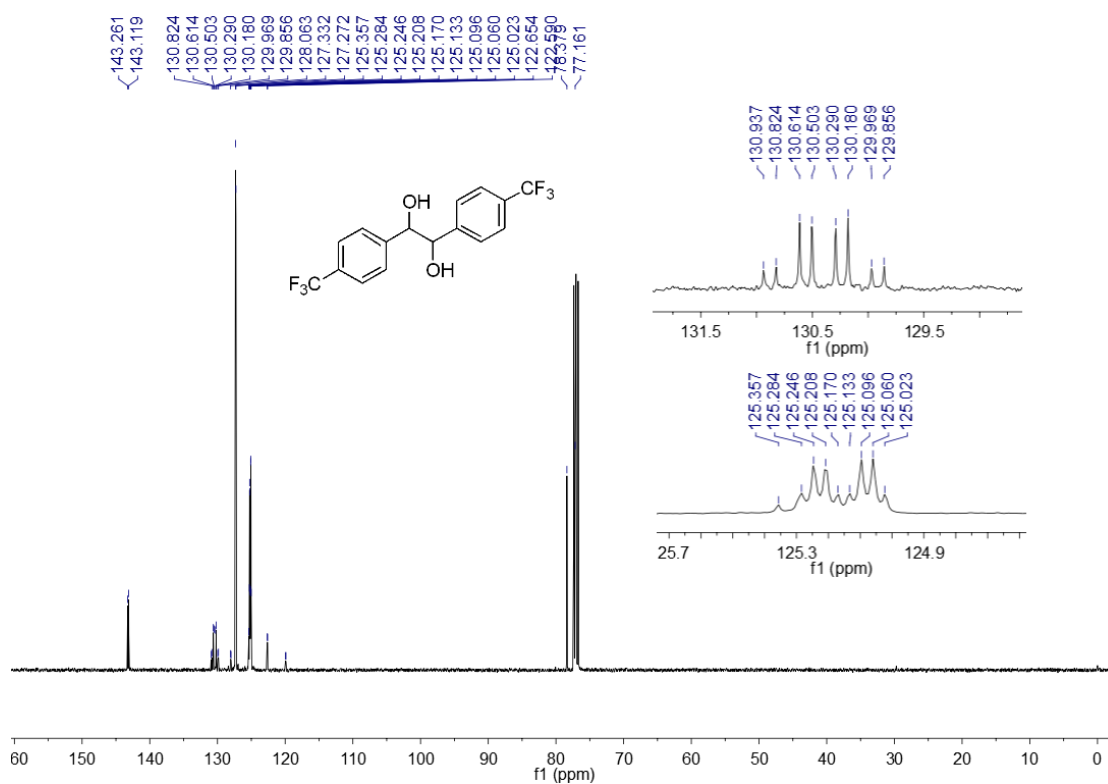
^{19}F NMR spectrum of compound **5b** (376 MHz) in CDCl_3



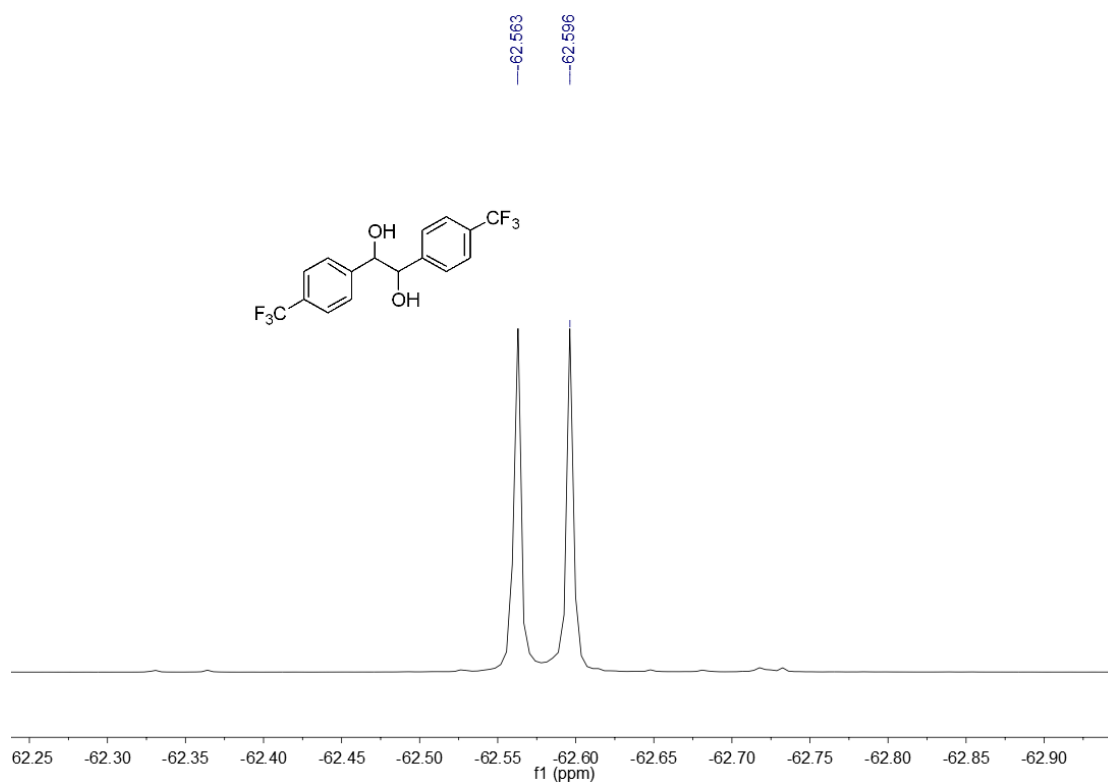
^1H NMR spectrum of compound **5c** (400 MHz) in CDCl_3



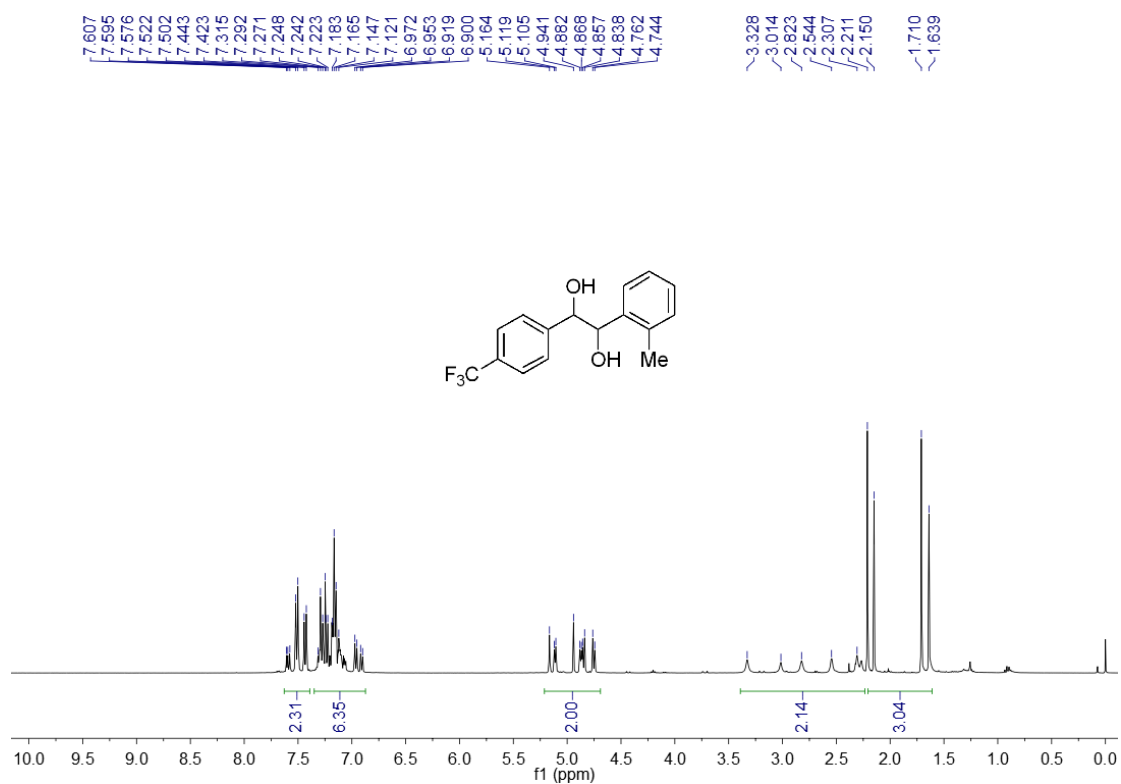
^{13}C NMR spectrum of compound **5c** (100 MHz) in CDCl_3



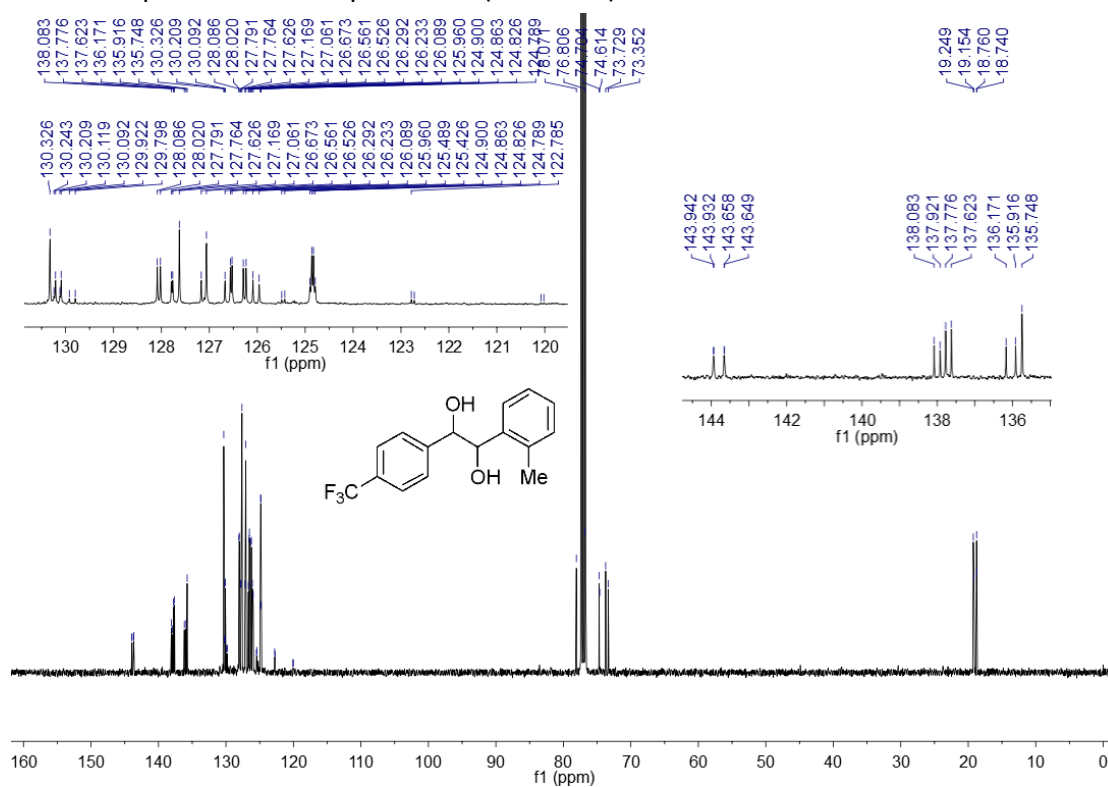
^{19}F NMR spectrum of compound **5c** (376 MHz) in CDCl_3



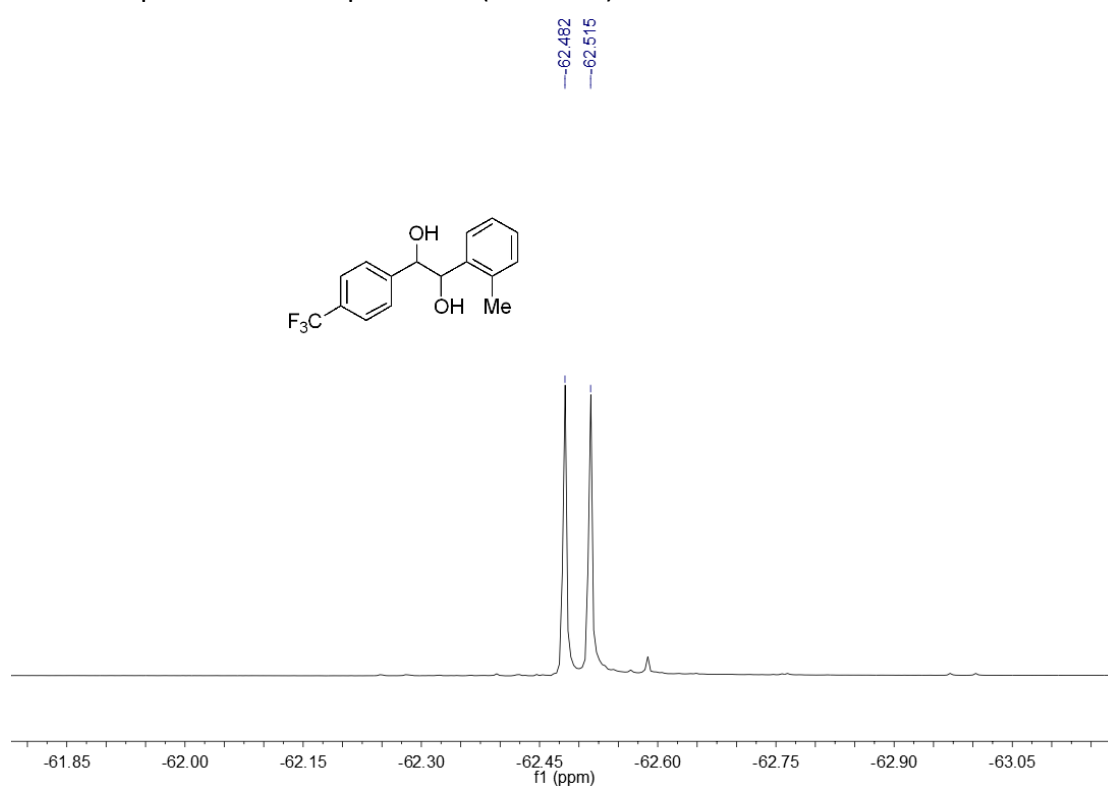
^1H NMR spectrum of compound **5d** (400 MHz) in CDCl_3



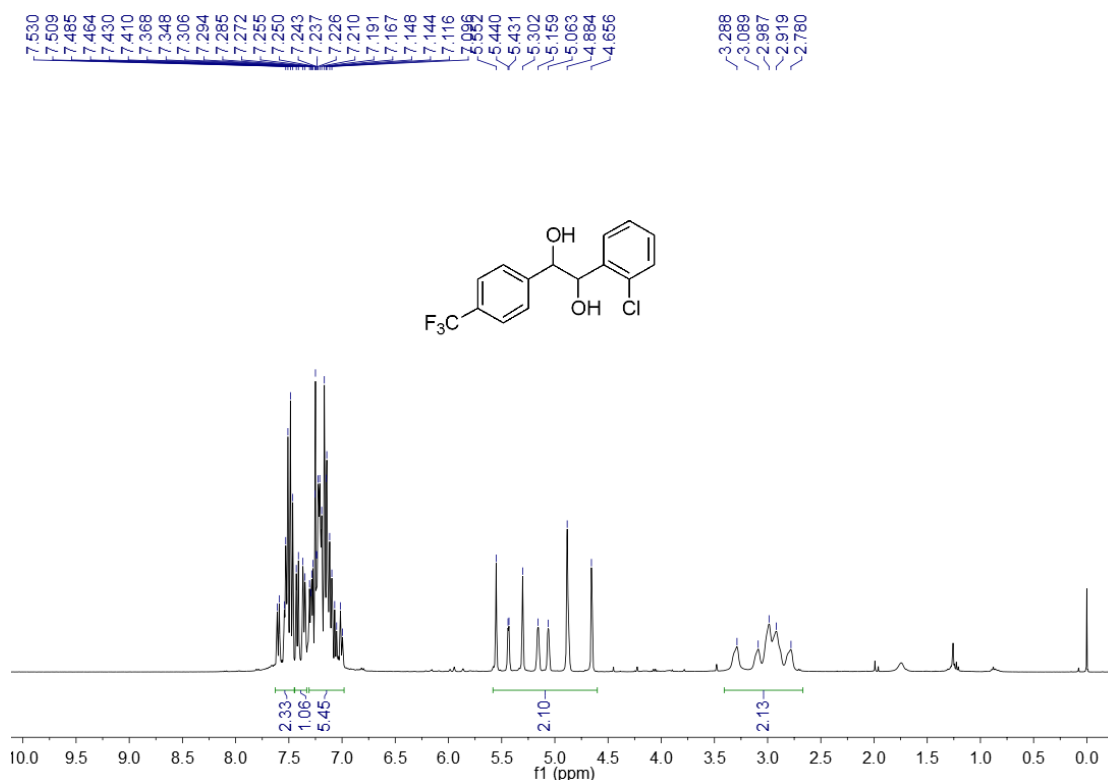
^{13}C NMR spectrum of compound **5d** (100 MHz) in CDCl_3



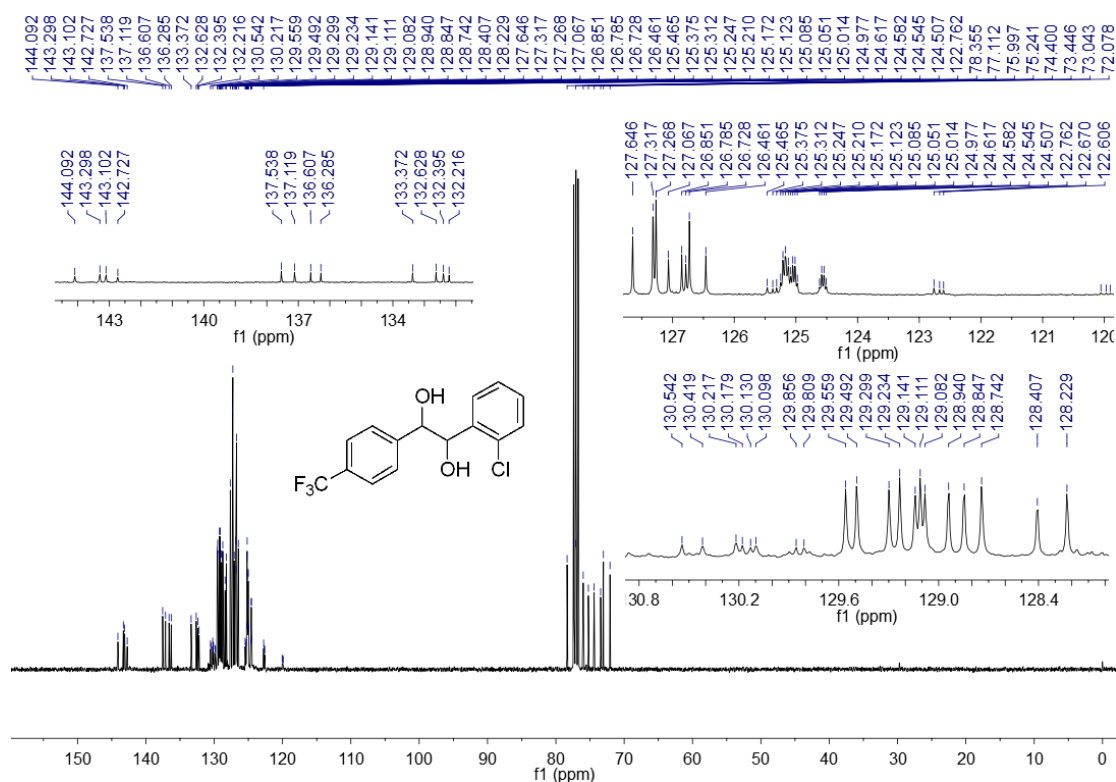
^{19}F NMR spectrum of compound **5d** (376 MHz) in CDCl_3



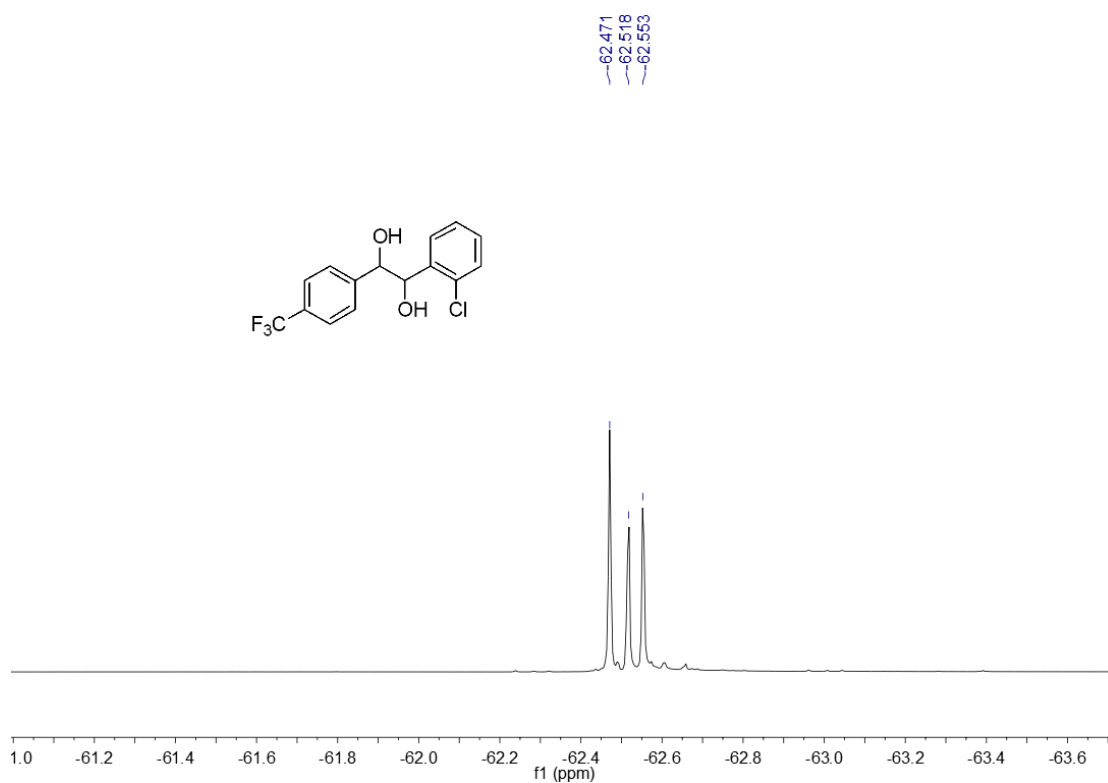
^1H NMR spectrum of compound **5e** (400 MHz) in CDCl_3



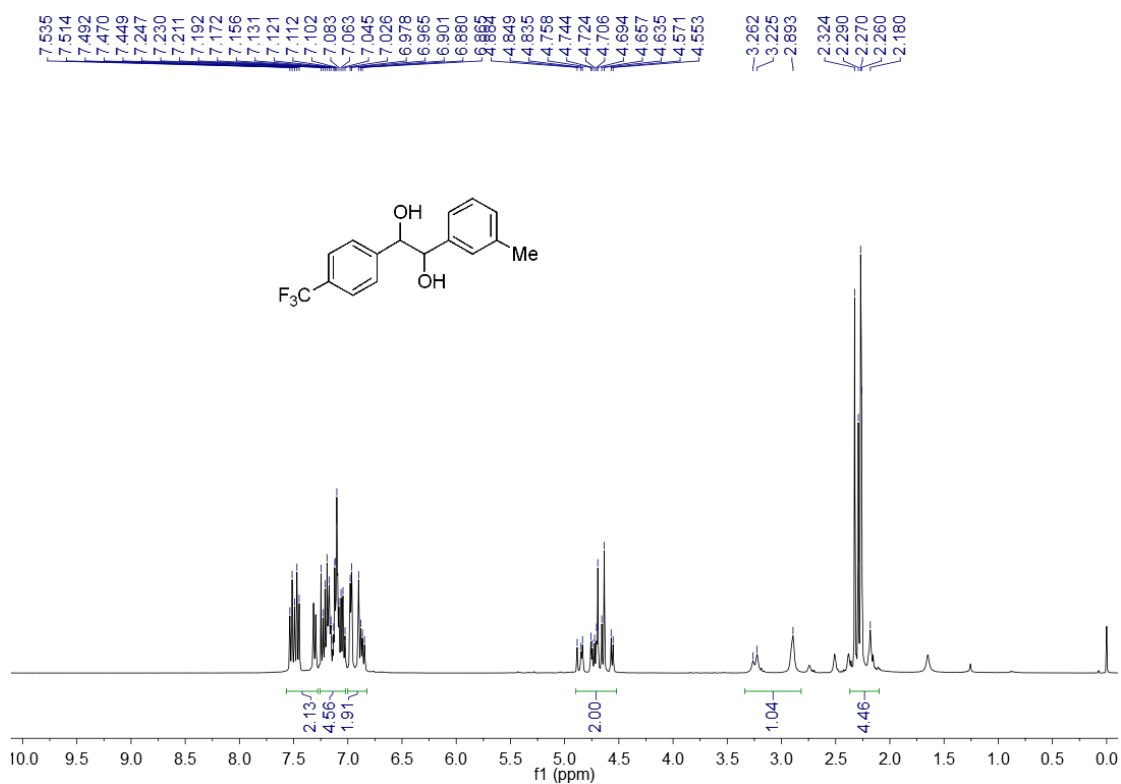
^{13}C NMR spectrum of compound **5e** (100 MHz) in CDCl_3



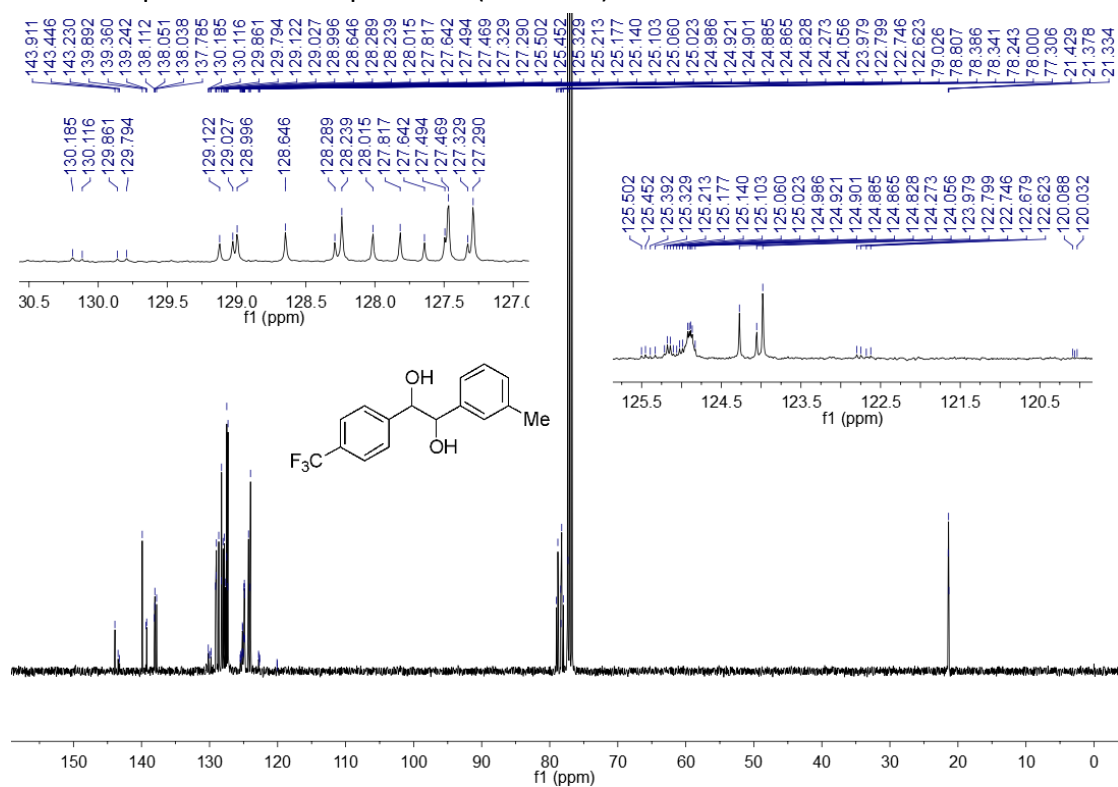
^{19}F NMR spectrum of compound **5e** (376 MHz) in CDCl_3



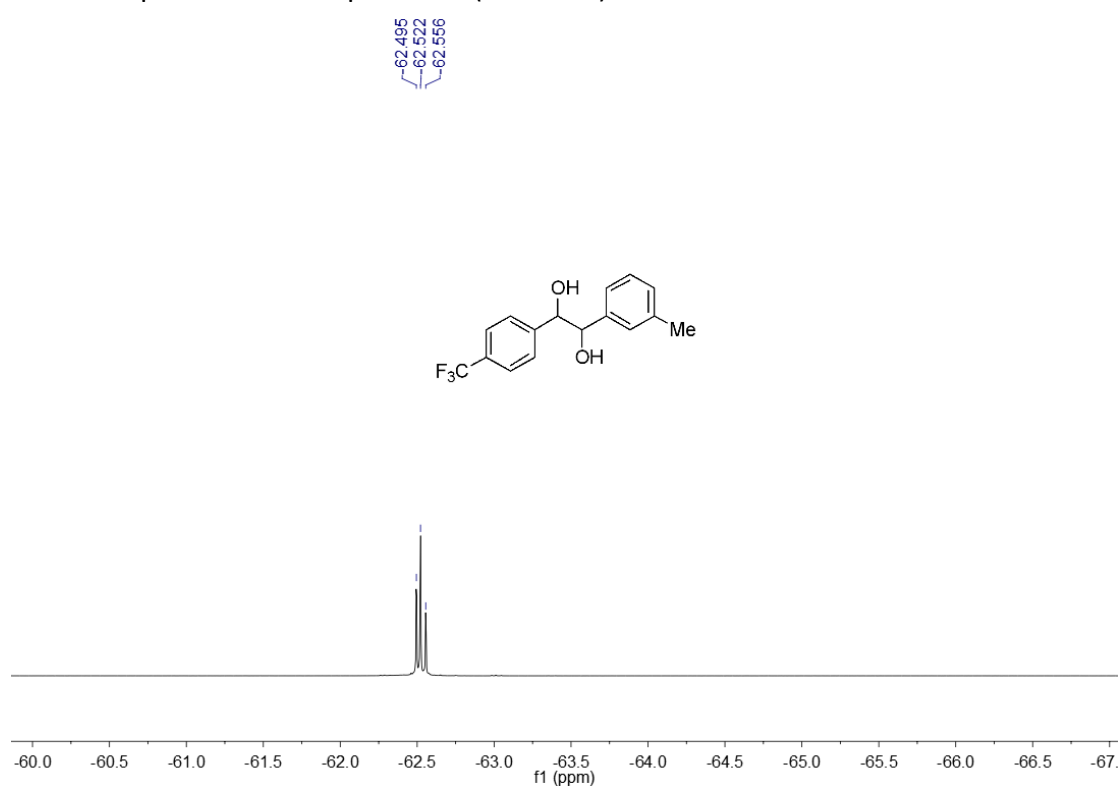
^1H NMR spectrum of compound **5f** (400 MHz) in CDCl_3



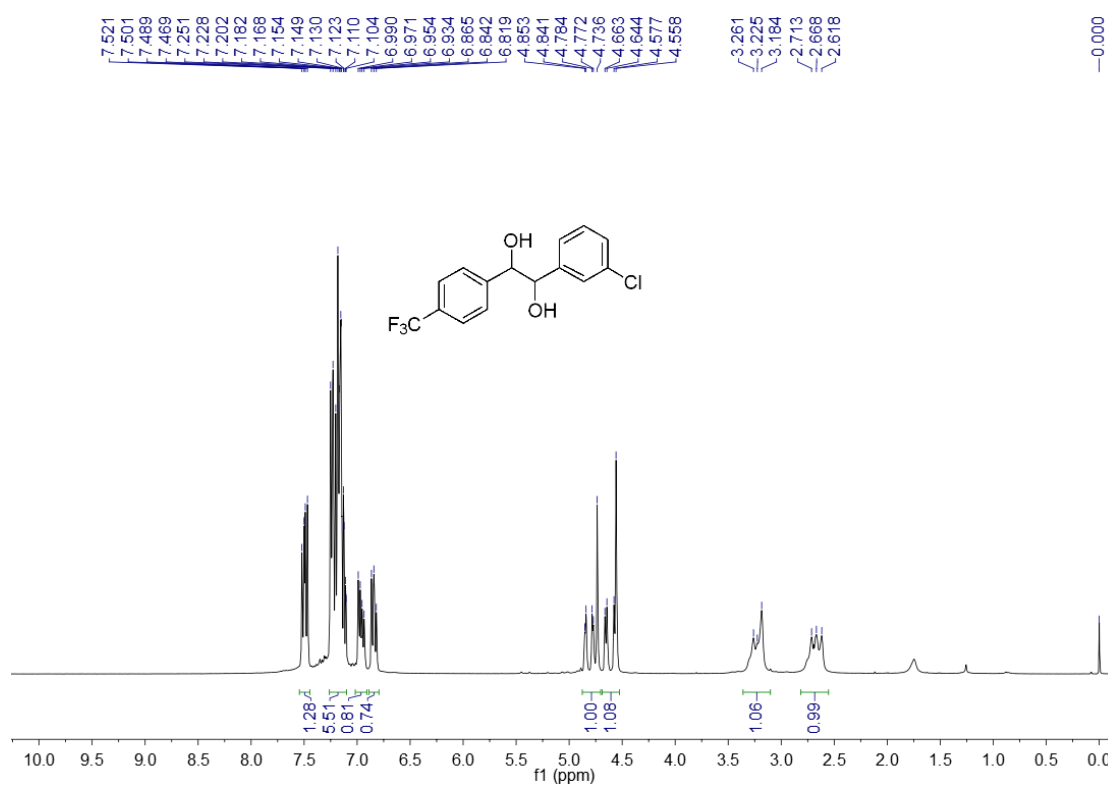
^{13}C NMR spectrum of compound **5f** (100 MHz) in CDCl_3



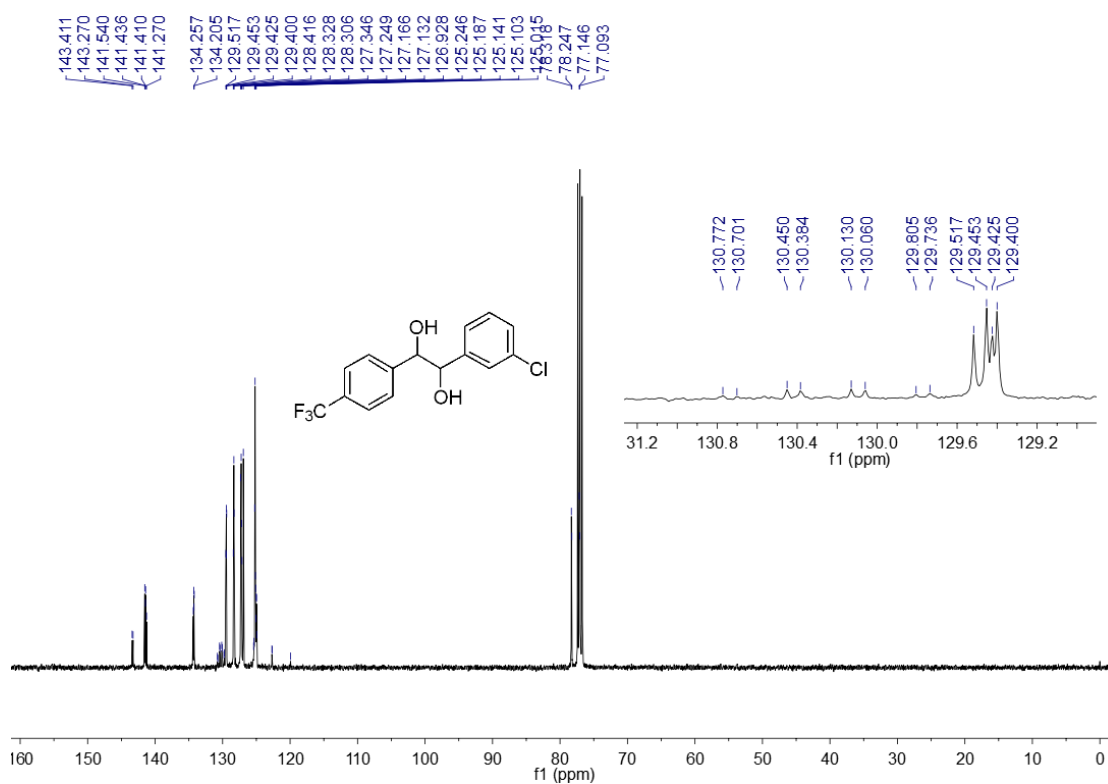
^{19}F NMR spectrum of compound **5f** (376 MHz) in CDCl_3



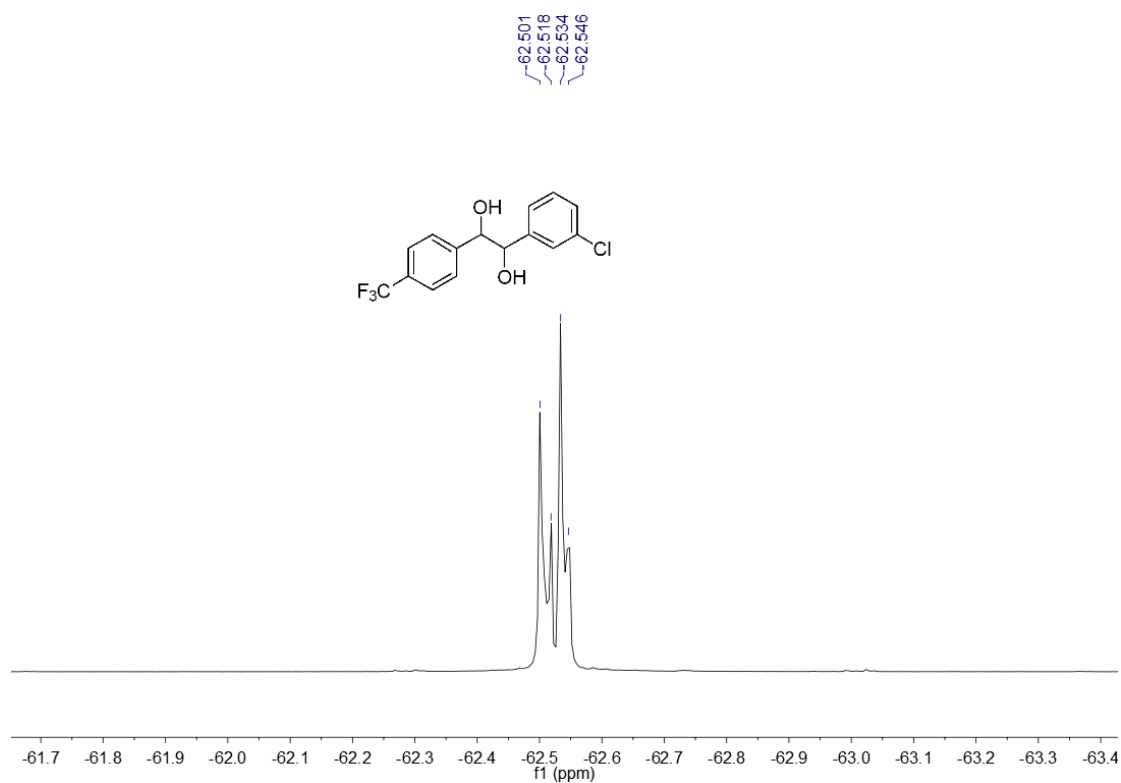
^1H NMR spectrum of compound **5g** (400 MHz) in CDCl_3



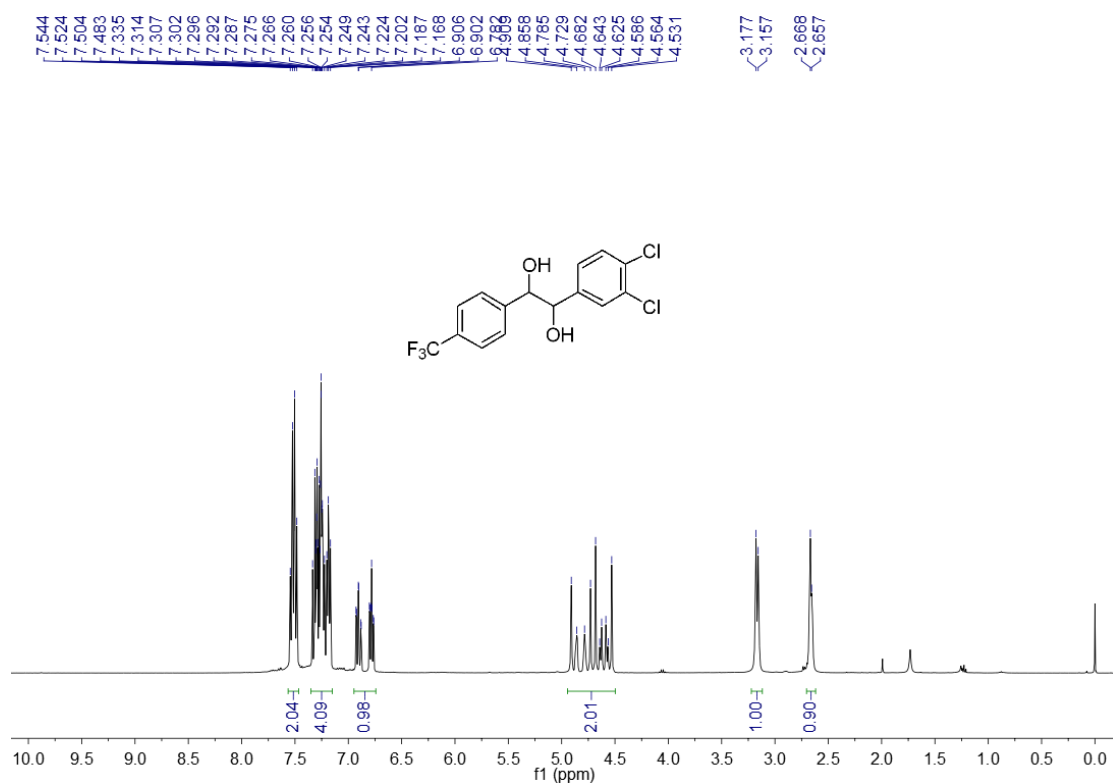
^{13}C NMR spectrum of compound **5g** (100 MHz) in CDCl_3



^{19}F NMR spectrum of compound **5g** (376 MHz) in CDCl_3



^1H NMR spectrum of compound **5h** (400 MHz) in CDCl_3



Chemical structure of (R)-1-(4-(trifluoromethyl)phenyl)-2-(2,4-dichlorophenyl)ethan-1-ol:

Clc1cc(Cl)ccc1C(O)(c2ccc(C(F)(F)F)cc2)CO

13C NMR Peaks (ppm):

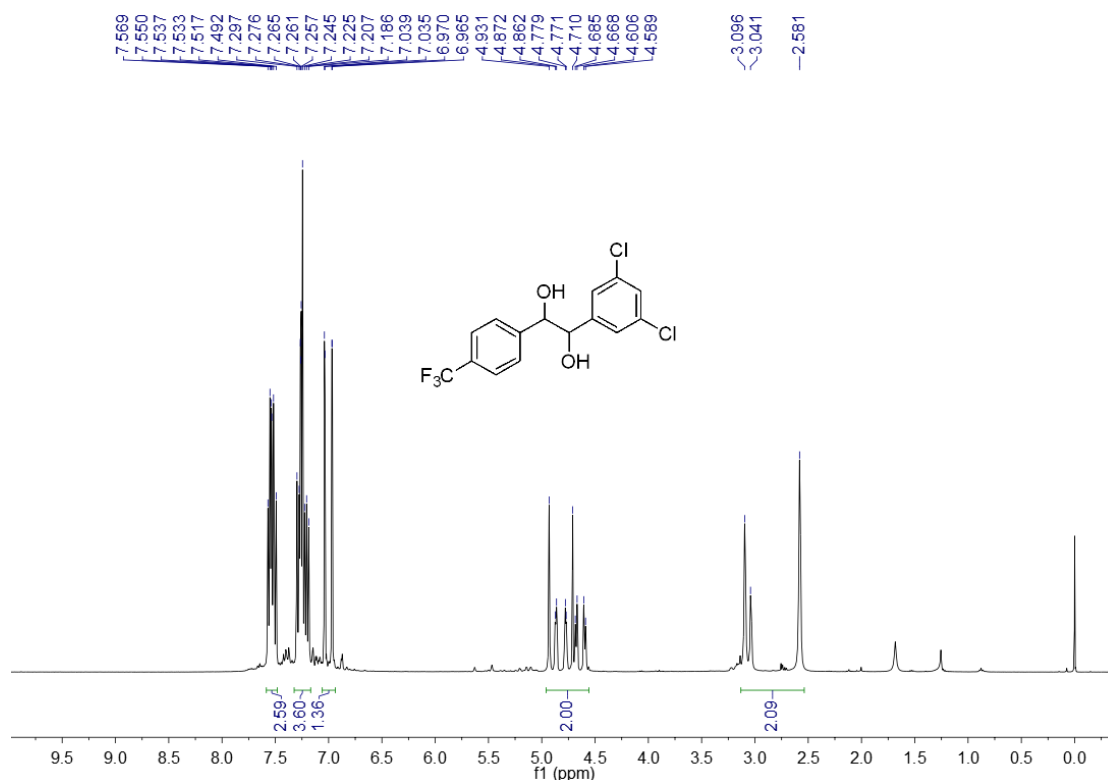
- 143.219, 143.139, 143.064, 139.537, 139.488, 139.435, 139.403
- 130.217, 130.167, 130.118, 130.042, 128.996, 128.960, 128.765, 128.735, 128.735
- 127.321, 127.298, 127.269, 126.367, 126.329, 125.296, 125.255, 125.243, 125.213
- 78.291, 77.763, 77.619, 77.139, 76.990, 76.564, 76.447
- 132.570, 132.463, 132.389, 132.311, 132.298, 132.278, 130.607, 130.548, 130.484, 130.354, 130.282, 130.217, 130.167, 130.118, 130.042, 128.996, 128.960, 128.765, 128.735
- 128.054, 127.992, 127.298, 127.269
- 119.943, 119.880

Chemical structure of (2S,3S)-2-(4-(trifluoromethyl)phenyl)-3-(2,4-dichlorophenyl)butane-2,3-diol is shown above the spectrum.

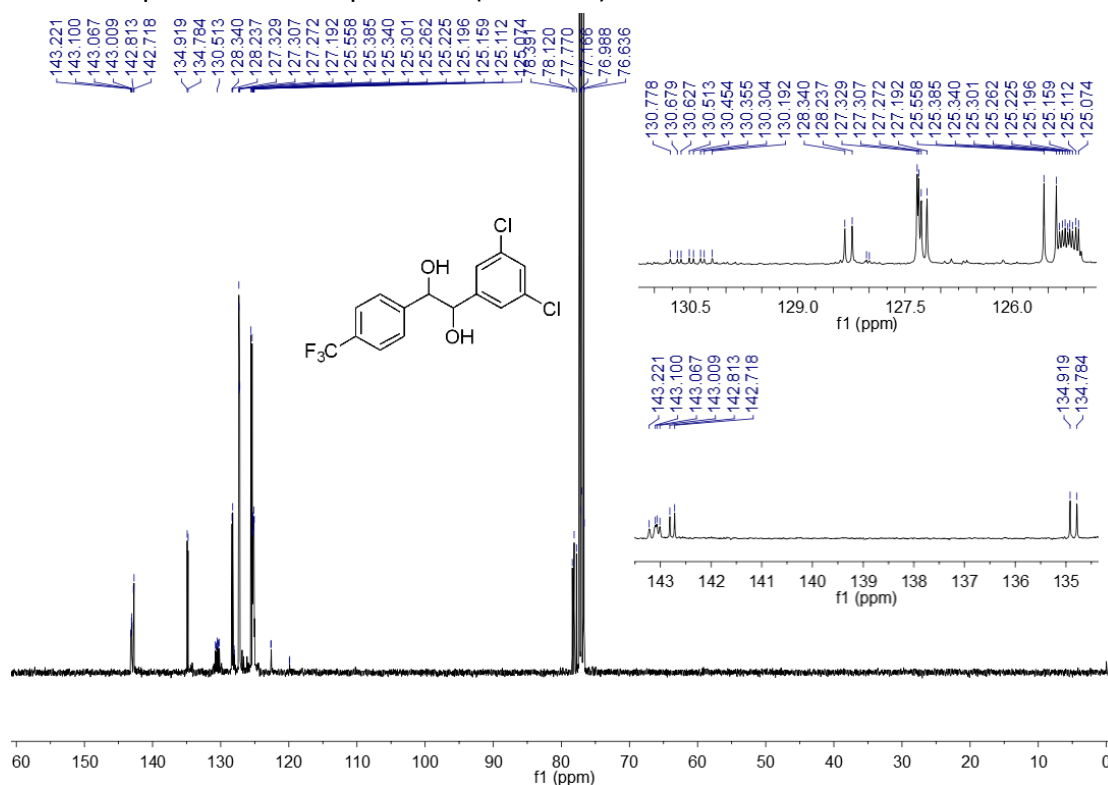
Peak list (ppm):

Peak Label	Chemical Shift (ppm)
62.515	-62.515
62.536	-62.536
62.552	-62.552
62.570	-62.570

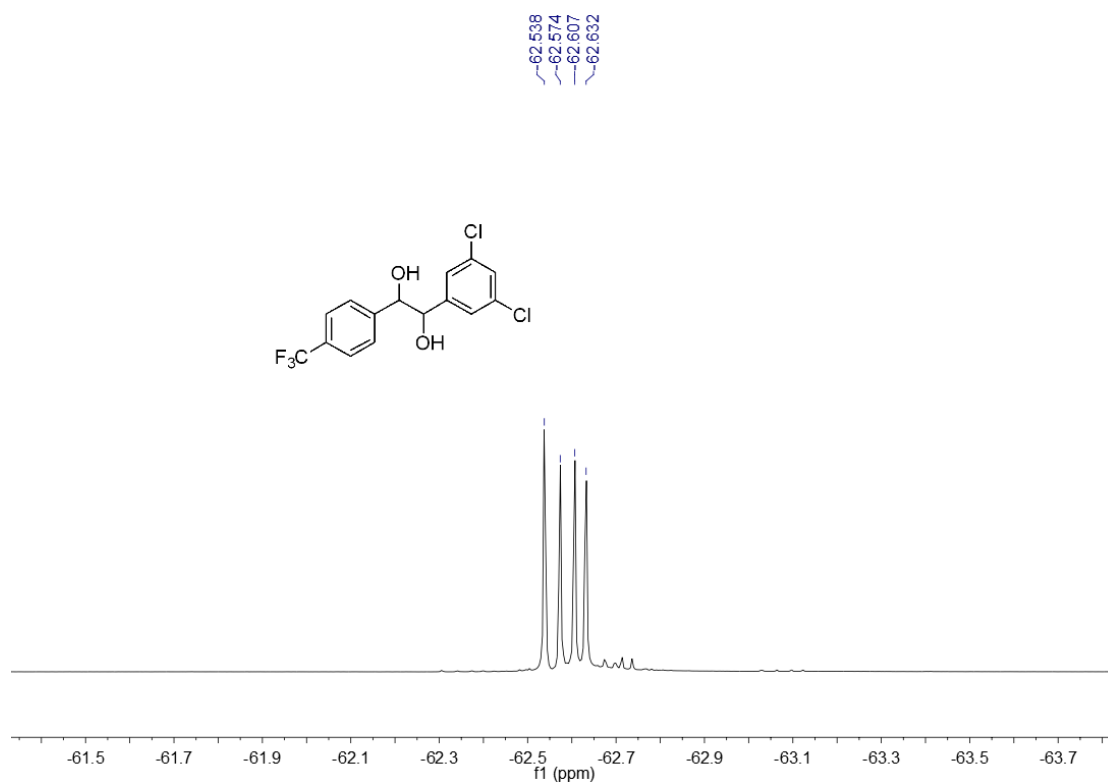
^1H NMR spectrum of compound **5i** (400 MHz) in CDCl_3



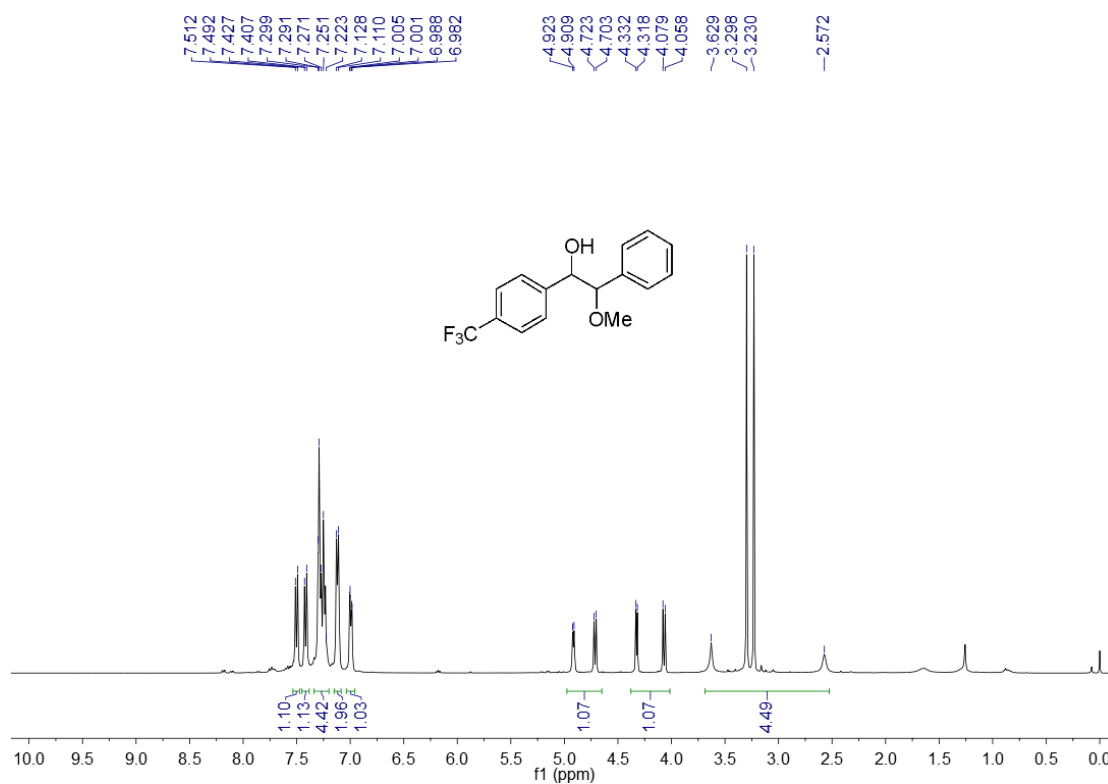
^{13}C NMR spectrum of compound **5i** (100 MHz) in CDCl_3



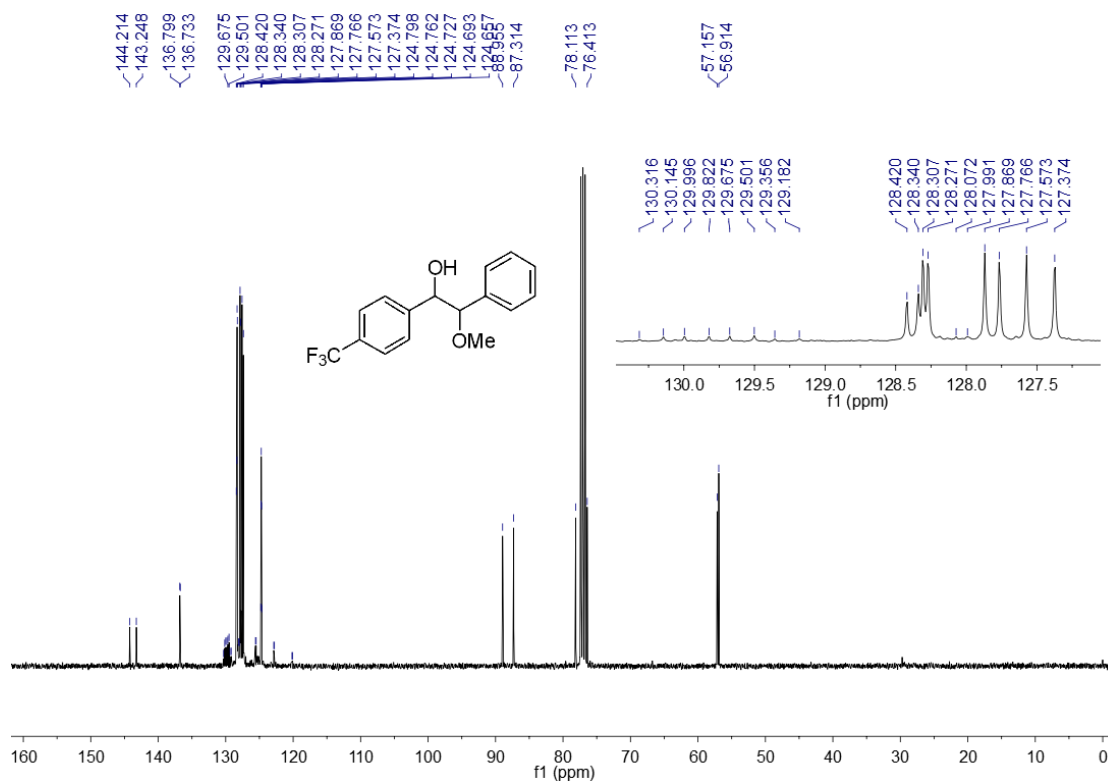
^{19}F NMR spectrum of compound **5i** (376 MHz) in CDCl_3



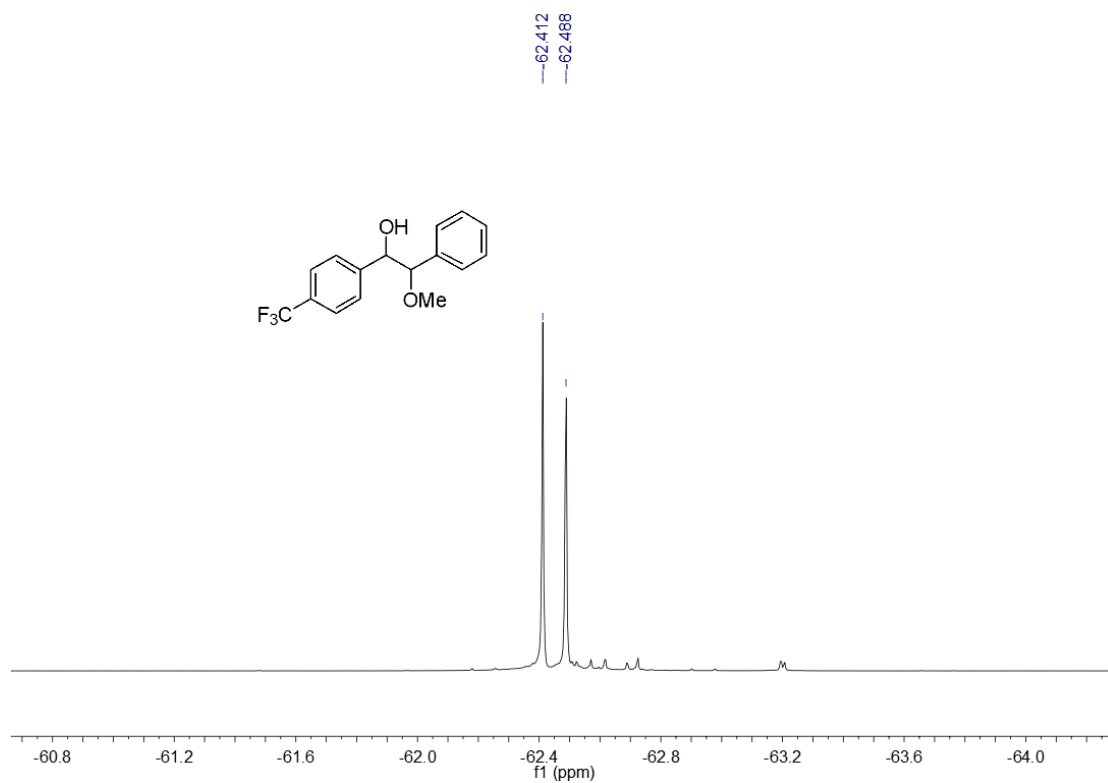
^1H NMR spectrum of compound **5j** (400 MHz) in CDCl_3



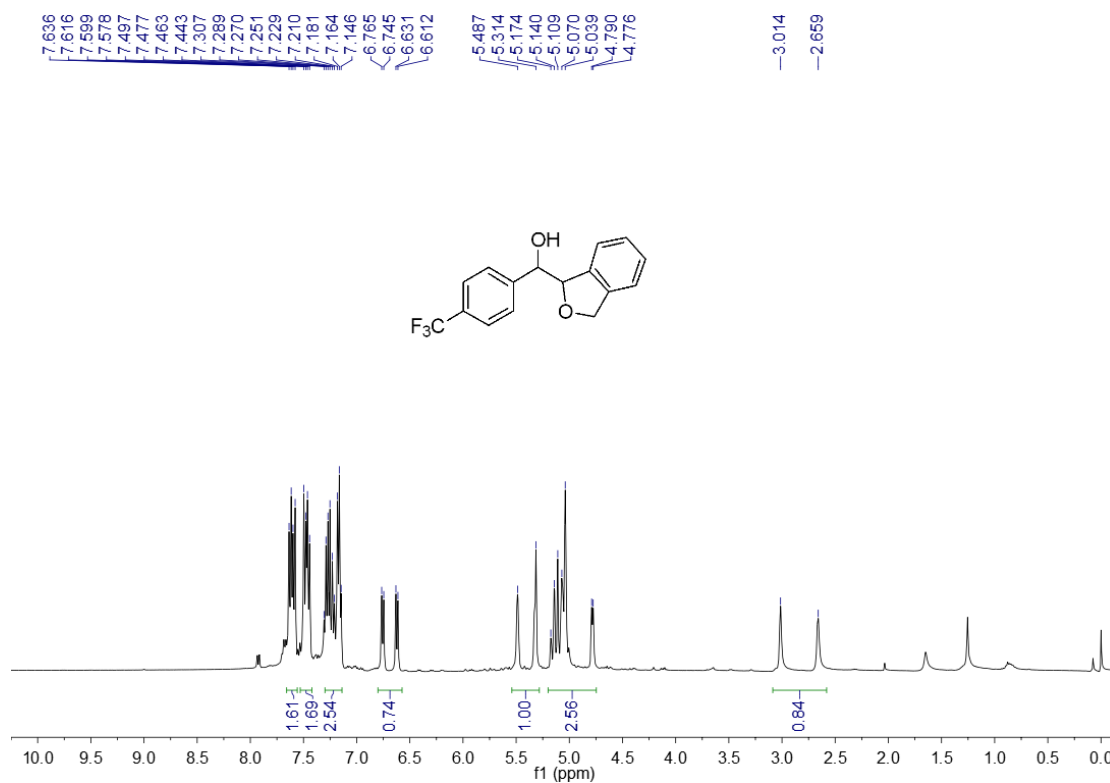
^{13}C NMR spectrum of compound **5j** (100 MHz) in CDCl_3



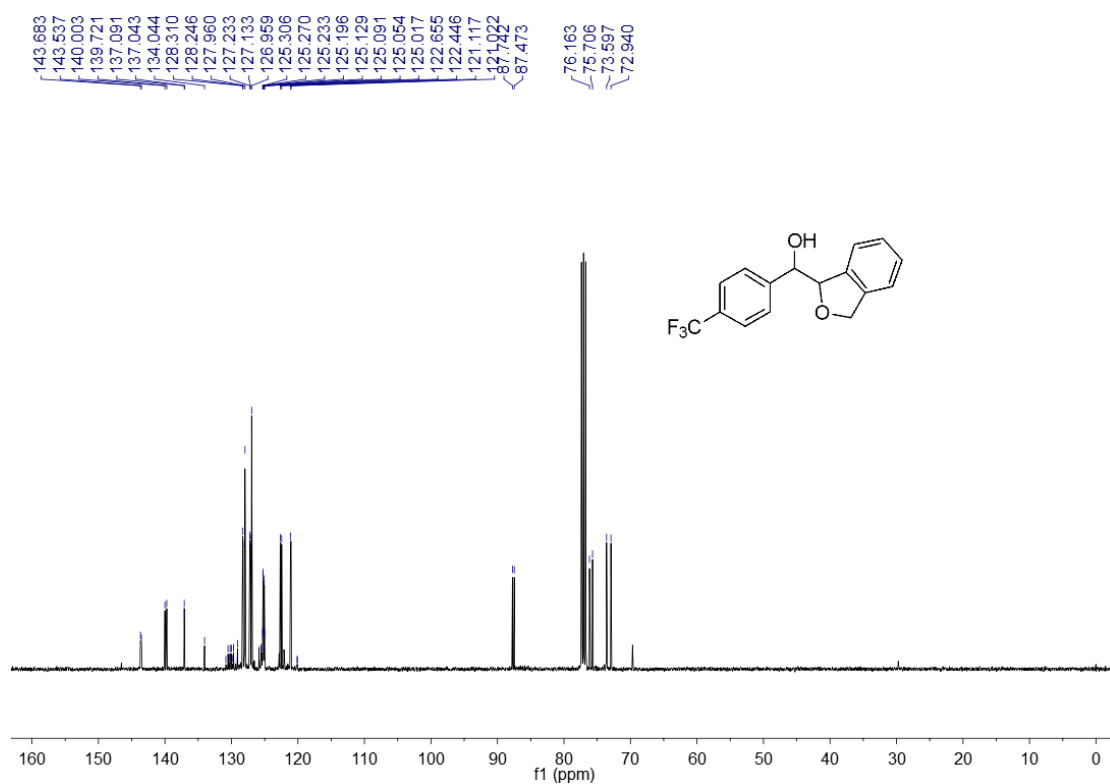
^{19}F NMR spectrum of compound **5j** (376 MHz) in CDCl_3



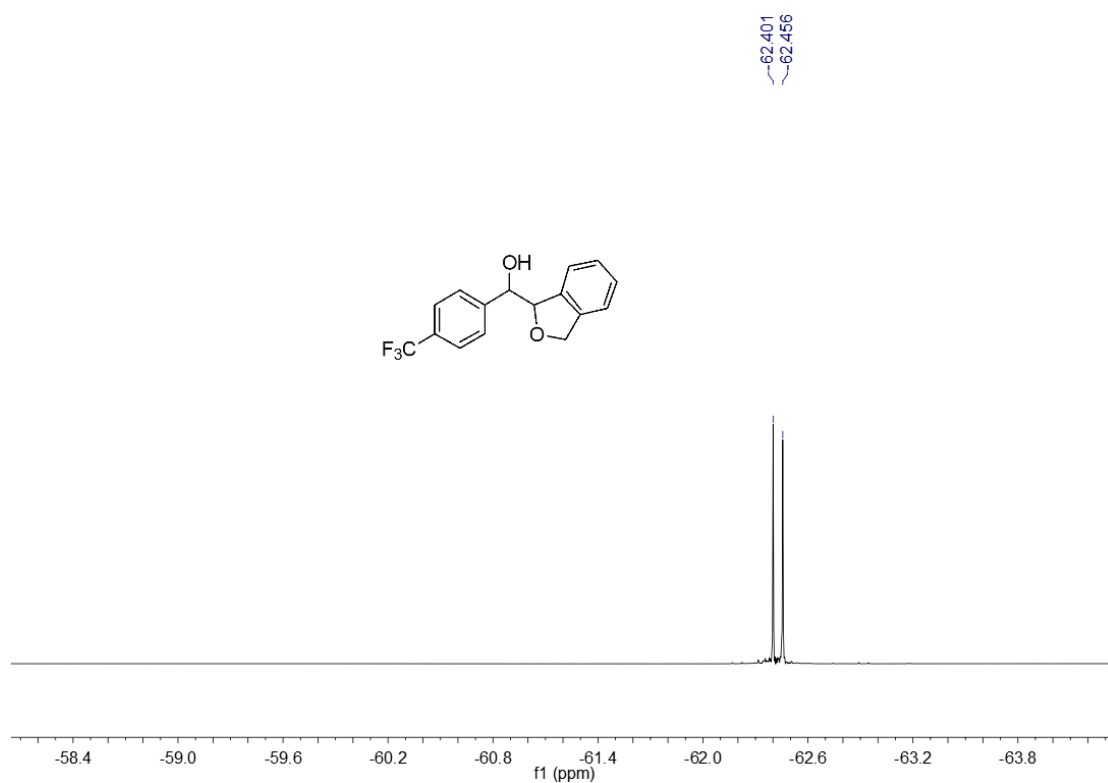
^1H NMR spectrum of compound **5k** (400 MHz) in CDCl_3



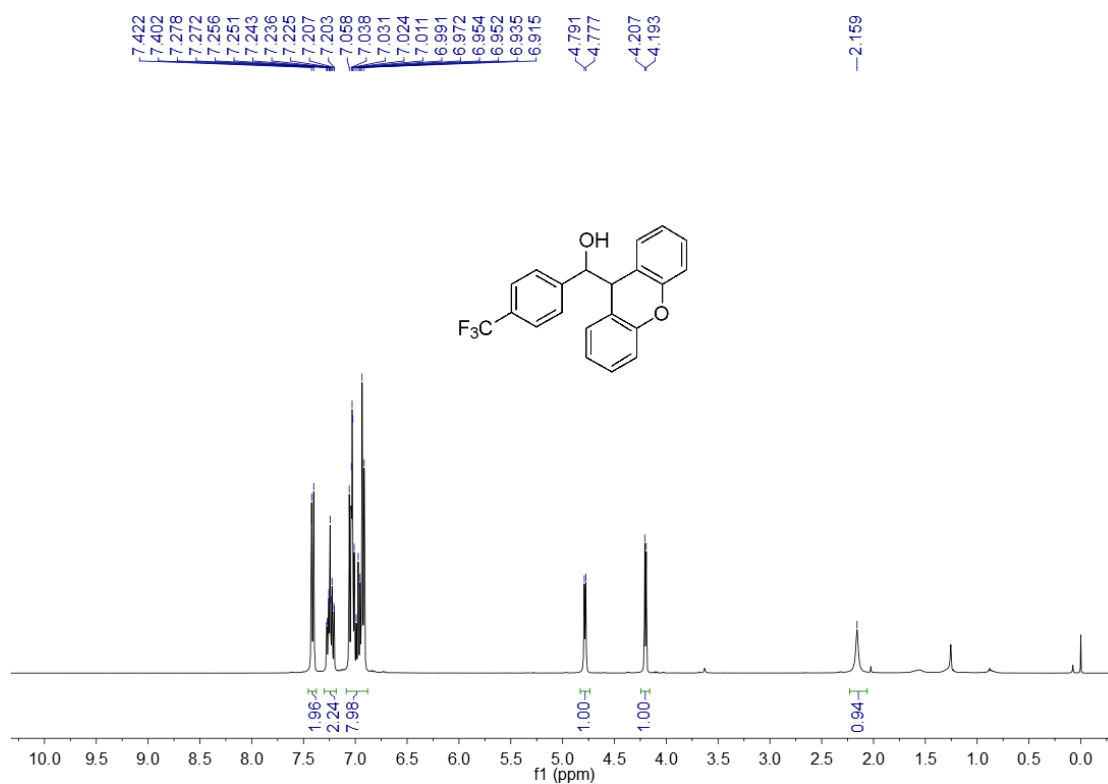
^{13}C NMR spectrum of compound **5k** (100 MHz) in CDCl_3



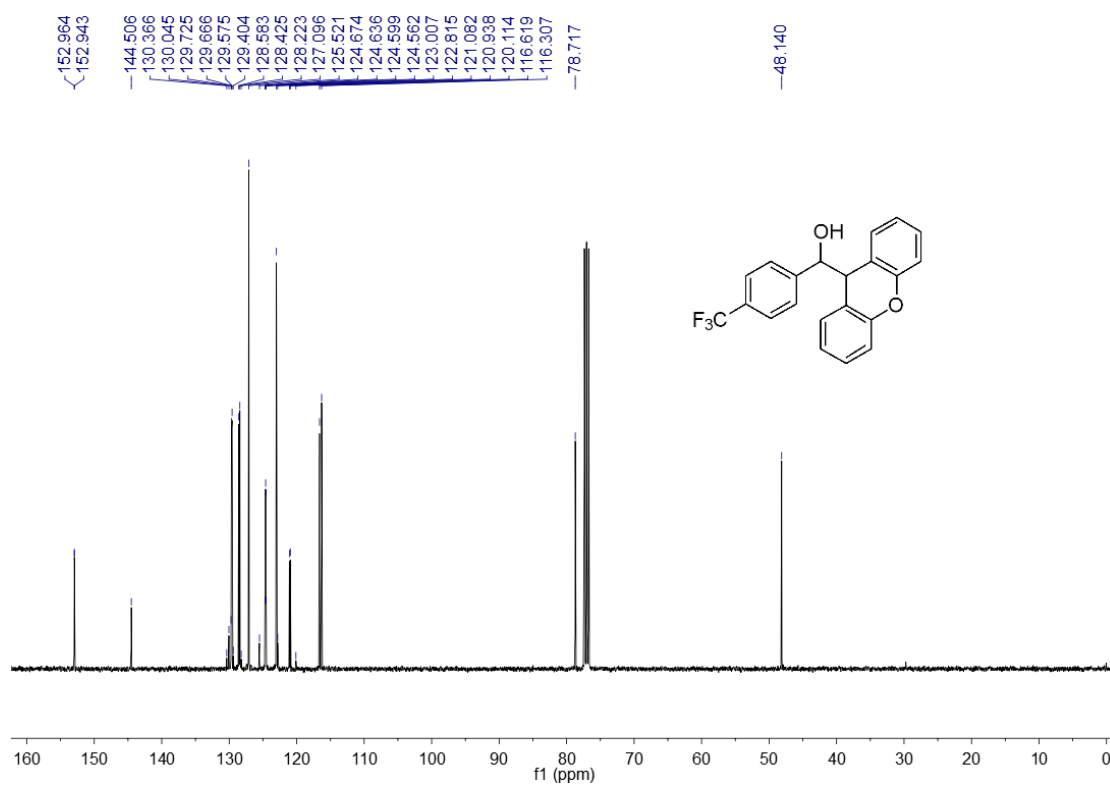
^{19}F NMR spectrum of compound **5k** (376 MHz) in CDCl_3



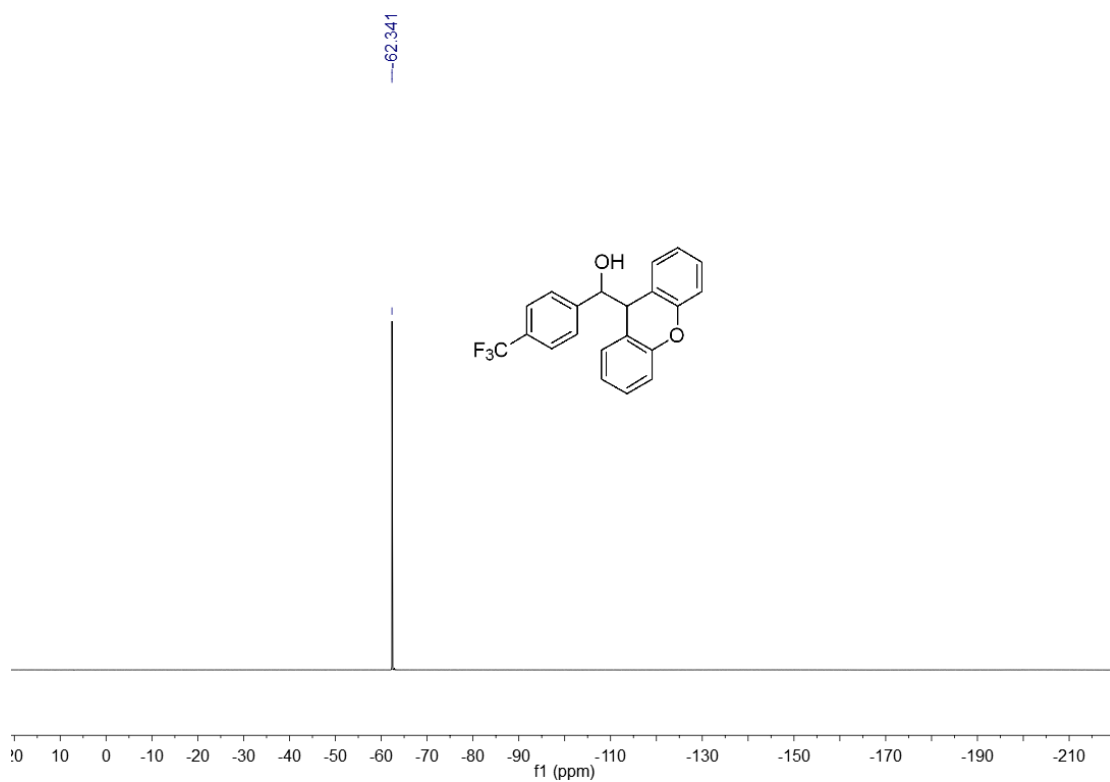
^1H NMR spectrum of compound **5l** (400 MHz) in CDCl_3



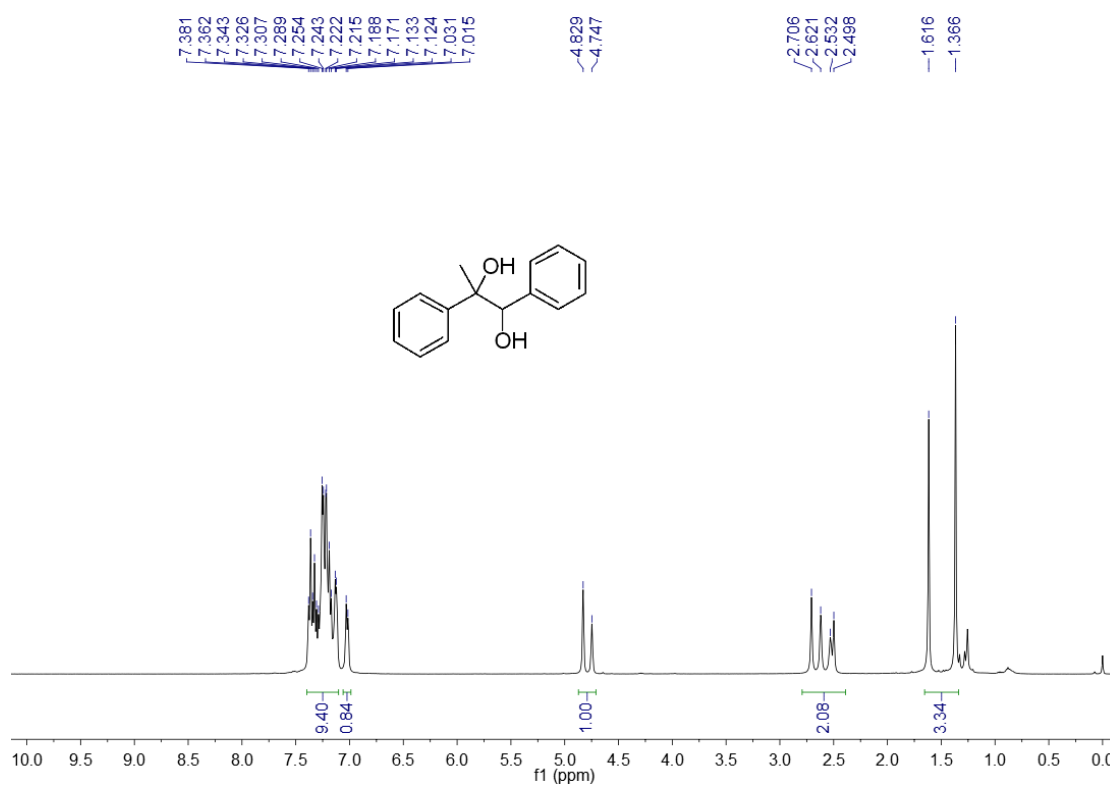
^{13}C NMR spectrum of compound **5l** (100 MHz) in CDCl_3



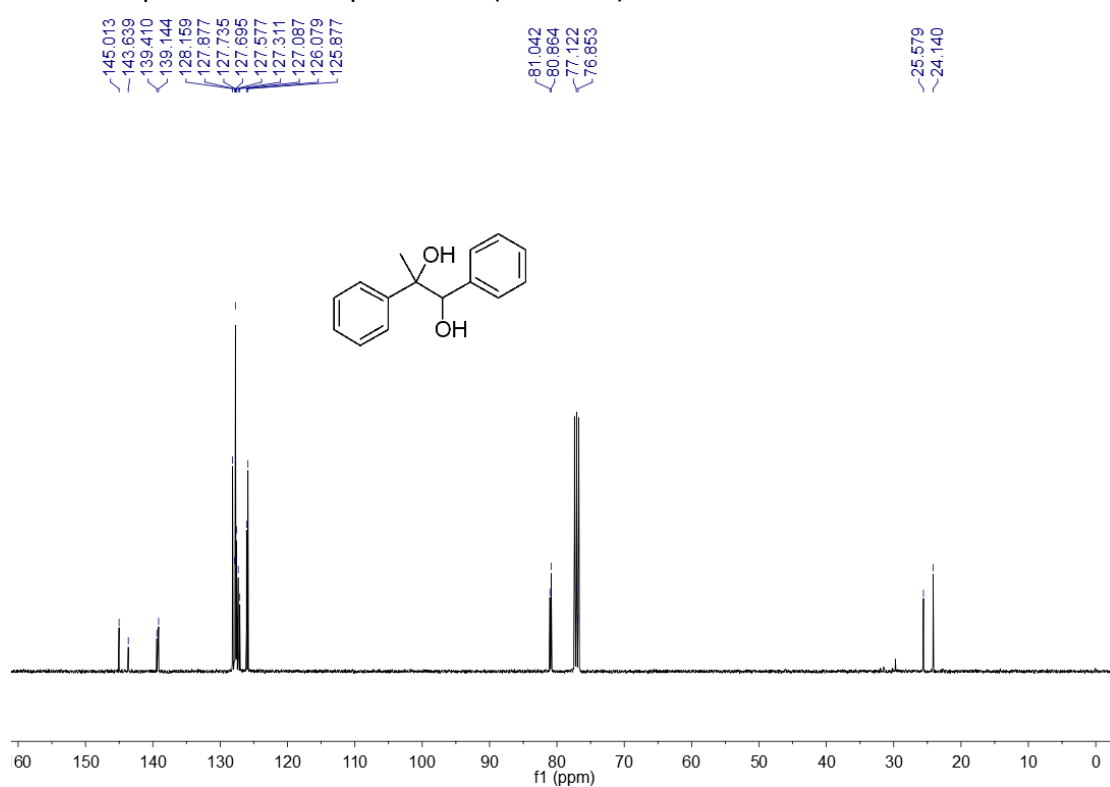
¹⁹F NMR spectrum of compound 5I (376 MHz) in CDCl₃



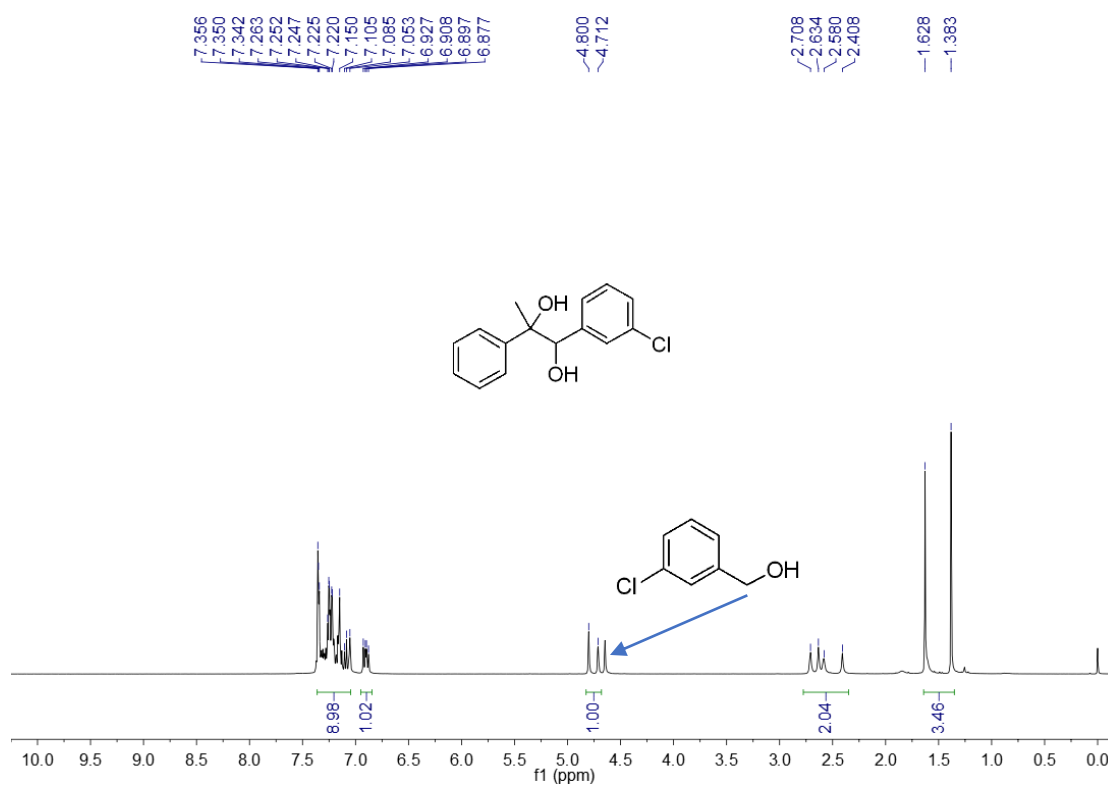
^1H NMR spectrum of compound **5m** (400 MHz) in CDCl_3



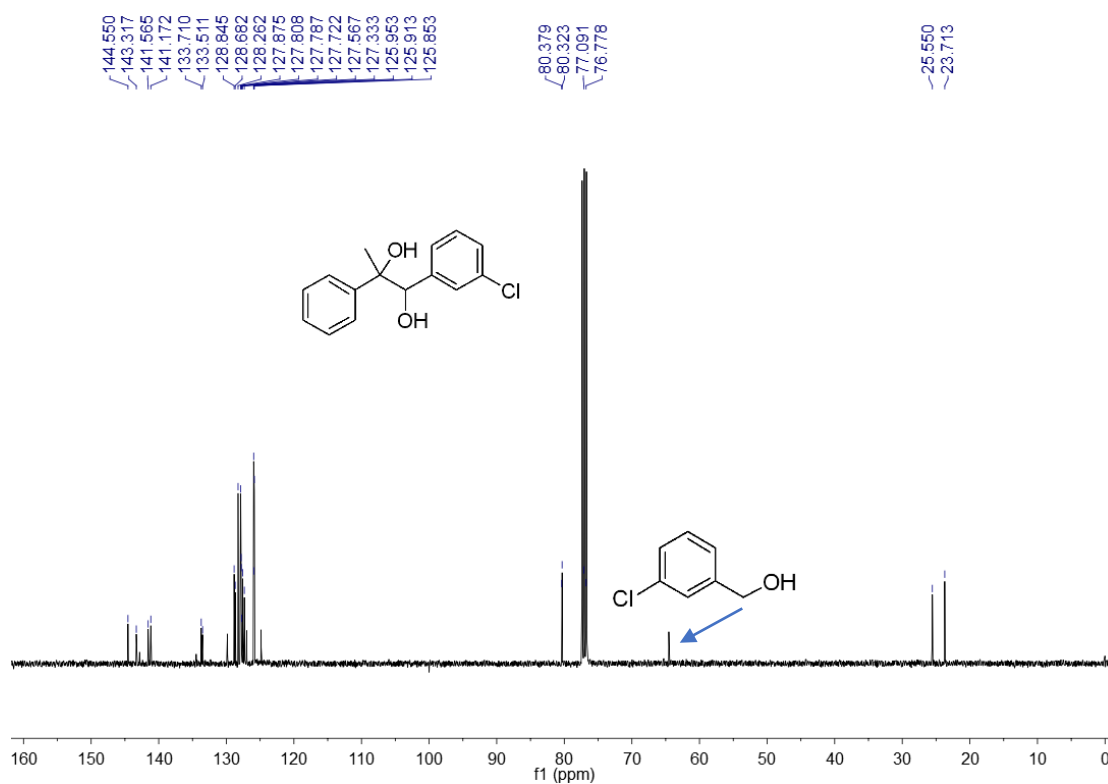
^{13}C NMR spectrum of compound **5m** (100 MHz) in CDCl_3



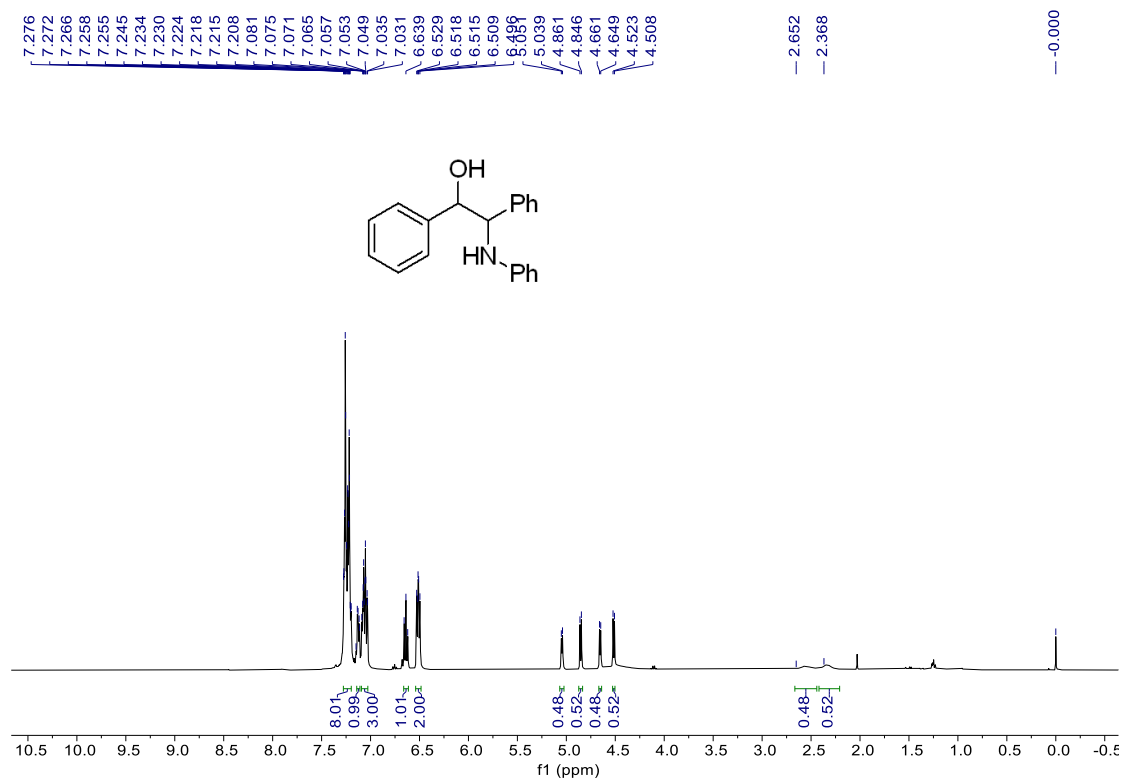
^1H NMR spectrum of compound **5n** (400 MHz) in CDCl_3



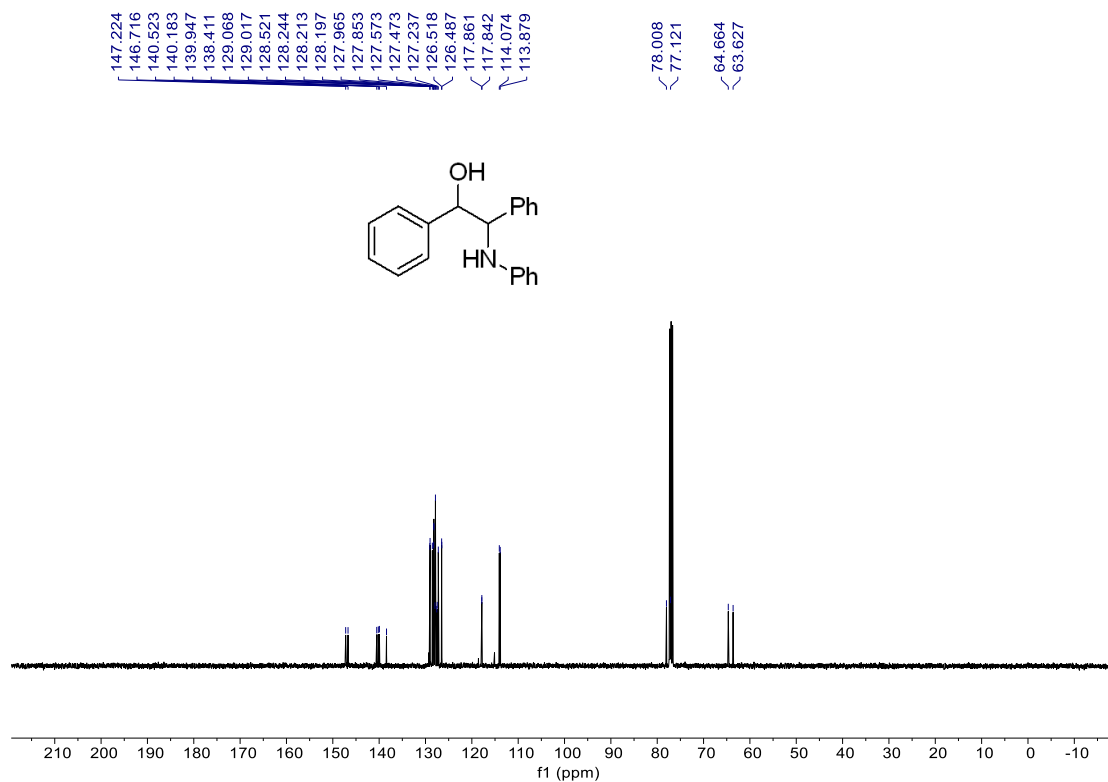
^{13}C NMR spectrum of compound **5n** (100 MHz) in CDCl_3



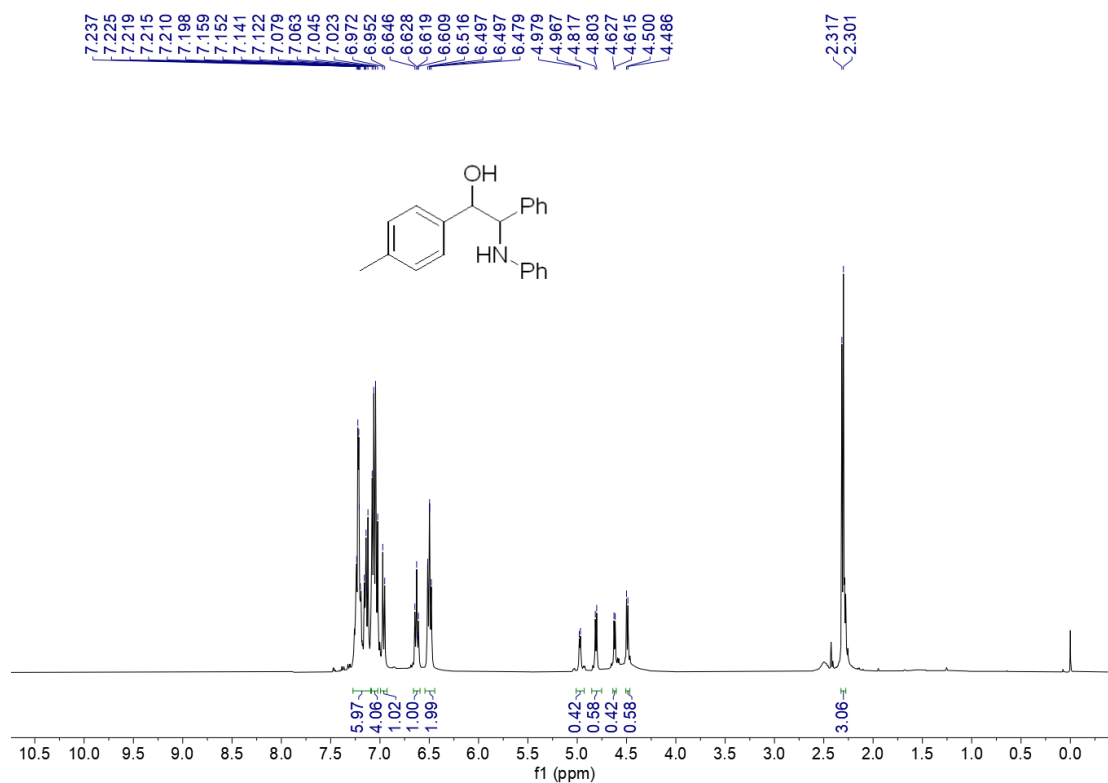
¹H NMR spectrum of compound **7a** (400 MHz) in CDCl₃



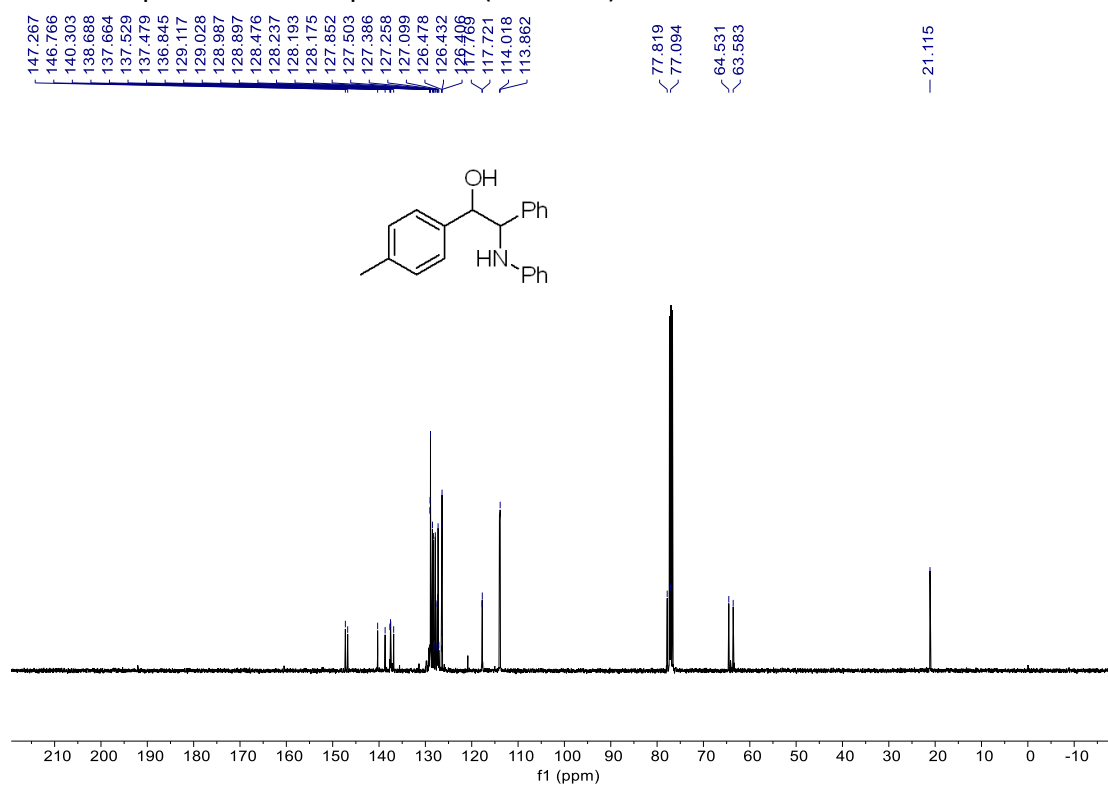
¹³C NMR spectrum of compound **7a** (100 MHz) in CDCl₃



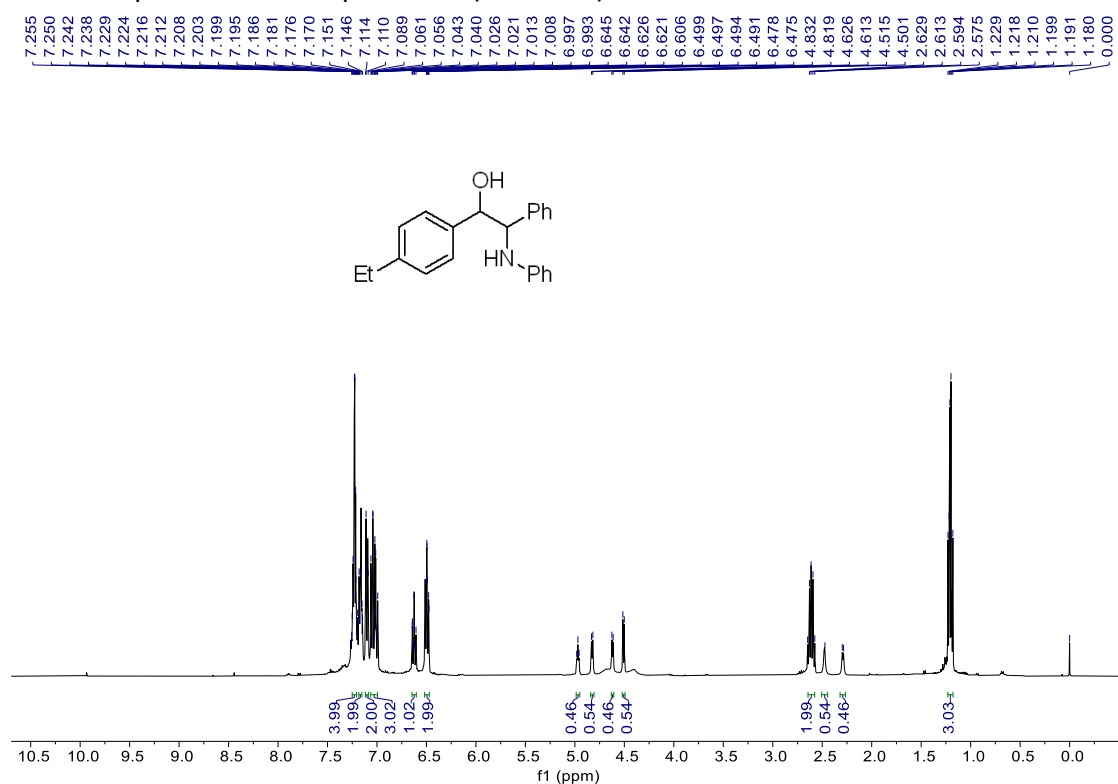
^1H NMR spectrum of compound **7b** (400 MHz) in CDCl_3



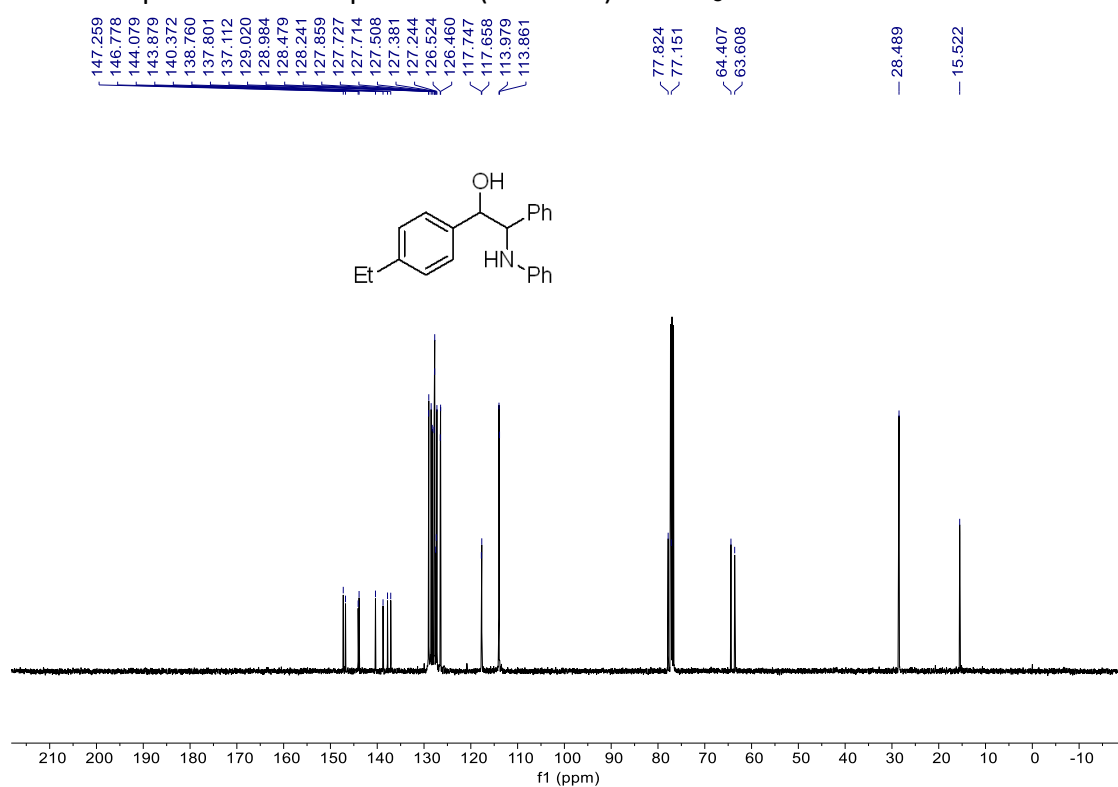
^{13}C NMR spectrum of compound **7b** (100 MHz) in CDCl_3



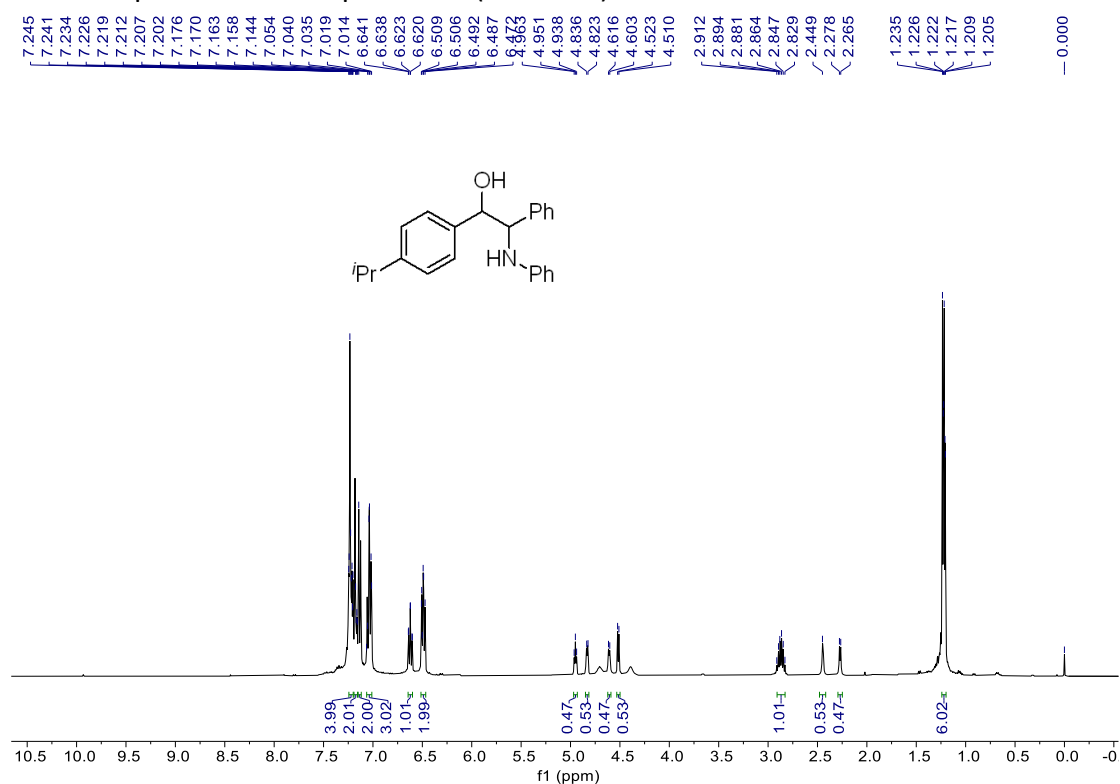
¹H NMR spectrum of compound **7c** (400 MHz) in CDCl₃



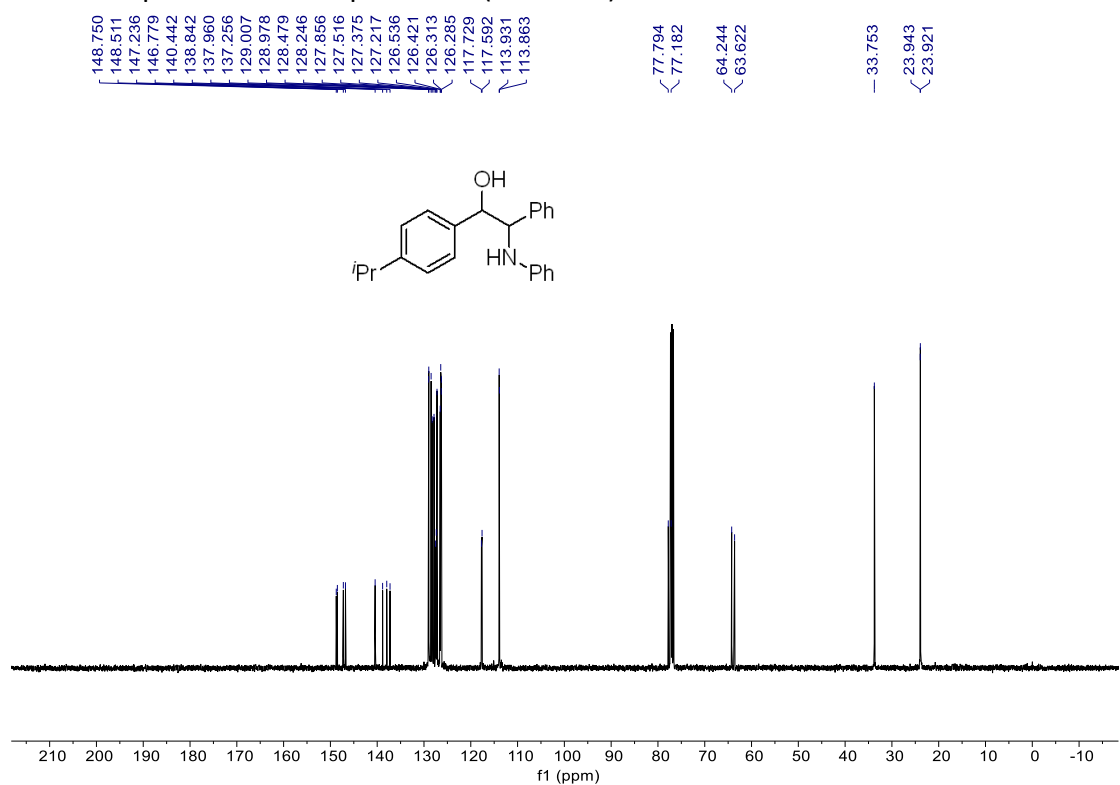
¹³C NMR spectrum of compound **7c** (100 MHz) in CDCl₃



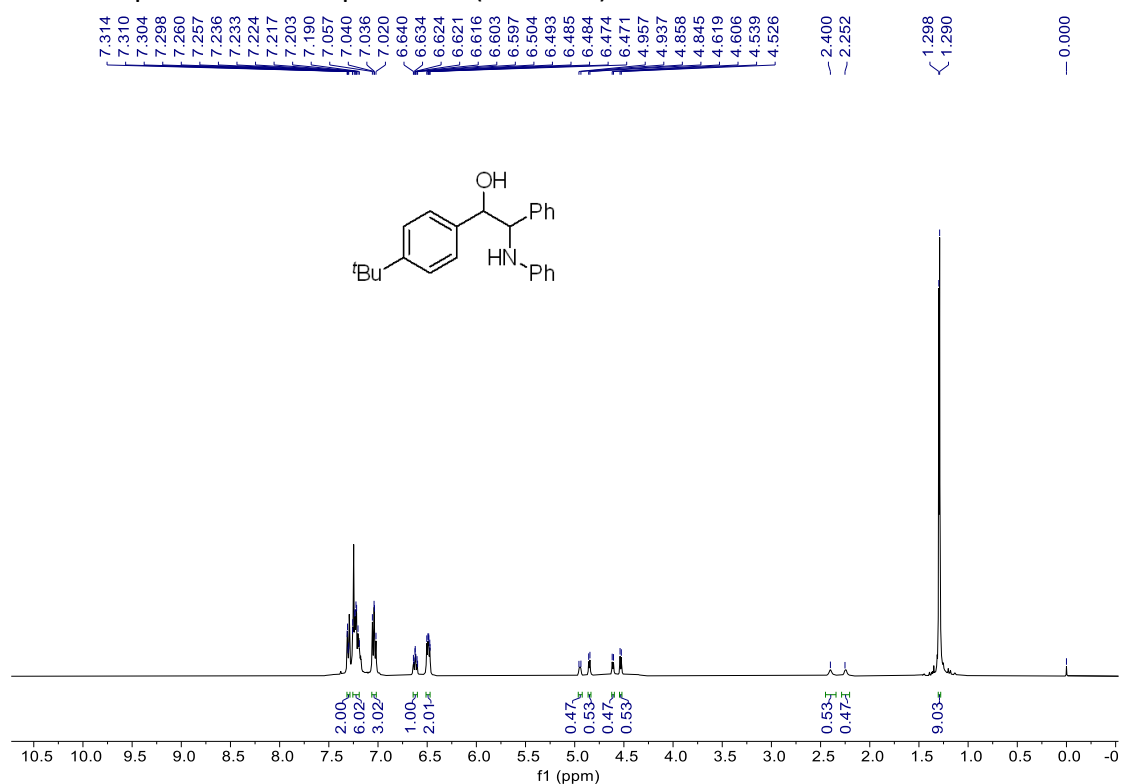
^1H NMR spectrum of compound **7d** (400 MHz) in CDCl_3



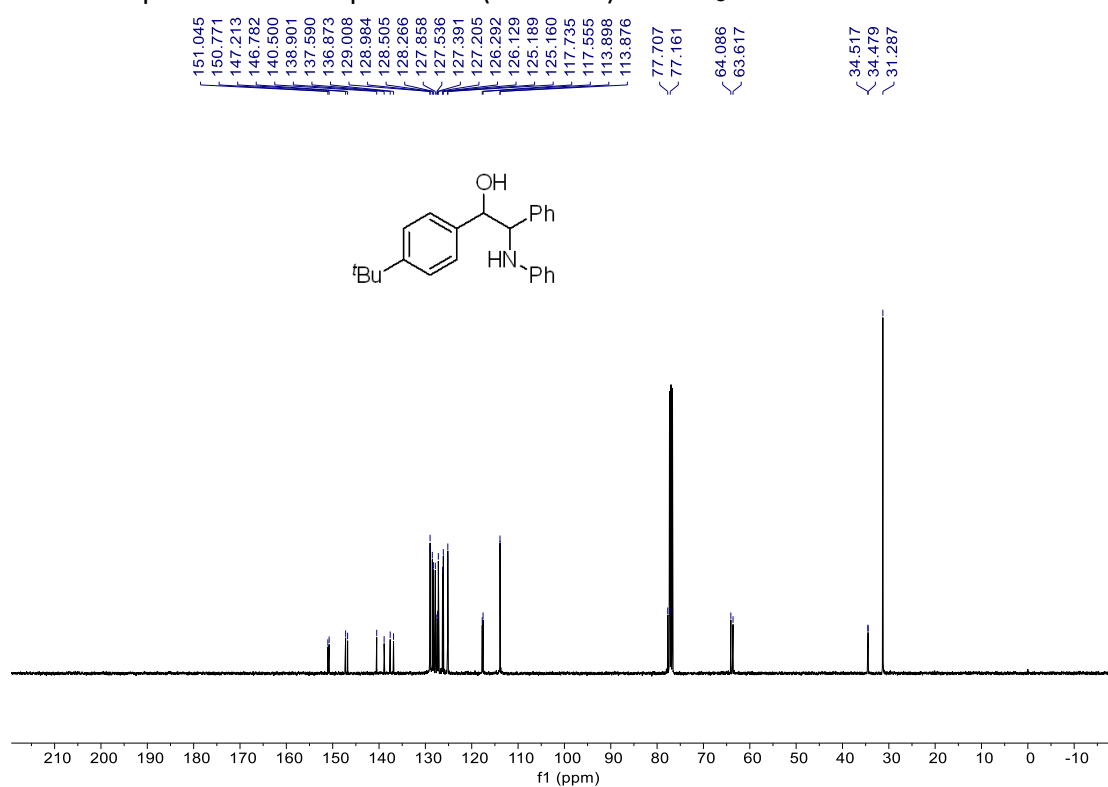
^{13}C NMR spectrum of compound **7d** (100 MHz) in CDCl_3



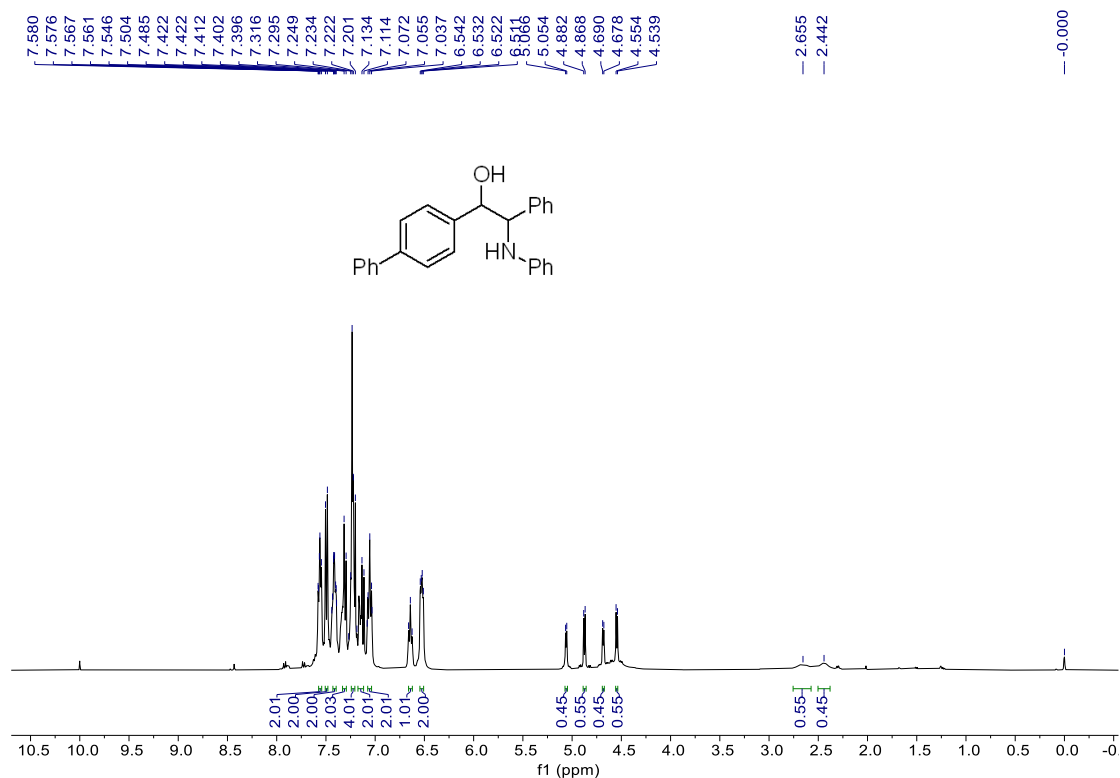
^1H NMR spectrum of compound **7e** (400 MHz) in CDCl_3



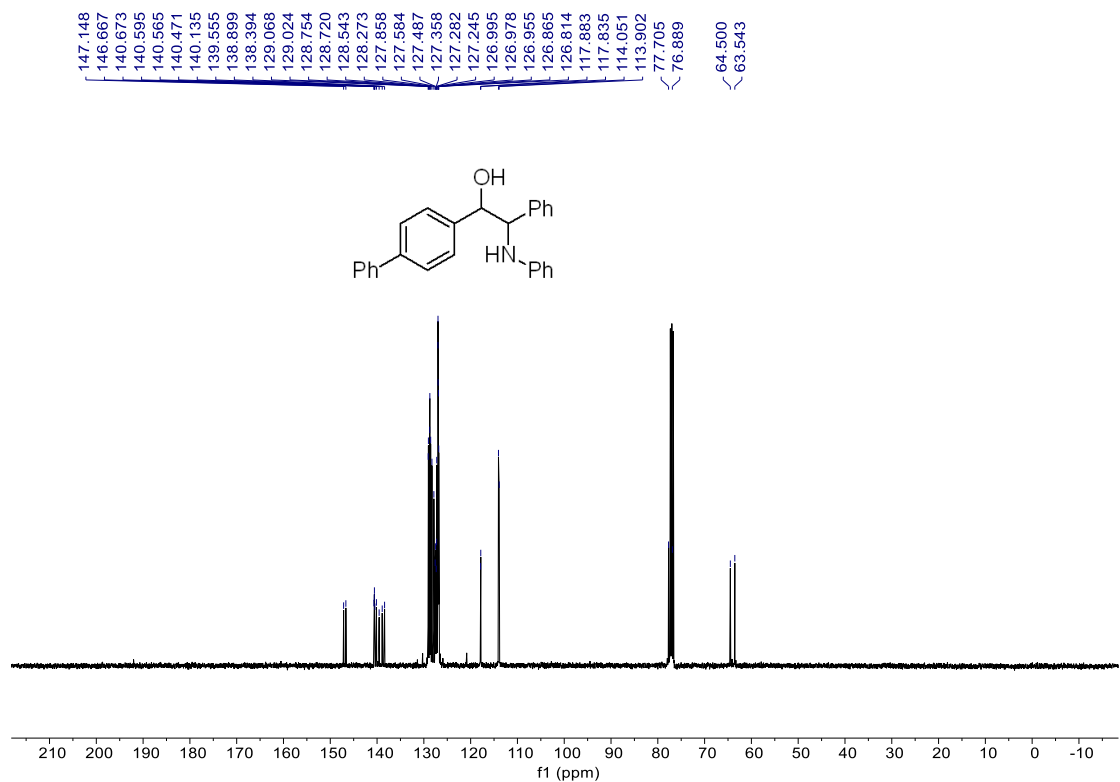
^{13}C NMR spectrum of compound **7e** (100 MHz) in CDCl_3



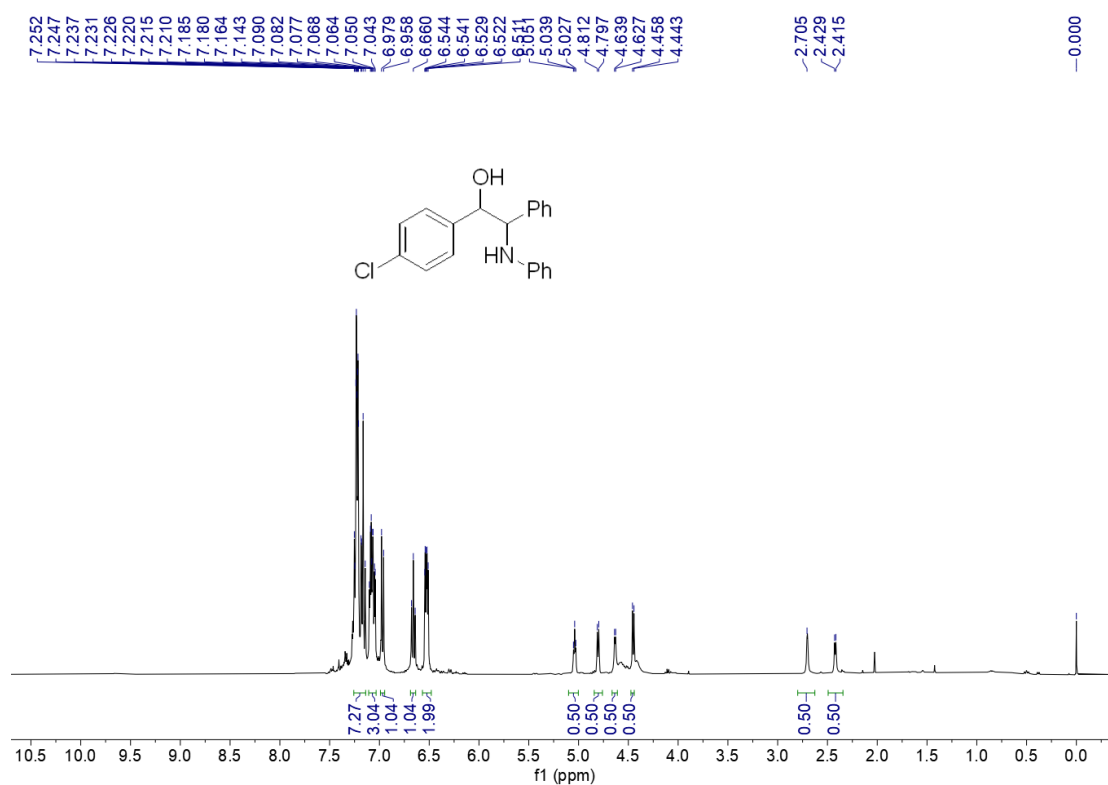
¹H NMR spectrum of compound **7f** (400 MHz) in CDCl₃



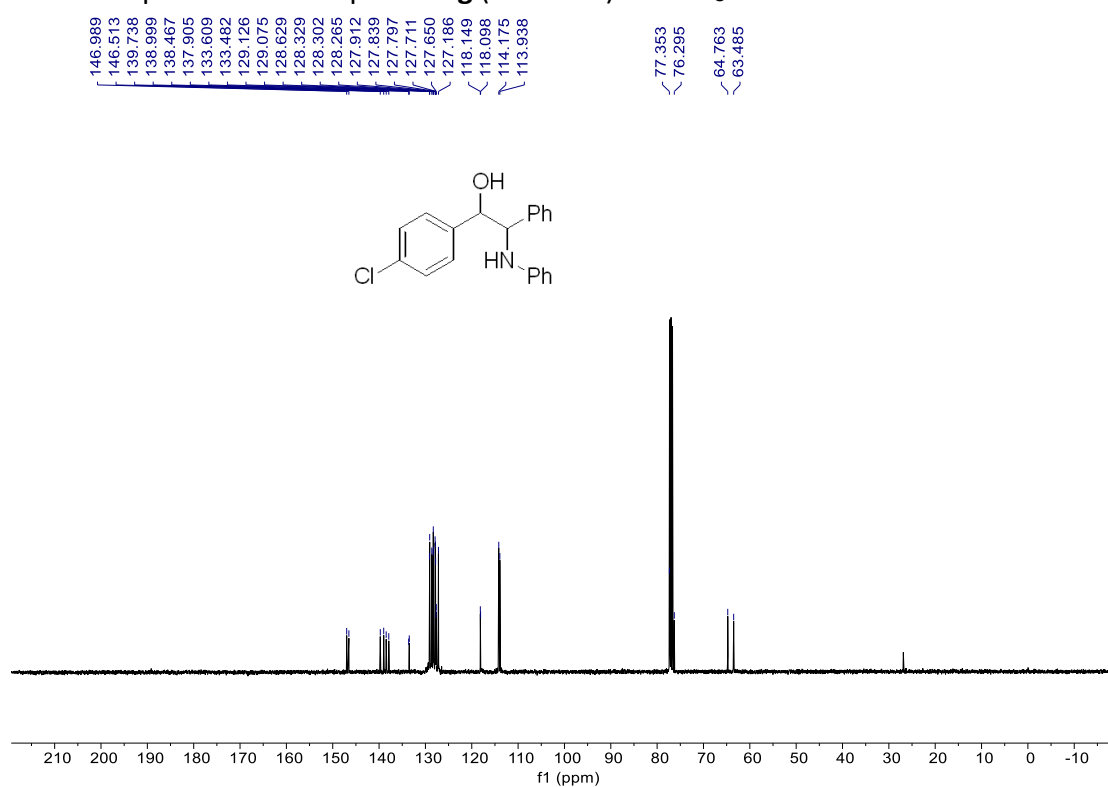
¹³C NMR spectrum of compound **7f** (100 MHz) in CDCl₃



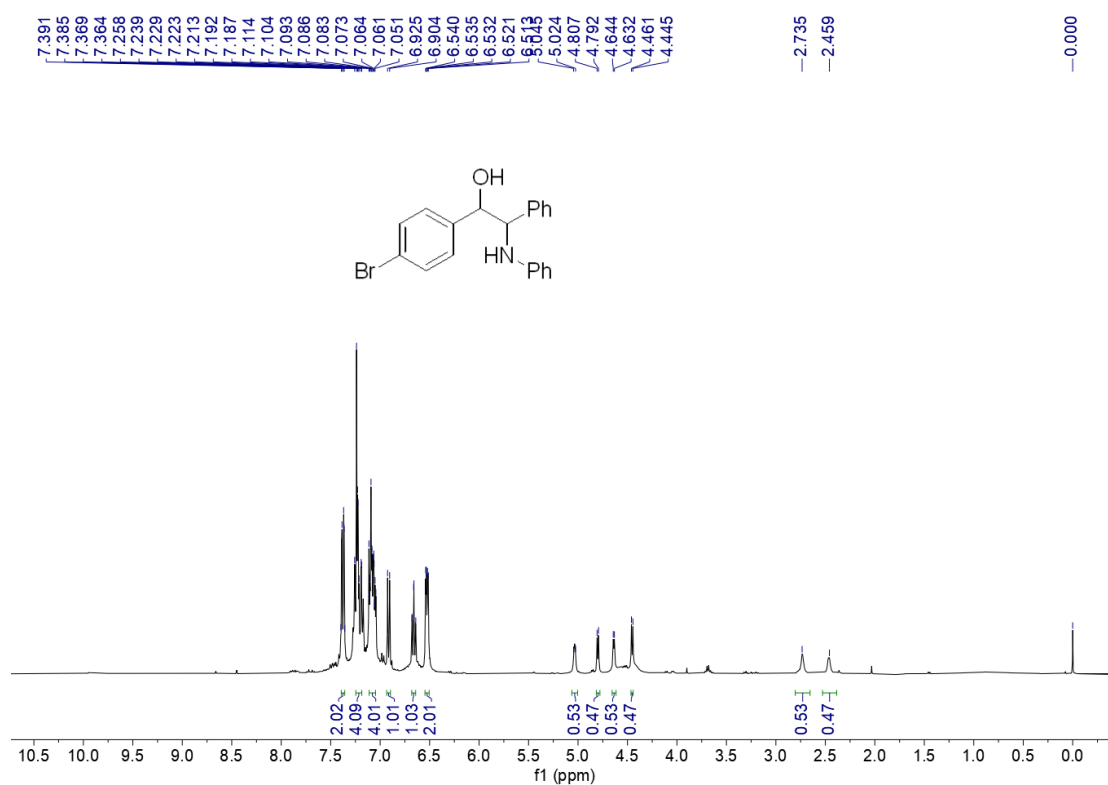
¹H NMR spectrum of compound **7g** (400 MHz) in CDCl₃



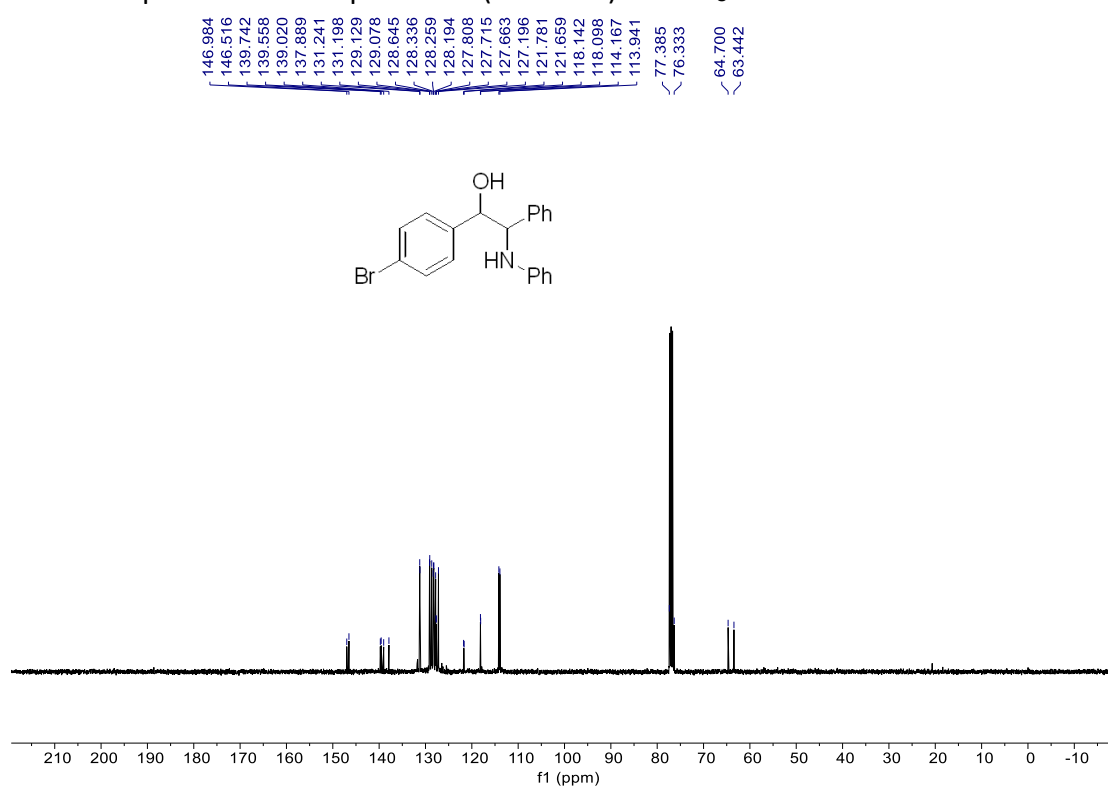
¹³C NMR spectrum of compound **7g** (100 MHz) in CDCl₃



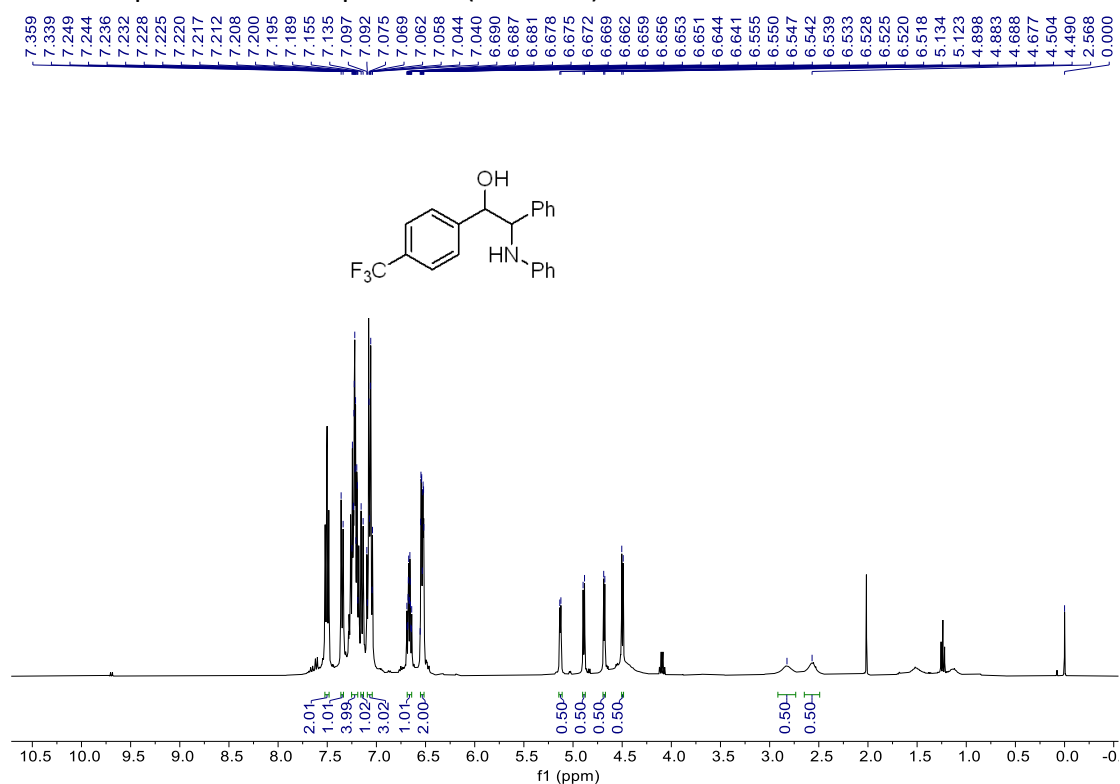
^1H NMR spectrum of compound **7h** (400 MHz) in CDCl_3



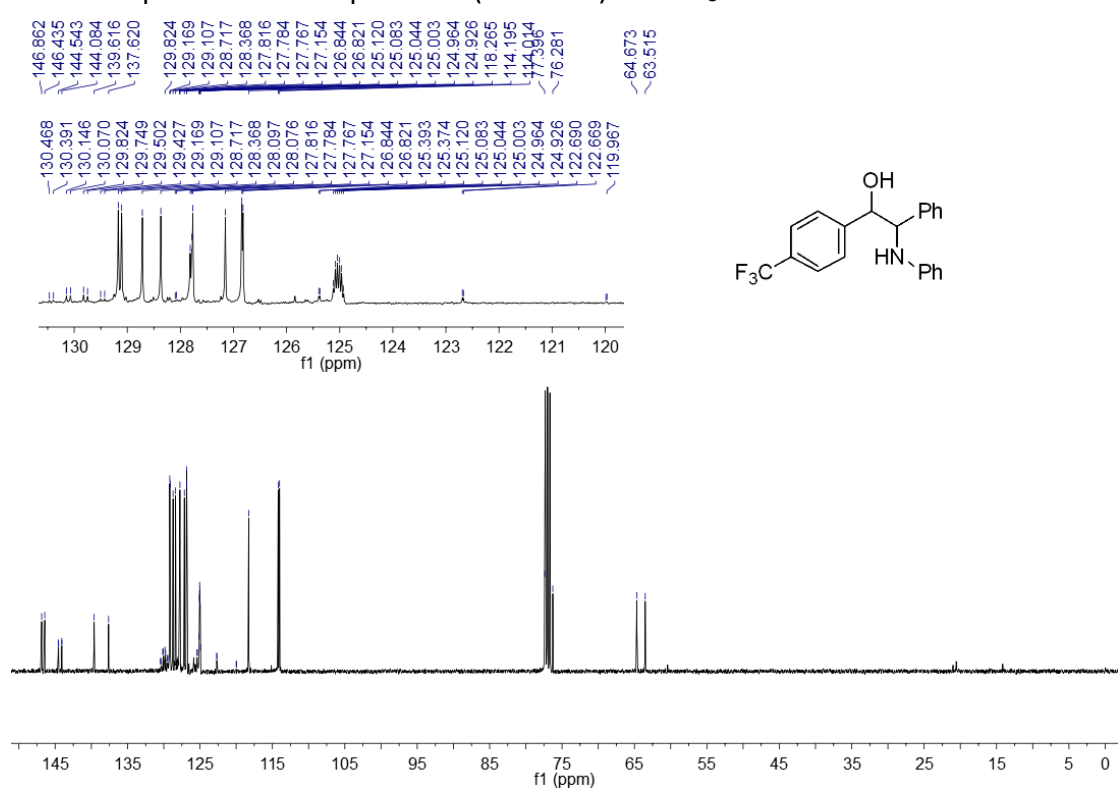
^{13}C NMR spectrum of compound **7h** (100 MHz) in CDCl_3



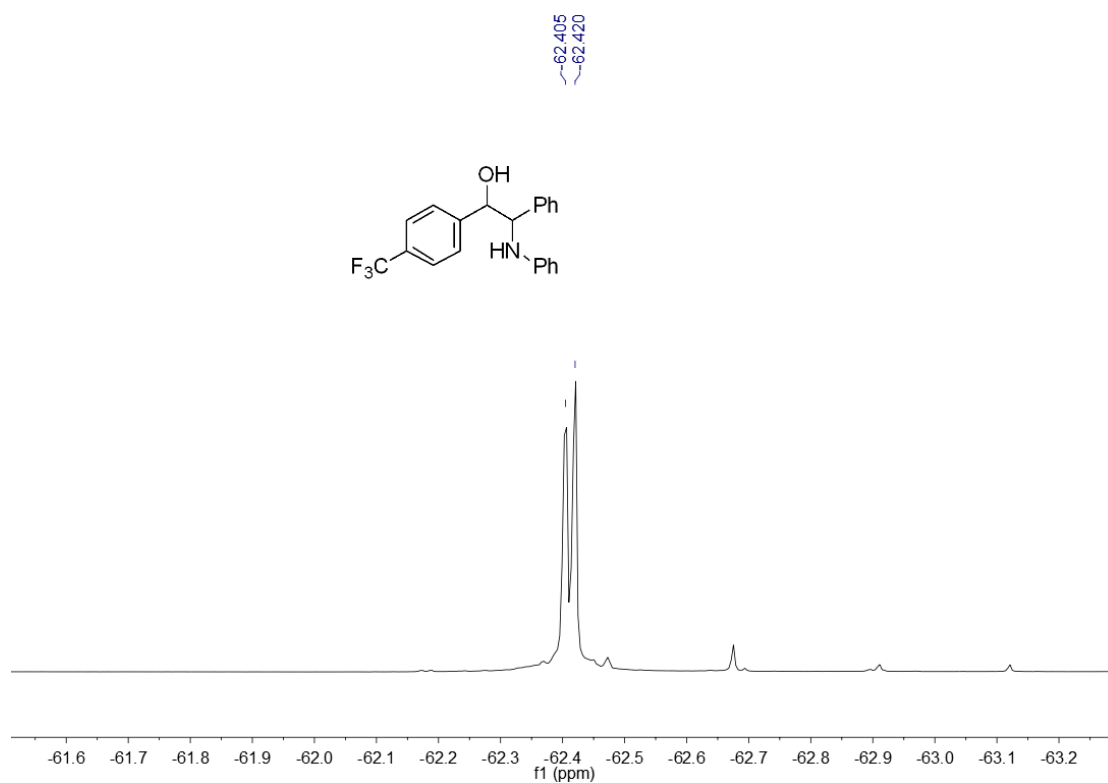
^1H NMR spectrum of compound **7i** (400 MHz) in CDCl_3



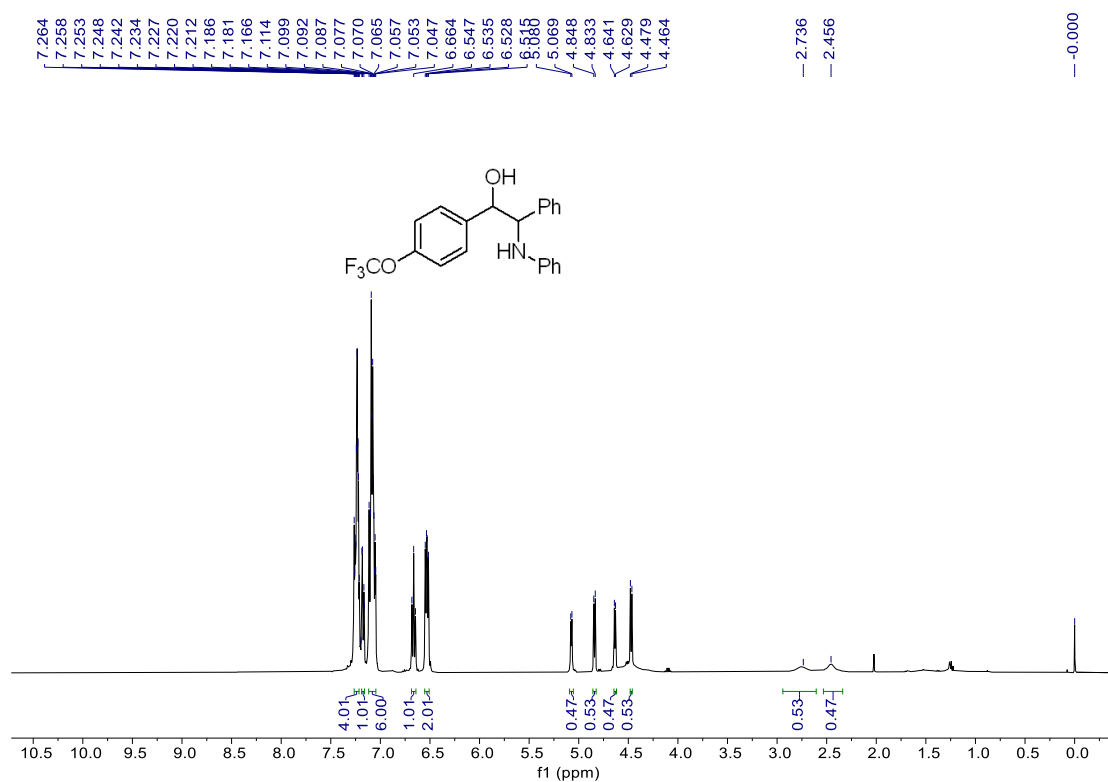
^{13}C NMR spectrum of compound **7i** (100 MHz) in CDCl_3



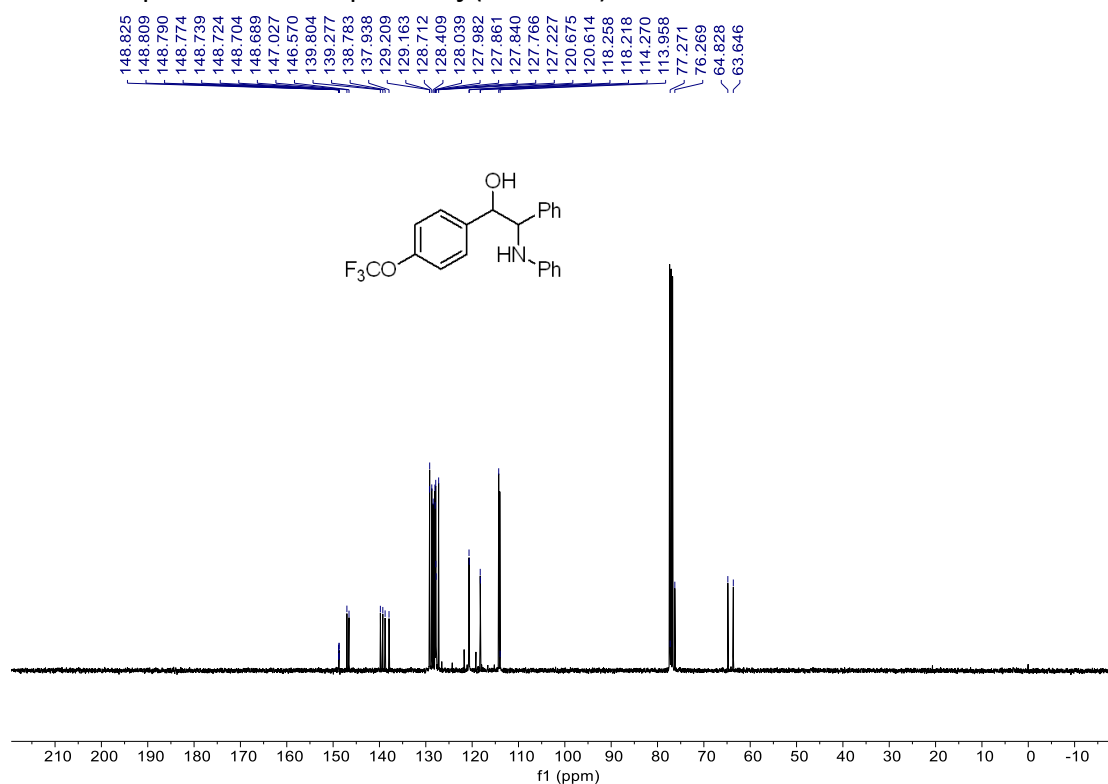
^{19}F NMR spectrum of compound **7i** (376 MHz) in CDCl_3



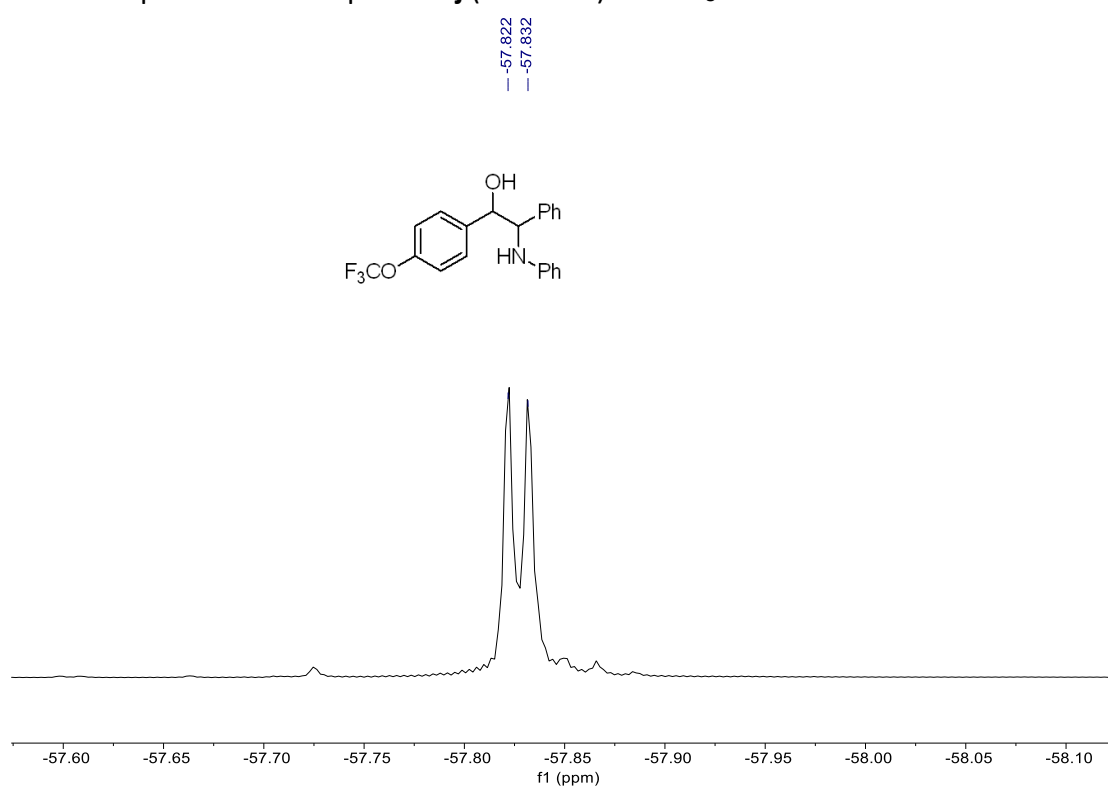
^1H NMR spectrum of compound **7j** (400 MHz) in CDCl_3



¹³C NMR spectrum of compound **7j** (100 MHz) in CDCl₃



¹⁹F NMR spectrum of compound **7j** (376 MHz) in CDCl₃



Chemical structure: Cc1ccc(cc1)C(O)(c2ccccc2)Nc3ccccc3

¹H NMR spectrum (ppm):

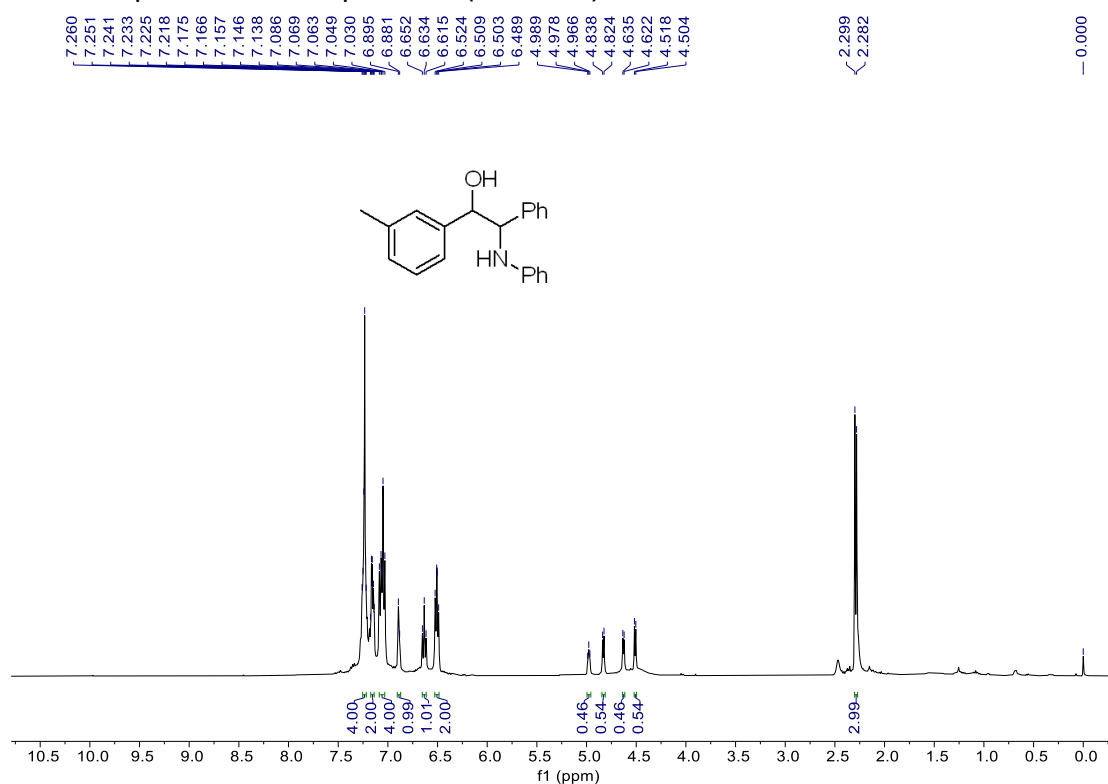
- 7.625, 7.609, 7.231, 7.227, 7.217, 7.214, 7.192, 7.181, 7.173, 7.088, 7.082, 7.075, 7.068, 7.064, 7.057, 7.054, 7.049, 7.046, 7.041, 7.036, 7.028, 7.022, 6.941, 6.922, 6.654, 6.635, 6.617, 6.524, 6.521, 6.518, 6.502, 6.499, 6.496, 5.289, 5.278, 5.267, 5.062, 5.046, 4.626, 4.615, 4.505, 4.490, 2.434, 2.426, 2.258, 2.203, 2.190, 1.960, -0.000

Integration values (from left to right): 0.47, 6.04, 4.02, 0.53, 0.99, 2.01, 0.48, 0.52, 0.48, 0.52, 0.52, 1.40, 0.48, 1.59.

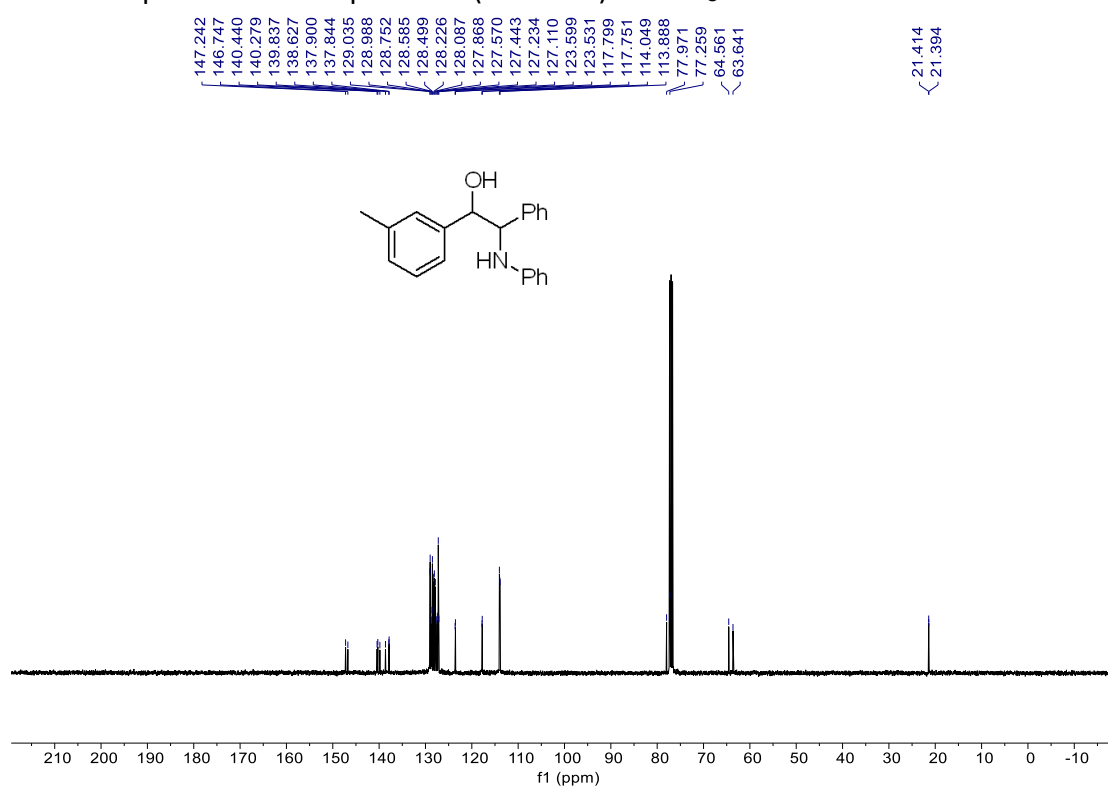
Cc1ccccc1C(O)C(Nc2ccccc2)c3ccccc3

¹³C NMR (CDCl₃) peaks (ppm): 147.335, 146.713, 140.181, 139.006, 138.310, 138.144, 135.434, 135.072, 130.362, 130.216, 129.060, 128.991, 128.451, 128.108, 128.078, 127.711, 127.671, 127.550, 127.429, 127.128, 126.306, 126.266, 126.137, 125.836, 117.751, 117.732, 114.087, 113.844, 74.050, 73.516, 63.781, 62.123, 19.164, 18.951.

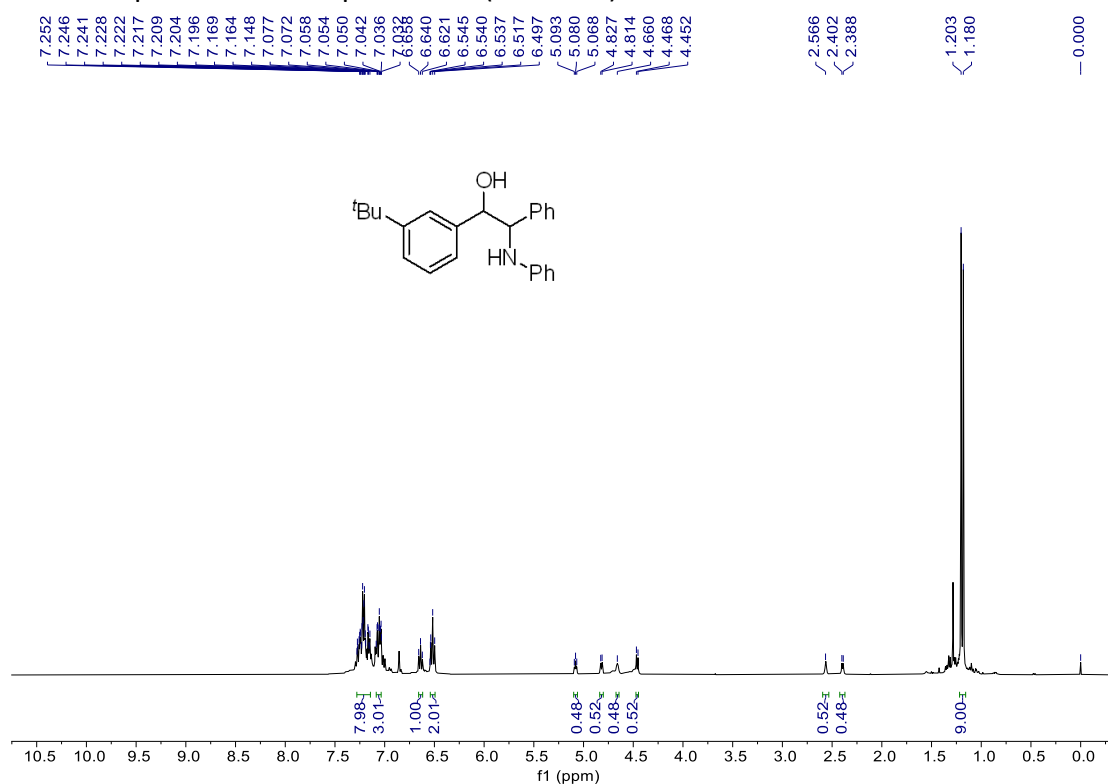
^1H NMR spectrum of compound **7l** (400 MHz) in CDCl_3



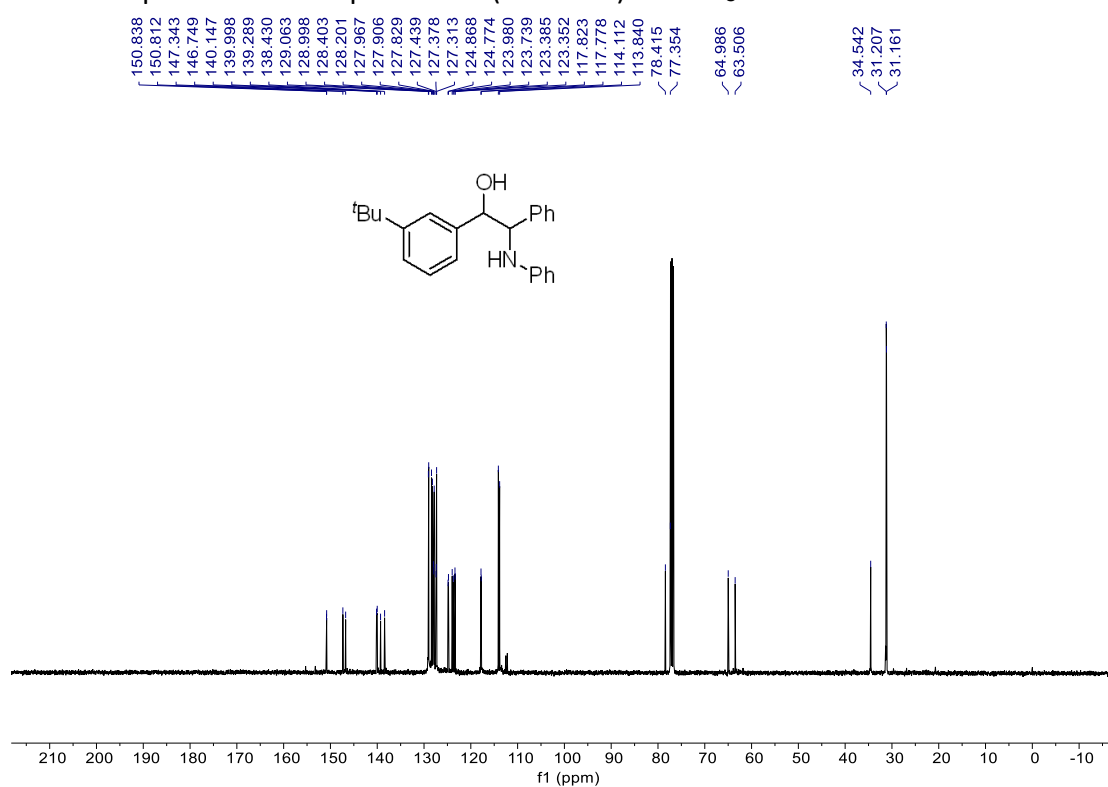
^{13}C NMR spectrum of compound **7l** (100 MHz) in CDCl_3



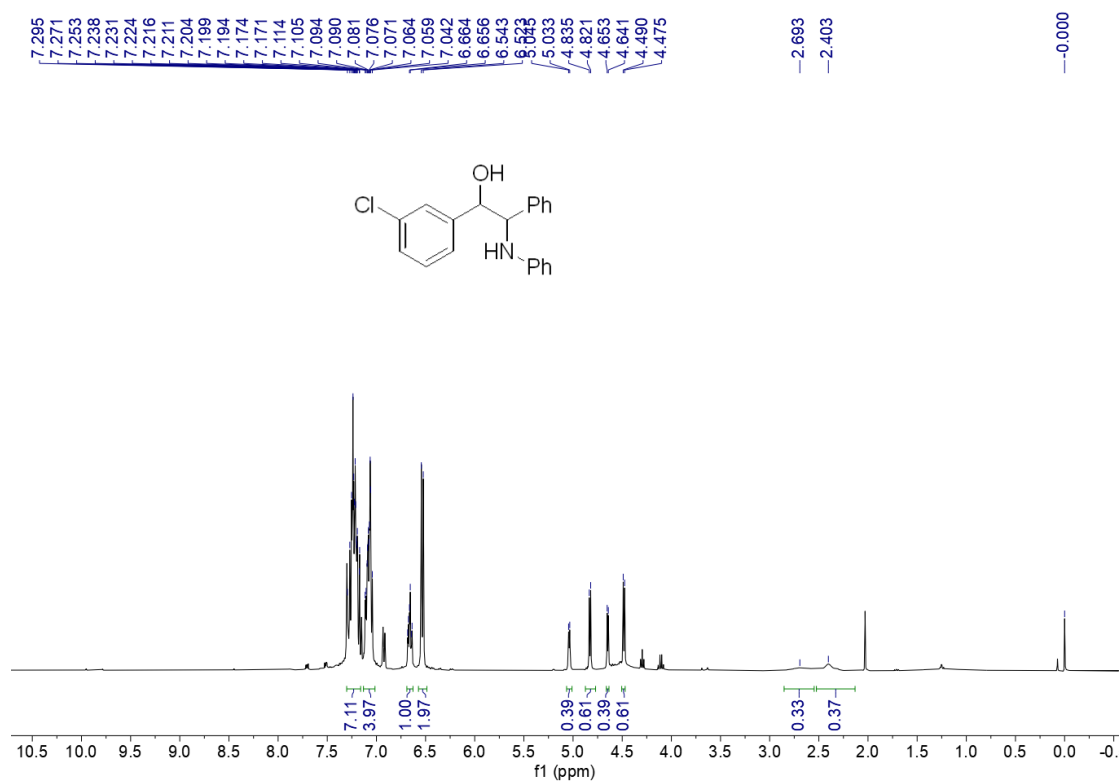
^1H NMR spectrum of compound **7m** (400 MHz) in CDCl_3



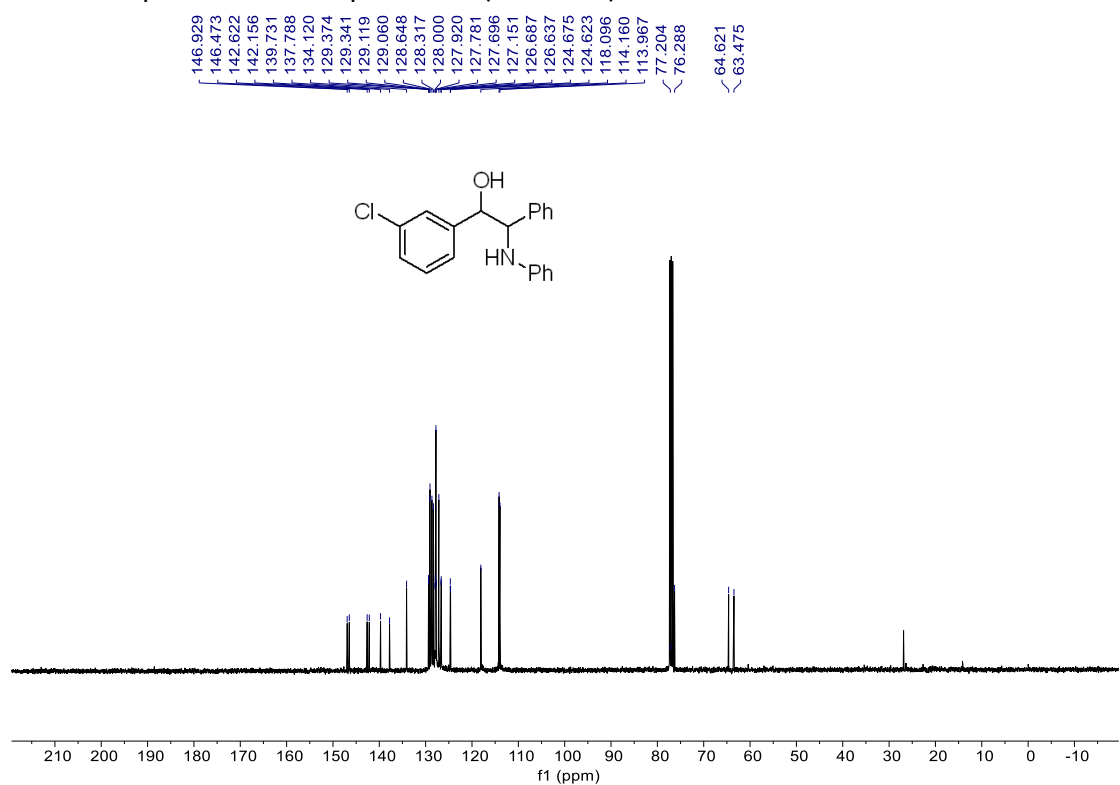
^{13}C NMR spectrum of compound **7m** (100 MHz) in CDCl_3



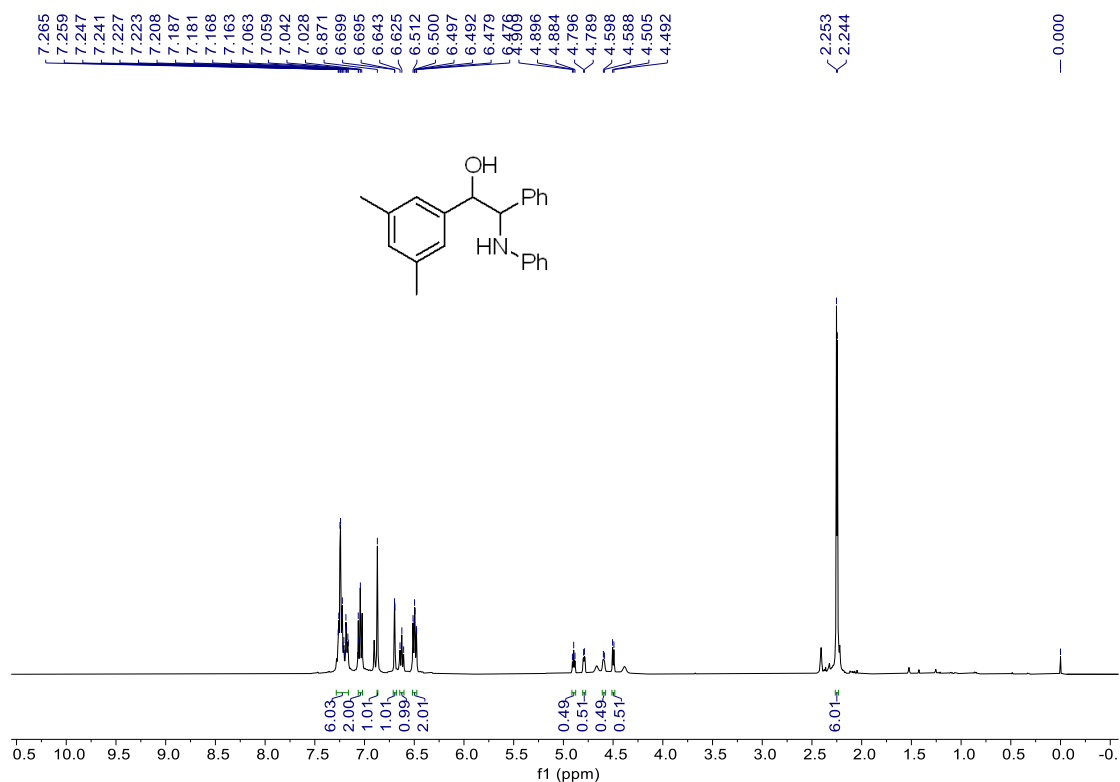
^1H NMR spectrum of compound **7n** (400 MHz) in CDCl_3



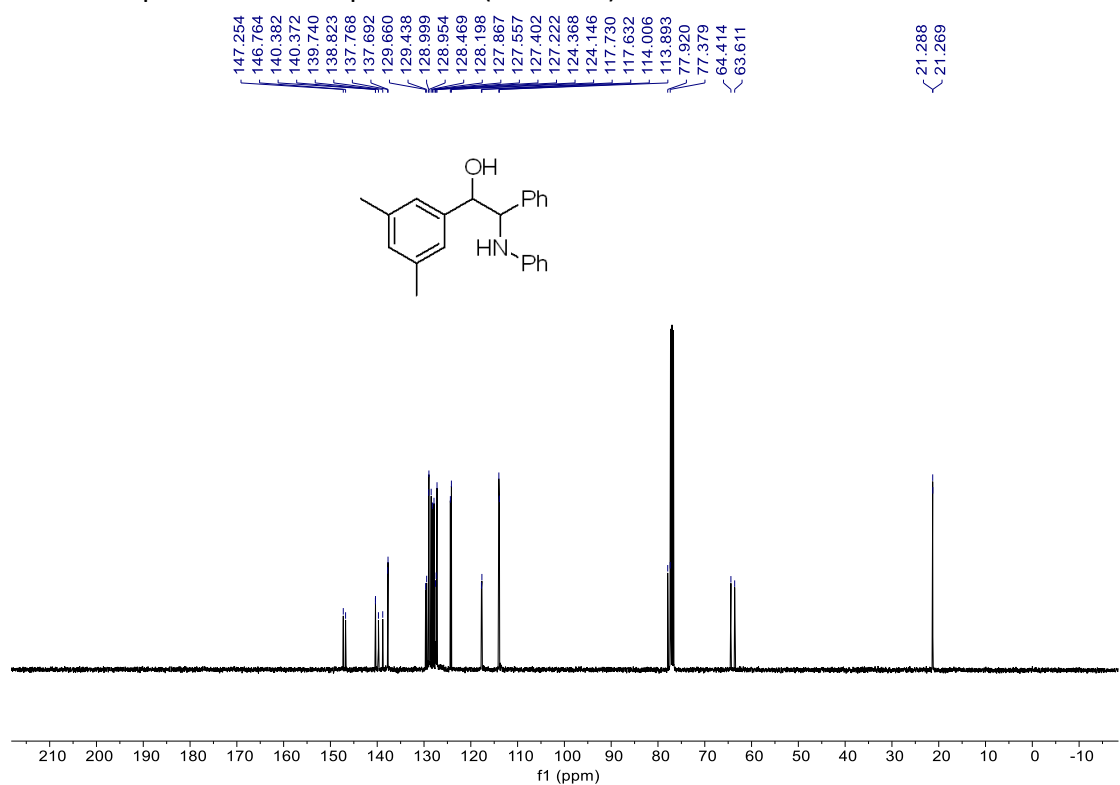
^{13}C NMR spectrum of compound **7n** (100 MHz) in CDCl_3



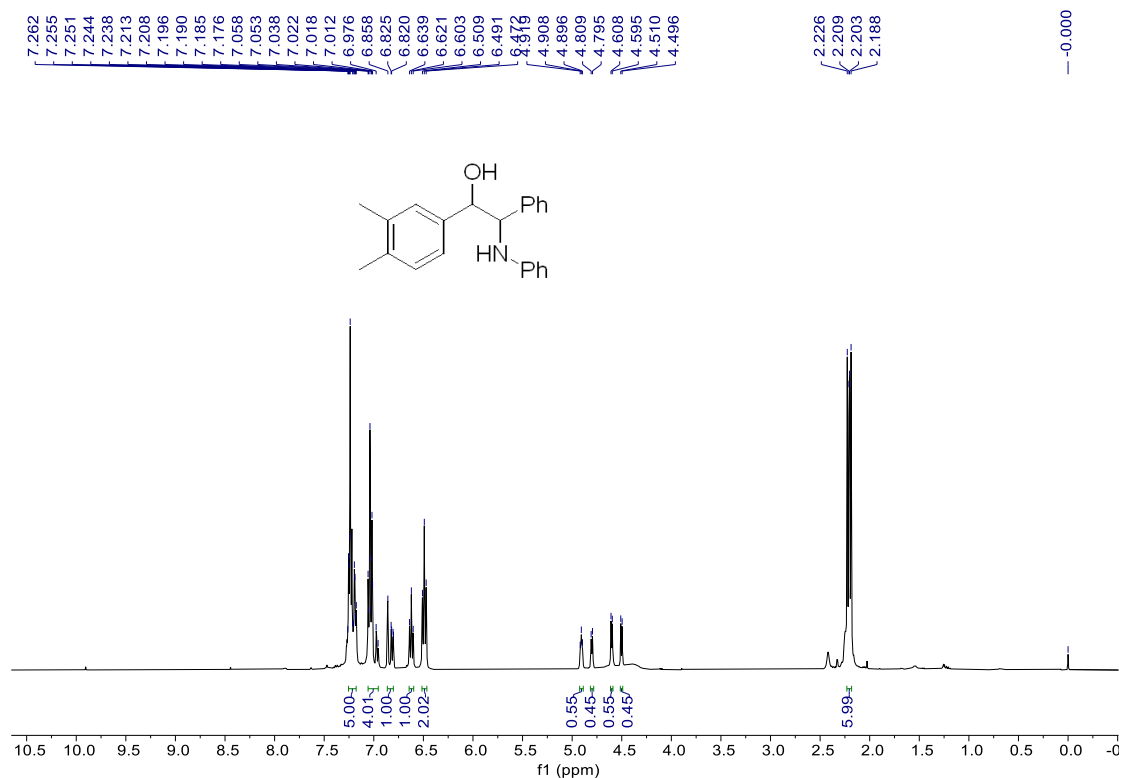
^1H NMR spectrum of compound **7o** (400 MHz) in CDCl_3



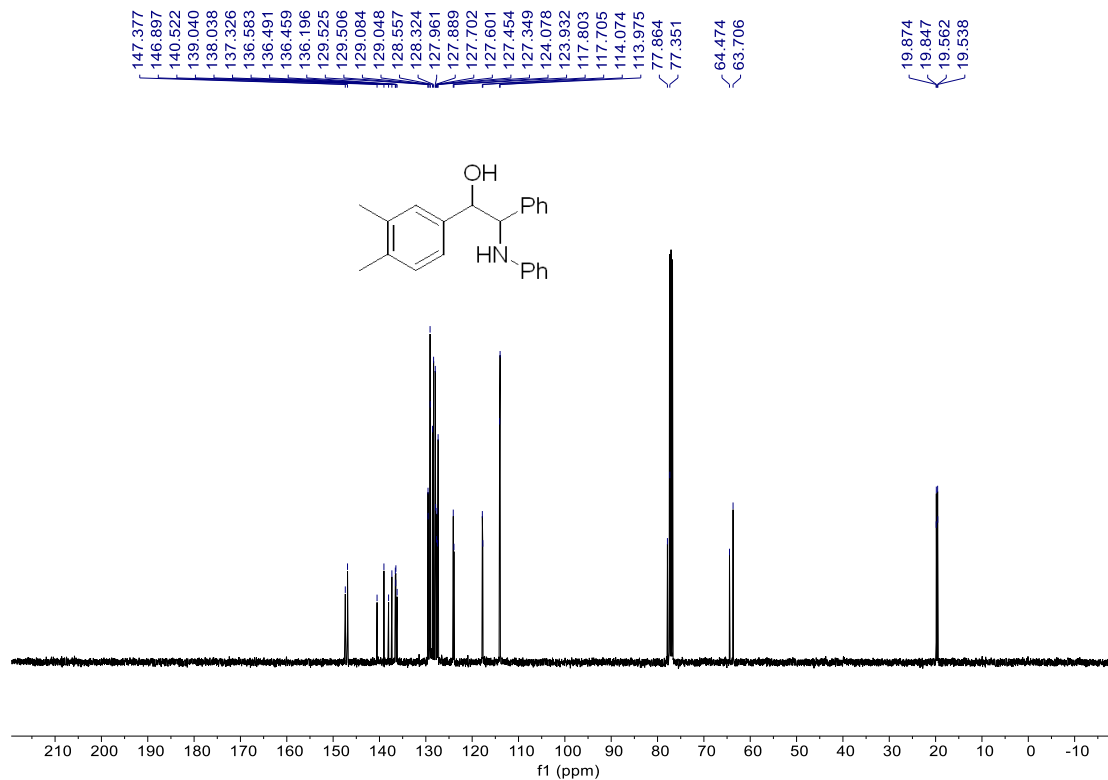
^{13}C NMR spectrum of compound **7o** (100 MHz) in CDCl_3



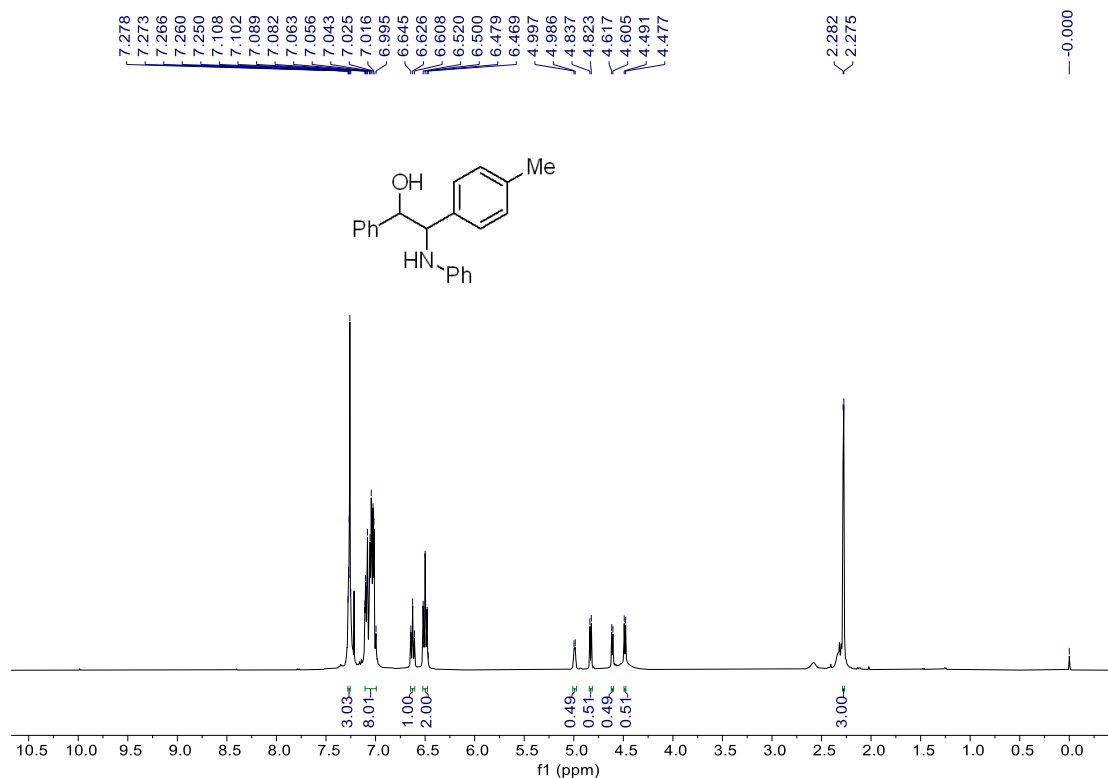
^1H NMR spectrum of compound **7p** (400 MHz) in CDCl_3



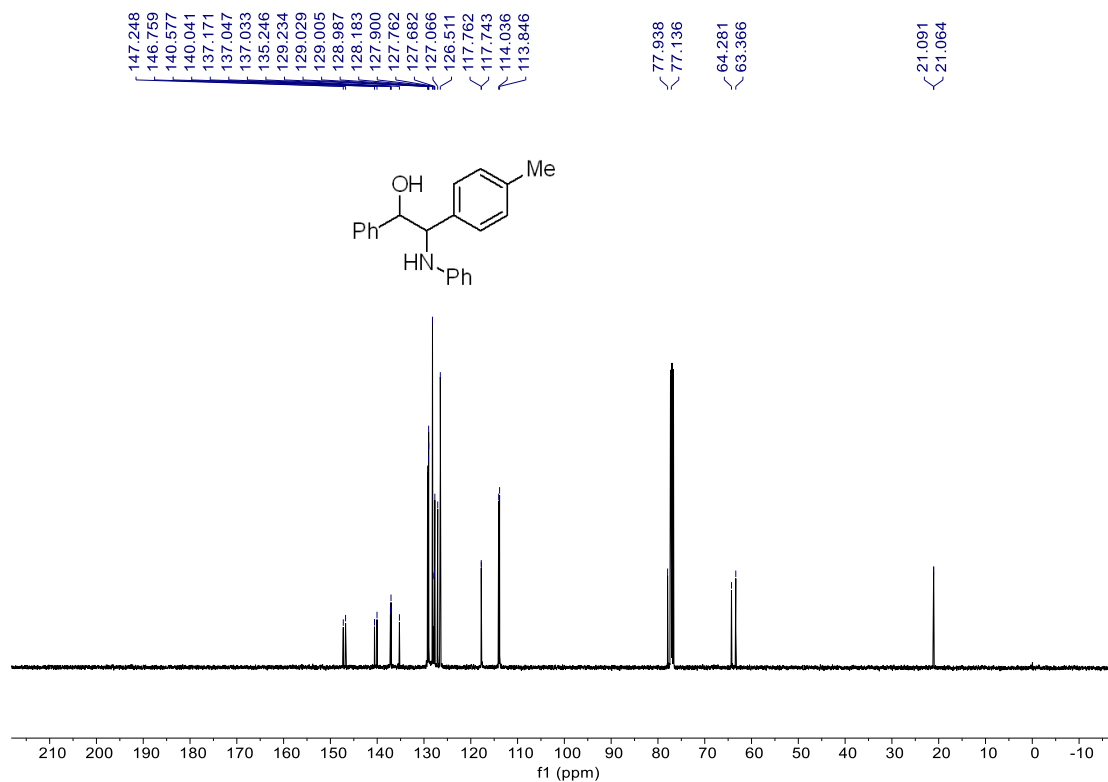
^{13}C NMR spectrum of compound **7p** (100 MHz) in CDCl_3



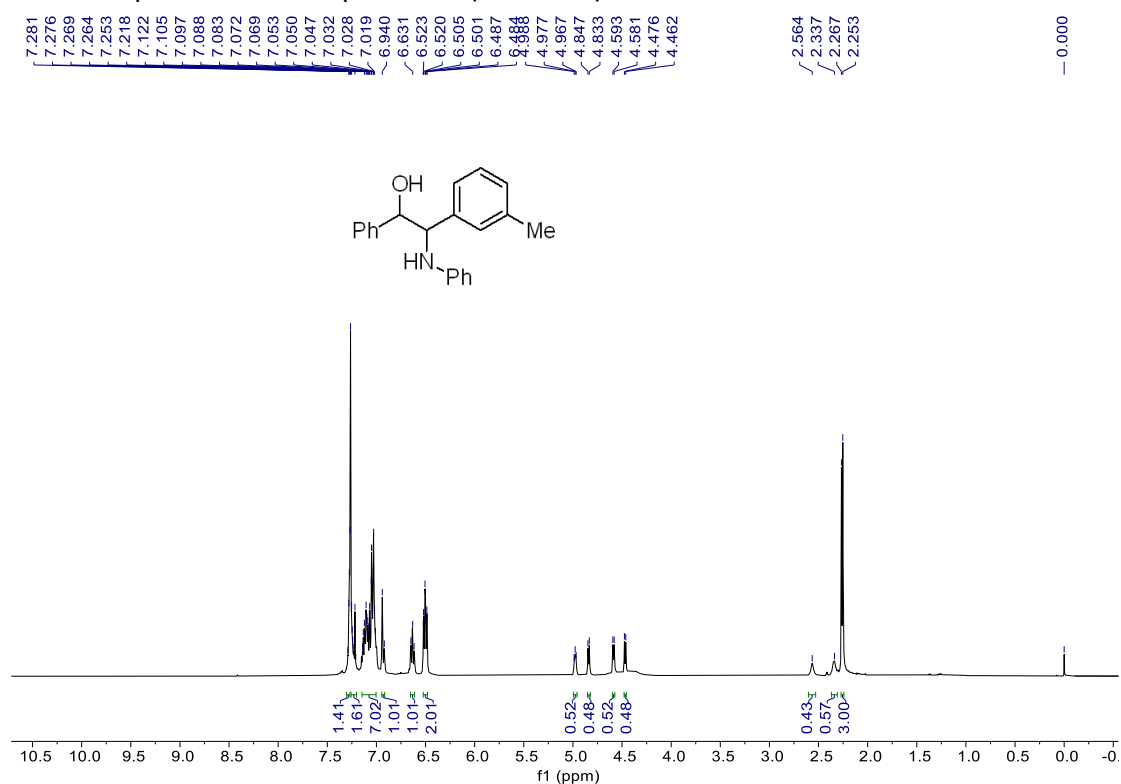
¹H NMR spectrum of compound **7q** (400 MHz) in CDCl₃



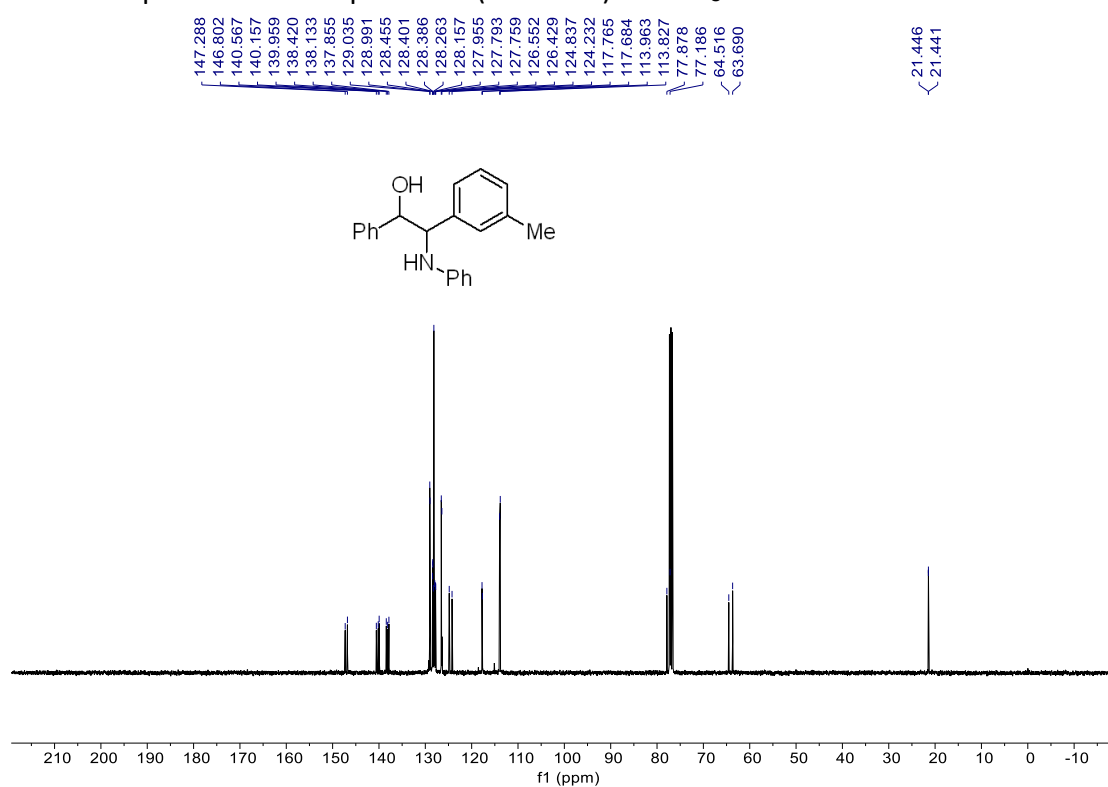
¹³C NMR spectrum of compound **7q** (100 MHz) in CDCl₃



¹H NMR spectrum of compound **7r** (400 MHz) in CDCl₃



¹³C NMR spectrum of compound **7r** (100 MHz) in CDCl₃



[illegible]

Chemical structure of the compound is shown above the spectrum:

CC1=CC=C(C=C1)C(NC(C2=CC=CC=C2)C(O)C3=CC=CC=C3)=CC=C1

The spectrum displays chemical shifts (f1) in ppm on the x-axis, ranging from -10 to 210. Key peaks are labeled with their corresponding chemical shift values:

- 147.234, 146.735, 140.503, 139.913, 138.390, 136.662, 136.549, 135.757, 130.646, 130.294, 129.098, 129.061, 128.136, 128.079, 127.715, 127.306, 126.906, 126.775, 126.443, 126.216, 126.096, 125.531, 117.813, 115.097, 113.800, 113.628, 76.644, 76.530, 60.406, 59.012, 19.275, 19.200.