

Metal-free Electrochemical Anti-Markovnikov Hydration of Aryl Alkenes

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Supporting Information

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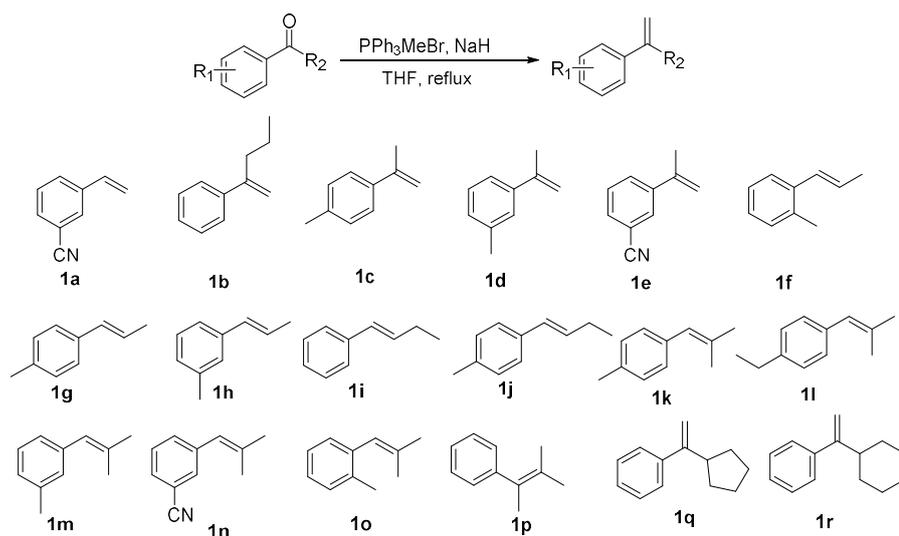
1. General Information

All solvents and reagents were obtained from commercial sources and were purified according to standard procedures before use (unless stated otherwise). Column chromatography was performed on silica gel (Qingdao, 300 - 400 mesh) using the indicated eluents. ^1H and ^{13}C NMR data were collected on a Varian Mercury 400 MHz or Agilent Mercury 600 MHz NMR spectrometer at room temperature using chloroform- d or DMSO- d_6 as a solvent and TMS as an internal standard, and chemical shift (δ) was expressed in parts per million (ppm). ^1H and ^{13}C NMR spectra were internally referenced to the proton (^1H) of the internal TMS signal at 0.00 ppm or the solvent residue of DMSO- d_6 at 2.50 ppm and the residual carbon nuclei (^{13}C) of the solvent at 77.0 or 39.5 ppm, respectively. The following abbreviations were used in expressing the multiplicity: s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet. High resolution mass spectra (HRMS-ESI) were recorded on a Bruker ESI-QTOF mass spectrometer. The course of the reactions was monitored by thin-layer chromatography (TLC).

2. Preparation of aryl alkenes

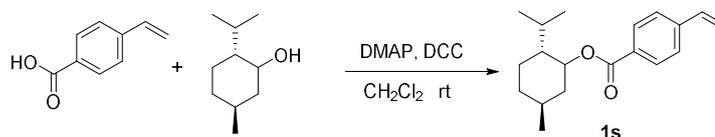
All of aryl alkenes are known compounds. Substrates **1a-1r** were prepared according to the published literature.¹ Other substrate were obtained from commercial sources or prepared according to known procedures as below:

Method A: To a solution of PPh₃MeBr (1.2 equiv., 4.8 mmol, 1.7 g) in THF (8 mL) was added NaH (60%, 1.1 equiv., 4.4 mmol, 105.6 mg), the reaction mixture was refluxed for 1 h. Then the corresponding ketones (4 mmol) in THF (2 mL) were added dropwise at 0 °C. The mixture was refluxed overnight. When the starting material was consumed (monitored by TLC), the reaction mixture was diluted by petroleum ether and filtered through a pad of silica gel. The filtrate was concentrated to give a crude product which was purified through flash column chromatography to obtain the desired product.

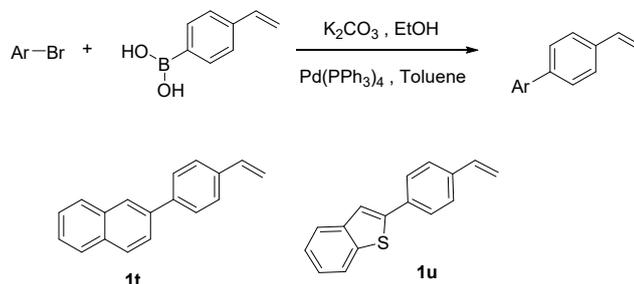


Method B: According to known procedures², the 4-vinylbenzoic acid (1.2 equiv., 6 mmol, 888.9 mg), DL-Menthol (1.0 equiv., 5 mmol, 781.4 mg), and 4-dimethylaminopyridine (5 mol %, 30.5 mg) were mixed in a flask, 30 mL CH₂Cl₂ was added. Then a solution of N, N'-dicyclohexylcarbodiimide (1.2 equiv., 6 mmol, 1237.9 mg) in CH₂Cl₂ (10 mL) was added slowly at room temperature. The reaction mixture was maintained at room temperature with stirring for 1-3 h, during which time the reaction mixture became cloudy and a white solid precipitated from the solution. The white solid was removed via vacuum filtration and the filtrate was

removed under reduced pressure. The residue was purified by flash column chromatography to give **1s**.



Method C: According to the published procedures³, a mixture of aryl bromide (3 mmol), 4-vinylphenylboronic acid (3.03 mmol, 448.3 mg), K_2CO_3 solution (2N, 3 mL), toluene (10 mL), ethanol (2.5 mL), $Pd(PPh_3)_4$ (1 mol%, 34.7 mg) were placed in a two-neck glass-reactor equipped with a magnetic stirring bar and reflux condenser. The suspension was heated in an oil bath at 85°C for 12 h. After the reaction was completed (monitored by TLC), the resulting mixture was transferred to a separatory funnel. The mixture was extracted with EtOAc (3 × 10 mL), and the resulting organic solution was washed with brine and dried over Na_2SO_4 . The solvent was evaporated under reduced pressure and the resulting residue was purified by silica gel chromatography (5% ethyl acetate in hexanes) provided the desired product **1t** or **1u**.



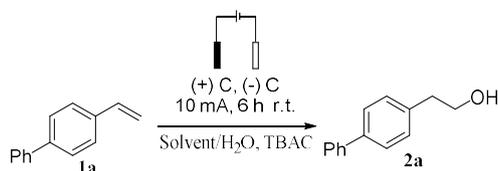
3. Optimization of the reaction conditions

3.1 Electrolysis general information

Electrochemical reactions were performed with ElectraSyn 2.0 package (IKA) using the constant current or constant voltage modes. The reactions were conducted in a 10 mL vial for 6 mL of solvent with a stir bar and a carbon graphite-SK-50 (5.0 × 0.8 × 0.2 cm) working electrode (anode and cathode) with a distance of 0.6 cm between the two electrodes.



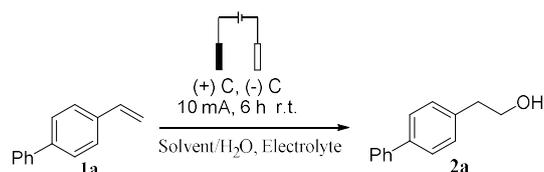
3.2 Screening of solvents, amounts of water ^a



Entry	Solvent	Electrolyte	H ₂ O	2a Yield (%)
1	TBAC	DMA	36 μL	36
2	TBAC	DMSO	36 μL	-
3	TBAC	DMF	36 μL	54
4	TBAC	DMF	108 μL	49
5	TBAC	DMF	500 μL	51
6 ^b	TBAC	DMF	1 mL	52
7 ^c	TBAC	DMF	3 mL	22

^a Conditions: undivided cell, carbon cloth anode and cathode, **1a** (0.2 mmol), TBAC (0.2 mmol), Solvent (6 mL), H₂O, CCE = 10 mA, T = 6 h, RT, under air. Yields were isolated yields. CCE = constant current electrolysis. ^b 5 mL DMF was used. ^c 3 mL DMF was used.

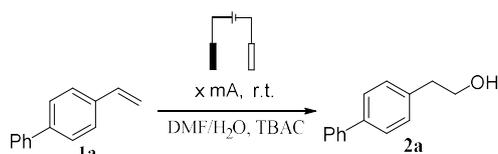
3.3 Screening of electrolyte ^a



Entry	Solvent	Electrolyte	H ₂ O (μL)	2a Yield (%)
1	DMF	ⁿ Bu ₄ NBr	36	22
2	DMF	ⁿ Bu ₄ NI	36	13
3	DMF	TBAC	36	58
4	DMF	NH ₄ Cl	36	-
5	DMF	ⁿ Bu ₄ NBF ₄	36	-
6 ^b	DMF	ⁿ Bu ₄ NBF ₄	36	28
7	DMF	(CH ₃) ₄ NCl	36	45
8	DMF	(C ₂ H ₅) ₄ NCl	36	44
9	DMF	(C ₃ H ₇) ₄ NCl	36	41
10	DMF	C ₁₆ H ₃₃ (CH ₃) ₃ NCl	36	55
11 ^c	DMF	TBAC	36	51
12 ^d	DMF	TBAC	36	54
13 ^e	DMF	TBAC	36	58

^a Conditions: undivided cell, carbon-cloth anode and cathode, **1a** (0.2 mmol), Electrolyte (0.4 mmol), DMF (6 mL), H₂O (36 μL), CCE = 10.0 mA, 6 h, RT, under air. Yields were isolated yields. CCE = constant current electrolysis. ^b 0.2 mmol NaCl was used. ^c 0.1 mmol TBAC was used. ^d 0.2 mmol TBAC was used. ^e 0.6 mmol TBAC was used.

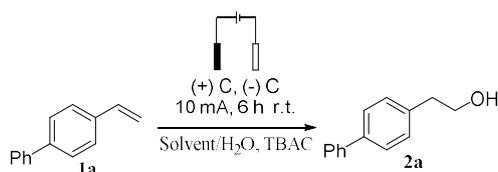
3.4 Screening of electrodes, electric current ^a



Entry	electrode	CCE (x mA)	T (h)	2a Yield (%)
1	(+)C, (-)C	5 mA	12	34
2	(+)C, (-)C	10 mA	6	58
3	(+)C, (-)C	20 mA	3	50
4	(+)C, (-)Pt	10 mA	6	51

^a Conditions: undivided cell, carbon-cloth anode and cathode, **1a** (0.2 mmol), TBAC, (0.4 mmol) DMF (6 mL), H₂O (36 μL), CCE, T, RT, under air. Yields are based on product isolated by column chromatography. CCE = constant current electrolysis.

3.5 Screening of additives ^a

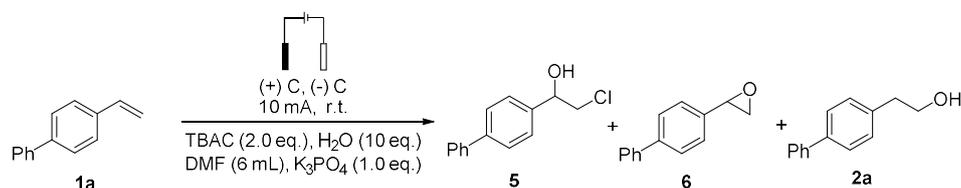


Entry	Electrolyte	Additives	H ₂ O (μL)	2a Yield (%)
1	TBAC	Bu ₄ NHSO ₄	36	39
2	TBAC	KH ₂ PO ₄	36	44
3	TBAC	K ₃ PO ₄	36	65
4	TBAC	K ₂ CO ₃	36	45
5	TBAC	t-BuOK	36	41
6	TBAC	KOH	36	63
7	TBAC	Na ₃ PO ₄	36	56
8	TBAC	NaOH	36	42
9	TBAC	AcONa	36	64
10	TBAC	CH ₃ CH ₂ ONa	36	30
11	TBAC	AcOK	36	39
12	TBAC	TFA	36	-
13	TBAC	Et ₃ N	36	-
14	TBAC	K ₂ HPO ₄	36	63
15	TBAC	(PhO) ₂ P(O)OH	36	26
16	TBAC	H ₃ PO ₄	36	16
17	TBAC	HCl	36	14
18 ^b	TBAC	K ₃ PO ₄	36	45
19 ^c	TBAC	K ₃ PO ₄	36	49

^a Conditions: undivided cell, carbon-cloth anode and cathode, **1a** (0.2 mmol), TBAC (0.4 mmol), DMF (6 mL), H₂O (36 μL), Additives (0.2 mmol), CCE = 10.0 mA, 6h, RT, under air. Yields are based on product isolated by column chromatography. CCE = constant current electrolysis. ^b 0.1 mmol K₃PO₄ was used. ^c 0.4 mmol K₃PO₄ was used.

4. Mechanistic studies

4.1 Kinetic experiments



Reaction procedure:

A mixture of **1a** (0.2 mmol, 36.1 mg), K₃PO₄ (0.2 mmol, 42.5 mg) and TBAC (0.4 mmol, 111.2 mg) were added in a 10 mL vial with a stir bar, then DMF (6 mL), H₂O (36 μ L) were added under air atmosphere. The vial was covered with the electrode holder. The electrolysis was carried out at RT using a constant current of 10.0 mA between a carbon graphite anode and carbon graphite cathode (1.4 x 0.8 x 0.2 cm submerged in solution) with stirring. The reactions were stopped respectively at 0.5 h, 1 h, 2 h, 4 h, 6 h. The reaction mixture was diluted with EtOAc. The organic phase was washed with brine, dried over Na₂SO₄ and concentrated under reduced pressure. Yields were determined by ¹H NMR with TTCE as internal standard. The plots of the percentage yield of the product displayed in Figure S2.

Entry	<i>t</i> (h)	Yield 5 (%)	Yield 6 (%)	Yield 2a (%)	Recoverd yield 1a (%)
1	0.5	6	19	13	52
2	1	4	18	28	37
3	2	3	12	49	4
4	4	2	7	61	1
5	6	0	0	74	0

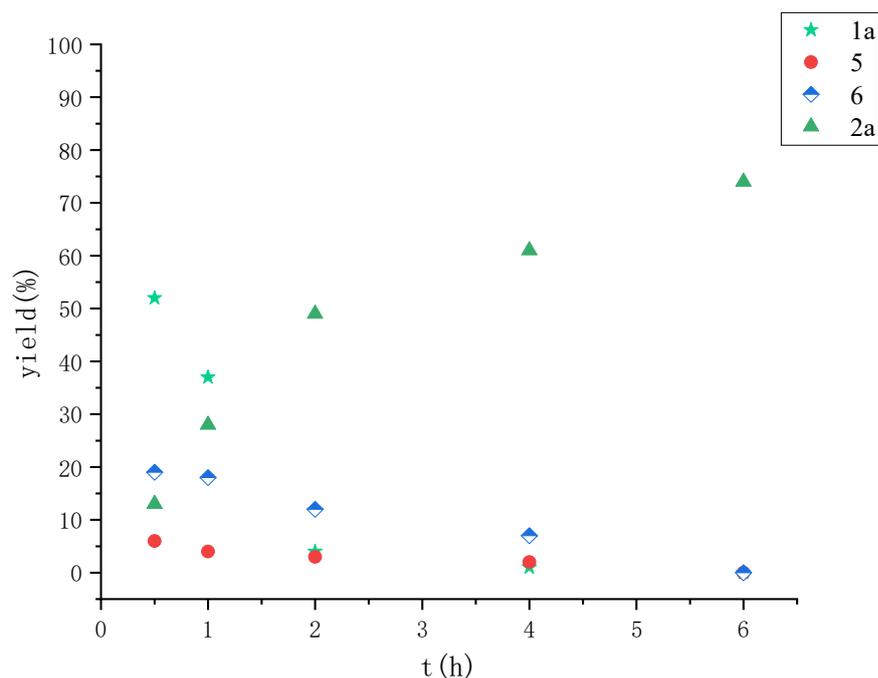
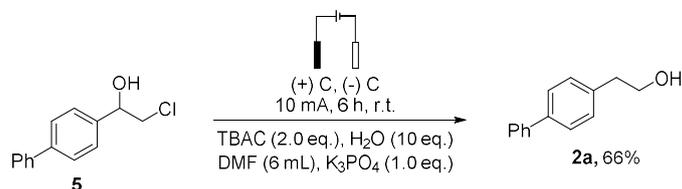
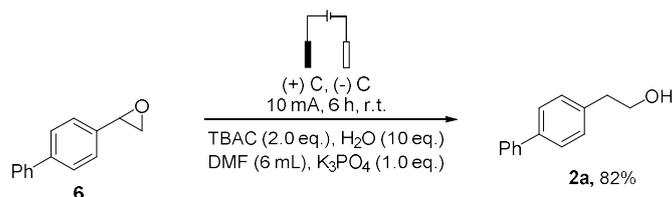


Figure S2 Results of kinetic experiments

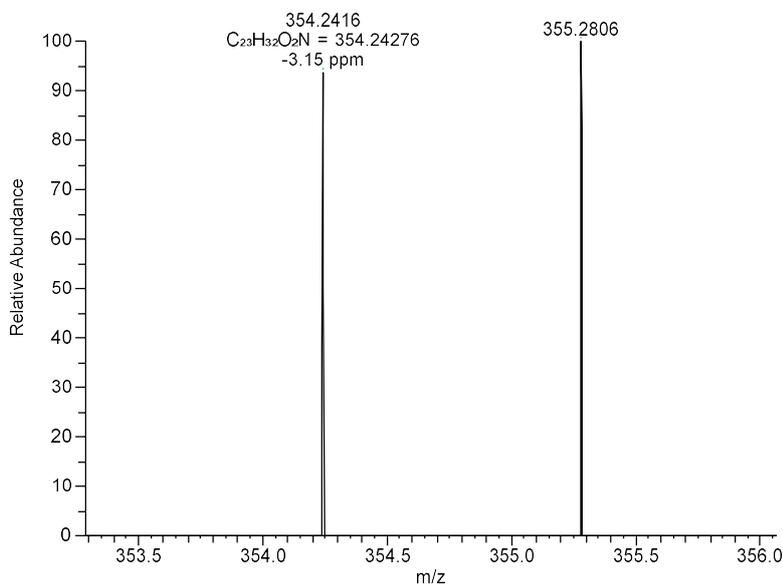
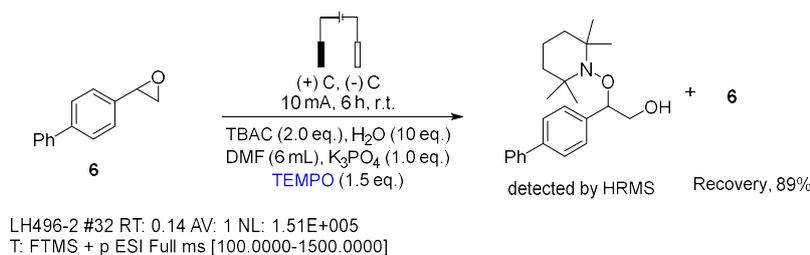
4.2 Control experiments



To a 10 mL flask, equipped with a magnetic stir bar and two graphite rods ($\Phi = 5$ mm) as anode and cathode, was added **5** (0.2 mmol, 46.5 mg), TBAC (0.4 mmol, 111.2 mg), K_3PO_4 (0.2 mmol, 42.5 mg), DMF (6ml), H_2O (10.0 equiv.). The mixture was stirred at room temperature and reacted at a constant current of 10 mA for 6 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent: Petroleum ether/ethyl acetate = 5:1, v/v) to afford. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel to afford **2a** (26.1 mg, 66% yield).

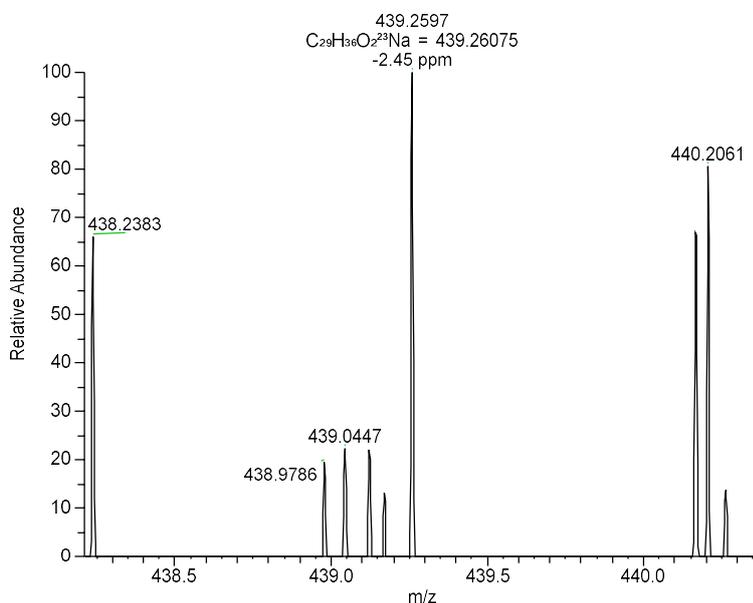
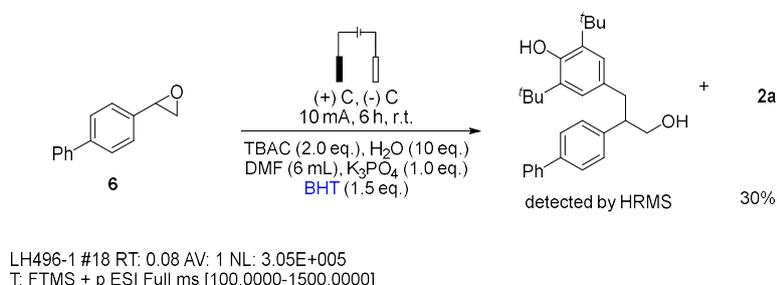


To a 10 mL flask, equipped with a magnetic stir bar and two graphite rods ($\Phi = 5$ mm) as anode and cathode, was added **6** (0.2 mmol, 39.3 mg), TBAC (0.4 mmol, 111.2 mg), K_3PO_4 (0.2 mmol, 42.5 mg), DMF (6 mL), H_2O (10.0 equiv.). The mixture was stirred at room temperature and reacted at a constant current of 10 mA for 6 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent: Petroleum ether/ethyl acetate = 5:1, v/v) to afford. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel to afford **2a** (32.5 mg, 82% yield).



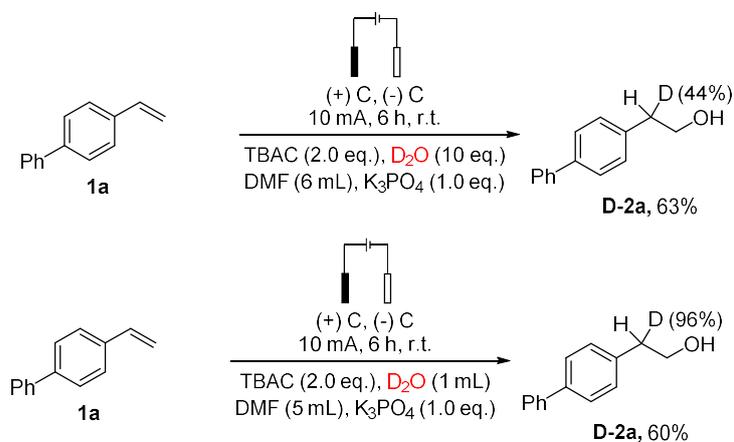
To a 10 mL flask, equipped with a magnetic stir bar and two graphite rods ($\Phi = 5$ mm) as anode and cathode, was added **6** (0.2 mmol, 39.3 mg), TBAC (0.4 mmol, 111.2 mg), K_3PO_4 (0.2 mmol, 42.5 mg), DMF (6 mL), H_2O (10.0 equiv.), TEMPO

(46.8 mg 1.5 equiv.). The mixture was stirred at room temperature and reacted at a constant current of 10 mA for 6 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent: Petroleum ether/ethyl acetate = 5:1, v/v) to afford. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel to recovery **6** (34.9 mg, 89% yield). We further investigated the possible intermediates generated in the reaction process by the use of high resolution mass spectrometry (HRMS) analysis, and the corresponding TEMPO adduct could be clearly detected. **HRMS** (ESI) m/z : $[M + H]^+$ Calcd for $C_{23}H_{32}O_2N$ 354.2428; Found 354.2416.



To a 10 mL flask, equipped with a magnetic stir bar and two graphite rods ($\Phi = 5$ mm) as anode and cathode, was added **6** (0.2 mmol, 39.3 mg), TBAC (0.4 mmol, 111.2 mg), K_3PO_4 (0.2 mmol, 42.5 mg), DMF (6 mL), H_2O (10.0 equiv.), BHT (66.0 mg 1.5 equiv.). The mixture was stirred at room temperature and reacted at a constant

current of 10 mA for 6 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent: Petroleum ether/ethyl acetate = 5:1, v/v) to afford. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel to afford **2a** (11.9 mg, 30% yield). We further investigated the possible intermediates generated in the reaction process by the use of high resolution mass spectrometry (HRMS) analysis, and the corresponding BHT adduct could be clearly detected. **HRMS** (ESI) m/z : $[M + Na]^+$ Calcd for $C_{29}H_{36}O_2Na$ 439.2608; Found 439.2597.



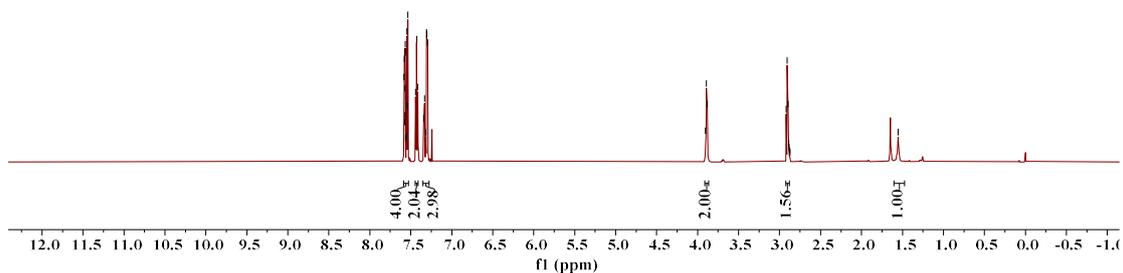
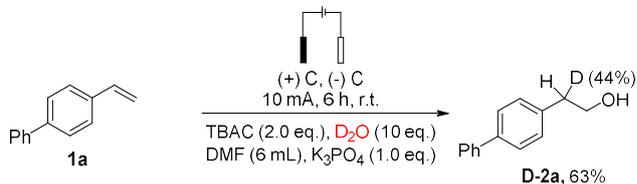
To a 10 mL flask, equipped with a magnetic stir bar and two graphite rods ($\Phi = 5$ mm) as anode and cathode, was added **1a** (0.2 mmol, 36.1 mg), TBAC (0.4 mmol, 111.2 mg), K_3PO_4 (0.2 mmol, 42.5 mg), DMF (6 or 5 mL), D_2O (36 μ L. or 1 mL), the mixture was stirred at room temperature and reacted at a constant current of 10 mA for 6 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent: Petroleum ether/ethyl acetate = 5:1, v/v) to afford. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel to afford **D-2a**.

7.582
7.580
7.577
7.571
7.570
7.568
7.566
7.563
7.553
7.549
7.546
7.539
7.535
7.441
7.428
7.415
7.344
7.332
7.320
7.307
7.306
7.293

3.904
3.893
3.882
2.918
2.907
2.896
2.888
2.885
2.877
2.874

— 1.554

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Nucleus	1H



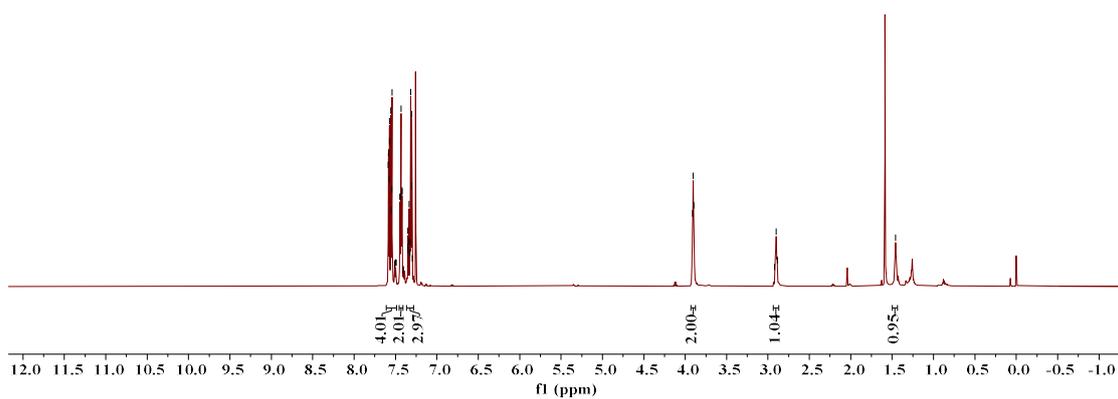
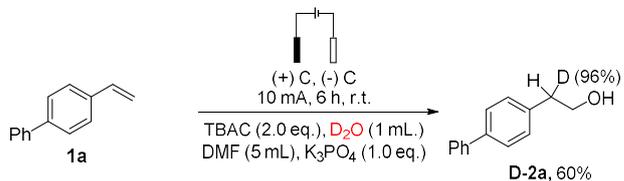
7.587
7.584
7.575
7.573
7.571
7.556
7.553
7.545
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7.492
7.446
7.436
7.433
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7.337
7.327
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7.316
7.305
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7.296

3.912
3.903
3.894

2.919
2.900
2.886

— 1.459

Parameters	
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Solvent	CDCl3
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Number of Scans	4
Acquisition Date	2025-03-04 12:11:35
Spectrometer Frequency	600 MHz
Nucleus	1H



4.3. General procedure for cyclic voltammetry (CV)

Cyclic voltammetry was performed with ElectroSyn 2.0 package (IKA). A glassy carbon disc (diameter 3 mm) working electrode, a platinum wire counter electrode and a saturated silver chloride electrode as reference electrode were used at scan rate of 400 mV/s. The experiments were conducted in a 10 mL vial without stirring in CH₃CN (5 mL) with **1a** (0.025 M) and/or TBAC (0.025 M), K₃PO₄ (0.025 M), H₂O (36 μL) and Et₄NBF₄ (0.025 M) at 25 °C.

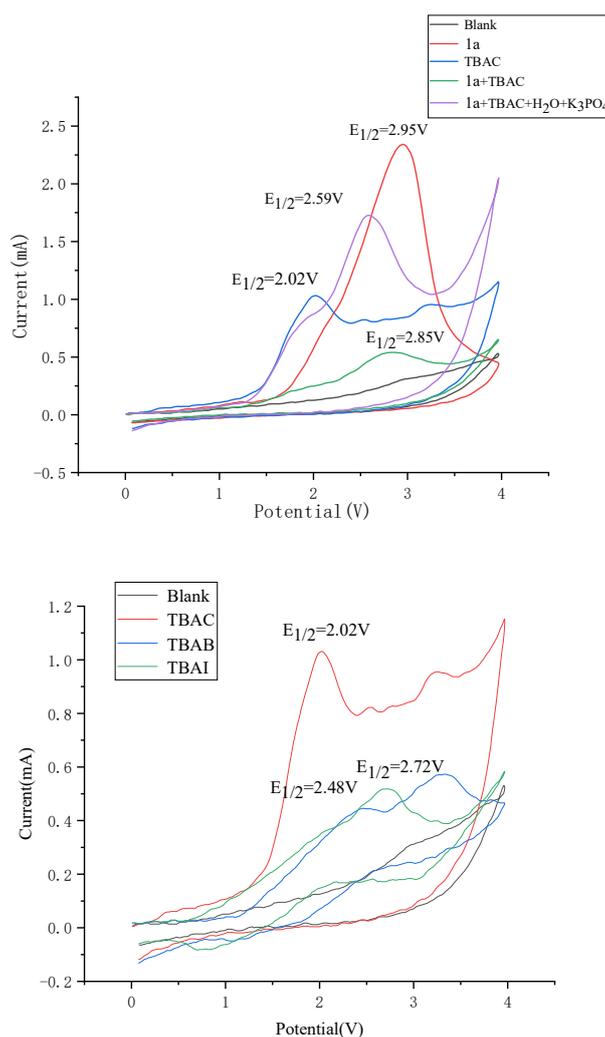
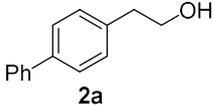


Figure S2. Cyclic voltammogram of Et₄NBF₄ (0.025 M) in CH₃CN

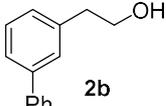
5. General procedure for 3-5 and product characterizations

To a 10 mL flask, equipped with a magnetic stir bar and two graphite rods ($\Phi = 5$ mm) as anode and cathode, was added aryl alkenes (0.2 mmol), TBAC (0.4 mmol, 111.2 mg), K_3PO_4 (0.2 mmol, 42.5 mg), DMF (6 mL), H_2O (10.0 equiv.), the mixture was stirred at room temperature and reacted at a constant current of 10 mA for 6 h. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel (eluent: Petroleum ether/ethyl acetate = 5:1, v/v) to afford. The reaction mixture was concentrated under reduced pressure and purified by column chromatography on silica gel to afford products.

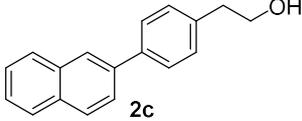
2-([1,1'-biphenyl]-4-yl) ethan-1-ol (2a)

 white solid, 6 h, 25.9 mg, 65% yield; 1H NMR (600 MHz, Chloroform-*d*) δ 7.60 (dd, $J = 22.8, 7.8$ Hz, 4H), 7.47 (t, $J = 7.8$ Hz, 2H), 7.38 (t, $J = 7.4$ Hz, 1H), 7.33 (d, $J = 7.7$ Hz, 2H), 3.91 (t, $J = 6.6$ Hz, 2H), 2.93 (t, $J = 6.6$ Hz, 2H), 1.87 (s, 1H). The spectral data is consistent with the literature.⁴

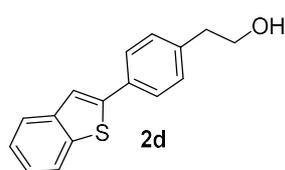
2-([1,1'-biphenyl]-3-yl) ethan-1-ol (2b)

 white solid, 6 h, 20.4 mg, 51% yield; 1H NMR (600 MHz, Chloroform-*d*) δ 7.37 – 7.32 (m, 2H), 7.28 – 7.25 (m, 3H), 3.90 (t, $J = 6.6$ Hz, 2H), 2.90 (t, $J = 6.6$ Hz, 2H), 1.62 (s, 1H). ppm. The spectral data is consistent with the literature.⁵

2-(4-(naphthalen-2-yl) phenyl) ethan-1-ol (2c)

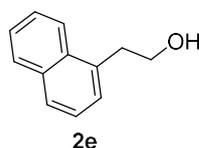
 white solid, 6 h, 27.3 mg, 55% yield; 1H NMR (600 MHz, Chloroform-*d*) δ 8.06 (s, 1H), 7.95 – 7.89 (m, 3H), 7.77 (dd, $J = 8.4, 1.8$ Hz, 1H), 7.72–7.71 (m, 2H), 7.55 – 7.51 (m, 2H), 7.40 – 7.38 (m, 2H), 3.96 (t, $J = 6.6$ Hz, 2H), 2.98 (t, $J = 6.4$ Hz, 2H), 1.53 (s, 1H). ppm. ^{13}C NMR (151 MHz, Chloroform-*d*) δ 139.4, 138.3, 137.7, 133.7, 132.6, 129.6, 128.4, 128.2, 127.7, 127.6, 126.3, 125.9, 125.6, 125.5, 63.7, 38.9. ppm. HRMS (ESI) m/z : $[M + Na]^+$ + Calcd for $C_{18}H_{16}ONa$ 271.1086; Found 271.1093.

2-(4-(benzo[b]thiophen-2-yl) phenyl) ethan-1-ol (2d)



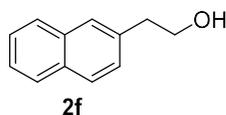
white solid, 6 h, 11.4 mg, 22% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.83 (d, $J = 7.8$ Hz, 1H), 7.77 (d, $J = 8.8$ Hz, 1H), 7.67 (d, $J = 8.2$ Hz, 2H), 7.52 (s, 1H), 7.37 (t, $J = 7.2$ Hz, 1H), 7.32 – 7.30 (m, 3H), 3.91 (t, $J = 6.6$ Hz, 2H), 2.92 (t, $J = 6.6$ Hz, 2H), 1.56 (s, 1H) ppm. $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 144.1, 140.7, 139.4, 138.9, 132.7, 129.6, 126.7, 124.5, 124.2, 123.5, 122.3, 119.2, 63.6, 38.9. ppm. HRMS (ESI) m/z : $[M + H]^+$ Calcd for $\text{C}_{16}\text{H}_{15}\text{OS}$ 255.0839; Found 255.0838.

2-(naphthalen-1-yl) ethan-1-ol (2e)



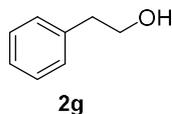
Colorless oil, 6 h, 18.4 mg, 53% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 8.05 (d, $J = 8.4$ Hz, 1H), 7.86 (d, $J = 7.8$ Hz, 1H), 7.75 (d, $J = 7.2$ Hz, 1H), 7.54 – 7.47 (m, 2H), 7.43 – 7.37 (m, 2H), 3.99 (d, $J = 6.8$ Hz, 2H), 3.35 (t, $J = 6.8$ Hz, 2H), 1.64 (s, 1H) ppm. The spectral data is consistent with the literature.⁴

2-(naphthalen-2-yl) ethan-1-ol (2f)



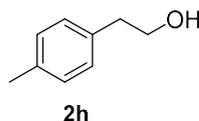
Colorless oil, 6 h, 16.8 mg, 49% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.82 – 7.79 (m, 3H), 7.69 (s, 1H), 7.48 – 7.43 (m, 2H), 7.37 (d, $J = 1.8$ Hz, 1H), 3.95 (t, $J = 10.4$ Hz, 2H), 3.04 (t, $J = 6.4$ Hz, 2H), 1.51 (s, 1H) ppm. The spectral data is consistent with the literature.⁶

2-phenylethan-1-ol (2g)



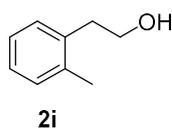
Colorless oil, 6 h, 8.6 mg, 35% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.37 – 7.35 (m, 2H), 7.29 – 7.26 (m, 3H), 3.86 (t, $J = 7.8$ Hz, 2H), 2.89 (t, $J = 6.6$ Hz, 2H), 2.03 (s, 1H) ppm. The spectral data is consistent with the literature.⁴

2-(p-tolyl)ethan-1-ol (2h)



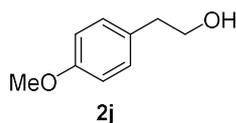
Colorless oil, 6 h, 10.1 mg, 37% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.17-7.14 (s, 4H), 3.87 (t, $J = 6.6$ Hz, 2H), 2.86 (t, $J = 6.6$ Hz, 2H), 2.35 (s, 3H), 1.41 (s, 1H) ppm. The spectral data is consistent with the literature.⁴

2-(*o*-tolyl) ethan-1-ol (2i)



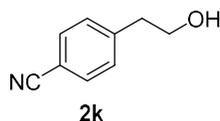
Colorless oil, 6 h, 10.5 mg, 39% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.21 – 7.17 (m, 4H), 3.87 (t, $J = 7.0$ Hz, 2H), 2.92 (t, $J = 7.0$ Hz, 2H), 2.37 (s, 3H), 1.65 (s, 1H) ppm. The spectral data is consistent with the literature.⁴

2-(4-methoxyphenyl) ethan-1-ol (2j)



Pale yellow oil, 6 h, 8.8 mg, 29% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.19-7.16 (m, 2H), 6.90-6.88 (m, 2H), 3.85 (t, $J = 6.6$ Hz, 2H), 3.82 (s, 3H), 2.84 (t, $J = 6.6$ Hz, 2H). 1.66 (s, 1H) ppm.

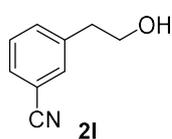
4-(2-hydroxyethyl) benzonitrile (2k)



Pale yellow oil, 6 h, 13.6mg, 46% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.60 (d, $J = 8.2$ Hz, 2H), 7.36 (d, $J = 8.2$ Hz, 2H), 3.90 (t, $J = 6.4$ Hz, 2H), 2.93 (t, $J = 6.6$ Hz, 2H), 1.74 (s, 1H) ppm.

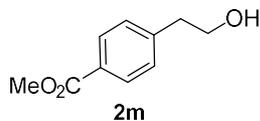
The spectral data is consistent with the literature.⁴

3-(2-hydroxyethyl) benzonitrile (2l)



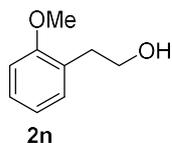
Pale yellow oil, 6 h, 18.5mg, 63% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.59 – 7.52 (m, 2H), 7.53 – 7.48 (m, 1H), 7.43 (t, $J = 7.8$ Hz, 1H), 3.90 (t, $J = 6.4$ Hz, 2H), 2.92 (t, $J = 6.6$ Hz, 2H), 1.76 (s, 1H) ppm. The spectral data is consistent with the literature.⁷

methyl 4-(2-hydroxyethyl) benzoate (2m)



Colorless oil, 6 h, 25.6 mg, 71% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.97 (d, $J = 8.4$ Hz, 2H), 7.30 (d, $J = 8.2$ Hz, 2H), 3.90 (s, 3H), 3.87 (t, $J = 6.6$ Hz, 4H), 2.91 (t, $J = 6.6$ Hz, 2H), 1.92 (s, 1H) ppm. The spectral data is consistent with the literature.⁸

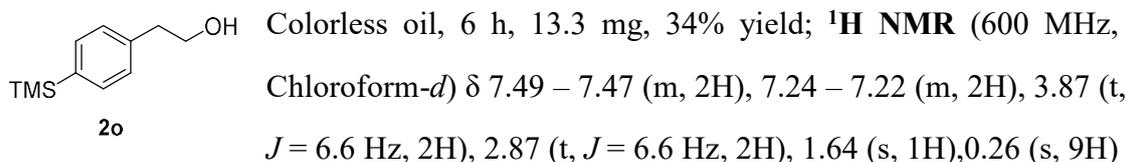
2-(2-methoxyphenyl) ethan-1-ol (2n)



Pale yellow oil, 6 h, 7.2 mg, 24% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.23 (t, $J = 7.8$ Hz, 1H), 7.17 (d, $J = 7.2$ Hz, 1H), 6.91 (t, $J = 7.6$ Hz, 1H), 6.88 (d, $J = 8.2$ Hz, 1H), 3.86 – 3.81 (m, 5H), 2.92–2.90 (t, $J = 6.3$ Hz, 2H), 1.81 (s, 1H) ppm. The spectral data is consistent with

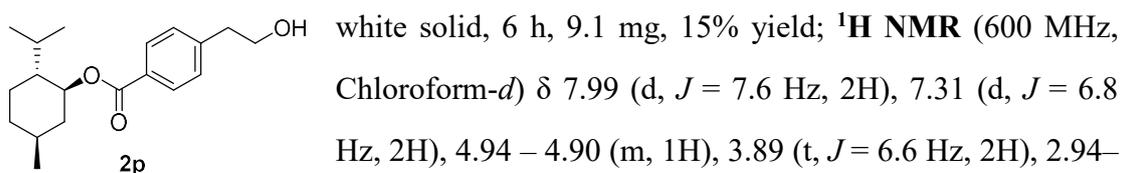
the literature.⁹

2-(4-(trimethylsilyl) phenyl) ethan-1-ol (2o)



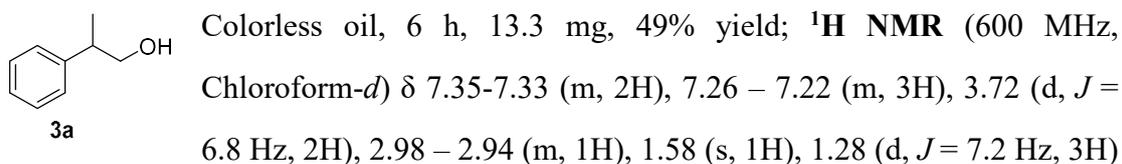
ppm. The spectral data is consistent with the literature.¹⁰

(1S,2R,5S)-2-isopropyl-5-methylcyclohexyl 4-(2-hydroxyethyl) benzoate (2p)



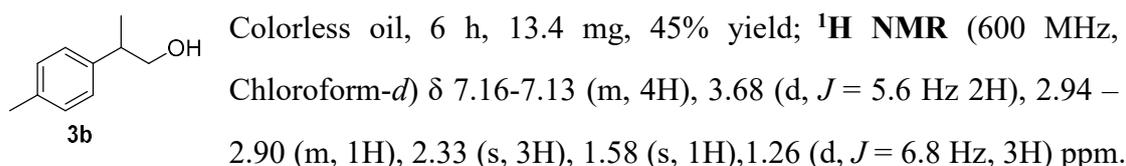
2.92 (m, 2H), 2.12 (d, *J* = 11.8 Hz, 1H), 1.98 – 1.93 (m, 1H), 1.74 – 1.72 (m, 2H), 1.66 (s, 1H), 1.59 – 1.52 (m, 3H), 1.14 – 1.06 (m, 2H), 0.93 – 0.90 (m, 6H), 0.79 (d, *J* = 7.0 Hz, 3H) ppm. The spectral data is consistent with the literature.¹¹

2-phenylpropan-1-ol (3a)



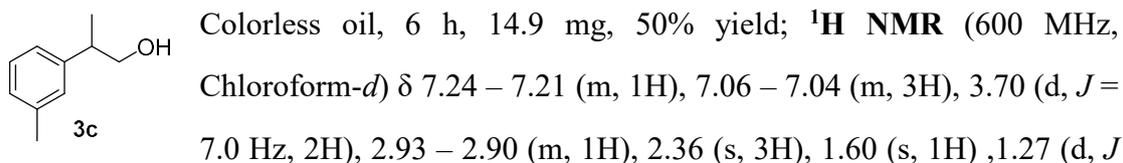
ppm. The spectral data is consistent with the literature.⁴

2-(p-tolyl) propan-1-ol (3b)



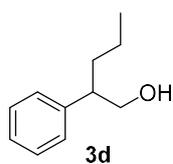
The spectral data is consistent with the literature.¹²

2-(m-tolyl) propan-1-ol (3c)



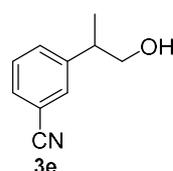
= 7.0 Hz, 3H) ppm. The spectral data is consistent with the literature.¹³

2-phenylpentan-1-ol (3d)



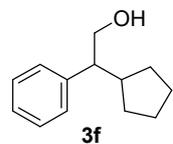
Colorless oil, 6 h, 15.1 mg, 46% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.34 – 7.32 (m, 2H), 7.26 – 7.20 (m, 3H), 3.79 – 3.70 (m, 2H), 2.83 – 2.78 (m, 1H), 1.69 – 1.64 (m, 1H), 1.59 (s, 1H), 1.57 – 1.54 (m, 1H), 1.25 – 1.20 (m, 2H), 0.87 (t, $J = 7.4$ Hz, 3H) ppm. The spectral data is consistent with the literature.¹⁴

3-(1-hydroxypropan-2-yl) benzonitrile (3e)



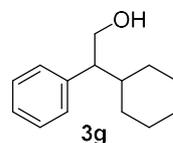
Pale yellow oil, 6 h, 18.3 mg, 57% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.54 – 7.49 (m, 3H), 7.45 – 7.42 (m, 1H), 3.72 (t, $J = 6.2$ Hz, 2H), 3.01 – 2.99 (m, 1H), 1.80 (s, 1H), 1.30 – 1.29 (d, $J = 7.0$ Hz, 3H). ppm. $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 145.6, 132.3, 131.2, 130.3, 129.3, 119.0, 112.4, 68.0, 42.0, 17.4. ppm. HRMS (ESI) m/z : [M + H]⁺ Calcd for C₁₀H₁₂ON 162.0909; Found 162.0913.

2-cyclopentyl-2-phenylethan-1-ol (3f)



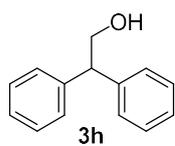
Colorless oil, 6 h, 12.4 mg, 33% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.32 (t, $J = 7.6$ Hz, 2H), 7.25 – 7.21 (m, 3H), 3.92 – 3.88 (m, 1H), 3.77 (t, $J = 10.0$ Hz, 1H), 2.58 – 2.54 (m, 1H), 2.10 – 2.02 (m, 1H), 1.94 – 1.89 (m, 1H), 1.71 – 1.68 (m, 1H), 1.59 (s, 1H), 1.54 – 1.49 (m, 1H), 1.45 – 1.39 (m, 2H), 1.29 – 1.23 (m, 2H), 1.02 – 0.98 (m, 1H) ppm. The spectral data is consistent with the literature.¹⁵

2-cyclohexyl-2-phenylethan-1-ol (3g)



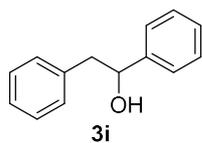
Colorless oil, 6 h, 17.3 mg, 42% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.34 – 7.31 (m, 2H), 7.25 – 7.23 (t, $J = 7.4$ Hz, 1H), 7.20 – 7.18 (m, 2H), 3.97 – 3.93 (m, 1H), 3.82 (t, $J = 10.6$ Hz, 1H), 2.59 – 2.56 (m, 1H), 1.90 – 1.87 (m, 1H), 1.77 – 1.73 (m, 1H), 1.63 – 1.61 (m, 1H), 1.60 (s, 1H), 1.43 – 1.39 (m, 1H), 1.29 – 1.22 (m, 2H), 1.14 – 1.09 (m, 3H), 1.06 – 0.99 (m, 1H), 0.85 – 0.79 (m, 1H) ppm. The spectral data is consistent with the literature.¹⁵

2,2-diphenylethan-1-ol (3h)



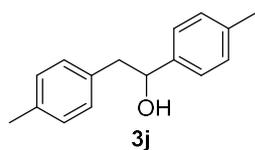
white solid, 6 h, 14.3 mg, 36% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.35 – 7.31 (m, 4H), 7.28 – 7.22 (m, 6H), 4.23 – 4.21 (m, 1H), 4.19-4.17 (m, 2H), 1.49 (s, 1H) ppm. The spectral data is consistent with the literature.⁴

1,2-diphenylethan-1-ol (3i)



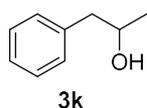
white solid, 6 h, 12.2 mg, 31% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.37 – 7.34 (m, 4H), 7.32 – 7.27 (m, 3H), 7.25 – 7.19 (m, 3H), 4.91 – 4.89 (m, 1H), 3.04 (dd, *J* = 13.6, 4.8 Hz, 1H), 2.99 (dd, *J* = 13.6, 8.6 Hz, 1H), 1.98 (s, 1H) ppm. The spectral data is consistent with the literature.¹⁶

1,2-di-p-tolyloethan-1-ol (3j)



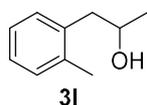
white solid, 6 h, 14.9 mg, 33% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.26 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 2H), 7.12-7.09 (m, 4H), 4.85 – 4.81 (m, 1H), 2.99 (dd, *J* = 13.8, 4.6 Hz, 1H), 2.92 (dd, *J* = 13.8, 8.8 Hz, 1H), 2.35 (s, 3H), 2.32 (s, 3H), 1.93 (s, 1H) ppm. The spectral data is consistent with the literature.¹⁷

1-phenylpropan-2-ol (3k)



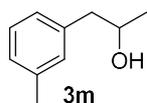
Colorless oil, 6 h, 15.7 mg, 58% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.33 – 7.30 (m, 2H), 7.25 – 7.20 (m, 3H), 4.05 – 3.99 (m, 1H), 2.80-2.77 (m, 1H), 2.67-2.71 m, 1H), 1.65 (s, 1H), 1.25 (d, *J* = 6.2 Hz, 3H) ppm. The spectral data is consistent with the literature.¹⁵

1-(o-tolyl) propan-2-ol (3l)



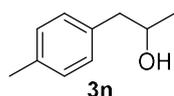
Colorless oil, 6 h, 13.1 mg, 44% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.18 – 7.14(m, 4H), 4.05 – 4.02 (m, 1H), 2.80 (dd, *J* = 13.6, 5.0 Hz, 1H), 2.73 (dd, *J* = 13.6, 8.0 Hz, 1H), 2.33 (s, 3H), 1.63 (s, 1H), 1.27 (d, *J* = 6.2 Hz, 3H) ppm. The spectral data is consistent with the literature.¹⁸

1-(*m*-tolyl) propan-2-ol (3m)



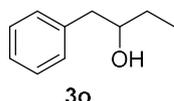
Colorless oil, 6 h, 15.7 mg, 52% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.20 (t, *J* = 7.6 Hz, 1H), 7.06 – 7.00 (m, 3H), 4.04 – 4.00(m, 1H), 2.77 – 2.74(m, 1H), 2.66– 2.63 (m, 1H), 2.34 (s, 3H), 1.63 (s, 1H), 1.25 (d, *J* = 6.2 Hz, 3H) ppm. The spectral data is consistent with the literature.¹⁵

1-(*p*-tolyl) propan-2-ol (3n)



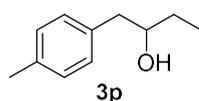
Colorless oil, 6 h, 12.6 mg, 42% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.13 – 7.09 (m, 4H), 4.02 – 3.97 (m, 1H), 2.76 (dd, *J* = 13.6, 4.8 Hz, 1H), 2.64 (dd, *J* = 13.6, 8.0 Hz, 1H), 2.33 (s, 3H), 1.61 (s, 1H), 1.24 (d, *J* = 6.2 Hz, 3H) ppm. The spectral data is consistent with the literature.¹⁹

1-phenylbutan-2-ol (3o)



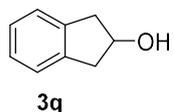
Colorless oil, 6 h, 15.6 mg, 52% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.33 – 7.30 (m, 2H), 7.26 – 7.21 (m, 3H), 3.77 – 3.74 (m, 1H), 2.84 (dd, *J* = 13.6, 4.4 Hz, 1H), 2.65 (dd, *J* = 13.6, 8.2 Hz, 1H), 1.64 (s, 1H), 1.57 – 1.50 (m, 2H), 1.00 (t, *J* = 7.5 Hz, 3H) ppm. The spectral data is consistent with the literature.²⁰

1-(*p*-tolyl) butan-2-ol (3p)



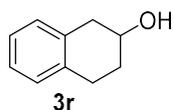
Colorless oil, 6 h, 13.6 mg, 41% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.13 – 7.10 (m, 4H), 3.74 – 3.70(m, 1H), 2.80 (dd, *J* = 13.6, 4.4 Hz, 1H), 2.60 (dd, *J* = 13.6, 8.4 Hz, 1H), 2.33 (s, 3H), 1.64 (s, 1H), 1.55 – 1.49 (m, 3H), 0.99 (t, *J* = 7.4 Hz, 3H) ppm. The spectral data is consistent with the literature.²¹

2,3-dihydro-1H-inden-2-ol (3q)



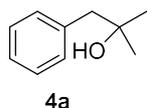
white solid, 6 h, 9.8 mg, 37% yield; ¹H NMR (600 MHz, Chloroform-*d*) δ 7.26 – 7.24 (m, 2H), 7.19 – 7.17 (m, 2H), 4.72 – 4.69 (m, 1H), 3.22 (dd, *J* = 16.4, 5.9 Hz, 2H), 2.92 (dd, *J* = 16.4, 3.2 Hz, 2H), 1.70 (s, 1H) ppm. The spectral data is consistent with the literature.¹⁵

1,2,3,4-tetrahydronaphthalen-2-ol (3r)



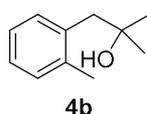
white solid, 6 h, 15.2 mg, 51% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.13 – 7.07 (m, 4H), 4.18 – 4.14 (m, 1H), 3.09 (dd, $J = 16.0, 4.8$ Hz, 1H), 2.98– 2.93 (m, 1H), 2.87 – 2.82 (m, 1H), 2.79 – 2.75 (m, 1H), 2.08 – 2.05 (m, 1H), 1.85 – 1.79 (m, 1H). 1.73 (s, 1H) ppm. The spectral data is consistent with the literature.¹⁵

2-methyl-1-phenylpropan-2-ol (4a)



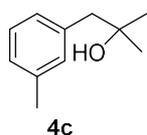
Colorless oil, 6 h, 24.7 mg, 82% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.33-7.21 (m, 5H), 2.77 (s, 2H), 1.64 (s, 1H), 1.23 (s, 6H) ppm. The spectral data is consistent with the literature.¹⁸

2-methyl-1-(o-tolyl) propan-2-ol (4b)



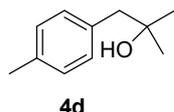
Colorless oil, 6 h, 19.6 mg, 60% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.23 – 7.22 (m, 2H), 7.21 – 7.16 (m, 2H), 2.86 (s, 2H), 2.39 (s, 3H), 1.65 (s, 1H), 1.29 (s, 6H) ppm. $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 137.5, 136.1, 131.4, 130.7, 126.6, 125.6, 71.8, 45.5, 29.5, 20.4. ppm. **HRMS** (ESI) m/z : $[M + H]^+$ Calcd for $\text{C}_{11}\text{H}_{17}\text{O}$ 165.1272; Found 165.1274.

2-methyl-1-(m-tolyl) propan-2-ol (4c)



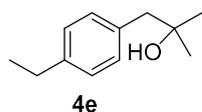
Colorless oil, 6 h, 23.1 mg, 70% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.20 (t, $J = 7.6$ Hz, 1H), 7.07 (d, $J = 7.6$ Hz, 1H), 7.03 – 7.00 (m, 2H), 2.73 (s, 2H), 2.34 (s, 3H), 1.68 (s, 1H), 1.23 (s, 6H). ppm. $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 137.8, 137.6, 131.3, 128.1, 127.5, 127.3, 49.7, 29.2, 21.4. ppm. **HRMS** (ESI) m/z : $[M + H]^+$ Calcd for $\text{C}_{11}\text{H}_{17}\text{O}$ 165.1274; Found 165.1274.

2-methyl-1-(p-tolyl) propan-2-ol (4d)



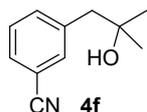
Colorless oil, 6 h, 20.1 mg, 61% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.11 (q, $J = 8.0$ Hz, 4H), 2.73 (s, 2H), 2.34 (s, 3H), 1.61 (s, 1H), 1.22 (s, 6H) ppm. The spectral data is consistent with the literature.²²

1-(4-ethylphenyl)-2-methylpropan-2-ol (4e)



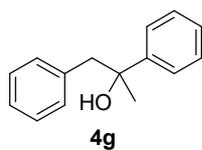
Colorless oil, 6 h, 24.2 mg, 68% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.16 – 7.13 (m, 4H), 2.74 (s, 2H), 2.64 (q, $J = 7.6$ Hz, 2H), 1.44 (s, 1H), 1.26 – 1.23 (m, 9H) ppm. The spectral data is consistent with the literature.²³

3-(2-hydroxy-2-methylpropyl) benzonitrile (4f)



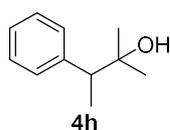
Pale yellow oil, 6 h, 24.2 mg, 69% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.56 – 7.55 (m, 2H), 7.50 – 7.49 (m, 1H), 7.44 – 7.41 (m, 1H), 2.81 (s, 2H), 1.76 (s, 1H), 1.25 (s, 6H). ppm. $^{13}\text{C NMR}$ (151 MHz, Chloroform-*d*) δ 139.5, 135.0, 133.9, 130.2, 128.8, 119.0, 112.1, 70.7, 49.1, 29.4. ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{11}\text{H}_{14}\text{ON}$ 176.1075; Found 176.1070.

1,2-diphenylpropan-2-ol (4g)



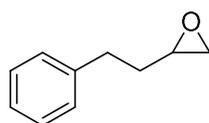
white solid, 6 h, 20.8 mg, 49% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.41 – 7.39 (m, 2H), 7.37 – 7.31 (m, 2H), 7.24 – 7.20 (m, 4H), 7.04 – 6.95 (m, 2H), 3.13 (d, $J = 13.2$ Hz, 1H), 3.03 (d, $J = 13.2$ Hz, 1H), 1.88 (s, 1H), 1.57 (s, 3H) ppm. The spectral data is consistent with the literature.²⁴

2-methyl-3-phenylbutan-2-ol (4h)



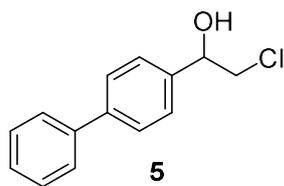
Colorless oil, 6 h, 4.6 mg, 14% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.31 (t, $J = 7.6$ Hz, 2H), 7.26 – 7.23 (m, 3H), 2.81 (q, $J = 7.2$ Hz, 1H), 1.59 (s, 1H), 1.35 (d, $J = 7.2$ Hz, 3H), 1.18 (s, 6H) ppm. The spectral data is consistent with the literature.²⁵

2-phenethyloxirane (4i')



Colorless oil, 6 h, 13.8 mg, 46% yield; $^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.34 – 7.31 (m, 2H), 7.25 – 7.22 (m, 3H), 3.00 – 2.97 (m, 1H), 2.88 – 2.83 (m, 1H), 2.81 – 2.76 (m, 2H), 2.50 (dd, $J = 4.9, 2.8$ Hz, 1H), 1.94 – 1.84 (m, 2H) ppm. The spectral data is consistent with the literature.²⁶

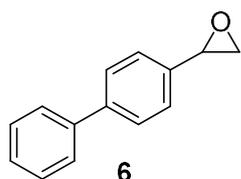
1-([1,1'-biphenyl]-4-yl)-2-chloroethan-1-ol (5)



$^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.65 – 7.61 (m, 4H), 7.50-7.46 (m, 4H), 7.41 – 7.38 (m, 1H), 4.96 (dd, $J = 8.6, 3.4$ Hz, 1H), 3.82 (dd, $J = 11.2, 3.4$ Hz, 1H), 3.73 (dd, $J = 11.2, 8.8$ Hz, 1H), 2.72 (d, $J = 3.2$ Hz, 1H) ppm. The spectral data

is consistent with the literature.²⁷

2-([1,1'-biphenyl]-4-yl)oxirane (6)



$^1\text{H NMR}$ (600 MHz, Chloroform-*d*) δ 7.63 – 7.61 (m, 4H), 7.49 – 7.46 (m, 2H), 7.40 – 7.37 (m, 3H), 3.95 (dd, $J = 4.2, 2.6$ Hz, 1H), 3.22 (dd, $J = 5.4, 4.2$ Hz, 1H), 2.89 (dd, $J = 5.6, 2.6$ Hz, 1H) ppm. The spectral data is consistent with the literature.²⁷

6. References

- [1] B. Yang, Z. Lu, *Chem. Commun.* **2017**, 53, 12634-12637.
- [2] F. Lian, F.-X. Luo, M. Wang, K. Xu, C.-C. Zeng, *Chin. J. Chem.* **2023**, 41, 1583-1588.
- [3] M. Majchrzak, G. Wilkowski, M. Kubicki, *J. Org. Chem.* **2017**, 29, 4291-4299.
- [4] C. Huang, W. Ma, X.-L. Zheng, M.-H. Xu, X.-T. Qi, *J. Am. Chem. Soc.* **2022**, 144, 1389-1395.
- [5] W. Pendergast, J.- V. Johnson, S.- H. Dickerson, I.- K. Dev, D.- S. Duch, R. Ferone, W.- R. Hall, J. Humphreys, J.- M. Kelly, D.- C. Wilson, *J. Med. Chem.* **1993** 36, 2279- 2291.
- [6] S. -N. Kessler, H. -A. Wegner, *Org. Lett.* **2010**, 12, 4062-4065.
- [7] B.- C. Lee, J.- Y. Paik, D.- Y. Chi, K.- H. Lee, Y.- S. Choe, *Bioconjugate Chem.* **2004**, 15, 104-111.
- [8] C. Chen, G. Nagy, A. -V. Waiker, K. Maurer, A. McShea, K.- D. Moeller, *J. Am. Chem. Soc.* **2006**, 128, 16020-16021.
- [9] N.- N.- H. Ton, B.- K. Mai, T.- V. Nguyen, *J. Org. Chem.* **2021**, 86, 9117-9133.
- [10] A. Graml, B. Konig, *Chem. Photo. Chem.* **2021**, 5, 362-368.
- [11] X. -C. Lin, Y. -M. Wang, X. Chen, P. -Y. You, K. -M. Mo, G. -H. Ning, D. Li, *Angew. Chem. Int. Ed.* **2023**, 62, e202306497.
- [12] S. M. A. Hakim Siddiki, A.- S. Touchy, M. A.- R. Jamil, T. Toyao, Ken-ichi Shimizu, *ACS Catal.* **2018**, 8, 3091-3103.
- [13] H.-Q. Geng, T. Meyer, R. Franke, X.-F. Wu, *Chem. Sci.* **2021**, 12, 14937-14943.
- [14] Y. Zhang, B. Han, S. -L. Zhu, *Angew. Chem. Int. Ed.* **2019**, 58, 13860-13864.
- [15] X. Hu, G.-T. Zhang, F.-X. Bu, A.-W. Lei, *ACS Catal.* **2017**, 7, 1432-1437.
- [16] S.- V. Ley, C. Mitchell, D. Pears, C. Ramarao, J.-Q. Yu, W- Z. Zhou, *Org Lett.* **2003**, 5, 4665-4668.
- [17] D.-K. Nielsen, A.-G. Doyle, *Angew. Chem. Int. Ed.* **2011**, 50. 6056-6059.
- [18] Y.- Zhao, D.- J. Weix, *J. Am. Chem. Soc.* **2014**, 136, 48-51.
- [19] X.- Y. Du, Y.- L. Zhang, D.- J. Peng, Z. Huang, *Angew. Chem. Int. Ed.* **2016**, 55,

6671-6675.

[20] T. Mahdi, D.- W. Stephan, *Angew. Chem. Int. Ed.* **2015**, *54*, 8511-8514.

[21] M. Parasram, B.- J. Shields, O. Ahmad, T. Knauber, A.- G. Doyle, *ACS Catal.* **2020**, *10*, 5821-5827.

[23] S.- C. Richter, M. Oestreich, *J. Org. Chem.*, **2021**, *14*, 2103-2106.

[24] C.- C. Li, X.-J. Dai, H.- N. Wang, D.- H. Zhu, J. Gao, C.- J. Li, *Org. Lett.* **2018**, *20*, 3801-3805.

[25] J. Harnedy, H.- A. Maashi, Albara A. M. A. El Gehani, M. Burns, L.- C. Morrill, *Org. Lett.* **2023**, *25*, 1486-1490.

[26] J. M. Mitchell and N. S. Finney, *J. Am. Chem. Soc.*, **2001**, *123*, 862-869.

[27] Y. Yuki, T. Touge, H. Nara, K. Matsumura, M. Fujiwhara, Y. Kayaki, T. Ikariya, *Adv. Synth. Catal.*, **2017**, *360*, 568-574.

7. ¹H NMR and ¹³C NMR Spectra of compounds

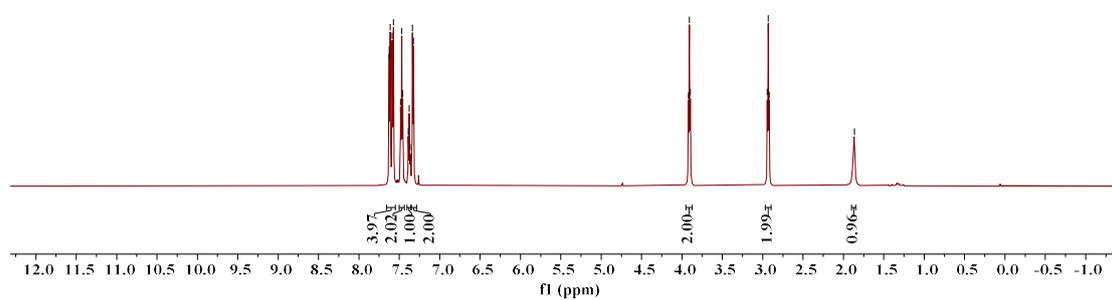
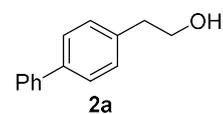
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7.364
7.338
7.325

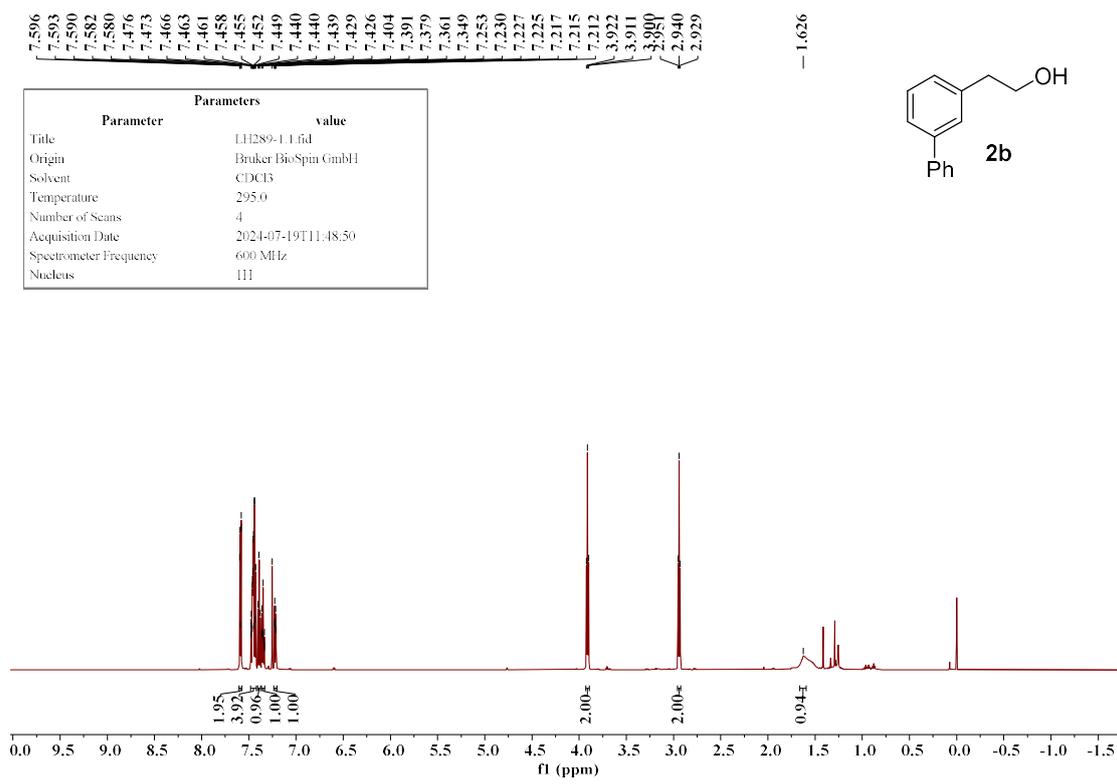
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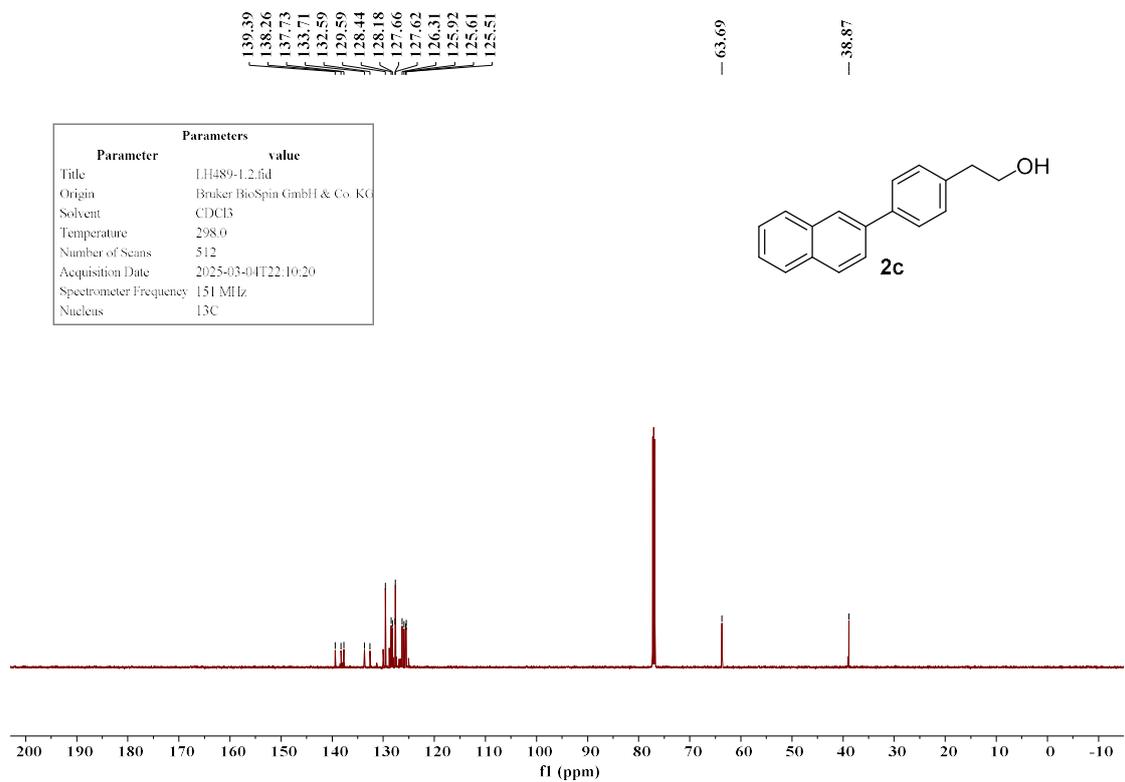
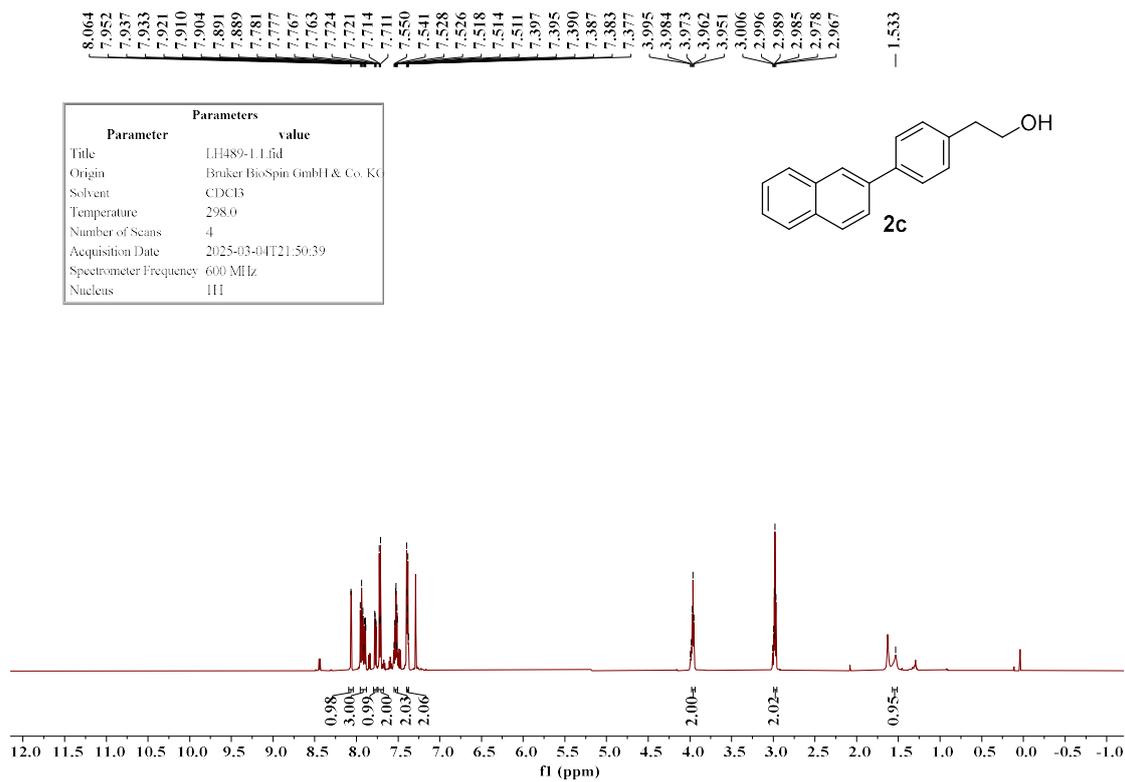
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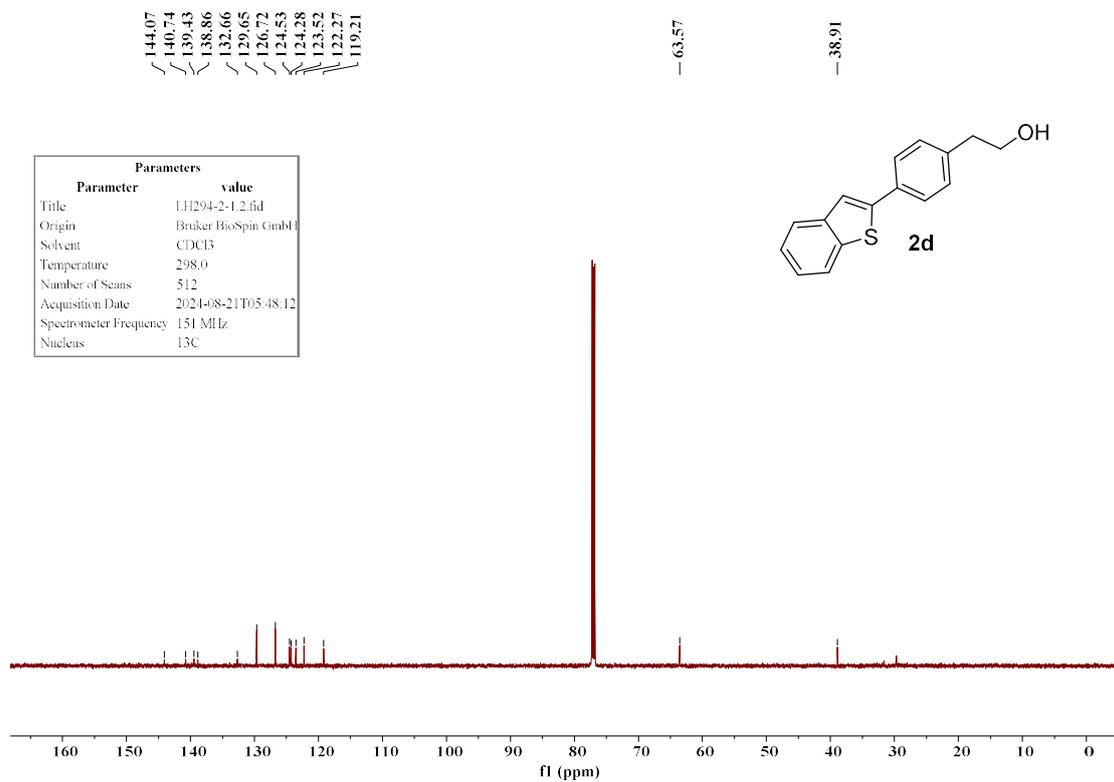
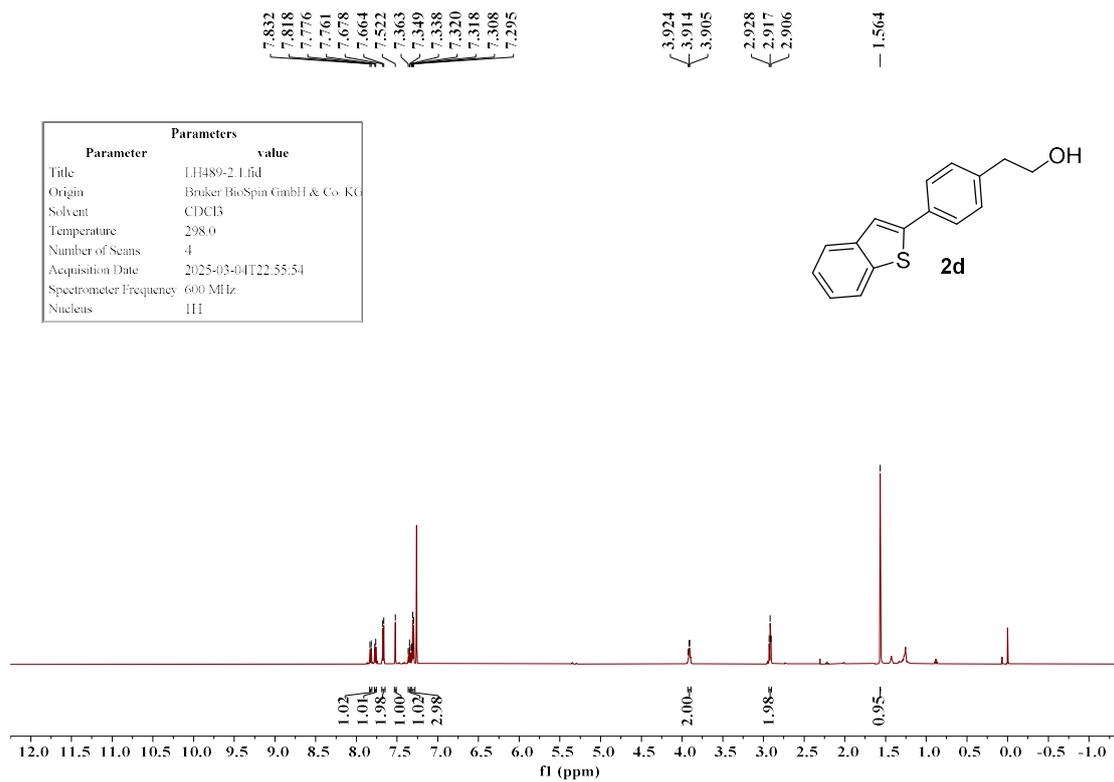
— 1.866

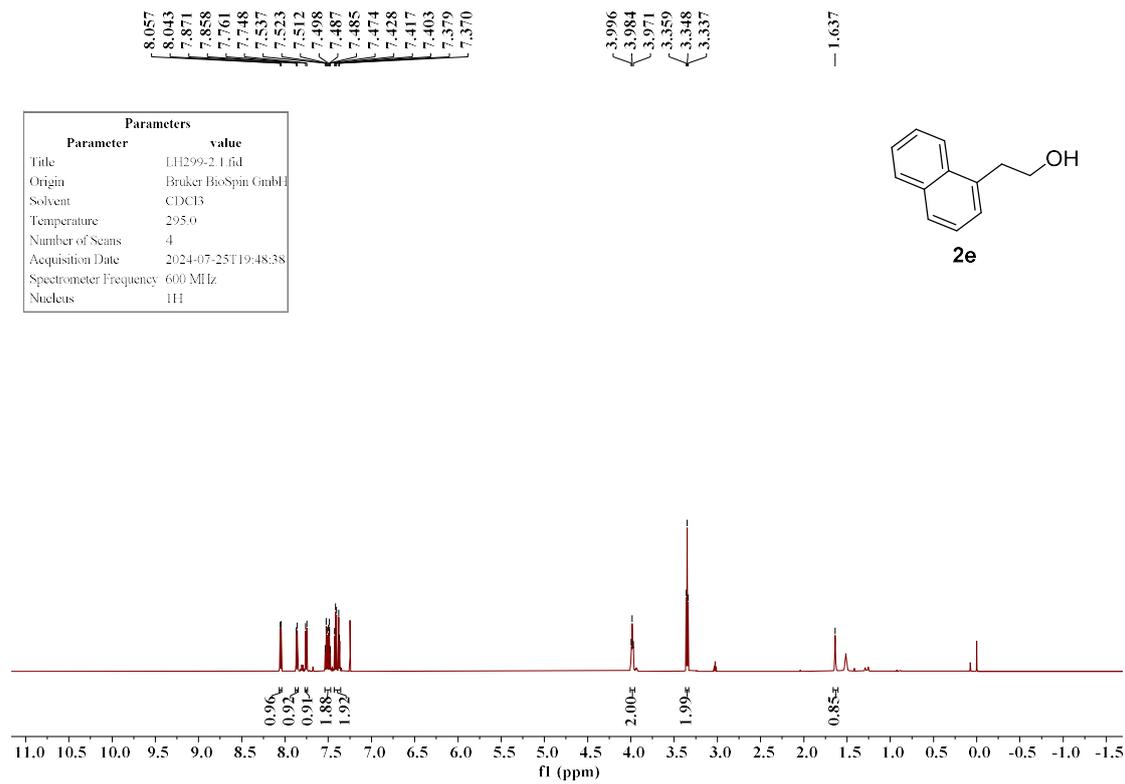
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Spectrometer Frequency	600 MHz	
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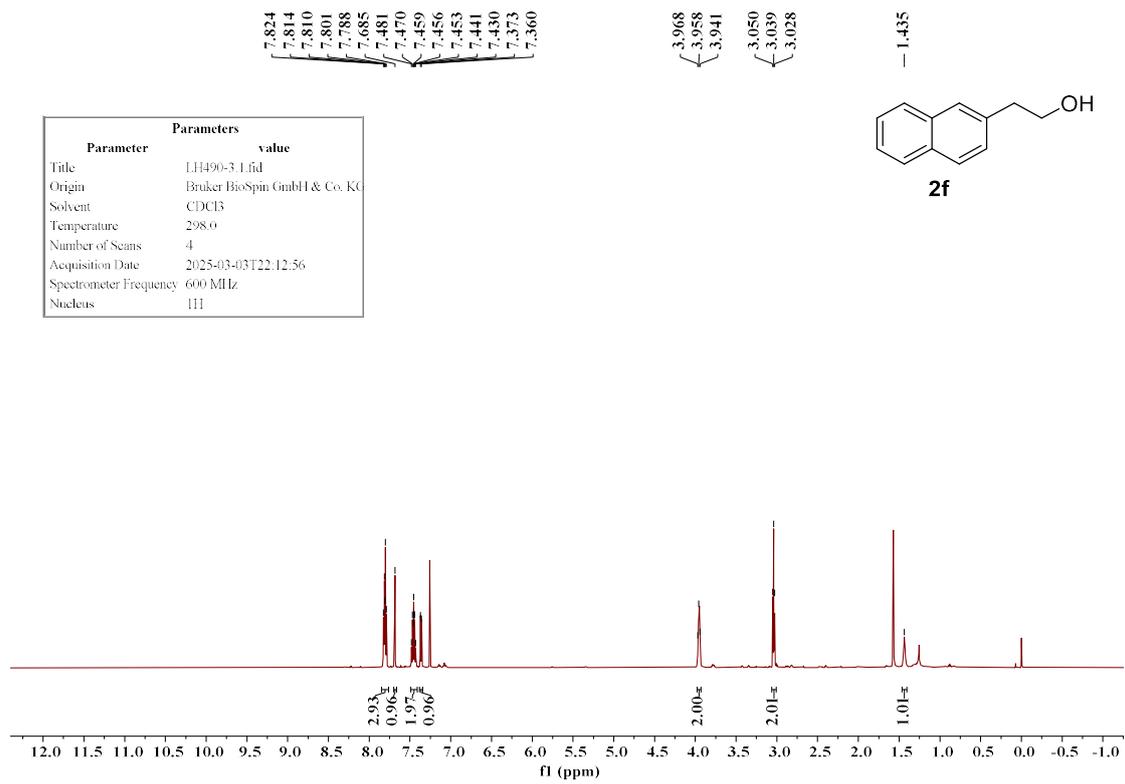


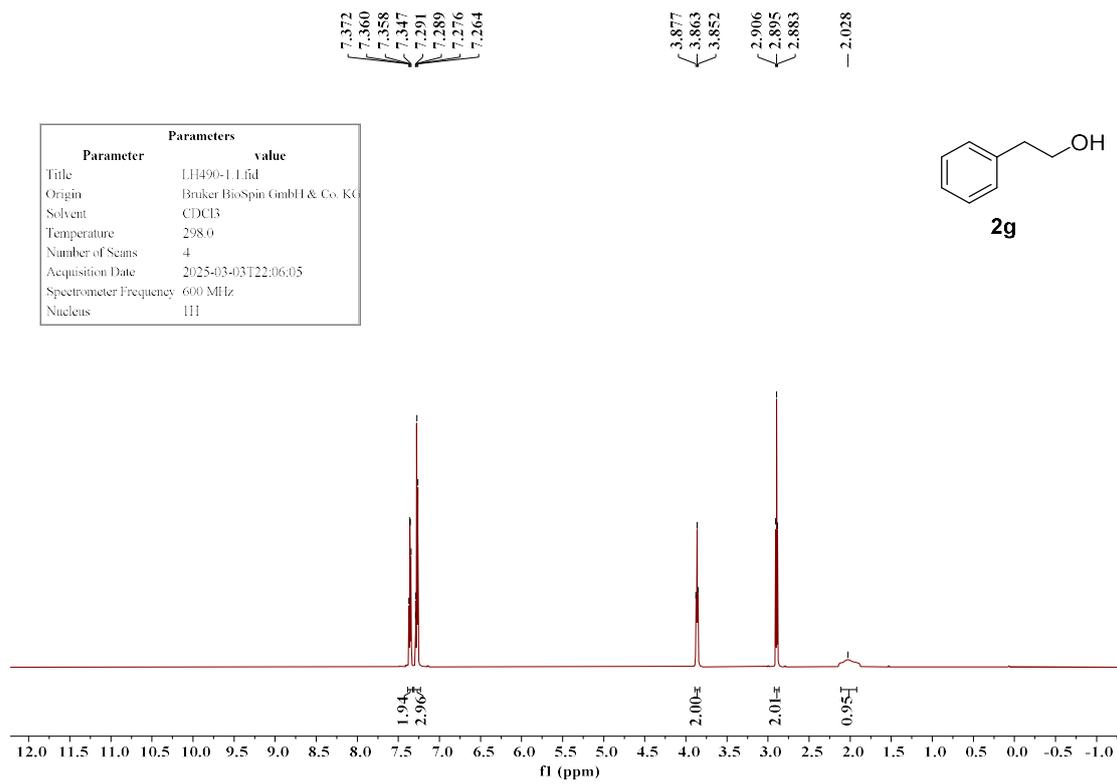




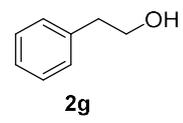


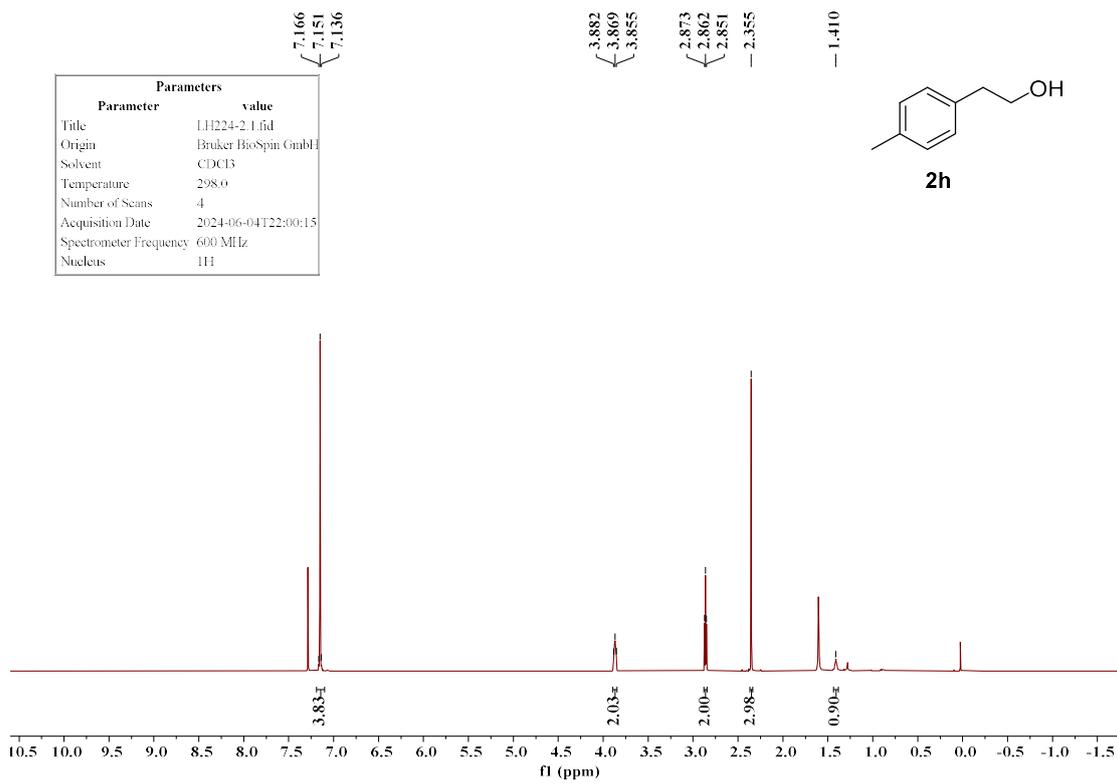


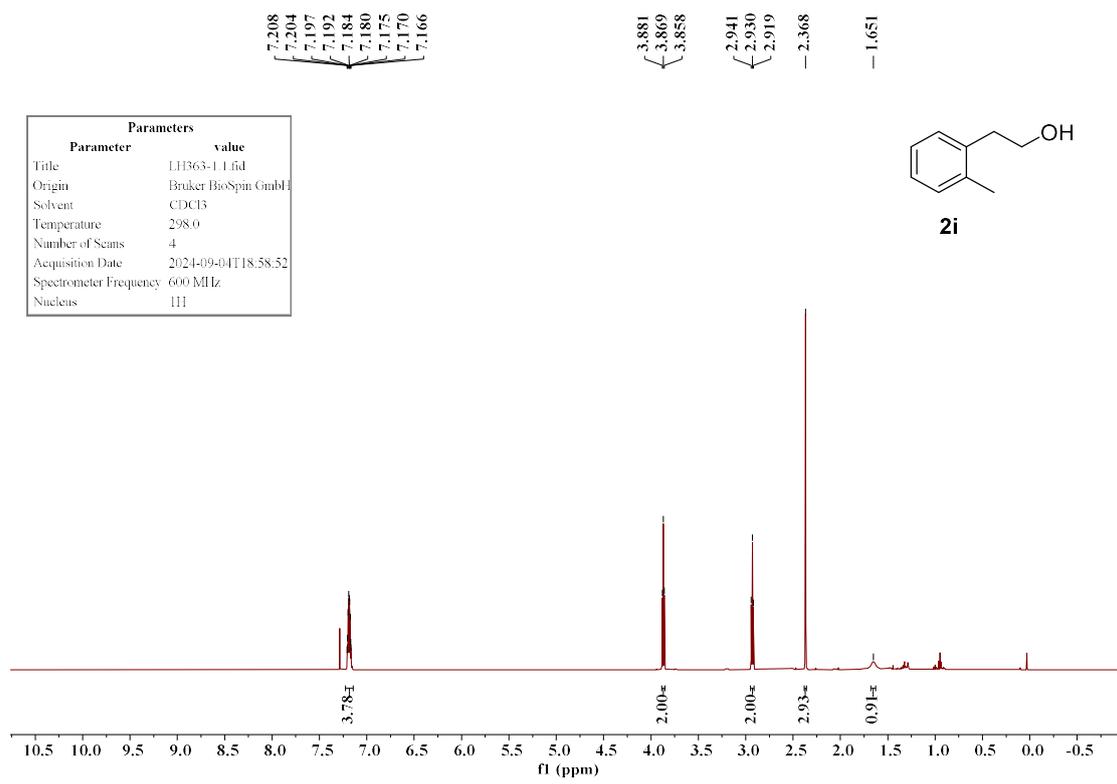




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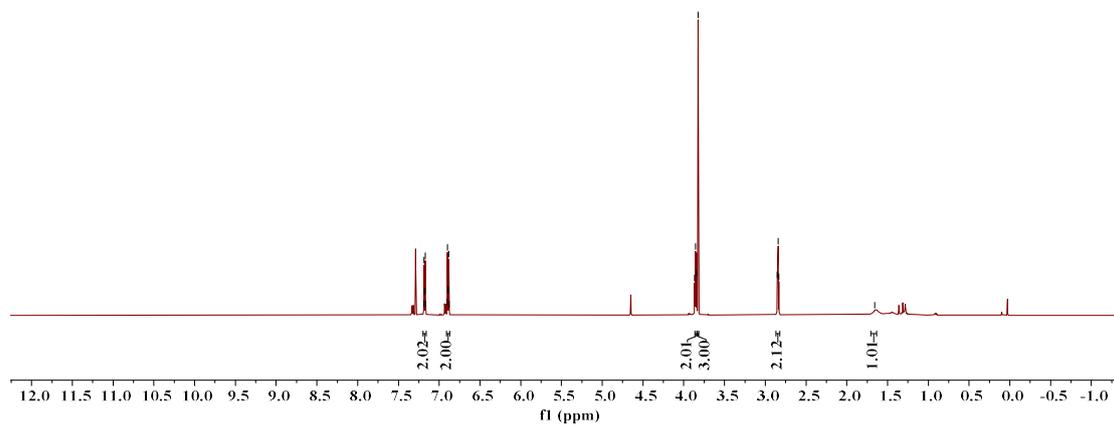
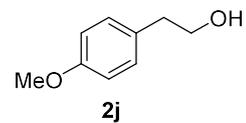
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3.844
3.823

2.854
2.844
2.833

— 1.657

Parameter	value
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Spectrometer Frequency	600 MHz
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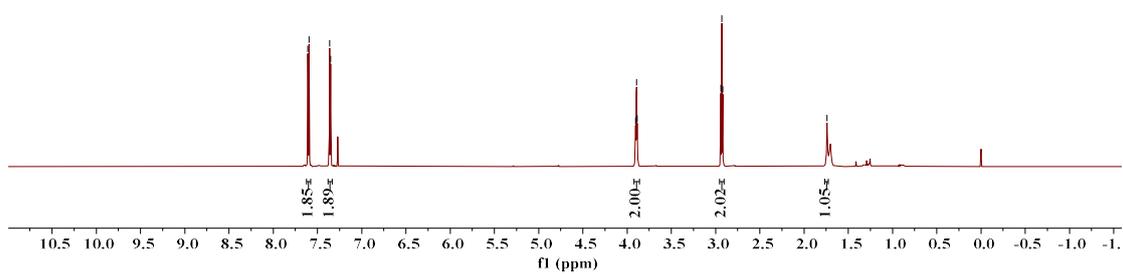
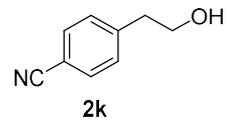
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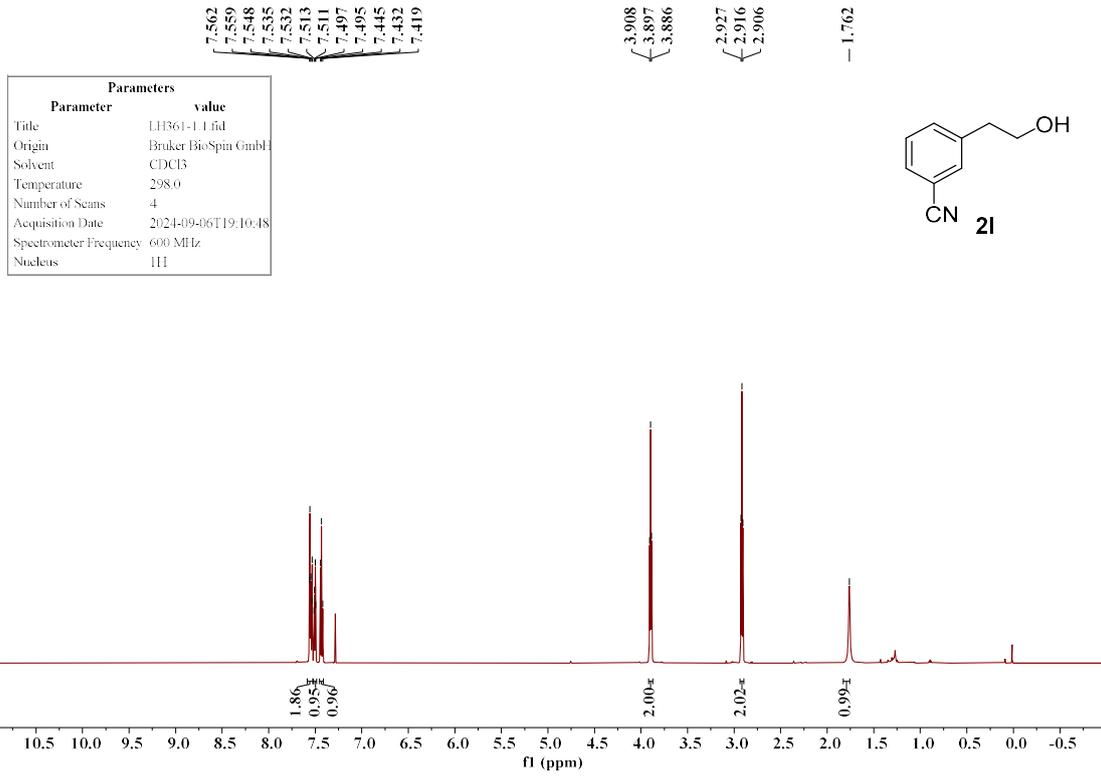
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2.919

— 1.741

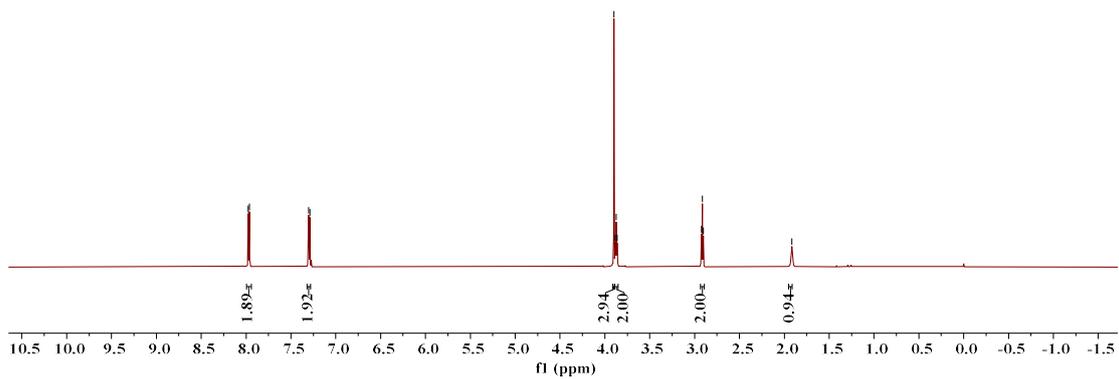
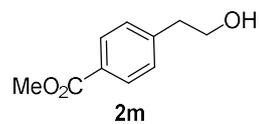
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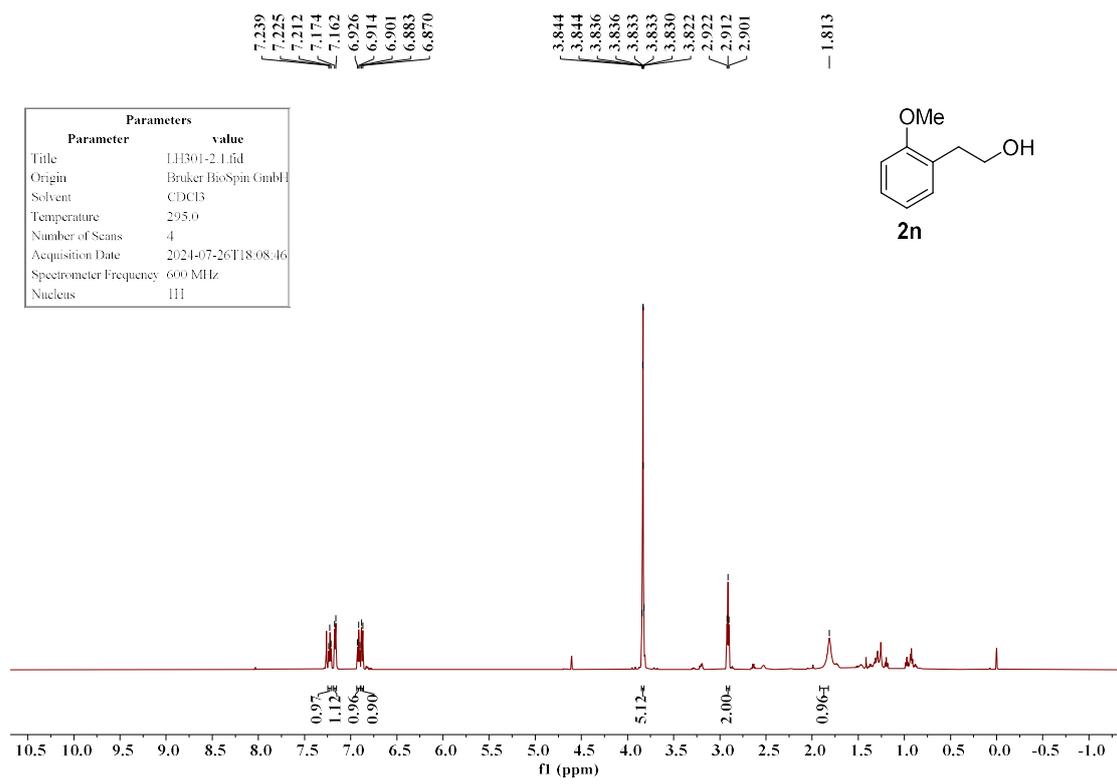




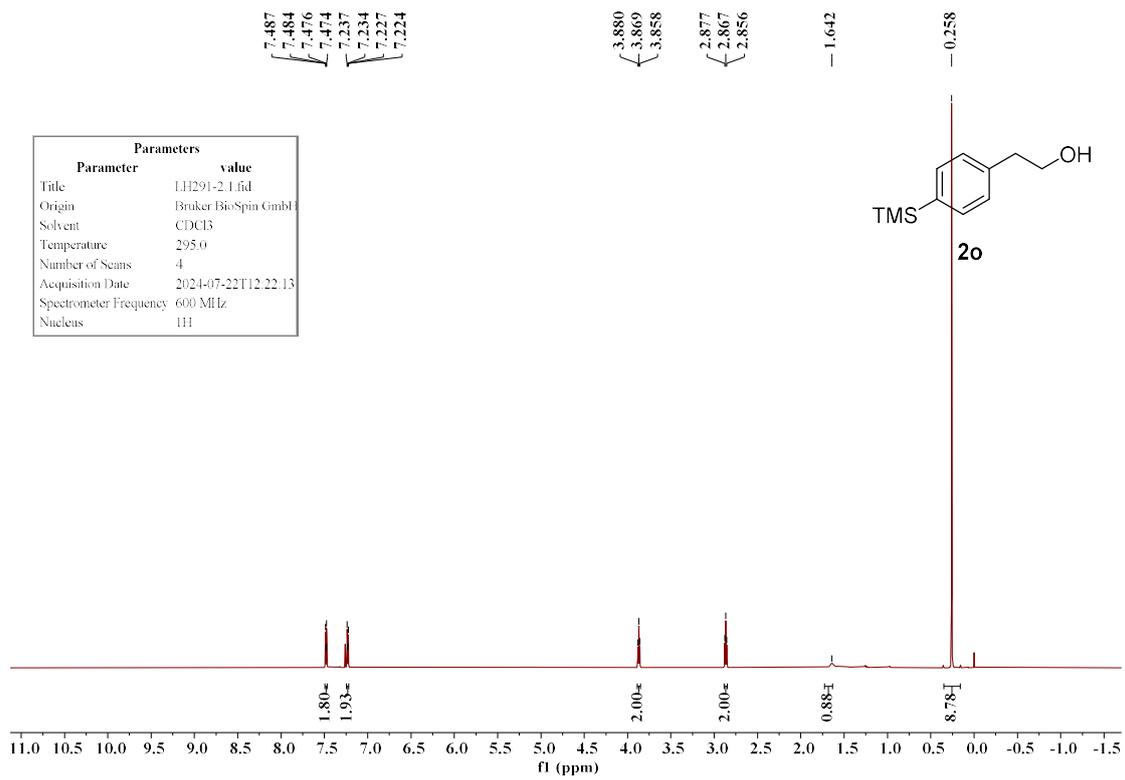
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 < 2.914
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 — 1.916

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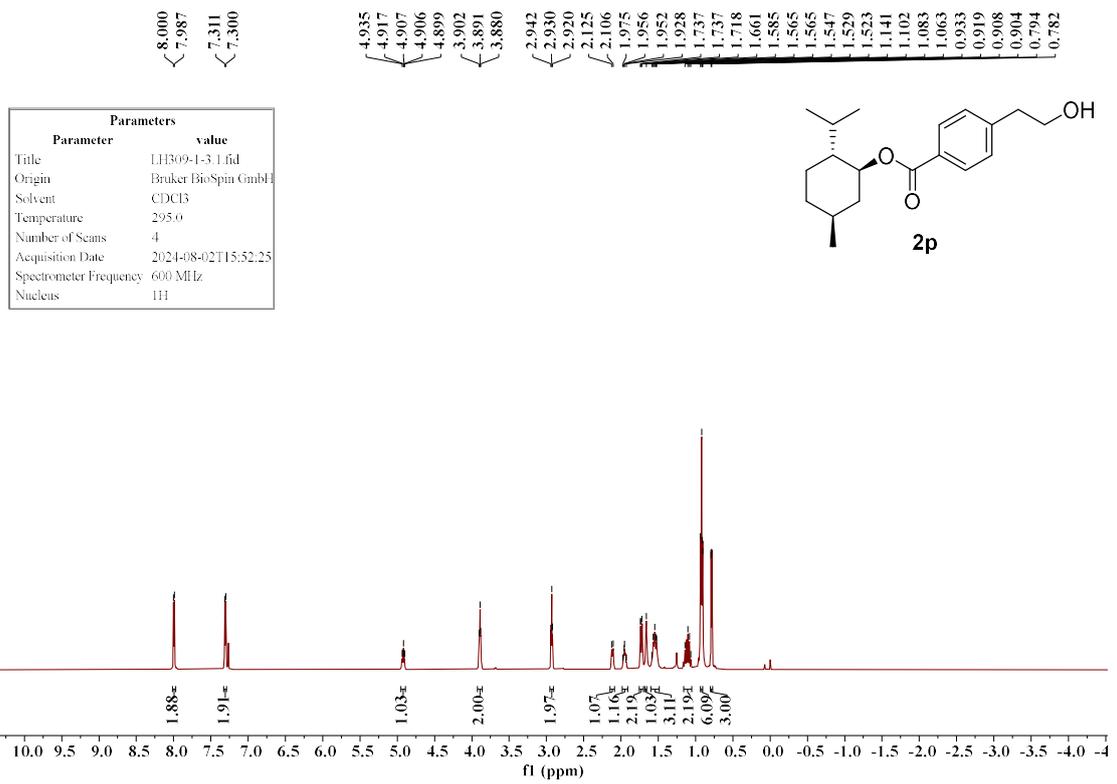




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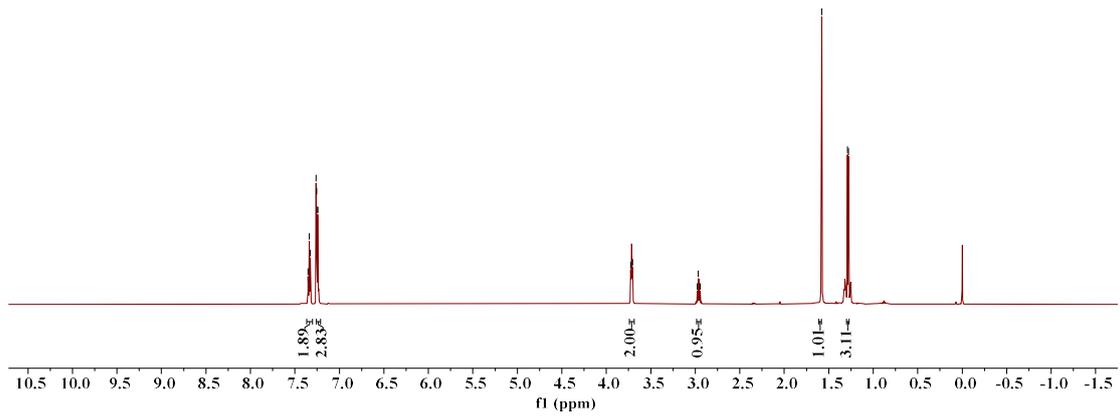
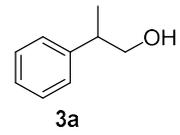


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7.243

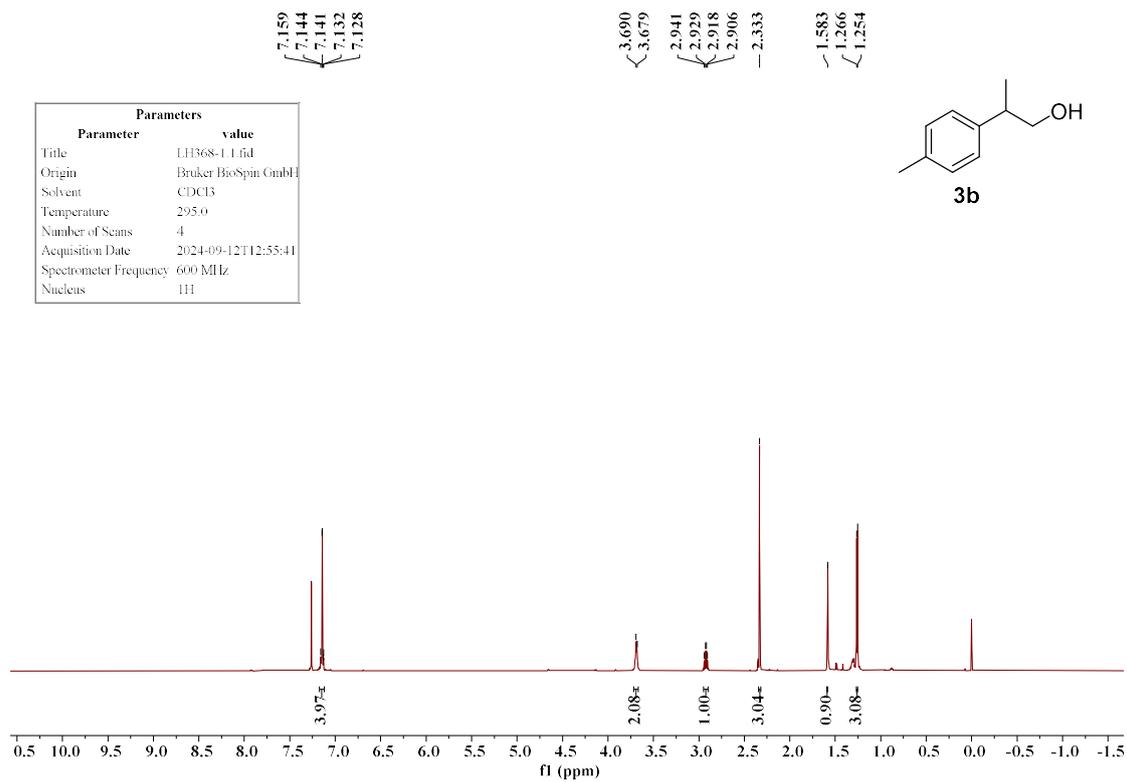
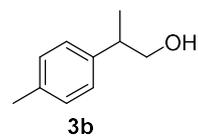
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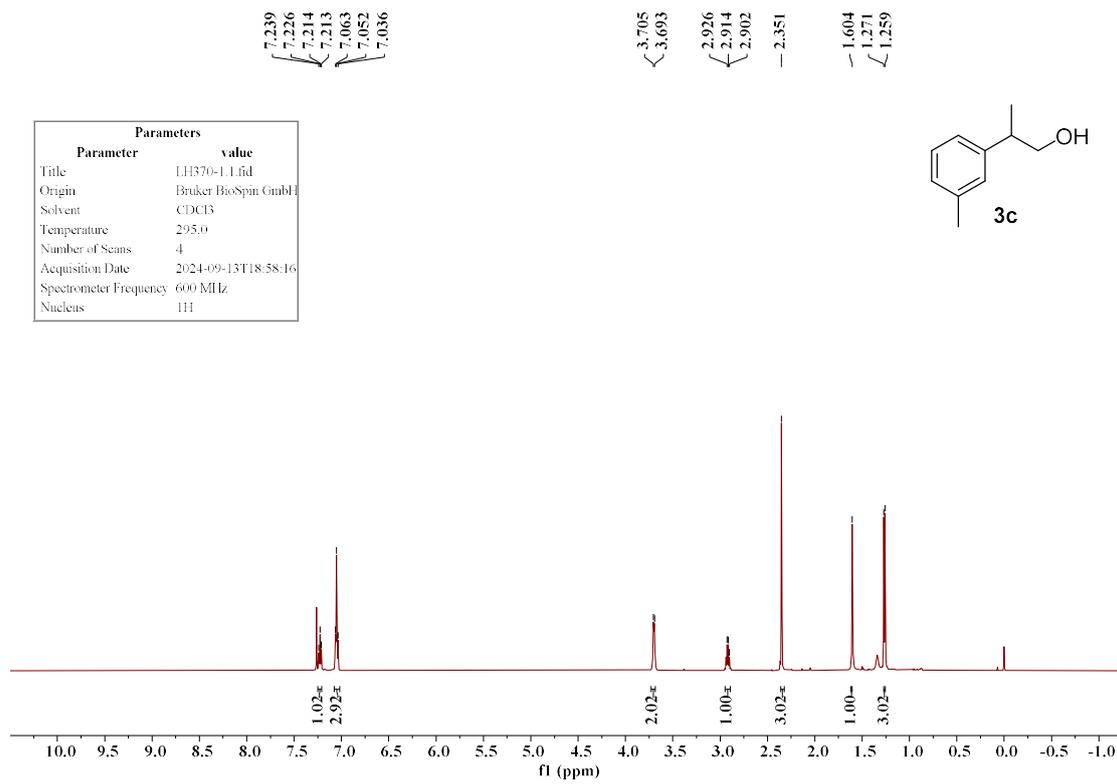
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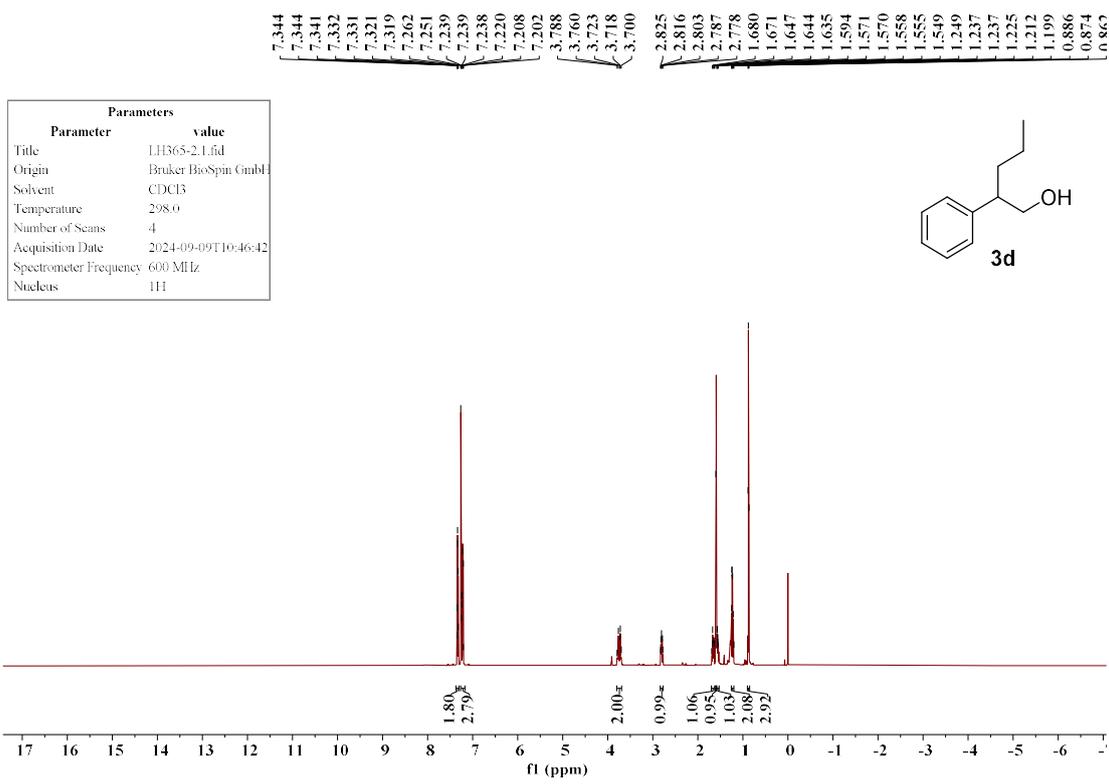
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Spectrometer Frequency	600 MHz
Nucleus	1H



Parameters	
Parameter	value
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Nucleus	¹ H





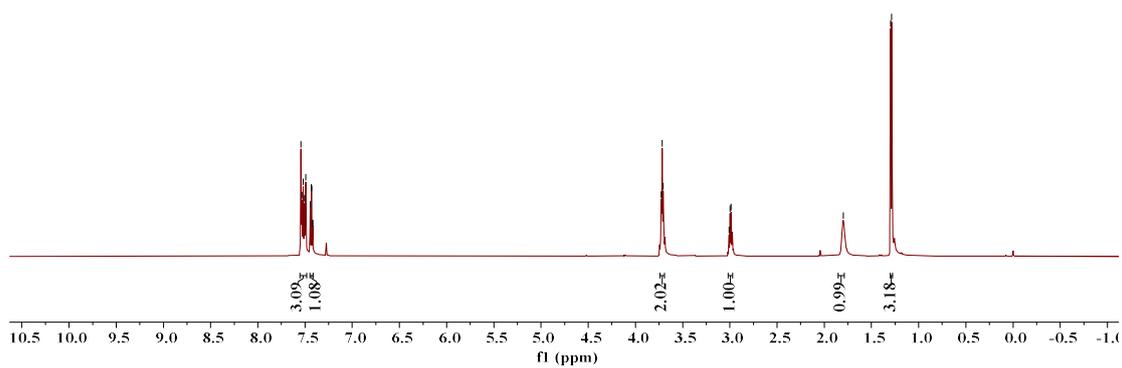
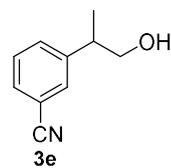


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Parameters	
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Nucleus	1H



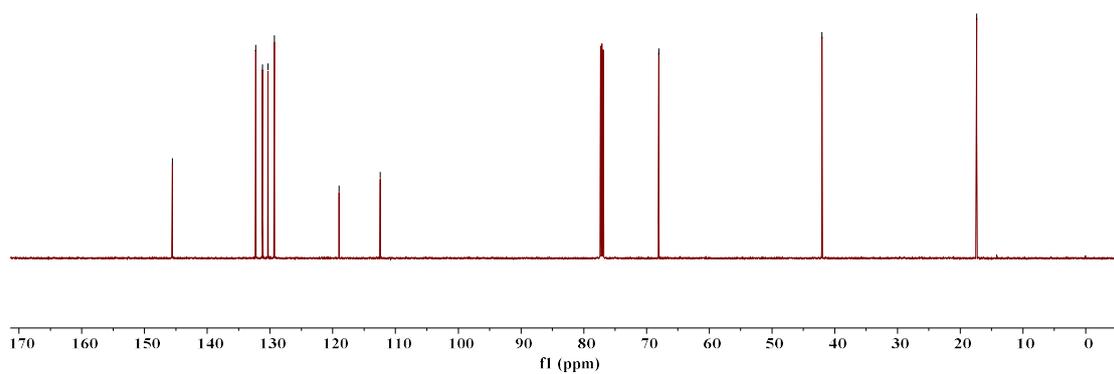
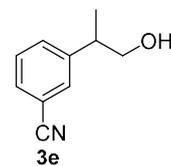
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112.45

68.03

42.03

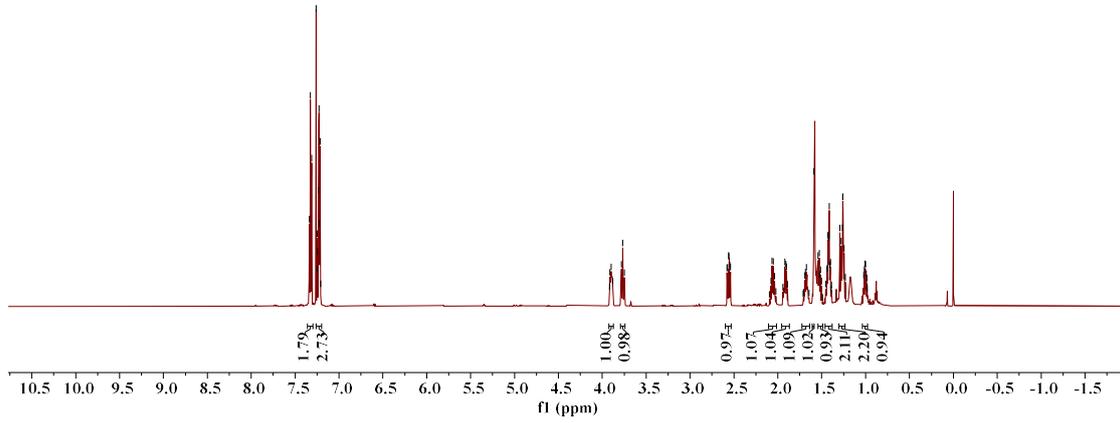
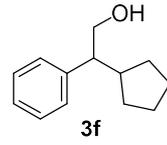
17.39

Parameters	
Parameter	value
Title	LH371-2.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Number of Scans	346
Acquisition Date	2024-09-19T22:20:38
Spectrometer Frequency	151 MHz
Nucleus	13C



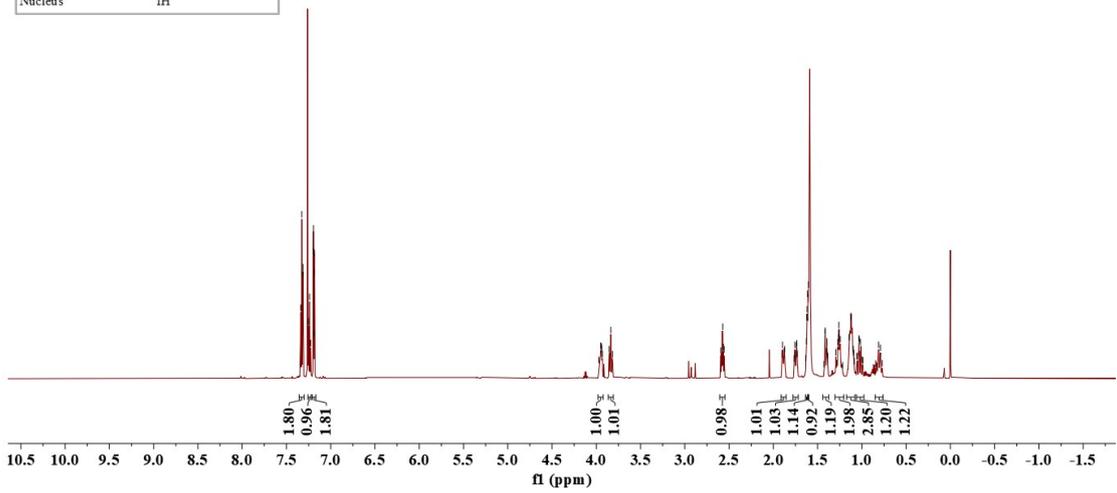
7.338
7.326
7.313
7.261
7.250
7.248
7.246
7.236
7.227
7.223
7.216
7.216
3.914
3.899
3.783
3.767
3.750
2.578
2.571
2.561
2.556
2.546
2.539
2.067
2.050
2.038
1.921
1.908
1.901
1.691
1.684
1.676
1.590
1.541
1.527
1.522
1.513
1.441
1.436
1.430
1.421
1.420
1.417
1.415
1.406
1.400
1.294
1.280
1.265
1.259
1.259
1.259
1.244
1.229
1.022
1.016
1.006
1.000
0.992
0.985

Parameters	
Parameter	value
Title	L11364-3-1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-09-05T18:38:15
Spectrometer Frequency	600 MHz
Nucleus	1H



7.339
7.326
7.314
7.314
7.252
7.239
7.227
7.227
7.198
7.195
7.192
7.184
7.182
3.950
3.944
3.938
3.930
3.885
3.885
3.817
2.594
2.571
2.564
2.556
2.556
1.902
1.896
1.871
1.756
1.750
1.738
1.734
1.729
1.627
1.622
1.618
1.614
1.602
1.602
1.414
1.414
1.399
1.394
1.393
1.385
1.293
1.271
1.265
1.259
1.255
1.243
1.143
1.123
1.096
1.091
1.050
1.035
1.029
1.016
0.810
-0.791

Parameters	
Parameter	value
Title	LH365-1.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-09-09T10:43:47
Spectrometer Frequency	600MHz
Nucleus	1H

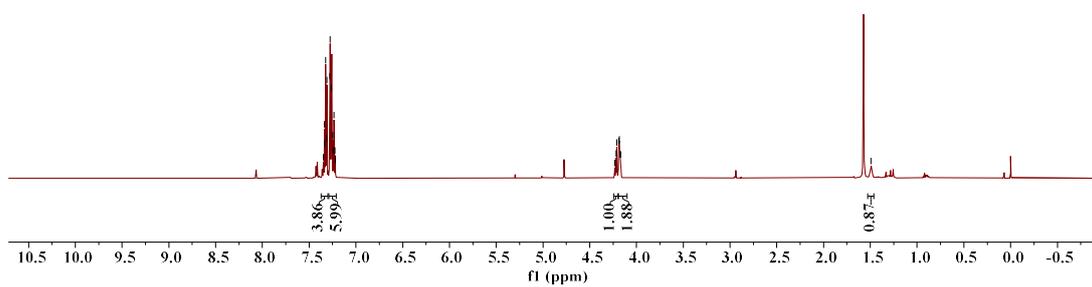
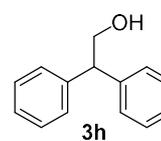


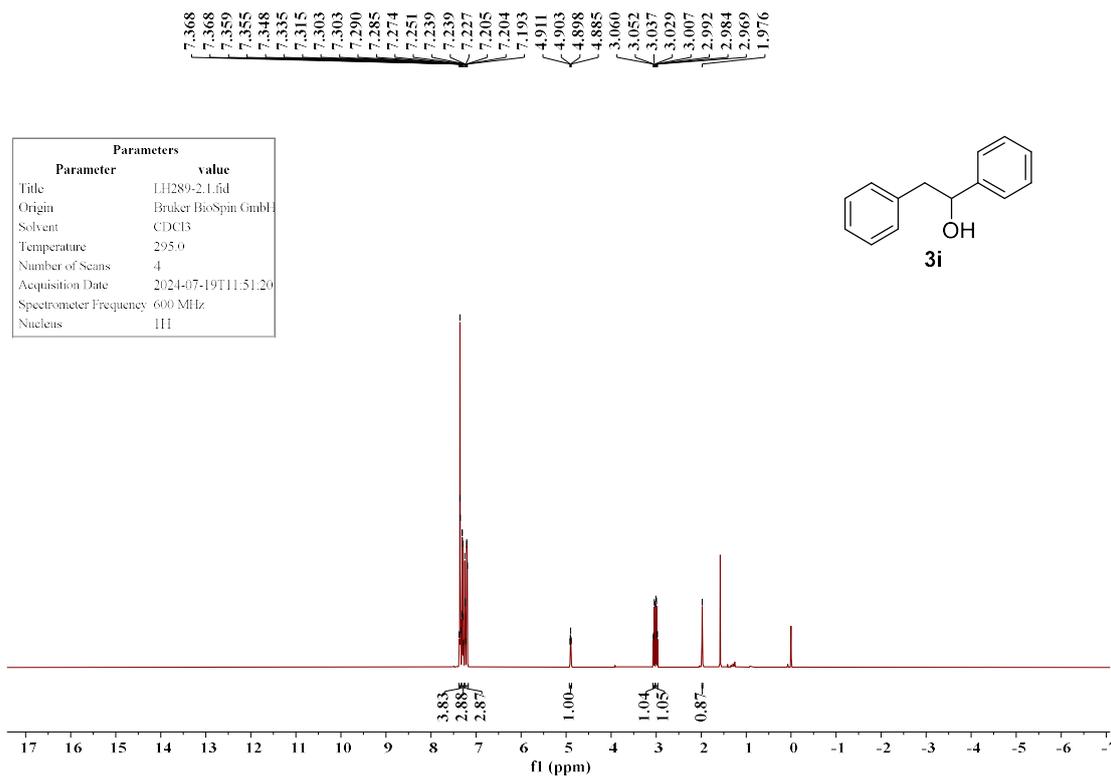
7.348
7.337
7.334
7.324
7.315
7.312
7.278
7.275
7.263
7.247
7.244
7.235
7.222
7.220

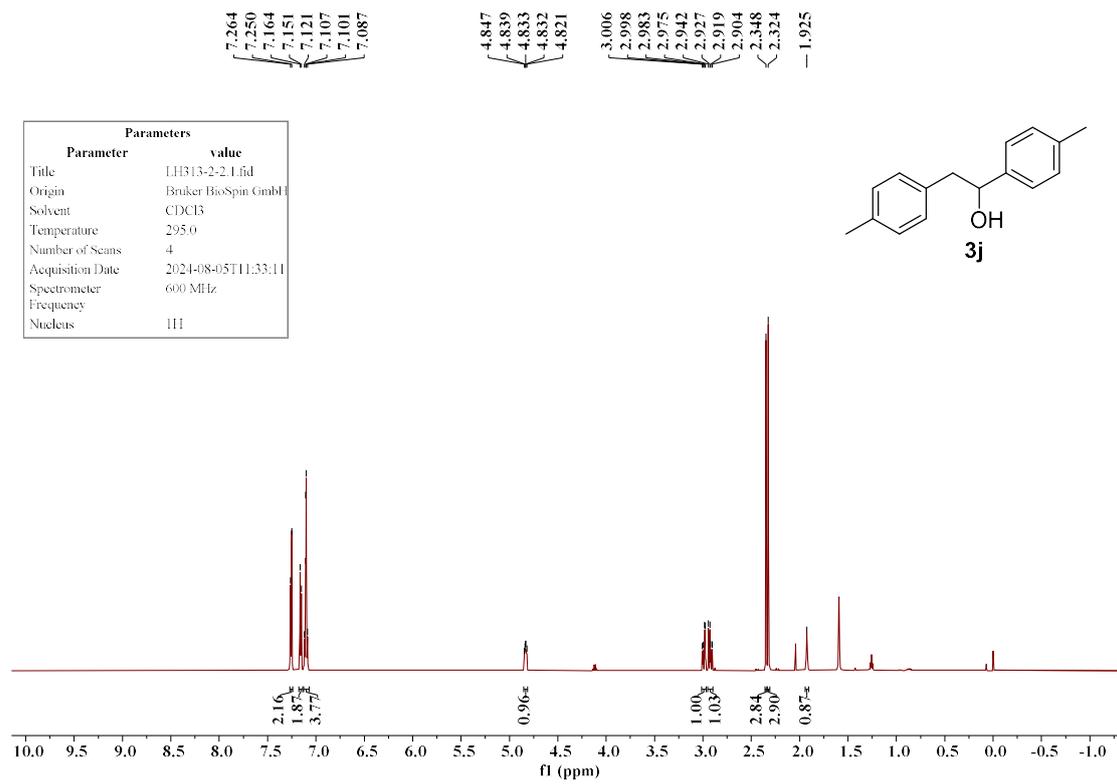
4.233
4.219
4.209
4.189
4.184
4.172
4.170

-1.492

Parameters	
Parameter	value
Title	L11254-1.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-06-27T18:39:20
Spectrometer Frequency	600 MHz
Nucleus	¹ H





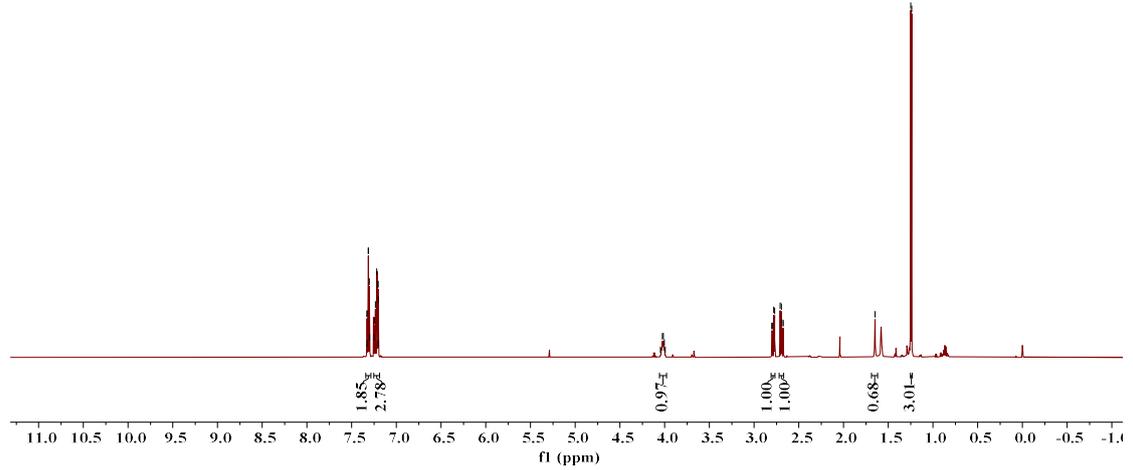
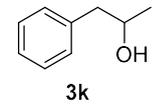


Parameters	
Parameter	value
Title	LH313-2-2-1.fid
Origin	Broker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Number of Scans	4
Acquisition Date	2024-08-05T11:33:11
Spectrometer	600 MHz
Frequency	
Nucleus	1H

7.330
7.327
7.326
7.314
7.314
7.302
7.299
7.249
7.247
7.235
7.219
7.206
7.206
7.203
7.200

4.047
4.026
4.015
4.005
3.994
2.803
2.795
2.780
2.772
2.710
2.696
2.687
2.674
1.650
1.250
1.240

Parameters	
Parameter	value
Title	LH225-3.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-06-06T19:12:04
Spectrometer Frequency	600 MHz
Nucleus	1H

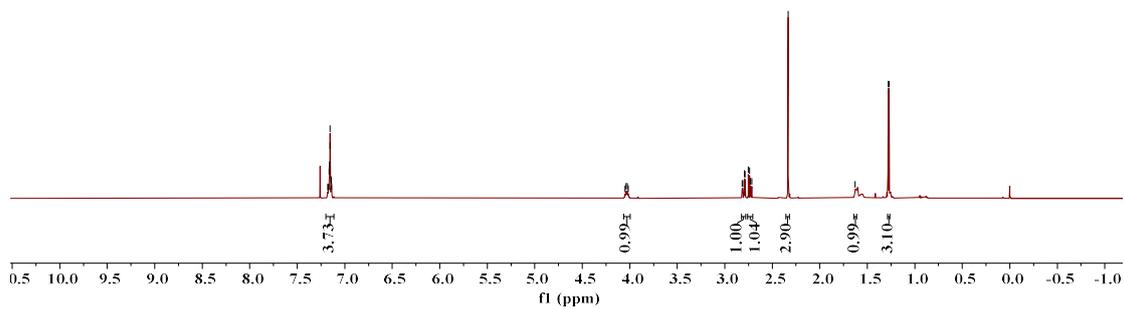
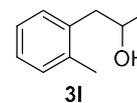


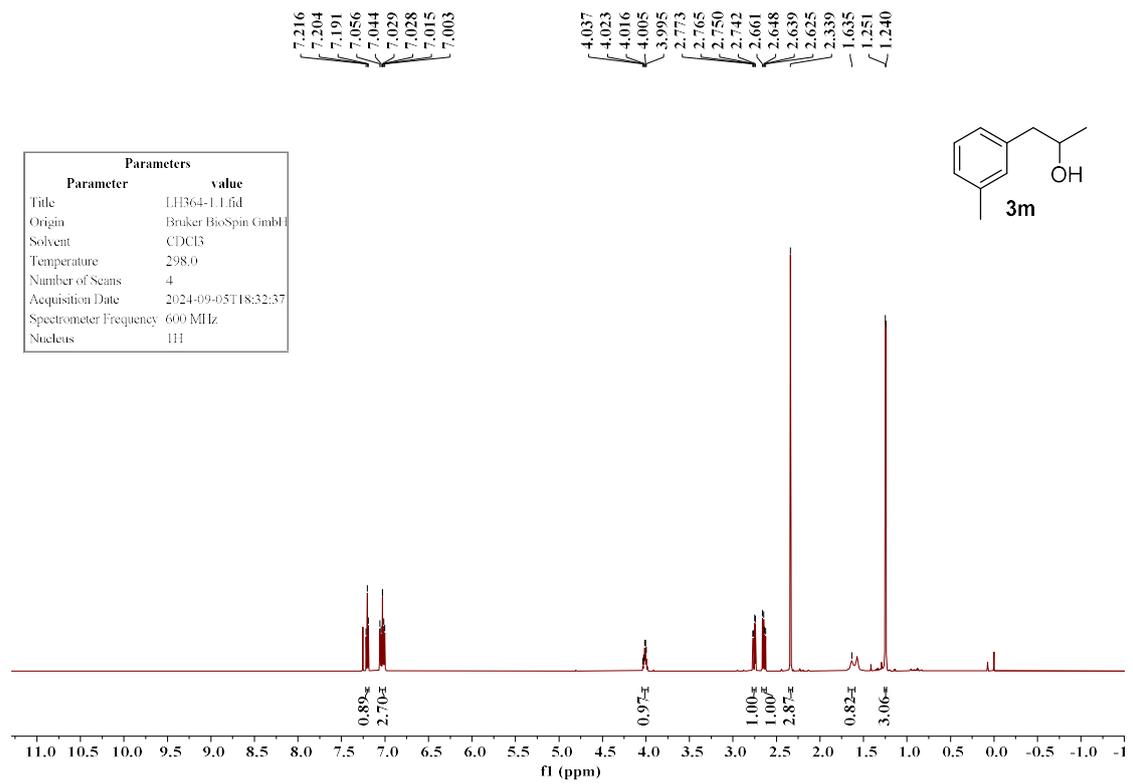
7.179
7.175
7.165
7.161
7.156
7.155
7.145
7.143
7.139

4.049
4.038
4.027
4.016

2.818
2.810
2.795
2.787
2.751
2.738
2.729
2.715
2.334
1.628
1.280
1.270

Parameters	
Parameter	value
Title	LH361-2-1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-09-03T19:37:56
Spectrometer Frequency	600 MHz
Nucleus	1H

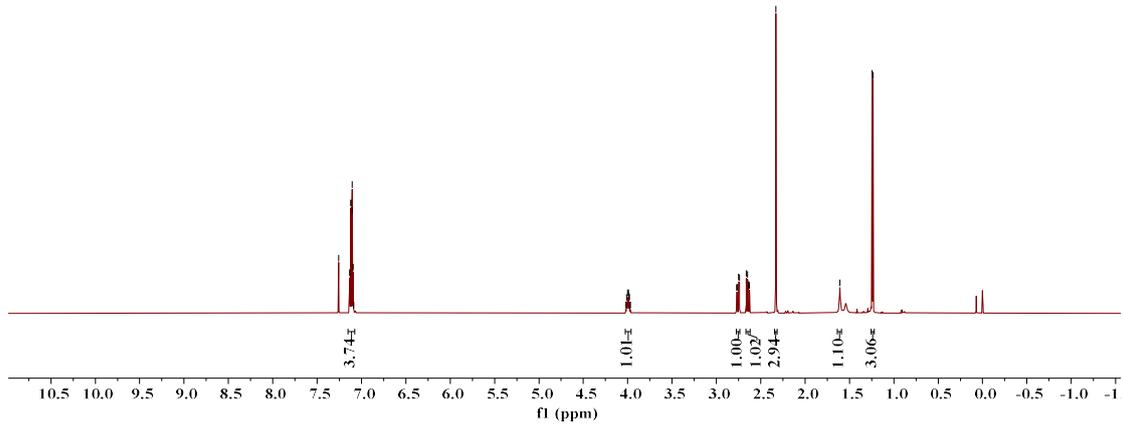
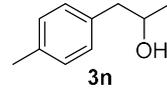




7.258
7.134
7.120
7.120
7.107
7.106
7.093
7.086

4.020
4.010
4.000
3.992
3.980
3.979
3.968
2.771
2.763
2.749
2.741
2.662
2.649
2.640
2.626
2.329
1.609
1.244
1.234

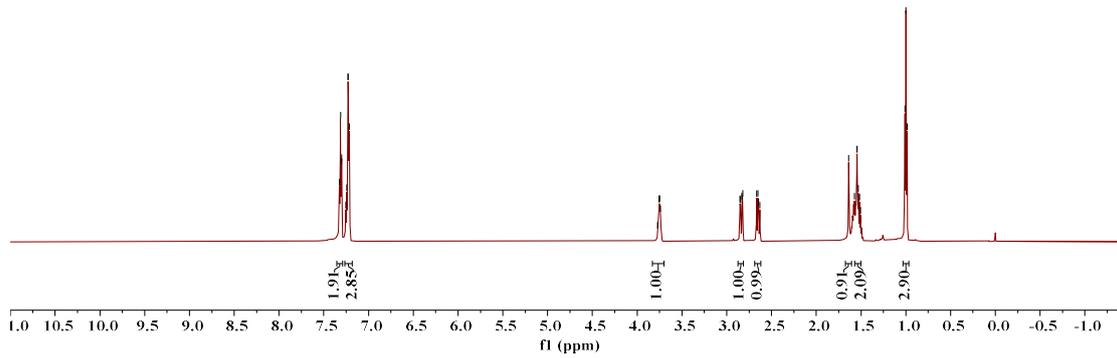
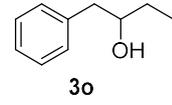
Parameters	
Parameter	value
Title	LH364-2.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-09-05T18:35:21
Spectrometer Frequency	600 MHz
Nucleus	1H



7.326
7.313
7.303
7.300
7.255
7.244
7.227
7.214

3.772
3.753
3.751
3.750
3.741
2.851
2.845
2.831
2.822
2.667
2.652
2.643
2.629
1.637
1.572
1.544
1.544
1.529
1.518
1.506
1.010
0.998
0.985

Parameters	
Parameter	value
Title	LH330-2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-08-15T08:48:22
Spectrometer Frequency	600 MHz
Nucleus	1H



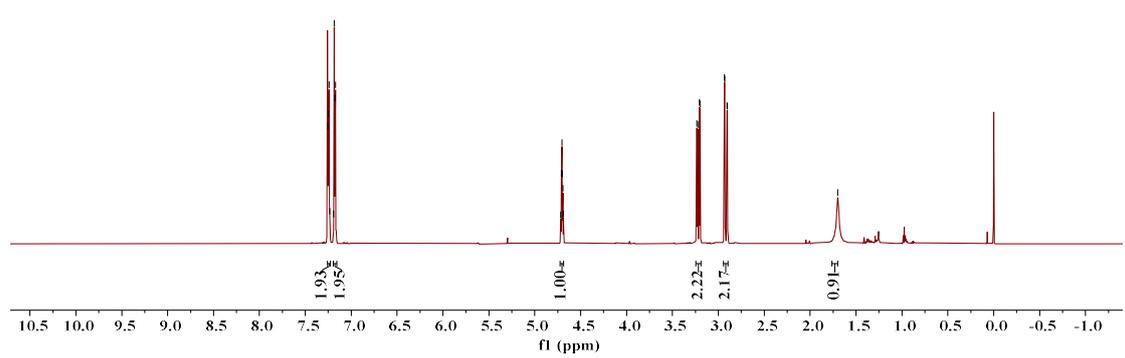
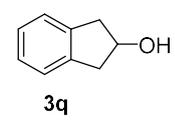
7.255
7.249
7.240
7.234
7.192
7.184
7.179
7.175
7.170

4.719
4.709
4.704
4.699
4.694
4.688

3.236
3.226
3.208
3.199
2.934
2.929
2.907
2.902

— 1.699

Parameters	
Parameter	value
Title	LH295-1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Number of Scans	4
Acquisition Date	2024-07-24T22:22:23
Spectrometer Frequency	600 MHz
Nucleus	1H

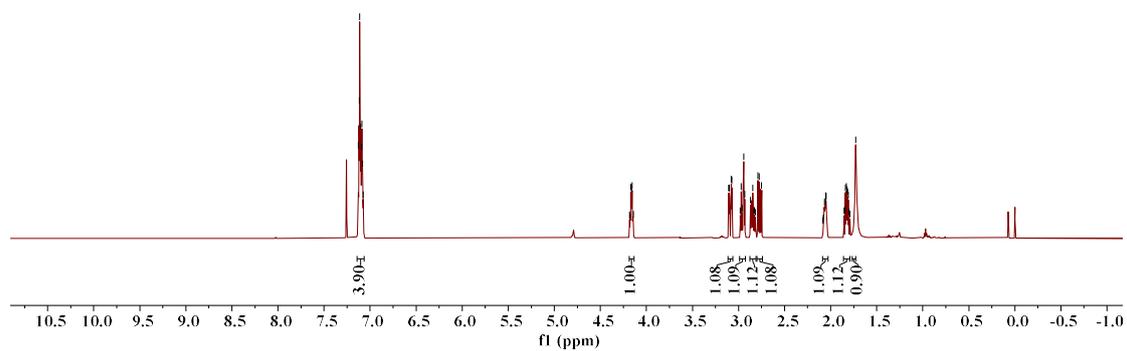
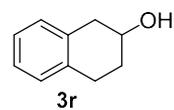


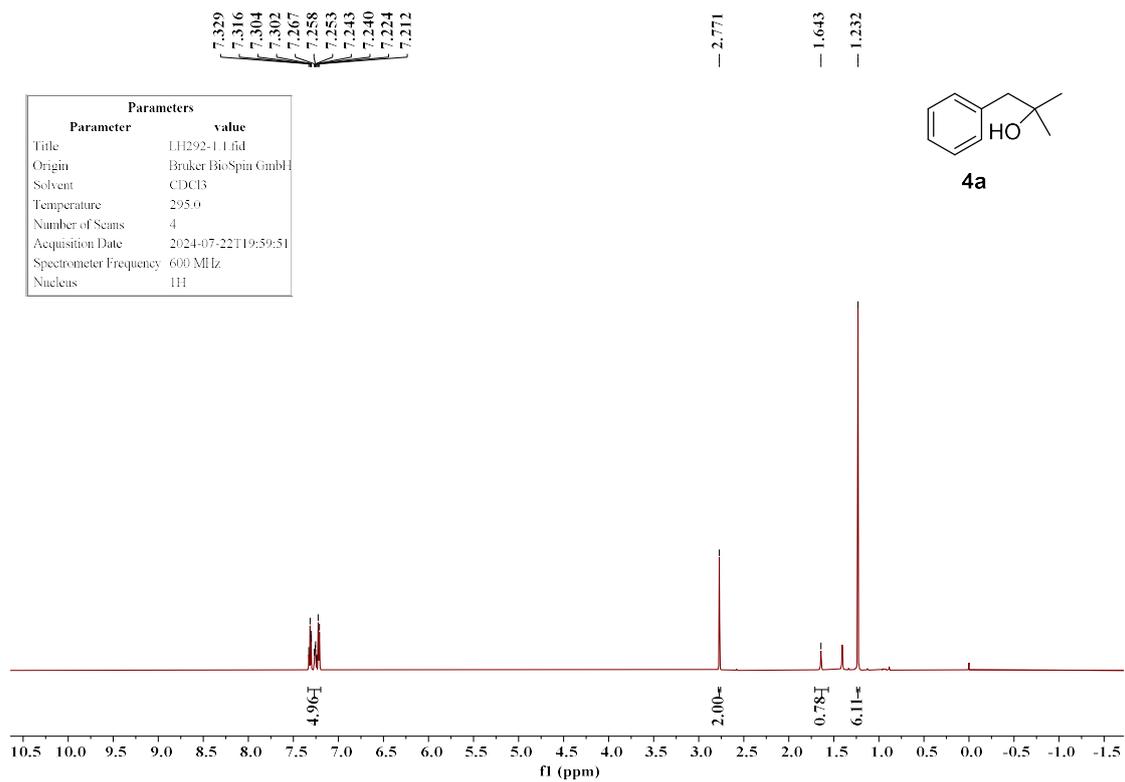
7.127
7.118
7.112
7.101
7.094
7.088
7.080
7.073

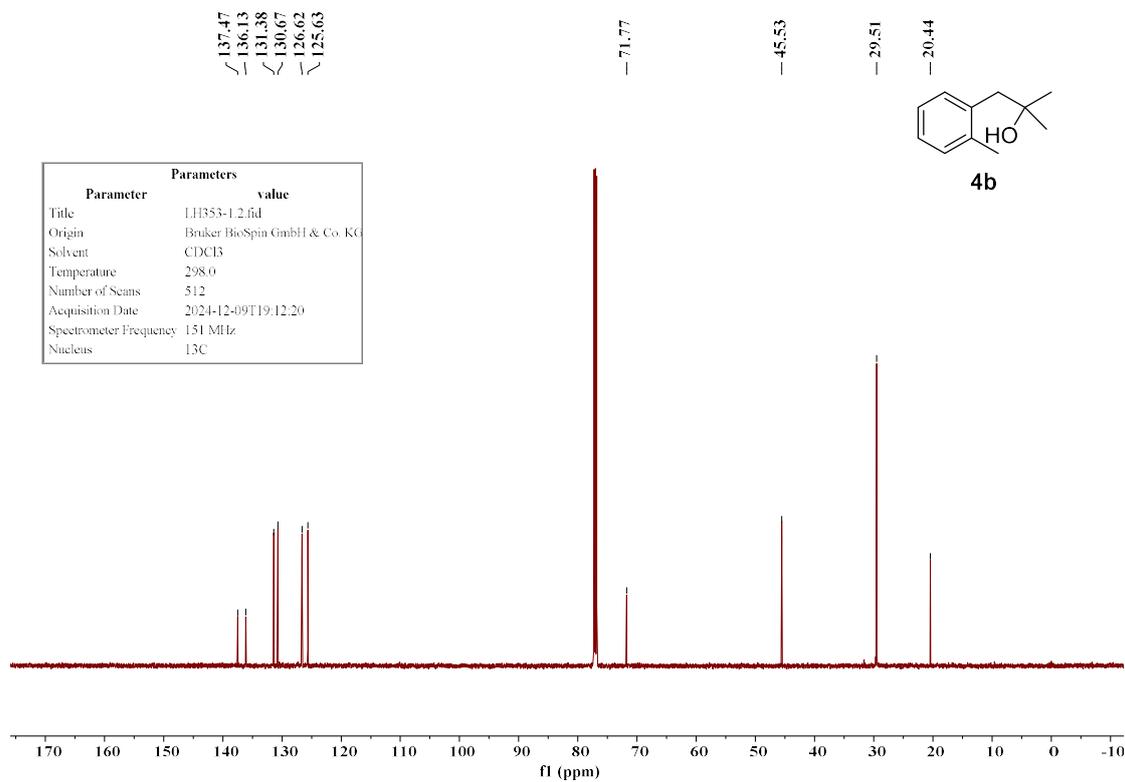
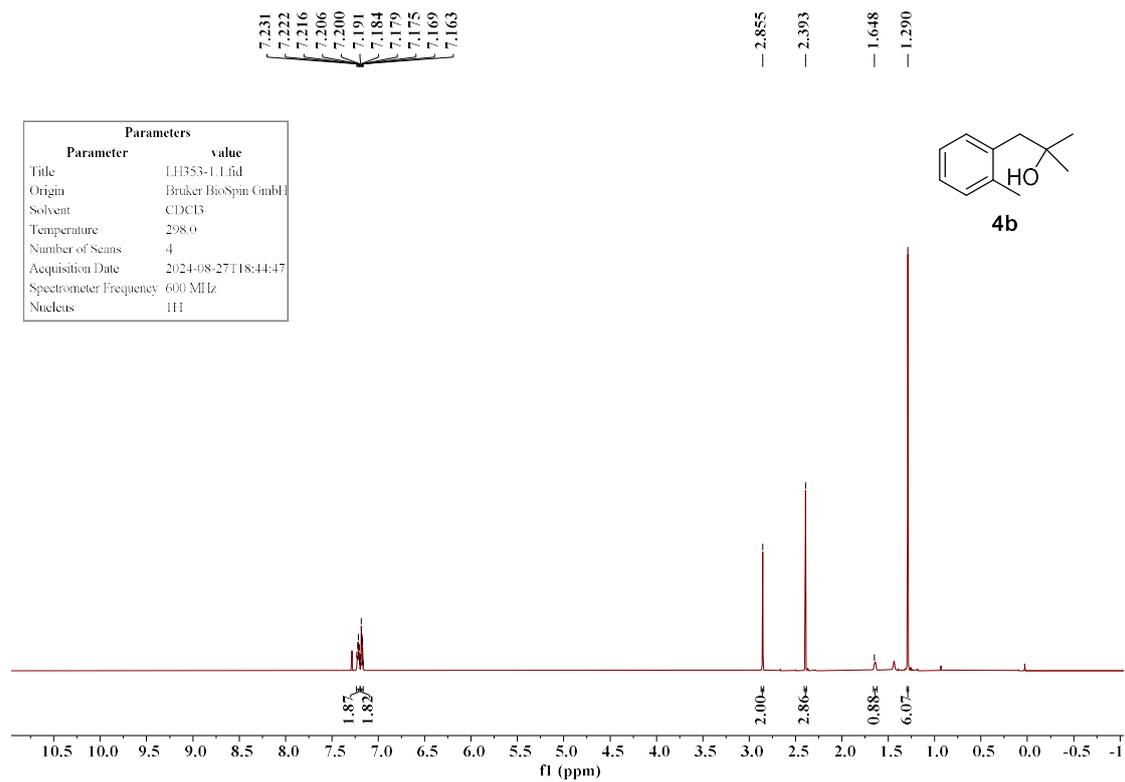
4.183
4.169
4.162
4.154
4.141

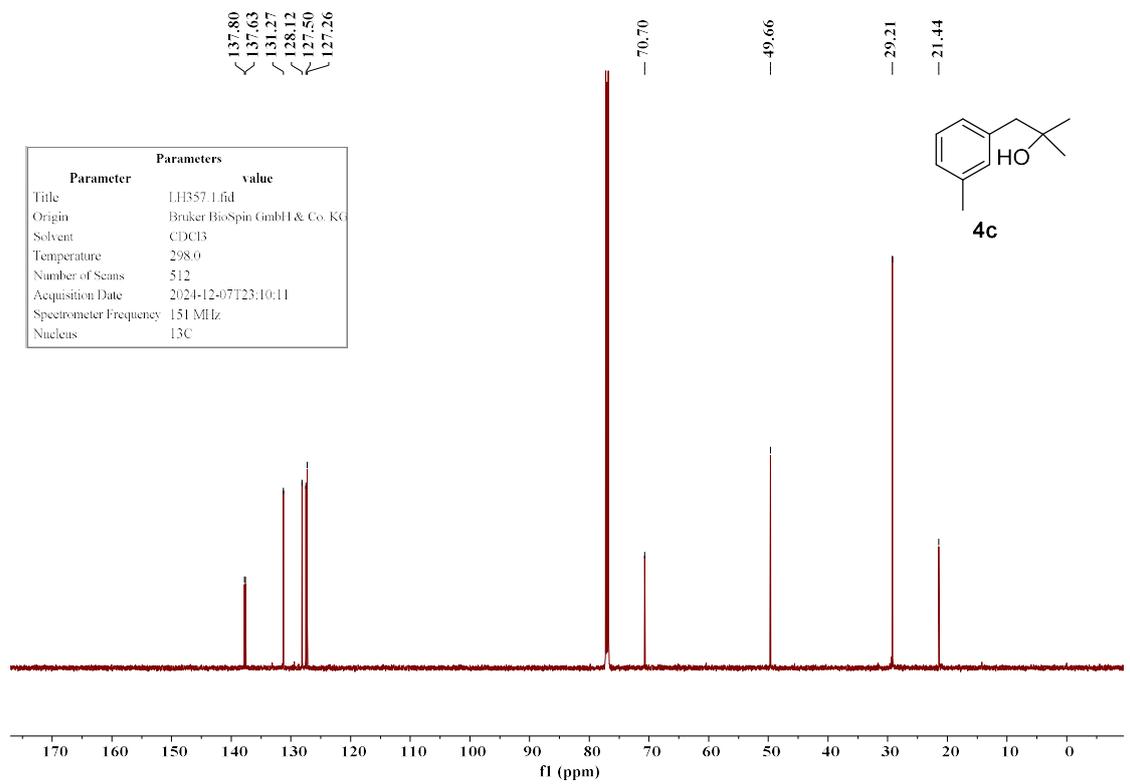
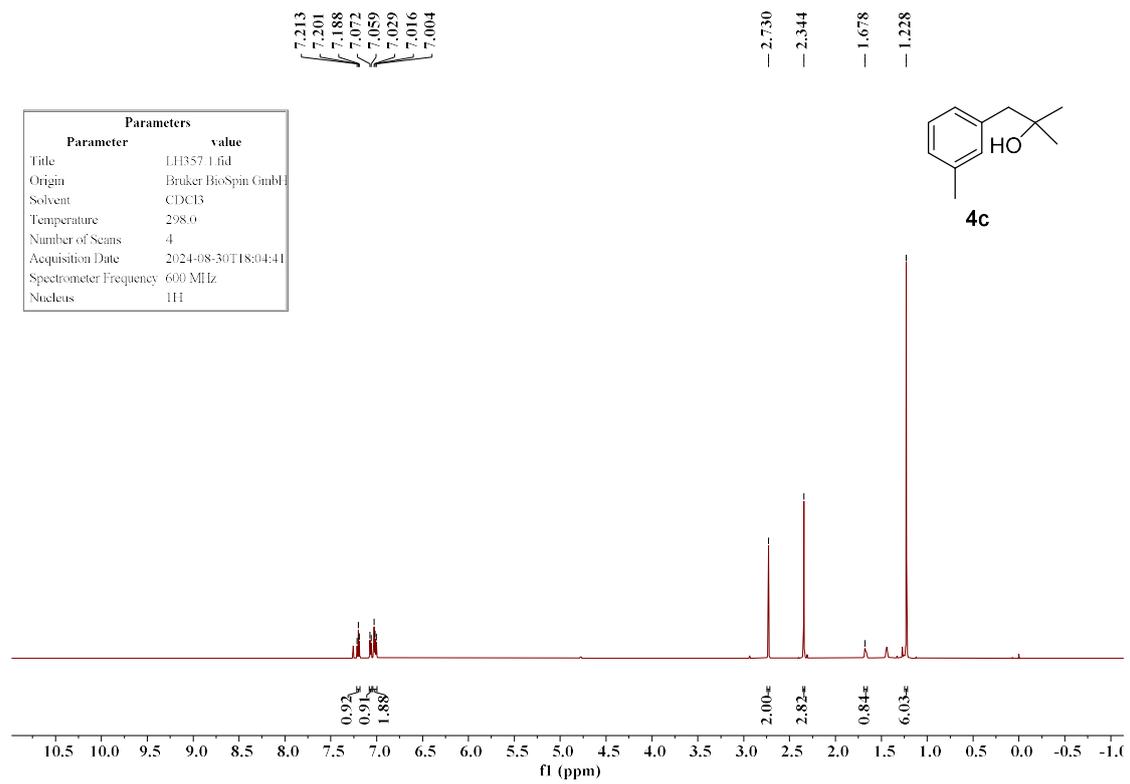
3.107
3.099
3.080
3.072
2.971
2.961
2.943
2.933
2.870
2.844
2.790
2.776
2.749
2.066
2.057
2.053
2.048
1.838
1.828
1.823
1.817
1.807
1.778

Parameters	
Parameter	value
Title	LH313-1-1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Number of Scans	4
Acquisition Date	2024-08-05T11:26:04
Spectrometer Frequency	600 MHz
Nucleus	1H





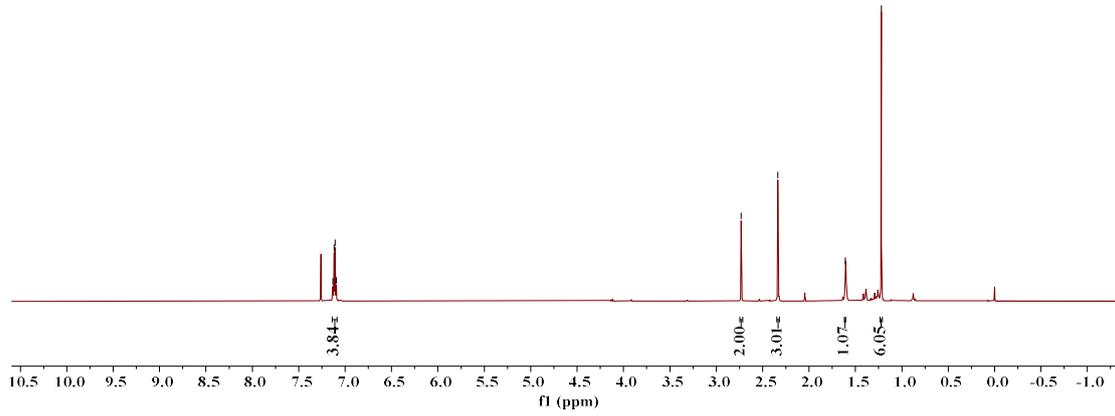
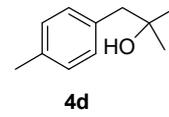


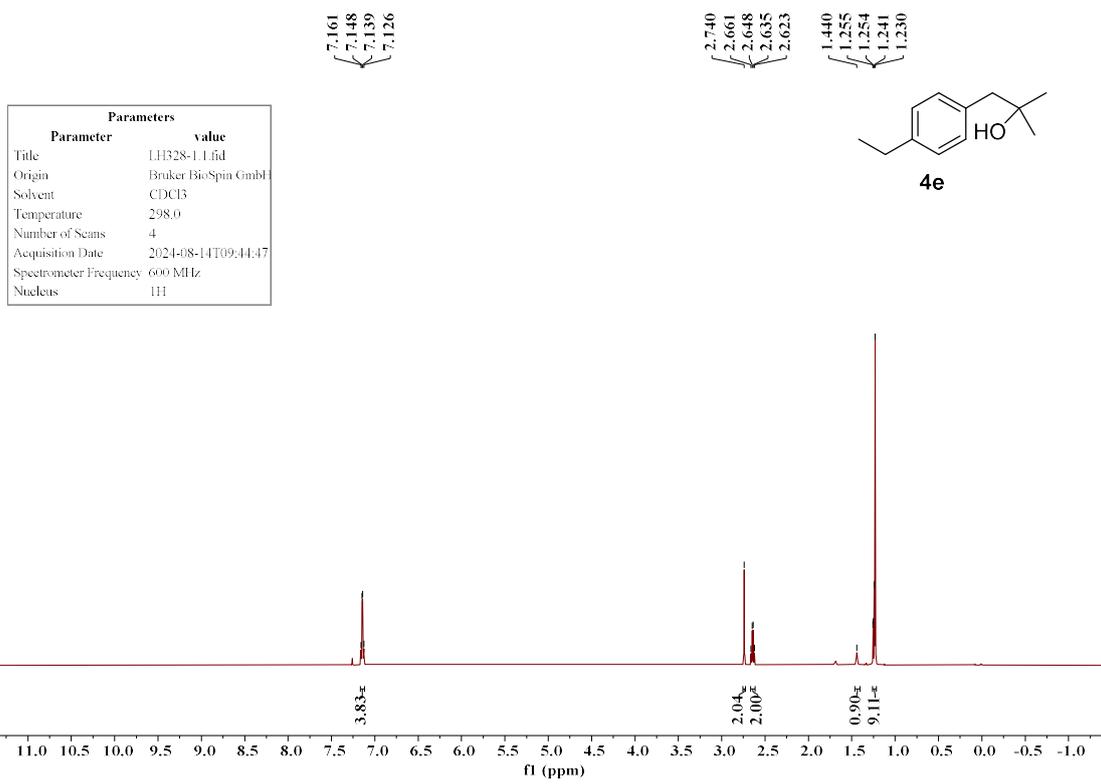


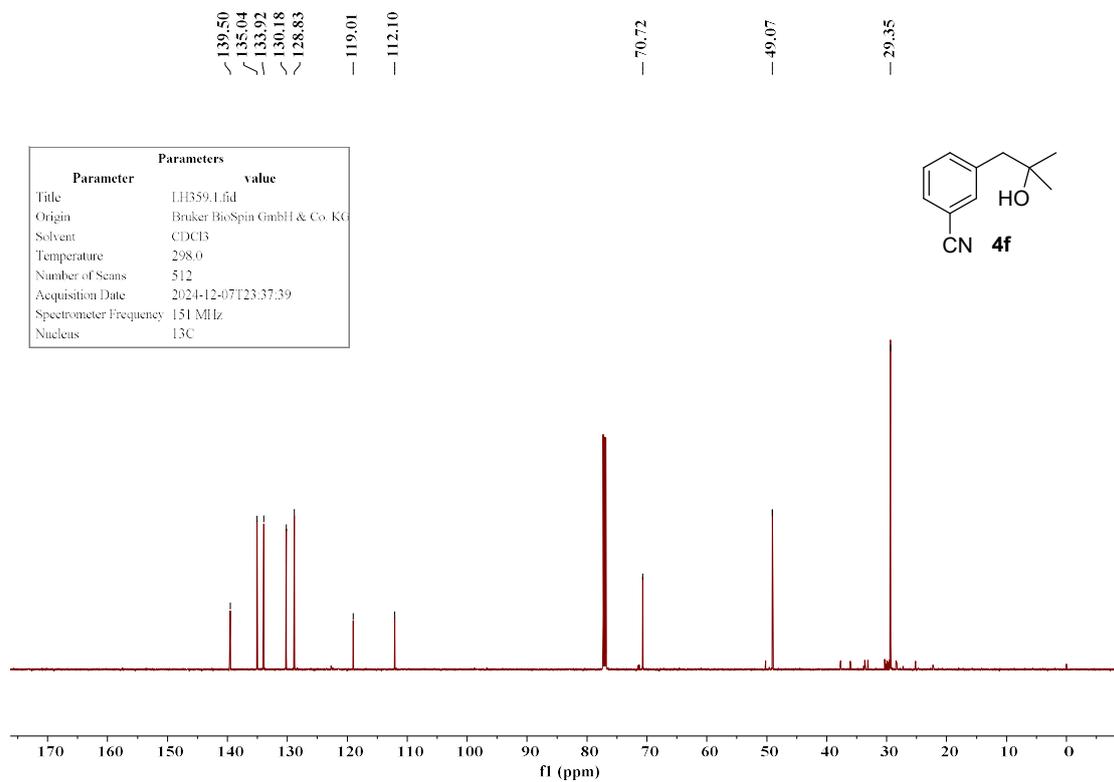
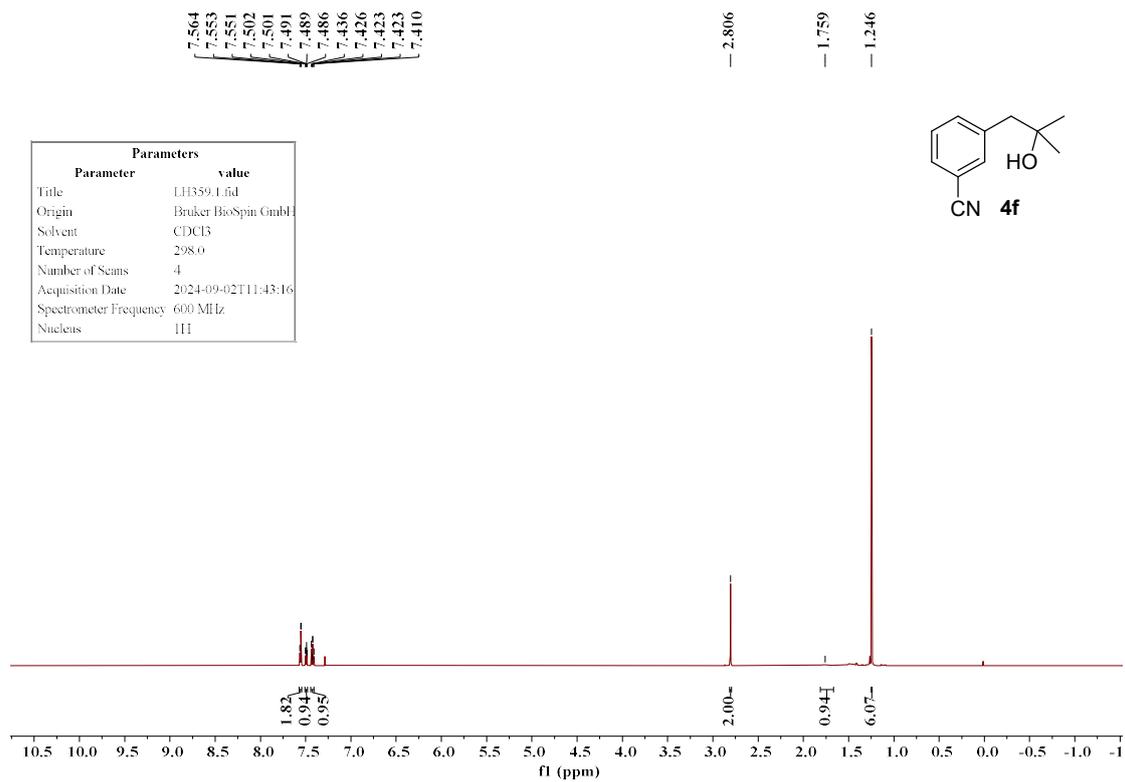
7.132
7.119
7.108
7.095

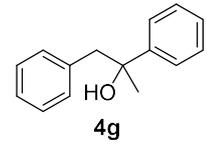
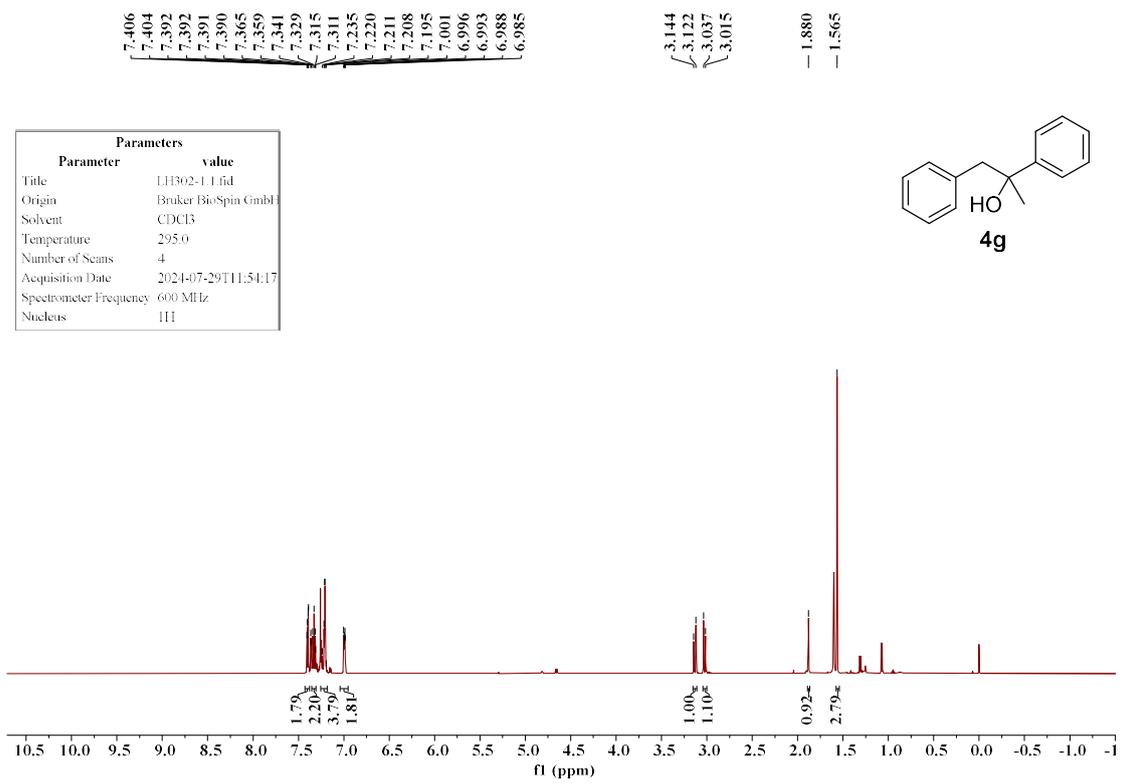
2.730
2.336
1.608
1.222

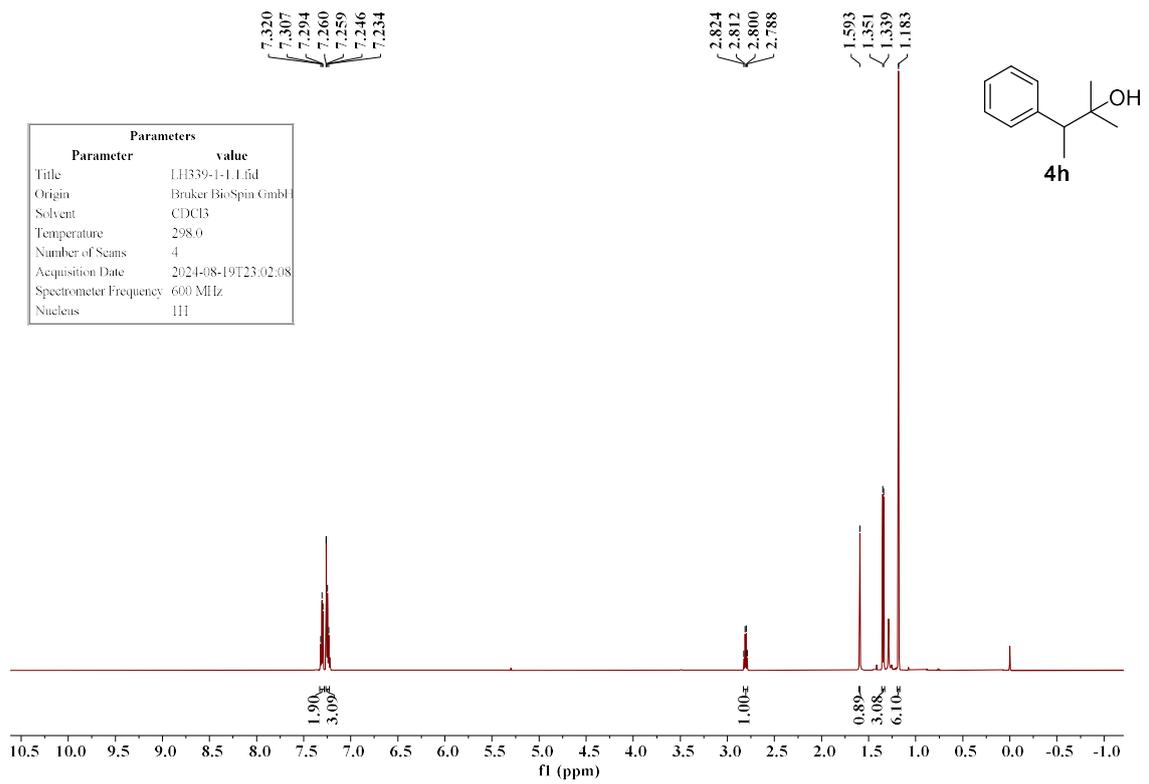
Parameters	
Parameter	value
Title	LH350-21.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-08-26T12:06:42
Spectrometer Frequency	600 MHz
Nucleus	¹ H







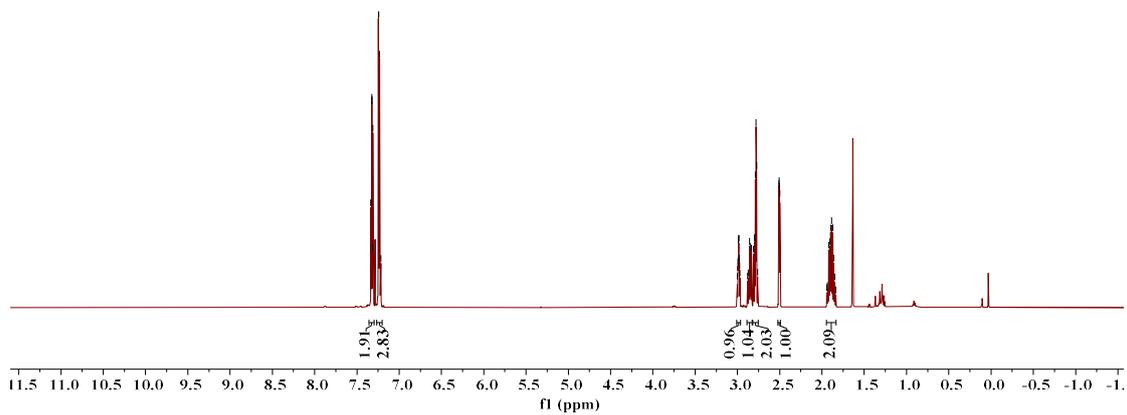
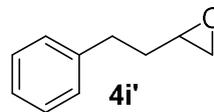




Parameters	
Parameter	value
Title	LH339-1-1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-08-19T23:02:08
Spectrometer Frequency	600 MHz
Nucleus	1H

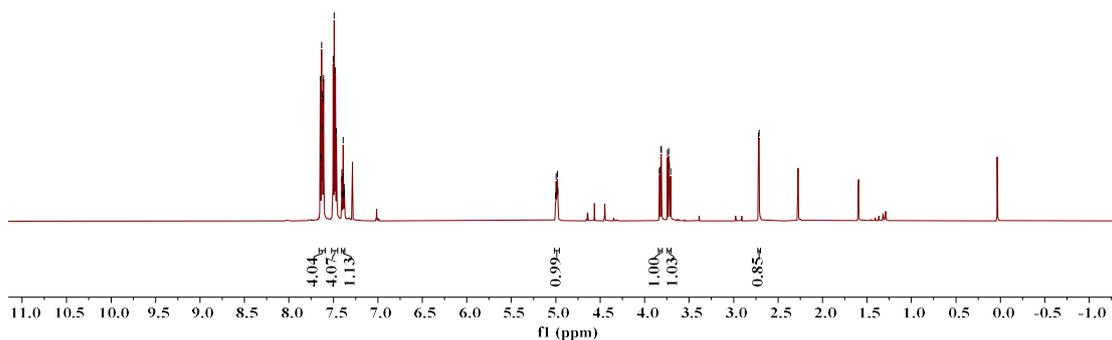
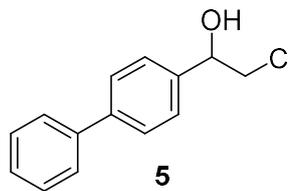
7.335
7.335
7.323
7.322
7.322
7.314
7.310
7.245
7.245
7.232
7.218
3.001
2.995
2.990
2.986
2.982
2.979
2.976
2.971
2.880
2.870
2.857
2.847
2.832
2.810
2.798
2.795
2.789
2.782
2.774
2.760
2.511
2.506
2.503
2.498
1.942
1.934
1.919
1.907
1.896
1.886
1.871
1.861
1.848
1.837

Parameters	
Parameter	value
Title	LH213-1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2024-05-29T18:32:20
Spectrometer Frequency	600 MHz
Nucleus	1H



7.647
7.637
7.634
7.626
7.624
7.612
7.610
7.503
7.489
7.477
7.464
7.406
7.403
7.401
7.394
7.391
7.381
7.379
7.377
5.001
4.996
4.981
4.976
3.835
3.829
3.816
3.811
3.742
3.727
3.723
3.708
2.719
2.714

Parameter	Parameters	value
Title	LH396 1 fid	
Origin	Broker BioSpin GmbH & Co. KG	
Solvent	CDCl3	
Temperature	298.0	
Number of Scans	4	
Acquisition Date	2024-12-12T18:39:42	
Spectrometer Frequency	600 MHz	
Nucleus	1H	



7.628
7.624
7.621
7.614
7.613
7.612
7.610
7.607
7.491
7.488
7.485
7.476
7.476
7.476
7.465
7.463
7.460
7.401
7.398
7.394
7.387
7.376
7.374
7.372

3.951
3.947
3.944
3.940
3.224
3.218
3.215
3.209
2.892
2.888
2.883
2.879

Parameter	Parameters	value
Title	LH397.1.fid	
Origin	Bruker BioSpin GmbH & Co. KG	
Solvent	CDCl3	
Temperature	298.0	
Number of Scans	4	
Acquisition Date	2024-11-06T19:02:57	
Spectrometer Frequency	600 MHz	
Nucleus	¹ H	

