

Supporting Information

**Electroreductive Divergent Hydrolysis and
Hydrogenation of 2-Oxazolidinones**

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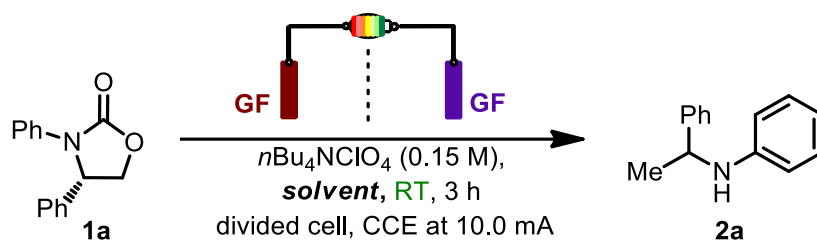
General Information

NMR spectra: ^1H -NMR spectra were recorded on 400 or 600 MHz spectrophotometers, ^{13}C NMR spectra were recorded on 100 or 150 MHz with complete proton decoupling spectrophotometers using CDCl_3 as solvent. Data were reported in the following order: chemical shift (δ) values are reported in ppm with the solvent resonance as internal standard (CDCl_3 : $\delta = 7.26$ ppm for ^1H , TMS: $\delta = 0$ ppm for ^1H , $\delta = 77.16$ ppm for ^{13}C); multiplicities are indicated brs (broadened singlet), s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet); coupling constants (J) are given in Hertz (Hz). **High Resolution Mass Spectrometry (HRMS):** All were recorded on Agilent 6210 ESI/TOF using a positive electrospray ionization (ESI^+). Measured values are reported to 4 decimal places of the calculated value. The calculated values are based on the most abundant isotope. **Chromatography:** Analytical thin layer chromatography was performed using Qingdao Puke Parting Materials Co. silica gel plates (Silicagel 60 F254). Visualisation was by ultraviolet fluorescence ($\lambda = 254$ nm) and/or staining with potassium permanganate (KMnO_4). Flash column chromatography was performed using 200-300 mesh silica gel.

All reactions were carried out under an air atmosphere using 5-5 mL H-type divided cell and Nafion 117 PFSA membranes. Electrolytes were dried in vacuum at $50\text{ }^\circ\text{C}$ for at least 4 h. If not noted, other commercial reagents were used without further purification. All starting materials are commercially available or prepared by the reported method.^[1] The electrochemical reactions were performed on a MESTEK DP3005B potentiostat (made in China) in constant current mode.

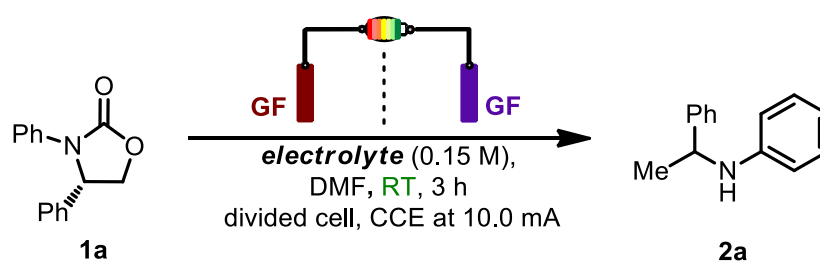
Optimization of the Reaction Conditions

Table S-1: Evaluation of Solvents



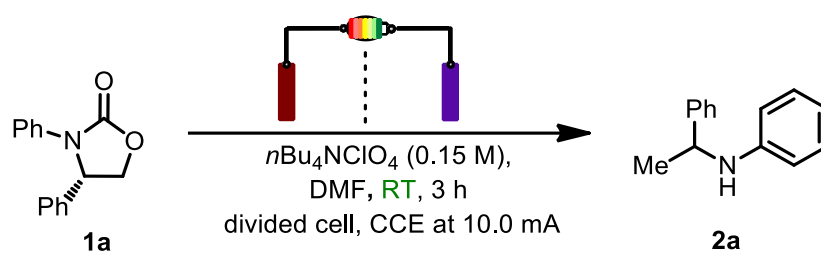
Entry	Solvent	Yield of 2a (%)
1	DMSO	66
2	Acetone	Trace
3	DMA	68
4	DMF	76
5	NMP	57
6	MeCN	Trace
7	MeOH	N.D.
8	DCM	N.D.

Reaction conditions: cathode: **1a** (0.3 mmol), $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in solvent (4.0 mL); anode: $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in solvent (4.0 mL). Graphite felt as anode and cathode in an H-type divided cell, room temperature, CCE = 10 mA, 3.0 h, air atmosphere. Yields of isolated products. DMA = *N,N*-dimethylacetamide; DMF = *N,N*-dimethylformamide; NMP = *N*-Methyl-2-pyrrolidone; GF = graphite felt; CCE = constant current electrolysis.

Table S-2: Evaluation of Electrolytes

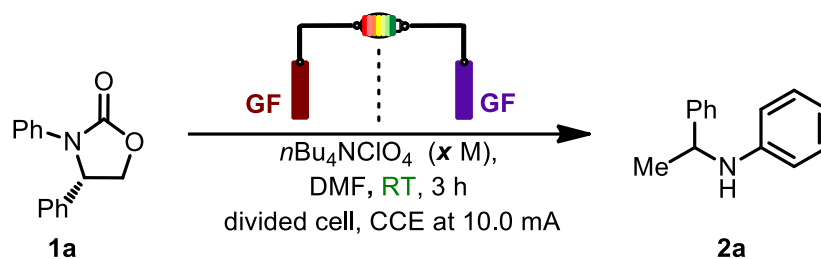
Entry	Electrolytes	Yield of 2a (%)
1	<i>n</i>Bu₄NClO₄	76
2	<i>n</i> Bu ₄ NI	45
3	<i>n</i> Bu ₄ NBr	59
4	<i>n</i> Bu ₄ NCl	54
5	<i>n</i> Bu ₄ NPF ₆	69
6	<i>n</i> Bu ₄ NBF ₄	24
7	NaClO ₄	7
8	Et ₄ NClO ₄	66

Reaction conditions: cathode: **1a** (0.3 mmol), electrolyte (0.15 M) in DMF (4.0 mL); anode: electrolyte (0.15 M) in DMF (4.0 mL). Graphite felt as anode and cathode in an H-type divided cell, room temperature, CCE = 10 mA, 3.0 h, air atmosphere. Yields of isolated products. GF = graphite felt; CCE = constant current electrolysis.

Table S-3: Evaluation of Electrodes

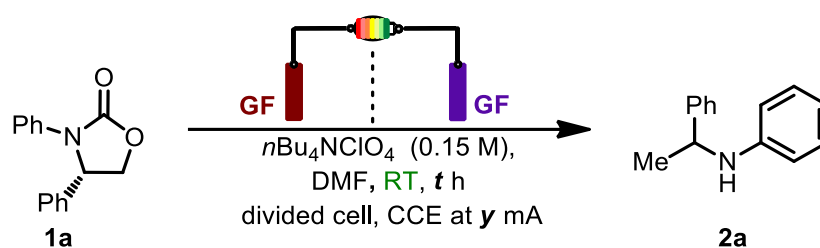
Entry	Anode	Cathode	Yield of 2a (%)
1	GF	Pt	59
2	GF	Graphite plate	54
3	GF	CC	63
4	GF	GF	76
5	Graphite plate	GF	58
6	CC	GF	59

Reaction conditions: cathode: **1a** (0.3 mmol), $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL); anode: $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL). Anode and cathode in an H-type divided cell, room temperature, CCE = 10 mA, 3.0 h, air atmosphere. Yields of isolated products. GF = graphite felt; CC = carbon cloth; CCE = constant current electrolysis.

Table S-4: Evaluation of Concentration of Electrolyte

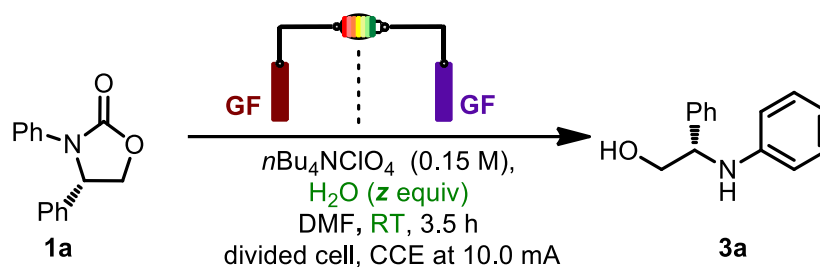
Entry	Concentration of electrolyte	Yield of 2a (%)
1	0.15	76
2	0.25	68
3	0.35	67

Reaction conditions: cathode: **1a** (0.3 mmol), $n\text{Bu}_4\text{NClO}_4$ (x M) in DMF (4.0 mL); anode: $n\text{Bu}_4\text{NClO}_4$ (x M) in DMF (4.0 mL). Graphite felt as anode and cathode in an H-type divided cell, room temperature, CCE = 10 mA, 3.0 h, air atmosphere. Yields of isolated products. GF = graphite felt; CCE = constant current electrolysis.

Table S-5: Evaluation of Current and Reaction Time

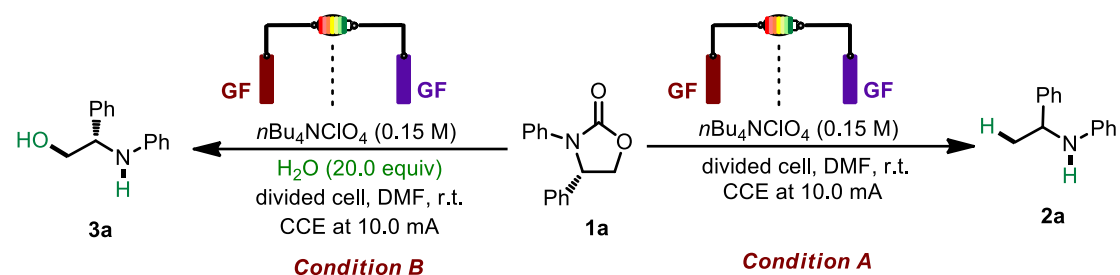
Entry	Constant current	t (h)	Yield of 2a (%)
1	0	3	N.D.
2	5	6	61
3	10	3	76
4	15	1.5	69

Reaction conditions: cathode: **1a** (0.3 mmol), $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL); anode: $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL). Graphite felt as anode and cathode in an H-type divided cell, room temperature, CCE = y mA, t h, air atmosphere. Yields of isolated products. GF = graphite felt; CCE = constant current electrolysis.

Table S-6: Evaluation of Equivalent of H₂O

Entry	Equivalent of H ₂ O	Yield of 3a (%)
1	5	71
2	10	73
3	20	92
4	30	81
5	DMF:H ₂ O=5:1	Trace
6	DMF:H ₂ O=1:1	N.D.
7	MeOH instead of H ₂ O	N.D.

Reaction conditions: cathode: **1a** (0.3 mmol), H₂O (z equiv), *n*Bu₄NClO₄ (0.15 M) in DMF (4.0 mL); anode: H₂O (z equiv), *n*Bu₄NClO₄ (0.15 M) in DMF (4.0 mL). Graphite felt as anode and cathode in an H-type divided cell, room temperature, CCE = 10 mA, 3.5 h, air atmosphere. Yields of isolated products. GF = graphite felt; CCE = constant current electrolysis.

Table S-7: Variation from Optimal Conditions

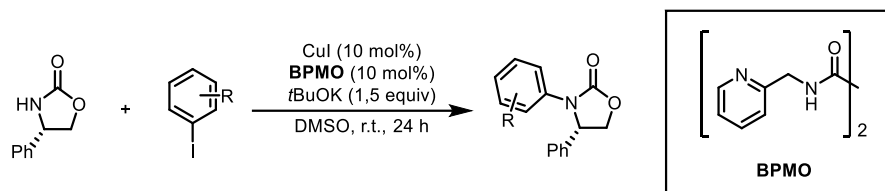
Entry	Variation from condition A ^a and B ^b	Yield of 2a (%)	Yield of 3a (%)
1	none	76	92
2	undivided cell	N.D.	N.D.
3	DMA as solvent	68	64
4	DMSO as solvent	66	39
5	CH_3CN as solvent	Trace	72
6	$n\text{Bu}_4\text{NI}$ as electrolyte	45	81
7	$n\text{Bu}_4\text{NPF}_6$ as electrolyte	69	64
8	Et_4NClO_4 as electrolyte	66	80
9	GF (+) / CC (-)	63	86
10	GF (+) / Pt (-)	59	63
11	5 mA (6.0 h)	66	65
12	15 mA (2.0 h)	69	69
13	N_2 atmosphere	72	83
14	no electricity	N.D.	N.D.

[a] Hydrogenation reaction conditions: divided cell, anode(GF), cathode (GF), cathode: **1a** (0.3 mmol), $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL), anode: $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL), CCE = 10 mA, room temperature, $t = 3$ h. Yields of isolated product. [b] Hydrolysis reaction conditions: divided cell, anode (GF), cathode (GF), cathode: **1a** (0.3 mmol), H_2O (20.0 equiv), $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL), Anode: H_2O (20.0 equiv), $n\text{Bu}_4\text{NClO}_4$ (0.15 M) in DMF (4.0 mL), CCE = 10 mA, room temperature, $t = 3.5$ h. Yields of isolated product. DMA = *N,N*-dimethylacetamide, DMF = *N,N*-dimethylformamide; GF = graphite felt; CC = carbon cloth; CCE = constant current electrolysis.

Preparation of Substrates

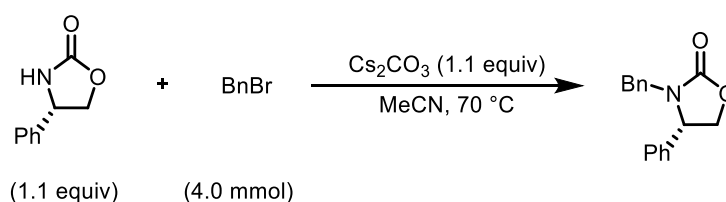
All starting materials were obtained from commercial sources or synthesized according to the following procedure^[1]:

N-aryl-2-oxazolidinones were obtained from following synthetic methods:



Procedure: In a flame-dried round bottom flask equipped with a magnetic stirring bar, *(S)*-4-benzyl-2-oxazolidinones (5.0 mmol), CuI (95.3 mg, 0.5 mmol) and BPMO (135.2 mg, 0.5 mmol) were dissolved in dry DMSO (5 mL) under nitrogen atmosphere, then *t*BuOK (7.5 mmol) and aryl iodide (5.0 mmol) were added to the mixture and the reaction mixture was stirred for 24 h. After completion of the reaction was monitored by TLC, water (10 mL) was added. The aqueous phase was extracted with EtOAc (15 mL×3). The combined organic phase was dried over Na₂SO₄ and filtered. Evaporation of the solvent and purification by column chromatography on silica gel (petroleum ether/EtOAc: 5:1) yielded the corresponding *N*-aryl-2-oxazolidinones.

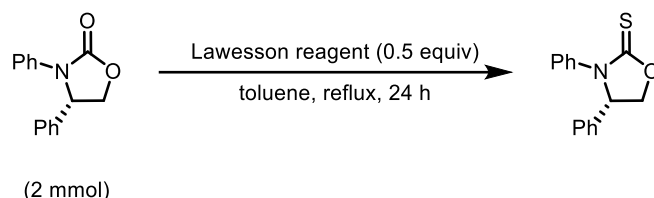
N-benzyl-2-oxazolidinone was obtained from following synthetic methods^[2]:



Procedure: In a flame-dried round bottom flask equipped with a magnetic stirring bar, *(S)*-4-benzyl-2-oxazolidinone (4.4 mmol), Cs₂CO₃ (4.4 mmol) and BnBr (4.0 mmol) were dissolved in dry MeCN (13 mL), then the reaction mixture was stirred at 70 °C overnight. After completion of the reaction was monitored by TLC, a saturated aqueous NH₄Cl solution (20 mL) was added after the mixture was cooled to room temperature. The aqueous phase was extracted with EtOAc (15 mL×3). The combined organic phase was dried over Na₂SO₄ and filtered. Evaporation of the solvent and purification by

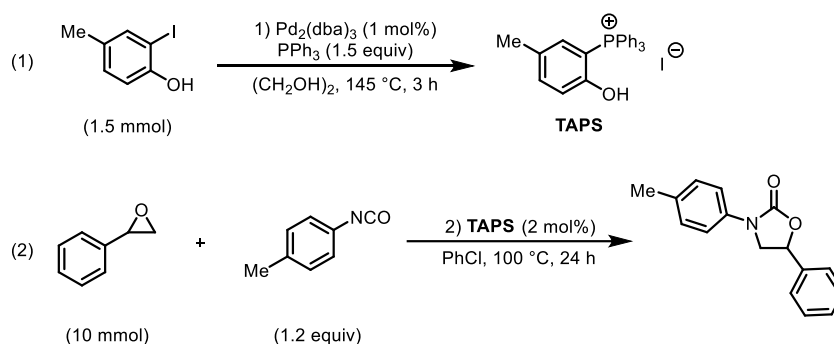
column chromatography on silica gel (petroleum ether/EtOAc: 10:1) yielded the corresponding *N*-benzyl-2-oxazolidinones.

(*S*)-3,4-diphenyloxazolidine-2-thione was obtained from following synthetic methods^[3]:



Procedure: In a flame-dried Schlenk tube equipped with a magnetic stirring bar, the dry toluene solution was added (*S*)-3,4-diphenyl-2-oxazolidinone (2.0 mmol) under nitrogen atmosphere, then Lawesson reagent (1.0 mmol) was added in portion and the mixture was stirred at 100 °C for 24 h. After completion of the reaction was monitored by TLC, Evaporation of the solvent under reduced pressure after the mixture was cooled to room temperature, then purification by column chromatography on silica gel (DCM) yielded the corresponding product.

5-phenyl-3-(*p*-tolyl)oxazolidin-2-one was obtained from following synthetic methods:



Procedure: 1) The PPh₃ (2.25 mmol) and Pd₂(dba)₂ (0.015 mmol) were added to a 5 mL Schlenk tube and dissolved in 1.0 mL of anhydrous ethylene glycol under nitrogen atmosphere. 2-iodo-4-methylphenol (1.5 mmol) was added, followed by stirring the reaction at 145 °C for 3h. Then the mixture was washed with cold THF solution after cooled to room temperature, filter out the white solid to obtain the target product TAPS.

2) To a 50 mL two-necked flask was added TAPS (0.2 mmol) and anhydrous chlorobenzene (10 mL) under nitrogen atmosphere. 2-phenyloxirane (10 mmol) and 1-isocyanato-4-methylbenzene (12 mmol) were added. followed by stirring the reaction at 100 °C for 24 h. After completion of the reaction monitored by TLC, purification of the mixture by column chromatography on silica gel (petroleum ether/EtOAc: 10:1) yielded the corresponding product.

General Procedure: Electroreduction of 2-Oxazolidinones

Hydrogenation reaction: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with 2-Oxazolidinones (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg each, 0.15 M), DMF (4.0 mL each). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 2-3 hours. Upon the reduction, the reaction mixture was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3 × 15 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to furnish the corresponding products.

Hydrolysis reaction: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with 2-Oxazolidinones (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg each, 0.15 M), DMF (4.0 mL each) and deionized H₂O (108 μL each, 20.0 equiv). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3-4 hours. Upon the reduction, the reaction mixture was quenched by 20mL of water. The aqueous phase was extracted with ethyl acetate. The combined organic portions were washed with H₂O (3 × 15 mL) and saturated brine. The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to furnish the corresponding products.

Photographic Guide for Electrochemical Reactions

Overview of materials used: from left to right: (a) Graphite felt electrode (15 mm×10 mm×3 mm). (b) Electrochemical cell.

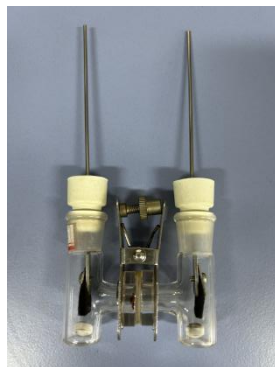
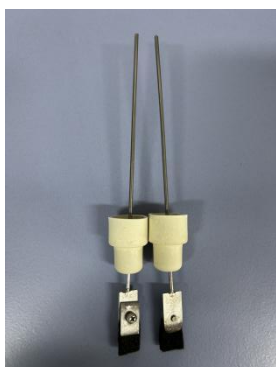


(a)

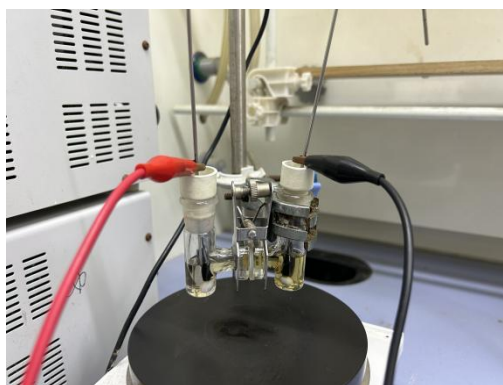


(b)

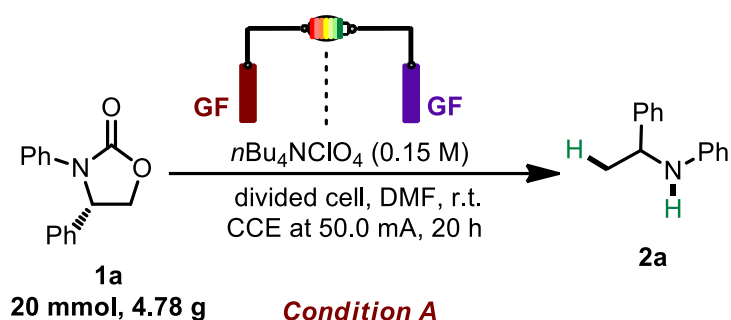
Assembling the cell



Electrolysis

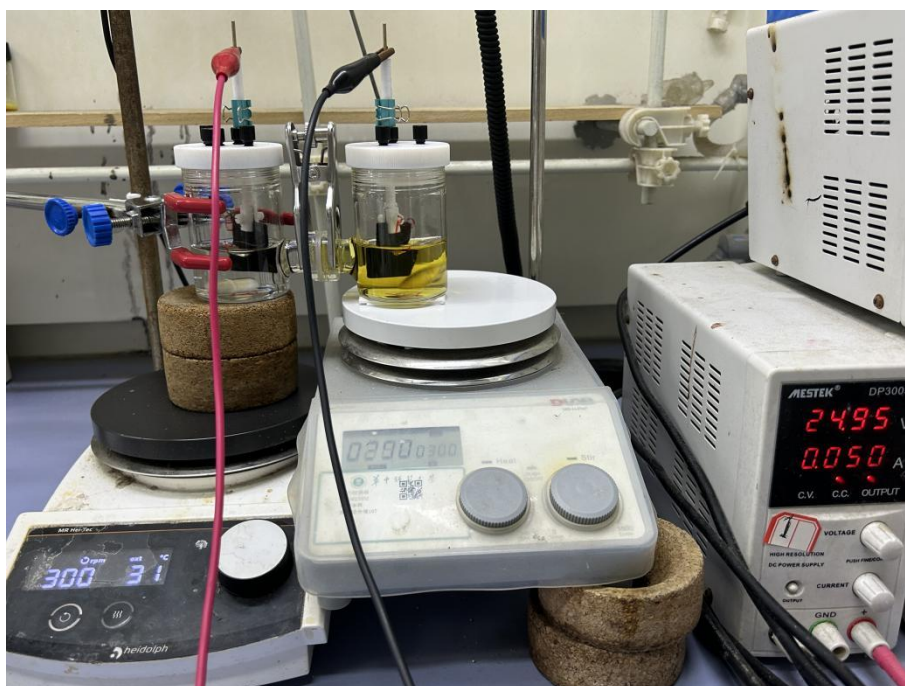


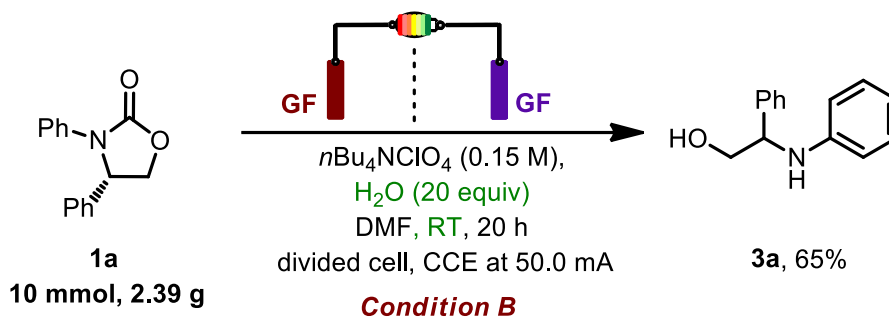
Scalability of Electrochemical Reduction



Hydrogenation reaction: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (20 mmol, 4.78 g, 1.0 equiv), and both chambers were charged with $n\text{Bu}_4\text{NClO}_4$ (3.08 g, each, 0.15 M), DMF (60 mL, each). Graphite felts (30 mm×30 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 50 mA for 20 h. Upon the reduction, the reaction mixture was transferred to 150 mL of ethyl acetate and subjected to washing with saturated brine (3×60 mL). The organic fractions were dried by Na_2SO_4 , filtered and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to furnish the desired product **2a** (2.24 g) in 57% yield.

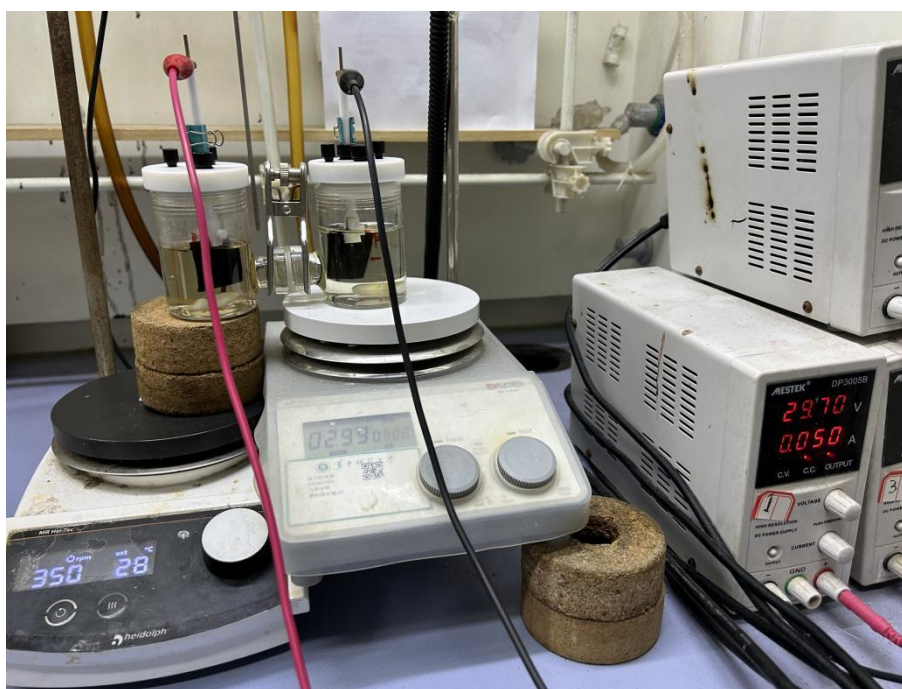
The Photographic Guide for Scale-up Electrochemical Hydrogenation Reaction



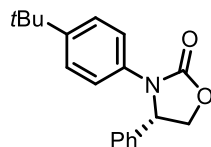


Hydrolysis reaction: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (10 mmol, 2.39 g, 1.0 equiv), and both chambers were charged with $n\text{Bu}_4\text{NClO}_4$ (4.10 g, each, 0.15 M), DMF (80 mL, each) and H_2O (3.6 mL each, 20.0 equiv). Graphite felts (30 mm×30 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 50 mA for 24 h. Upon the reduction, the reaction mixture was transferred to 150 mL of ethyl acetate and subjected to washing with saturated brine (3 × 80 mL). The organic fractions were dried by Na_2SO_4 , filtered and concentrated in vacuo. The crude product was purified by column chromatography on silica gel to furnish the desired product **3a** (1.39 g) in 65% yield.

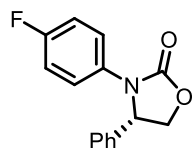
The Photographic Guide for Scale-up Electrochemical Hydrolysis Reaction:



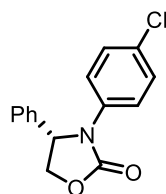
Characterization Data of Substrates



(S)-3-(4-(tert-butyl)phenyl)-4-phenyloxazolidin-2-one (1c): Prepared from (S)-4-phenyloxazolidin-2-one and 1-(*tert*-butyl)-4-iodobenzene following the reported literature procedure^[1c] and obtained as a white solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.40–7.30 (m, 5H), 7.30–7.24 (m, 4H), 5.36 (dd, *J* = 8.8, 5.9 Hz, 1H), 4.77 (t, *J* = 8.7 Hz, 1H), 4.19 (dd, *J* = 8.6, 5.9 Hz, 1H), 1.24 (s, 9H). ¹³C-NMR (100 MHz, CDCl₃) δ 156.2, 147.6, 138.7, 134.5, 129.5, 128.9, 126.4, 125.9, 120.6, 70.0, 60.9, 34.4, 31.3. **HR-MS** (ESI) *m/z* calcd for C₁₉H₂₁NO₂ [M+H]⁺ 296.1645, found 296.1636.

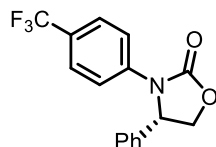


(S)-3-(4-fluorophenyl)-4-phenyloxazolidin-2-one (1f): Prepared from (S)-4-phenyloxazolidin-2-one and 1-fluoro-4-iodobenzene following the reported literature procedure^[1c] and obtained as a yellow solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.34–7.34 (m, 7H), 6.94 (t, *J* = 8.7 Hz, 2H), 5.34 (dd, *J* = 8.8, 6.3 Hz, 1H), 4.79 (t, *J* = 8.7 Hz, 1H), 4.22 (dd, *J* = 8.7, 6.3 Hz, 1H). ¹³C-NMR (100 MHz, CDCl₃) δ 159.7 (d, *J* = 244.7 Hz), 156.2, 137.9, 133.0 (d, *J* = 2.9 Hz), 129.5, 129.1, 126.5, 123.0 (d, *J* = 8.1 Hz), 115.77 (d, *J* = 22.5 Hz), 69.9, 61.1. ¹⁹F-NMR (377 MHz, CDCl₃) δ -117.26 (m, 1F). **HR-MS** (ESI) *m/z* calcd for C₁₅H₁₂FNO₂ [M+H]⁺ 258.0925, found 258.0917.

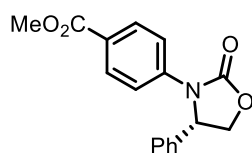


(S)-3-(4-chlorophenyl)-4-phenyloxazolidin-2-one (1'e): Prepared from (S)-4-phenyloxazolidin-2-one and 1-chloro-4-iodobenzene following the reported literature procedure^[1c] and obtained as a yellow solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.39 – 7.24

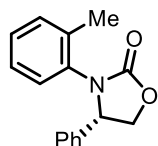
(m, 7H), 7.22 – 7.17 (m, 2H), 5.34 (dd, $J = 8.8, 6.1$ Hz, 1H), 4.77 (t, $J = 8.7$ Hz, 1H), 4.19 (dd, $J = 8.7, 6.1$ Hz, 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 155.8, 137.8, 135.7, 129.9, 129.5, 129.1, 129.0, 126.3, 122.0, 69.9, 60.6. **HR-MS** (ESI) m/z calcd for $\text{C}_{15}\text{H}_{12}\text{ClNO}_2$ $[\text{M}+\text{H}]^+$ 274.0629, found 274.0620.



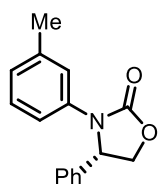
(S)-4-phenyl-3-(4-(trifluoromethyl)phenyl)oxazolidin-2-one (1g): Prepared from (S)-4-phenyloxazolidin-2-one and 1-iodo-4-(trifluoromethyl)benzene following the reported literature procedure^[1c] and obtained as a yellow solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) ^1H NMR (400 MHz, Chloroform-*d*) δ 7.53 (q, $J = 8.8$ Hz, 4H), 7.42–7.32 (m, 3H), 7.29 (d, $J = 6.2$ Hz, 2H), 5.43 (dd, $J = 8.7, 5.8$ Hz, 1H), 4.82 (t, $J = 8.7$ Hz, 1H), 4.23 (dd, $J = 8.6, 5.8$ Hz, 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 159.7 (d, $J = 244.7$ Hz), 156.2, 137.9, 133.0 (d, $J = 2.9$ Hz), 129.5, 129.1, 126.5, 123.0 (d, $J = 8.1$ Hz), 115.77 (d, $J = 22.5$ Hz), 69.9, 61.1. $^{19}\text{F-NMR}$ (377 MHz, CDCl_3) δ -62.28 (s, 3F). **HR-MS** (ESI) m/z calcd for $\text{C}_{16}\text{H}_{12}\text{F}_3\text{NO}_2$ $[\text{M}+\text{H}]^+$ 308.0893, found 308.0882.



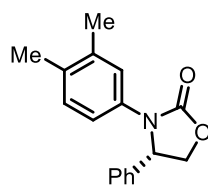
methyl (S)-4-(2-oxo-4-phenyloxazolidin-3-yl)benzoate (1i): Prepared from (S)-4-phenyloxazolidin-2-one and methyl 4-iodobenzoate following the reported literature procedure^[1c] and obtained as a white solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.92 (d, $J = 8.9$ Hz, 2H), 7.51 (d, $J = 8.9$ Hz, 2H), 7.41–7.26 (m, 5H), 5.44 (dd, $J = 8.7, 5.6$ Hz, 1H), 4.80 (t, $J = 8.7$ Hz, 1H), 4.22 (dd, $J = 8.6, 5.6$ Hz, 1H), 3.85 (s, 3H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 166.5, 155.5, 141.2, 137.9, 130.6, 129.7, 129.1, 126.1, 125.8, 119.5, 69.9, 60.4, 52.2. **HR-MS** (ESI) m/z calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_4$ $[\text{M}+\text{H}]^+$ 298.1074, found 298.1063.



(S)-4-phenyl-3-(*o*-tolyl)oxazolidin-2-one (1j): Prepared from (S)-4-phenyloxazolidin-2-one and 1-iodo-2-methylbenzene following the reported literature procedure^[1c] and obtained as a yellow solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.35–7.29 (m, 5H), 7.18 (d, *J* = 7.0 Hz, 1H), 7.11 (t, *J* = 7.5 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 7.8 Hz, 1H), 5.23 (dd, *J* = 8.7, 7.4 Hz, 1H), 4.83 (t, *J* = 8.8 Hz, 1H), 4.42 (dd, *J* = 8.9, 7.4 Hz, 1H), 2.30 (s, 3H). **¹³C-NMR** (100 MHz, CDCl₃) δ 156.5, 137.6, 136.2, 134.8, 131.5, 129.4, 129.2, 129.2, 128.0, 127.5, 126.7, 70.2, 62.9, 18.3. **HR-MS** (ESI) *m/z* calcd for C₁₆H₁₅NO₂ [M+H]⁺ 254.1176, found 254.1166.

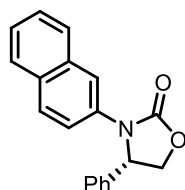


(S)-4-phenyl-3-(*m*-tolyl)oxazolidin-2-one (1l): Prepared from (S)-4-phenyloxazolidin-2-one and 1-iodo-3-methylbenzene following the reported literature procedure^[1c] and obtained as a gray solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.40–7.24 (m, 6H), 7.14–7.04 (m, 2H), 6.88 (d, *J* = 7.2 Hz, 1H), 5.38 (dd, *J* = 8.6, 6.0 Hz, 1H), 4.77 (t, *J* = 8.7 Hz, 1H), 4.19 (dd, *J* = 8.6, 6.0 Hz, 1H), 2.27 (s, 3H). **¹³C-NMR** (100 MHz, CDCl₃) δ 156.1, 139.0, 138.4, 137.0, 129.4, 128.9, 128.7, 126.4, 125.7, 121.9, 118.0, 69.9, 60.8, 21.7. **HR-MS** (ESI) *m/z* calcd for C₁₆H₁₅NO₂ [M+H]⁺ 254.1176, found 254.1166.

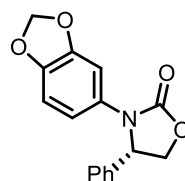


(S)-3-(3,4-dimethylphenyl)-4-phenyloxazolidin-2-one (1m): Prepared from (S)-4-phenyloxazolidin-2-one and 4-iodo-1,2-dimethylbenzene following the reported

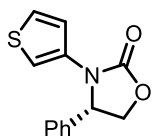
literature procedure^[1c] and obtained as a yellow solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.38–7.28 (m, 5H), 7.24 (s, 1H), 6.98 (s, 2H), 5.35 (dd, *J* = 8.6, 6.2 Hz, 1H), 4.76 (t, *J* = 8.7 Hz, 1H), 4.19 (dd, *J* = 8.6, 6.2 Hz, 1H), 2.17 (s, 3H), 2.15 (s, 3H). **¹³C-NMR** (100 MHz, CDCl₃) δ 156.3, 138.6, 137.4, 134.7, 133.5, 130.0, 129.4, 128.8, 126.5, 122.8, 118.7, 69.9, 61.0, 20.1, 19.2. **HR-MS** (ESI) *m/z* calcd for C₁₇H₁₇NO₂ [M+H]⁺ 268.1332, found 268.1322.



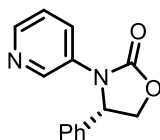
(S)-3-(naphthalen-2-yl)-4-phenyloxazolidin-2-one (1n): Prepared from (S)-4-phenyloxazolidin-2-one and 2-iodonaphthalene following the reported literature procedure^[1c] and obtained as a orange solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.79–7.71 (m, 3H), 7.70–7.63 (m, 2H), 7.45–7.36 (m, 2H), 7.36–7.27 (m, 5H), 5.52 (dd, *J* = 8.6, 6.1 Hz, 1H), 4.82 (t, *J* = 8.7 Hz, 1H), 4.25 (dd, *J* = 8.6, 6.1 Hz, 1H). **¹³C-NMR** (100 MHz, CDCl₃) δ 156.2, 138.2, 134.7, 133.4, 130.7, 129.5, 129.0, 128.9, 127.7, 127.6, 126.6, 126.4, 125.6, 120.3, 118.5, 70.0, 60.9. **HR-MS** (ESI) *m/z* calcd for C₁₉H₁₅NO₂ [M+H]⁺ 290.1176, found 290.1167.



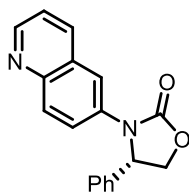
(S)-3-(benzo[*d*][1,3]dioxol-5-yl)-4-phenyloxazolidin-2-one (1o): Prepared from (S)-4-phenyloxazolidin-2-one and 5-iodobenzo[*d*][1,3]dioxole following the reported literature procedure^[1c] and obtained as a white solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.41–7.25 (m, 5H), 6.98 (s, 1H), 6.65 (s, 2H), 5.88 (d, *J* = 2.6 Hz, 2H), 5.32–5.24 (m, 1H), 4.76 (t, *J* = 8.7 Hz, 1H), 4.24–4.15 (m, 1H). **¹³C-NMR** (100 MHz, CDCl₃) δ 156.4, 148.0, 145.1, 138.1, 131.1, 129.4, 129.0, 126.6, 115.4, 108.1, 104.3, 101.5, 69.9, 61.6. **HR-MS** (ESI) *m/z* calcd for C₁₆H₁₃NO₄ [M+H]⁺ 284.0917, found 284.0909.



(S)-4-phenyl-3-(thiophen-3-yl)oxazolidin-2-one (1p): Prepared from (S)-4-phenyloxazolidin-2-one and 3-iodothiophene following the reported literature procedure^[1c] and obtained as a brown solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.45–7.32 (m, 3H), 7.31 (d, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 5.3 Hz, 1H), 7.17 (t, *J* = 4.4 Hz, 1H), 6.93 (s, 1H), 5.26 (dd, *J* = 9.0, 5.6 Hz, 1H), 4.78 (t, *J* = 8.8 Hz, 1H), 4.20 (dd, *J* = 8.7, 5.6 Hz, 1H). **¹³C-NMR** (100 MHz, CDCl₃) δ 155.60, 138.63, 135.66, 129.62, 129.09, 126.17, 125.08, 120.86, 110.01, 70.24, 61.37. **HR-MS** (ESI) *m/z* calcd for C₁₃H₁₁NO₄S [M+H]⁺ 246.0583, found 246.0576.

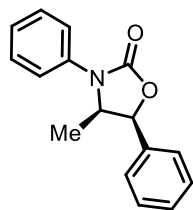


(S)-4-phenyl-3-(pyridin-3-yl)oxazolidin-2-one (1q): Prepared from (S)-4-phenyloxazolidin-2-one and 3-iodopyridine following the reported literature procedure^[1c] and obtained as a yellow solid. **¹H-NMR** (400 MHz, CDCl₃) δ 8.50 (d, *J* = 2.7 Hz, 1H), 8.30 (dd, *J* = 4.7, 1.5 Hz, 1H), 7.98 (m, *J* = 8.5, 2.8, 1.5 Hz, 1H), 7.42 – 7.27 (m, 5H), 7.23 (dd, *J* = 8.4, 4.7 Hz, 1H), 5.43 (dd, *J* = 8.8, 6.0 Hz, 1H), 4.83 (t, *J* = 8.7 Hz, 1H), 4.26 (dd, *J* = 8.7, 6.0 Hz, 1H). **¹³C-NMR** (100 MHz, CDCl₃) δ 155.8, 145.6, 141.7, 137.4, 133.9, 129.7, 129.3, 127.9, 126.3, 123.6, 70.2, 60.2. **HR-MS** (ESI) *m/z* calcd for C₁₄H₁₂N₂O₂ [M+H]⁺ 241.0972, found 241.0963.

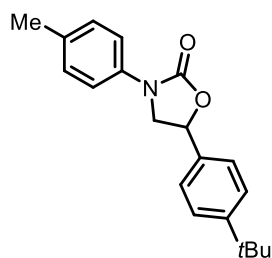


(S)-3-(naphthalen-2-yl)-4-phenyloxazolidin-2-one (1r): Prepared from (S)-4-phenyloxazolidin-2-one and 6-iodoquinoline following the reported literature procedure^[1c] and obtained as a yellow solid. **¹H-NMR** (400 MHz, CDCl₃) δ 8.80 (d, *J*

= 4.2 Hz, 1H), 8.05–7.95 (m, 2H), 7.89–7.75 (m, 2H), 7.40–7.29 (m, 6H), 5.54 (dd, J = 8.7, 6.0 Hz, 1H), 4.85 (t, J = 8.7 Hz, 1H), 4.26 (dd, J = 8.6, 6.0 Hz, 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 156.0, 150.1, 145.5, 138.0, 135.9, 135.2, 130.3, 129.7, 129.2, 128.4, 126.3, 123.3, 121.7, 118.0, 70.0, 60.8. **HR-MS** (ESI) m/z calcd for $\text{C}_{18}\text{H}_{14}\text{N}_2\text{O}_2$ $[\text{M}+\text{H}]^+$ 291.1128, found 291.1119.



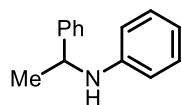
(4*R*,5*S*)-4-methyl-3,5-diphenyloxazolidin-2-one (1t): Prepared from (4*R*,5*S*)-4-methyl-5-phenyloxazolidin-2-one and iodobenzene following the reported literature procedure^[1c] and obtained as a white solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.52 (d, J = 8.1 Hz, 1H), 7.44 – 7.36 (m, 3H), 7.23 – 7.15 (m, 1H), 5.78 (d, J = 5.7 Hz, 0H), 4.71 (t, J = 6.2 Hz, 0H), 0.83 (d, J = 4.4 Hz, 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 155.20, 136.97, 134.72, 129.34, 128.72, 128.70, 126.10, 125.28, 121.84, 78.29, 57.01, 14.66. **HR-MS** (ESI) m/z calcd for $\text{C}_{16}\text{H}_{15}\text{NO}_2$ $[\text{M}+\text{H}]^+$ 254.1176, found 254.1167.



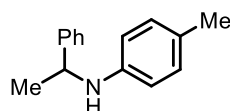
5-(4-(*tert*-butyl)phenyl)-3-(*p*-tolyl)oxazolidin-2-one (1w): Prepared from 1-isocyanato-4-methylbenzene and 2-phenyloxirane following the reported literature procedure^[1c] and obtained as a yellow solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 8.80 (d, J = 4.2 Hz, 1H), 8.05–7.95 (m, 2H), 7.89–7.75 (m, 2H), 7.40–7.29 (m, 6H), 5.54 (dd, J = 8.7, 6.0 Hz, 1H), 4.85 (t, J = 8.7 Hz, 1H), 4.26 (dd, J = 8.6, 6.0 Hz, 1H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3) δ 156.0, 150.1, 145.5, 138.0, 135.9, 135.2, 130.3, 129.7, 129.2, 128.4,

126.3, 123.3, 121.7, 118.0, 70.0, 60.8. **HR-MS** (ESI) m/z calcd for $C_{20}H_{23}NO_2$ $[M+H]^+$
310.1802, found 310.1792.

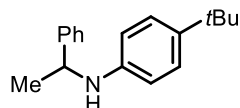
Characterization Data of Products



***N*-(1-phenylethyl)aniline (2a):** The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2a** (75.1 mg, 76%) as a yellow oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.35 (d, *J* = 7.3 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.4 Hz, 1H), 7.07 (t, *J* = 7.7 Hz, 2H), 6.63 (t, *J* = 7.3 Hz, 1H), 6.49 (d, *J* = 8.4 Hz, 2H), 4.47 (q, *J* = 6.7 Hz, 1H), 4.00 (s, 1H), 1.49 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[1]

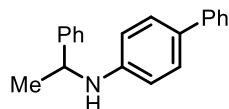


4-methyl-*N*-(1-phenylethyl)aniline (2b): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2b** (68.8 mg, 65%) as a yellow solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.35 (d, *J* = 7.3 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 2H), 6.42 (d, *J* = 8.1 Hz, 2H), 4.44 (q, *J* = 6.7 Hz, 1H), 3.89 (s, 1H), 2.17 (s, 3H), 1.49 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[2]

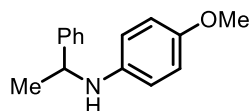


4-(*tert*-butyl)-*N*-(1-phenylethyl)aniline (2c): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2c** (106.4 mg, 84%) as a yellow solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.38 (d, *J* = 6.8 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.24–7.20 (m, 1H), 7.12 (d, *J* = 8.7 Hz, 2H), 6.46 (d, *J* = 8.6 Hz, 2H), 4.44 (q, *J* = 6.7 Hz, 1H), 3.93 (s, 1H), 1.59 (d, *J* = 6.7 Hz, 3H), 1.23 (s, 9H). The product is known and

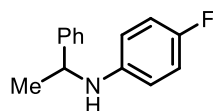
the characterization is in consistence with the reported literature.^[3]



***N*-(1-phenylethyl)-[1,1'-biphenyl]-4-amine (2d)**: The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2d** (66.7 mg, 78%) as a yellow solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.48 (d, *J* = 7.2 Hz, 2H), 7.39 (d, *J* = 7.2 Hz, 2H), 7.35 (d, *J* = 4.1 Hz, 3H), 7.33 (t, *J* = 5.4 Hz, 3H), 7.24 (t, *J* = 6.6 Hz, 2H), 6.57 (d, *J* = 8.6 Hz, 2H), 4.52 (q, *J* = 6.7 Hz, 1H), 4.13 (s, 1H), 1.53 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[4]

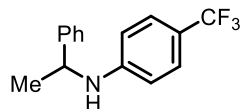


4-methoxy-*N*-(1-phenylethyl)aniline (2e): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2e** (60.6 mg, 53%) as a brown solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.36 (d, *J* = 6.6 Hz, 2H), 7.30 (t, *J* = 7.7 Hz, 2H), 7.21 (t, *J* = 7.9 Hz, 1H), 6.68 (d, *J* = 8.9 Hz, 2H), 6.46 (d, *J* = 8.9 Hz, 2H), 4.40 (q, *J* = 6.7 Hz, 1H), 3.78 (s, 1H), 3.68 (s, 3H), 1.49 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[1]

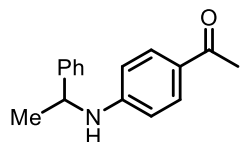


4-fluoro-*N*-(1-phenylethyl)aniline (2f): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2f** (75.8 mg, 71%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.39–7.28 (m, 4H), 7.28–7.19 (m, 1H), 6.80 (t, *J* = 8.8 Hz, 2H), 6.43 (dd, *J* = 9.0, 4.4 Hz, 2H), 4.42 (q, *J* = 6.7 Hz, 1H), 3.93 (s, 1H), 1.51 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the

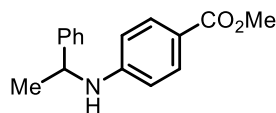
reported literature.^[2]



N-(1-phenylethyl)-4-(trifluoromethyl)aniline (2g): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2g** (73.5 mg, 56%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.36–7.27 (m, 6H), 7.24 (d, *J* = 5.5 Hz, 1H), 6.50 (d, *J* = 8.3 Hz, 2H), 4.51 (q, *J* = 6.8 Hz, 1H), 4.37 (s, 1H), 1.54 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[5]

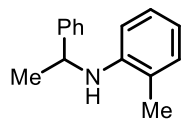


1-(4-((1-phenylethyl)amino)phenyl)ethan-1-one (2h): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2h** (43.5 mg, 36%) as a yellow solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.74 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 4.3 Hz, 4H), 7.25 (q, *J* = 4.1 Hz, 1H), 6.48 (d, *J* = 8.8 Hz, 2H), 4.62 (s, 1H), 4.61–4.53 (m, 1H), 2.44 (s, 3H), 1.55 (d, *J* = 6.6 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[6]

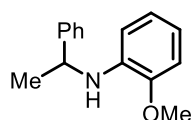


methyl 4-((1-phenylethyl)amino)benzoate (2i): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2i** (63.8 mg, 50%) as a yellow solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.82 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 4.4 Hz, 4H), 7.28 (q, *J* = 4.1 Hz, 1H), 6.51 (d, *J* = 8.6 Hz, 2H), 4.63–4.58 (m, 1H), 4.58 (s, 1H), 3.85 (s, 3H), 1.58 (d, *J* = 6.2 Hz, 3H). The product is known and the characterization is in

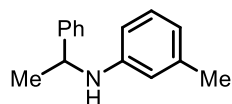
consistence with the reported literature.^[7]



2-methyl-N-(1-phenylethyl)aniline (2j): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2j** (53.0 mg, 50%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.42 (d, *J* = 6.9 Hz, 2H), 7.37 (t, *J* = 7.5 Hz, 2H), 7.29 (d, *J* = 6.7 Hz, 1H), 7.11 (d, *J* = 7.3 Hz, 1H), 7.01 (t, *J* = 7.7 Hz, 1H), 6.66 (t, *J* = 7.3 Hz, 1H), 6.42 (d, *J* = 8.1 Hz, 1H), 4.59 (q, *J* = 6.7 Hz, 1H), 3.91 (s, 1H), 2.28 (s, 3H), 1.61 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[3]

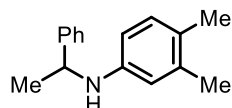


2-methoxy-N-(1-phenylethyl)aniline (2k): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2k** (41.7 mg, 37%) as a white solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.36 (d, *J* = 7.0 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.22 (d, *J* = 8.4 Hz, 1H), 6.76 (d, *J* = 7.8 Hz, 1H), 6.69 (t, *J* = 7.6 Hz, 1H), 6.60 (t, *J* = 7.7 Hz, 1H), 6.34 (d, *J* = 7.8 Hz, 1H), 4.63 (s, 1H), 4.47 (q, *J* = 6.7 Hz, 1H), 3.88 (s, 3H), 1.64 (d, *J* = 6.8 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[1]

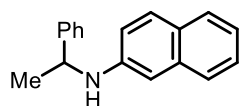


3-methyl-N-(1-phenylethyl)aniline (2l): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2l** (73.1 mg, 69%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.35 (d, *J* = 6.8 Hz, 2H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.20 (t, *J* = 7.2

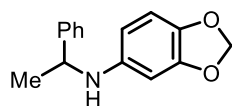
Hz, 1H), 6.96 (t, $J = 7.8$ Hz, 1H), 6.46 (d, $J = 7.4$ Hz, 1H), 6.35 (s, 1H), 6.29 (d, $J = 8.0$ Hz, 1H), 4.46 (q, $J = 6.7$ Hz, 1H), 3.94 (s, 1H), 2.20 (s, 3H), 1.48 (d, $J = 6.7$ Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[3]



3,4-dimethyl-*N*-(1-phenylethyl)aniline (2m): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2m** (81.6 mg, 73%) as a brown oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.36 (d, $J = 6.8$ Hz, 2H), 7.30 (t, $J = 7.6$ Hz, 2H), 7.26–7.19 (m, 1H), 6.83 (d, $J = 8.1$ Hz, 1H), 6.37 (d, $J = 2.5$ Hz, 1H), 6.25 (dd, $J = 8.1, 2.6$ Hz, 1H), 4.45 (q, $J = 6.7$ Hz, 1H), 3.85 (s, 1H), 2.12 (s, 3H), 2.09 (s, 3H), 1.49 (d, $J = 6.7$ Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[8]

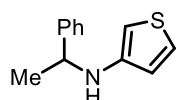


***N*-(1-phenylethyl)naphthalen-2-amine (2n):** The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2n** (93.8 mg, 76%) as a brown oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.60 (dd, $J = 13.9, 8.5$ Hz, 2H), 7.46 (d, $J = 8.3$ Hz, 1H), 7.40 (d, $J = 7.0$ Hz, 2H), 7.36–7.20 (m, 4H), 7.18–7.09 (m, 1H), 6.87 (dd, $J = 8.8, 2.4$ Hz, 1H), 6.62 (s, 1H), 4.61 (q, $J = 6.7$ Hz, 1H), 4.21 (s, 1H), 1.56 (d, $J = 6.7$ Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[9]

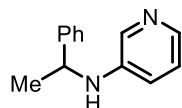


***N*-(1-phenylethyl)benzo[*d*][1,3]dioxol-5-amine (2o):** The general procedure A for the

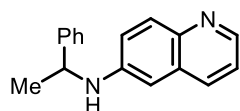
electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2o** (92.9 mg, 77%) as a black oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.35–7.29 (m, 4H), 7.25–7.22 (m, 1H), 6.56 (d, *J* = 8.3 Hz, 1H), 6.14 (d, *J* = 2.3 Hz, 1H), 5.93 (dd, *J* = 8.3, 2.3 Hz, 1H), 5.79 (q, *J* = 1.4 Hz, 2H), 4.38 (q, *J* = 6.7 Hz, 1H), 3.85 (s, 1H), 1.48 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[8]



N-(1-phenylethyl)thiophen-3-amine (2p): The general procedure A for the electrolysis was followed. 36by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2p** (44.6 mg, 65%) as a brown oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.36 (d, *J* = 6.7 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 1H), 7.07 (dd, *J* = 5.1, 3.0 Hz, 1H), 6.59 (dd, *J* = 5.1, 1.6 Hz, 1H), 5.68 (dd, *J* = 3.0, 1.6 Hz, 1H), 4.34 (q, *J* = 6.7 Hz, 1H), 4.02 (s, 1H), 1.48 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[10]

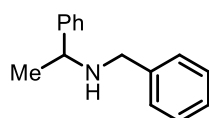


N-(1-phenylethyl)pyridin-3-amine (2q): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 6/1 to 3/1) yielded **2q** (79.7 mg, 81%) as a brown oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.99 (s, 1H), 7.89 (d, *J* = 4.8 Hz, 1H), 7.42–7.88 (m, 4H), 7.28–7.19 (m, 1H), 6.96 (dd, *J* = 8.3, 4.6 Hz, 1H), 6.70 (d, *J* = 8.3 Hz, 1H), 4.52–4.41 (m, 1H), 4.18 (s, 1H), 1.53 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[10]

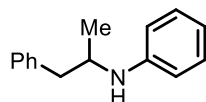


N-(1-phenylethyl)quinolin-6-amine (2r): The general procedure A for the electrolysis

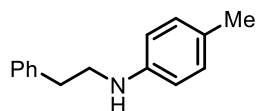
was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 2/1 to 1/1) yielded **2r** (98.0 mg, 79%) as a brown oil. ¹H-NMR (400 MHz, CDCl₃) δ 8.56, *J* = 2.8 Hz, 1H), 7.84 (d, *J* = 9.0 Hz, 1H), 7.76 (d, *J* = 6.7 Hz, 1H), 7.40 (d, *J* = 6.8 Hz, 2H), 7.33 (t, *J* = 7.6 Hz, 2H), 7.25 (d, *J* = 6.9 Hz, 1H), 7.18 (dd, *J* = 8.3, 4.2 Hz, 1H), 7.11 (dd, *J* = 9.1, 2.6 Hz, 1H), 6.49 (d, *J* = 2.6 Hz, 1H), 4.64–4.57 (m, 1H), 4.39 (s, 1H), 1.58 (d, *J* = 6.7 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[8]



***N*-benzyl-1-phenylethan-1-amine (2s)**: The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 20/1 to 10/1) yielded **2s** (57.1 mg, 54%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.37–7.30 (m, 5H), 7.29–7.22 (m, 5H), 3.80 (q, *J* = 6.6 Hz, 1H), 3.61 (q, *J* = 13.1 Hz, 1H), 1.36 (d, *J* = 6.6 Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[2]

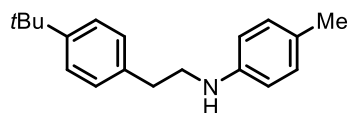


***N*-(1-phenylpropan-2-yl)aniline (2t)**: The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2t** (44.4 mg, 42%) as a yellow oil. The product is known and the characterization is in consistence with the reported literature.^[11]

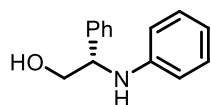


4-methyl-*N*-phenethylaniline (2v): The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2v** (33.8 mg, 32%) as a brown oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.35 – 7.28 (m, 2H), 7.23 (t, *J* = 7.4 Hz, 3H), 6.99 (d, *J* = 8.0 Hz, 2H), 6.54

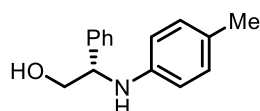
(d, $J = 8.4$ Hz, 2H), 3.38 (t, $J = 7.0$ Hz, 2H), 2.90 (t, $J = 7.0$ Hz, 2H), 2.24 (s, 3H). The product is known and the characterization is in consistence with the reported literature.^[12]



***N*-(4-(*tert*-butyl)phenethyl)-4-methylaniline (2w)**: The general procedure A for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 60/1 to 30/1) yielded **2w** (65.0 mg, 49%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.33 (d, $J = 8.2$ Hz, 2H), 7.15 (d, $J = 8.2$ Hz, 2H), 6.98 (d, $J = 8.1$ Hz, 2H), 6.54 (d, $J = 8.4$ Hz, 2H), 3.36 (t, $J = 7.0$ Hz, 2H), 2.87 (t, $J = 7.0$ Hz, 2H), 2.24 (s, 3H), 1.32 (s, 9H). The product is known and the characterization is in consistence with the reported literature.^[12]

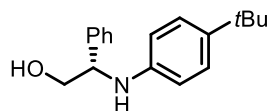


(S)-2-phenyl-2-(phenylamino)ethan-1-ol (3a): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3a** (58.8 mg, 92%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.39–7.32 (m, 4H), 7.329–7.25 (m, 1H), 7.12–7.08 (m, 2H), 6.68 (t, $J = 7.3$ Hz, 1H), 6.57 (d, $J = 7.5$ Hz, 2H), 4.51 (dd, $J = 7.0, 4.2$ Hz, 1H), 3.95 (dd, $J = 11.2, 4.1$ Hz, 1H), 3.76 (dd, $J = 11.1, 6.9$ Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[13]

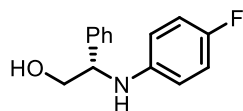


(S)-2-phenyl-2-(*p*-tolylamino)ethan-1-ol (3b): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3b** (49.4 mg, 79%) as a yellow solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.41 – 7.31 (m, 4H), 7.30 – 7.27 (m, 1H), 6.93 (d, $J =$

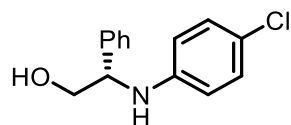
8.4 Hz, 2H), 6.52 (d, $J = 8.4$ Hz, 2H), 4.50 (dd, $J = 7.2, 4.2$ Hz, 1H), 4.38 (br, 1H), 3.99 – 3.92 (m, 1H), 3.76 (dd, $J = 11.1, 7.2$ Hz, 1H), 2.21 (s, 3H). The product is known and the characterization is in consistence with the reported literature.^[13]



(S)-2-((4-(*tert*-butyl)phenyl)amino)-2-phenylethan-1-ol (3c): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3c** (66.5 mg, 82%) as a white solid. **¹H-NMR** (400 MHz, CDCl₃) δ 7.39–7.31 (m, 5H), 7.29–7.26 (m, 4H), 5.36 (dd, $J = 8.7, 5.9$ Hz, 1H), 4.77 (t, $J = 8.7$ Hz, 1H), 4.19 (dd, $J = 8.6, 5.9$ Hz, 1H), 1.24 (s, 9H). **¹³C-NMR** (100 MHz, CDCl₃) δ 145.02, 140.80, 140.52, 128.95, 127.72, 126.88, 126.09, 113.68, 67.54, 60.35, 33.96, 31.62. **HR-MS** (ESI) m/z calcd for C₁₈H₂₃NO [M+H]⁺ 270.1852, found 270.1843.

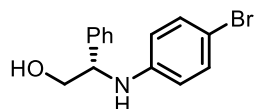


(S)-2-((4-fluorophenyl)amino)-2-phenylethan-1-ol (3d): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3d** (56.7 mg, 82%) as a yellow oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.37 – 7.32 (m, 4H), 7.31 – 7.23 (m, 1H), 6.80 (dt, $J = 9.1, 4.5$ Hz, 2H), 6.49 (dt, $J = 9.1, 4.5$ Hz, 2H), 4.42 (dd, $J = 7.1, 3.5$ Hz, 1H), 3.97 – 3.89 (m, 1H), 3.73 (t, $J = 8.0$ Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[14]

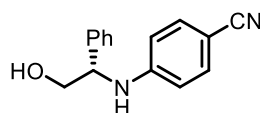


(S)-2-((4-chlorophenyl)amino)-2-phenylethan-1-ol (3e): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel

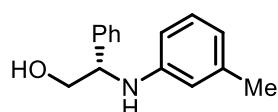
(petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3e** (56.5 mg, 82%) as a yellow solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.40 – 7.32 (m, 4H), 7.31 – 7.27 (m, 1H), 7.03 (d, $J = 8.8$ Hz, 2H), 6.47 (d, $J = 8.8$ Hz, 2H), 4.46 (dd, $J = 6.8, 4.1$ Hz, 1H), 3.96 (dd, $J = 11.1, 4.1$ Hz, 1H), 3.76 (dd, $J = 11.1, 6.8$ Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[15]



(S)-2-((4-bromophenyl)amino)-2-phenylethan-1-ol (3f): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3f** (59.3 mg, 68%) as a white solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.36–7.32 (m, 4H), 7.29–7.27 (m, 1H), 7.16 (d, $J = 8.7$ Hz, 2H), 6.42 (d, $J = 8.7$ Hz, 2H), 4.59 (br, 1H), 4.45 (dd, $J = 6.8, 4.1$ Hz, 1H), 3.95 (dd, $J = 11.1, 4.0$ Hz, 1H), 3.76 (dd, $J = 11.2, 6.8$ Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[16]

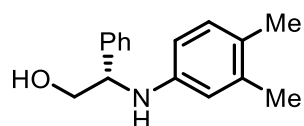


(S)-4-((2-hydroxy-1-phenylethyl)amino)benzotrile (3g): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 3/1) yielded **3g** (50.5 mg, 71%) as a yellow oil. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.41 – 7.27 (m, 7H), 6.51 (d, $J = 8.8$ Hz, 2H), 4.53 (dd, $J = 6.2, 4.0$ Hz, 1H), 4.00 (dd, $J = 11.3, 4.0$ Hz, 1H), 3.82 (dd, $J = 11.2, 6.1$ Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[15]

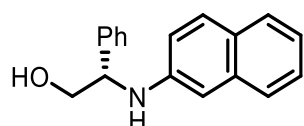


(S)-2-phenyl-2-(m-tolylamino)ethan-1-ol(3h): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel

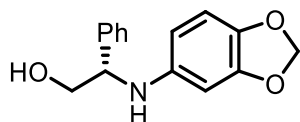
(petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3h** (44.5 mg, 65%) as a yellow oil. ¹H-NMR (400 MHz, CDCl₃) δ 7.41 – 7.32 (m, 4H), 7.31–7.25 (m, 1H), 6.99 (t, *J* = 7.8 Hz, 1H), 6.52 (d, *J* = 7.4 Hz, 1H), 6.44 (s, 1H), 6.37 (d, *J* = 8.0 Hz, 1H), 4.51 (dd, *J* = 7.1, 4.0 Hz, 1H), 3.94 (dd, *J* = 11.3, 4.1 Hz, 1H), 3.75 (dd, *J* = 11.1, 6.8 Hz, 1H), 2.22 (s, 3H). The product is known and the characterization is in consistence with the reported literature.^[17]



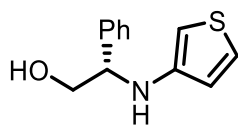
(S)-2-((3,4-dimethylphenyl)amino)-2-phenylethan-1-ol (3i): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3i** (53.7 mg, 74%) as a brown solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.37–7.31 (m, 4H), 7.27 (d, *J* = 7.6 Hz, 1H), 6.85 (d, *J* = 8.1 Hz, 1H), 6.45 (d, *J* = 2.6 Hz, 1H), 6.31 (dd, *J* = 8.2, 2.5 Hz, 1H), 4.48 (dd, *J* = 7.3, 4.3 Hz, 1H), 3.91 (dd, *J* = 11.1, 4.2 Hz, 1H), 3.71 (dd, *J* = 11.1, 7.3 Hz, 1H), 2.13 (s, 3H), 2.10 (s, 3H). The product is known and the characterization is in consistence with the reported literature.^[18]



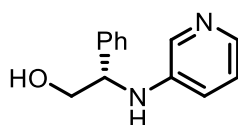
(S)-2-(naphthalen-2-ylamino)-2-phenylethan-1-ol (3j): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3j** (39.4mg, 50%) as a brown solid. ¹H-NMR (400 MHz, CDCl₃) δ 7.63 (t, *J* = 8.7 Hz, 2H), 7.47 (d, *J* = 8.3 Hz, 1H), 7.42 (d, *J* = 7.6 Hz, 2H), 7.35 (t, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 2H), 7.21 – 7.12 (m, 1H), 6.96 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.67 (d, *J* = 2.4 Hz, 1H), 4.65 (dd, *J* = 6.7, 4.2 Hz, 1H), 4.02 (dd, *J* = 11.1, 4.2 Hz, 1H), 3.84 (dd, *J* = 11.1, 6.7 Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[19]



(S)-2-(benzo[d][1,3]dioxol-5-ylamino)-2-phenylethan-1-ol (3k): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 5/1) yielded **3k** (40.3 mg, 52%) as a black oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.34 (d, *J* = 4.4 Hz, 4H), 7.29– 7.25 (m, 1H), 6.56 (d, *J* = 8.3 Hz, 1H), 6.19 (d, *J* = 2.3 Hz, 1H), 5.99 (dd, *J* = 8.3, 2.3 Hz, 1H), 5.80 (d, *J* = 2.1 Hz, 2H), 4.40 (dd, *J* = 7.3, 4.1 Hz, 1H), 3.89 (dd, *J* = 11.1, 4.1 Hz, 1H), 3.68 (dd, *J* = 11.1, 7.3 Hz, 1H). **¹³C-NMR** (100 MHz, CDCl₃) δ 148.3, 143.0, 140.2, 140.0, 129.0, 127.8, 126.8, 108.6, 105.9, 100.7, 97.1, 67.4, 60.9. **HR-MS** (ESI) *m/z* calcd for C₁₅H₁₅NO₃ [M+H]⁺ 258.1125, found 258.1125.

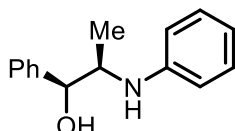


(S)-2-phenyl-2-(thiophen-3-ylamino)ethan-1-ol (3l): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3l** (33.5 mg, 51%) as a black oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.40 – 7.32 (m, 4H), 7.29 (d, *J* = 6.8 Hz, 1H), 7.10 (dd, *J* = 5.2, 3.0 Hz, 1H), 6.66 (dd, *J* = 5.1, 1.6 Hz, 1H), 5.75 (dd, *J* = 3.2, 1.5 Hz, 1H), 4.39 (dd, *J* = 7.8, 4.2 Hz, 1H), 3.91 (dd, *J* = 11.1, 4.2 Hz, 1H), 3.71 (dd, *J* = 11.1, 7.8 Hz, 1H). **¹³C-NMR** (100 MHz, CDCl₃) δ 147.36, 140.13, 128.90, 127.79, 126.94, 124.99, 120.52, 98.01, 67.36, 62.45. **HR-MS** (ESI) *m/z* calcd for C₁₂H₁₂NOS [M+H]⁺ 220.0791, found 220.0791.

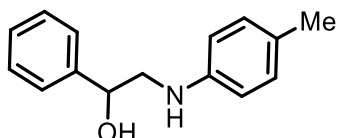


(S)-2-phenyl-2-(pyridin-3-ylamino)ethan-1-ol (3n): The general procedure B for the

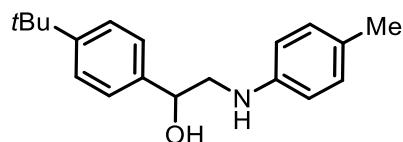
electrolysis was followed and obtained as a black oil. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.90 (dd, $J = 29.2, 3.5$ Hz, 2H), 7.34–7.29 (m, 4H), 7.28–7.25 (m, 1H), 6.97 (dd, $J = 8.3, 4.6$ Hz, 1H), 6.74 (dd, $J = 8.0, 2.4$ Hz, 1H), 4.84 (s, 1H), 4.43 (dd, $J = 7.3, 4.0$ Hz, 1H), 3.95 (dd, $J = 11.3, 4.0$ Hz, 1H), 3.74 (dd, $J = 11.3, 7.4$ Hz, 1H). The product is known and the characterization is in consistence with the reported literature.^[20]



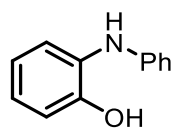
(1*S*,2*R*)-2-(benzylamino)-1-phenylpropan-1-ol (3o): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3o** (55.4 mg, 81%) as a yellow solid. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.39 (d, $J = 4.1$ Hz, 4H), 7.33–7.28 (m, 1H), 7.24–7.17 (m, 2H), 6.79–6.69 (m, 3H), 5.01 (d, $J = 3.1$ Hz, 1H), 3.80 (qd, $J = 6.6, 3.0$ Hz, 1H), 1.03 (d, $J = 6.6$ Hz, 3H). The product is known and the characterization is in consistence with the reported literature.^[21]



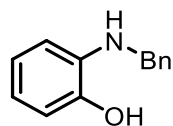
1-phenyl-2-(*p*-tolylamino)ethan-1-ol (3q): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3q** (33.6 mg, 49%) as a yellow oil. $^1\text{H-NMR}$ (400 MHz, CDCl_3) δ 7.46–7.36 (m, 4H), 7.34–7.30 (m, 1H), 7.01 (d, $J = 8.0$ Hz, 2H), 6.62 (d, $J = 8.4$ Hz, 2H), 4.92 (dd, $J = 8.7, 3.8$ Hz, 1H), 3.42 (dd, $J = 13.1, 3.8$ Hz, 1H), 3.28 (dd, $J = 13.1, 8.7$ Hz, 1H), 2.25 (s, 3H). The product is known and the characterization is in consistence with the reported literature.^[22]



1-(4-(*tert*-butyl)phenyl)-2-(*p*-tolylamino)ethan-1-ol (3r): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3r** (45.0 mg, 53%) as a yellow oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.40 (d, *J* = 8.4 Hz, 4H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.00 (d, *J* = 8.1 Hz, 2H), 6.60 (d, *J* = 8.4 Hz, 2H), 4.88 (dd, *J* = 8.7, 3.9 Hz, 1H), 3.40 (dd, *J* = 13.0, 3.9 Hz, 1H), 3.28 (dd, *J* = 13.0, 8.7 Hz, 1H), 2.25 (s, 3H), 1.33 (s, 9H). **¹³C-NMR** (100 MHz, CDCl₃) δ 151.1, 145.7, 139.2, 129.9, 127.5, 125.8, 125.6, 113.8, 72.4, 52.2, 34.7, 31.5, 20.5. **HR-MS** (ESI) *m/z* calcd for C₁₉H₂₆NO [M+H]⁺ 284.2009, found 284.2018.

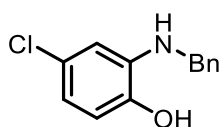


2-(phenylamino)phenol (3s): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3s** (40.7 mg, 73%) as a black oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.27 – 7.17 (m, 3H), 7.09 (t, *J* = 7.7 Hz, 1H), 6.98 (d, *J* = 8.0 Hz, 1H), 6.88 (q, *J* = 7.0 Hz, 2H), 6.78 (d, *J* = 7.9 Hz, 2H). The product is known and the characterization is in consistence with the reported literature.^[23]



2-(benzylamino)phenol (3t): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3t** (31.4 mg, 53%) as a black oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.39–7.32 (m, 4H), 7.28 (d, *J* = 7.0 Hz, 1H), 6.82 (t, *J* = 7.7 Hz, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 6.67–6.60 (m, 2H), 4.34 (s, 2H). The product is known and the characterization

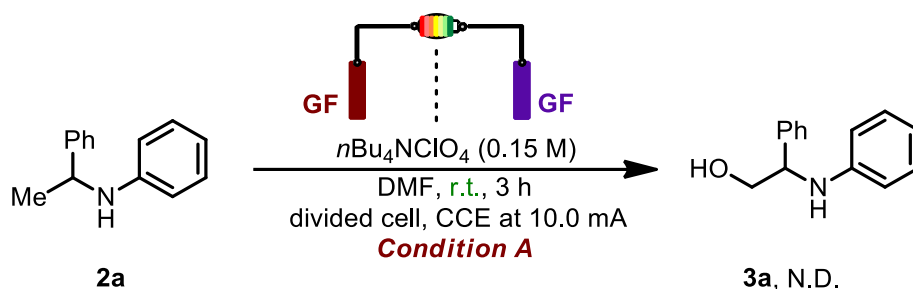
is in consistence with the reported literature.^[23]



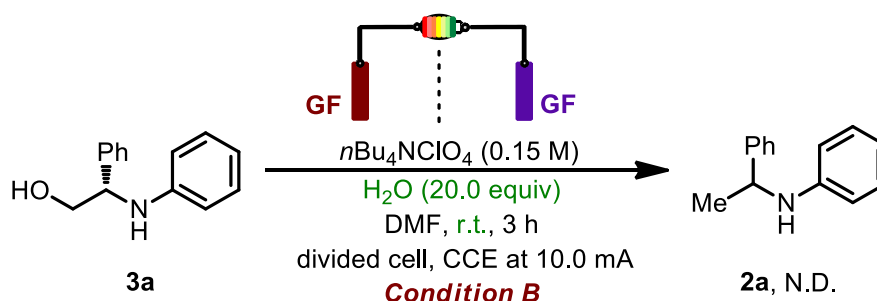
2-(benzylamino)-4-chlorophenol (3u): The general procedure B for the electrolysis was followed. Purification by column chromatography on silica gel (petroleum ether/ethyl acetate: 10/1 to 5/1) yielded **3u** (50.3 mg, 72%) as a black oil. **¹H-NMR** (400 MHz, CDCl₃) δ 7.39 – 7.24 (m, 5H), 6.60 (d, *J* = 7.7 Hz, 2H), 6.55 (d, *J* = 6.2 Hz, 1H), 4.31 (s, 2H). The product is known and the characterization is in consistence with the reported literature.^[24]

Mechanistic Studies

a) Validation experiments:

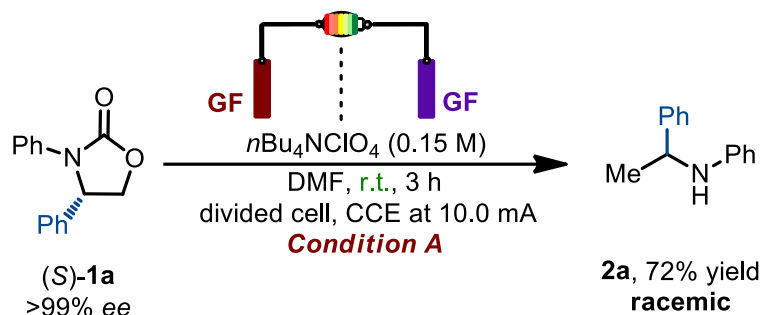


Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **2a** (0.3 mmol, 1.0 equiv), and both chambers were charged with $n\text{Bu}_4\text{NClO}_4$ (205 mg, 0.15 M), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. No any product **3a** was detected under the reaction condition.

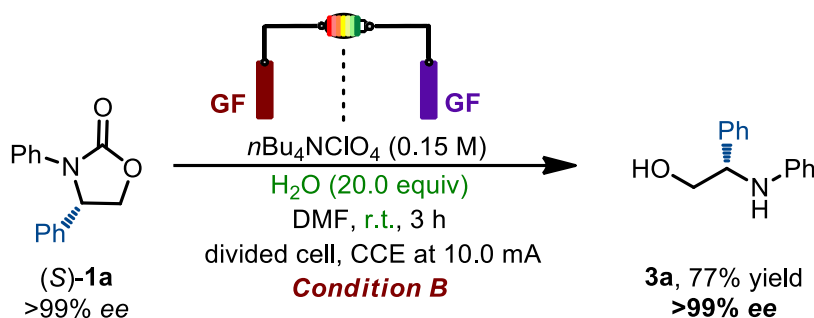


Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **3a** (0.3 mmol, 1.0 equiv), and both chambers were charged with $n\text{Bu}_4\text{NClO}_4$ (205 mg, 0.15 M), H_2O (20 equiv), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. No any product **2a** was detected under the reaction condition.

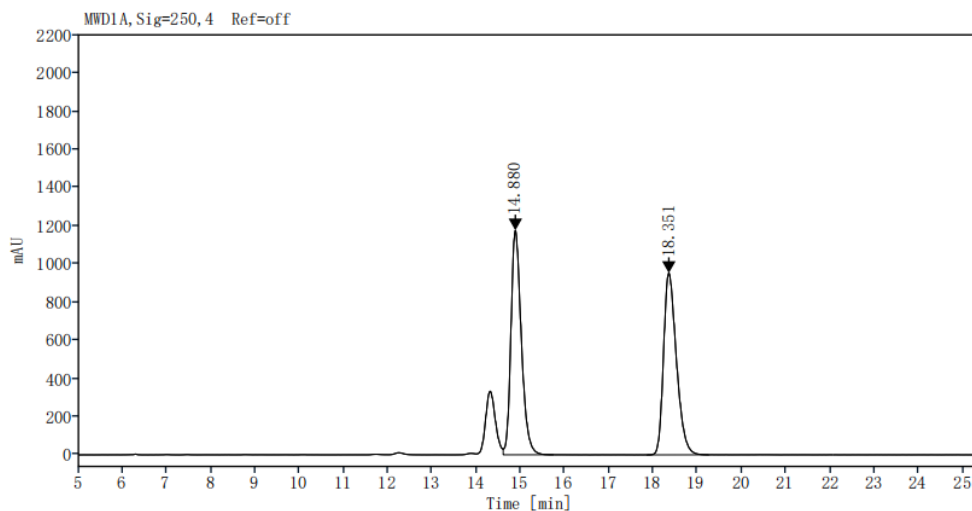
b) Reaction using chiral substrate:



Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with (*S*)-**1a** (0.3 mmol, 1.0 equiv, >99% ee), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. the obtained product **2a** is racemic under the reaction condition.



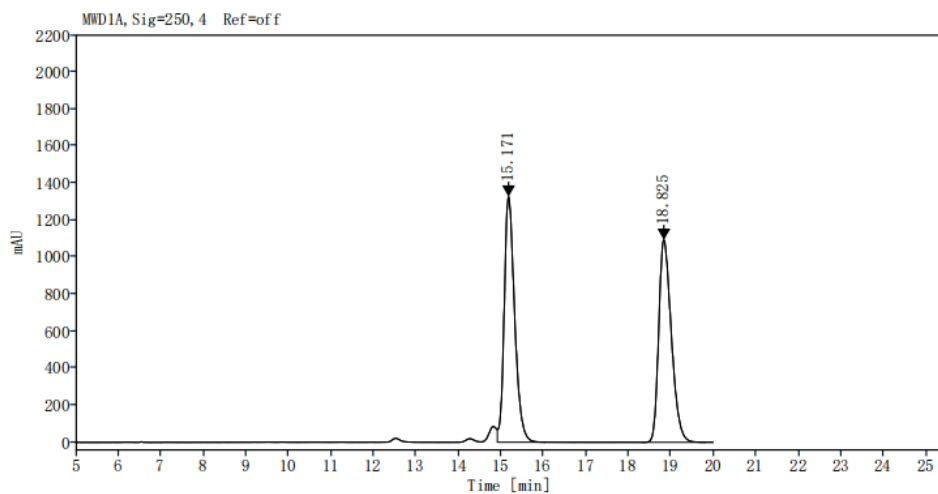
Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with (*S*)-**1a** (0.3 mmol, 1.0 equiv, >99% ee), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), H₂O (20 equiv), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. the obtained product **3a** can keep stable enantioselectivity under the reaction condition.



MWD1A, Sig=250, 4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
14.880	19188.08	1176.38	49.88	55.24
18.351	19276.74	953.05	50.12	44.76

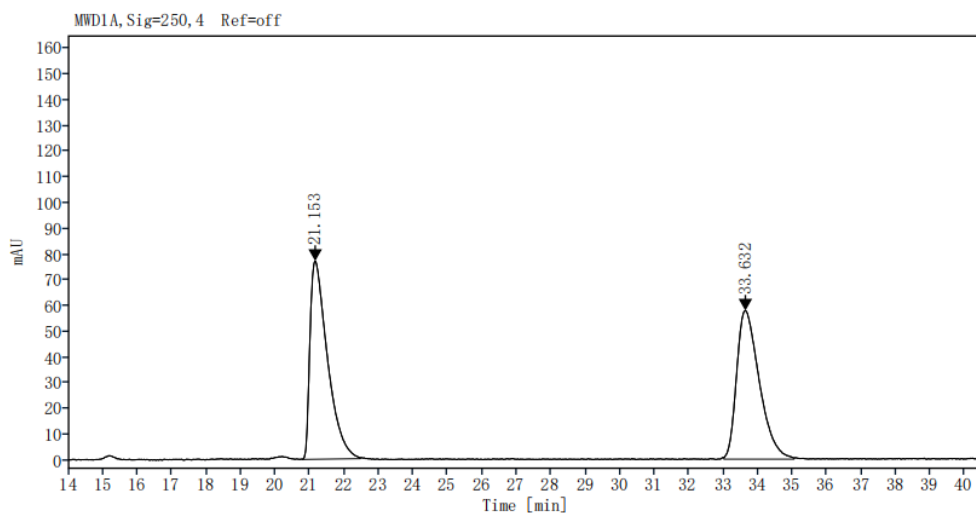
HPLC spectra of 2a-rac



MWD1A, Sig=250, 4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
15.171	22678.36	1326.23	50.10	54.82
18.825	22585.31	1093.01	49.90	45.18

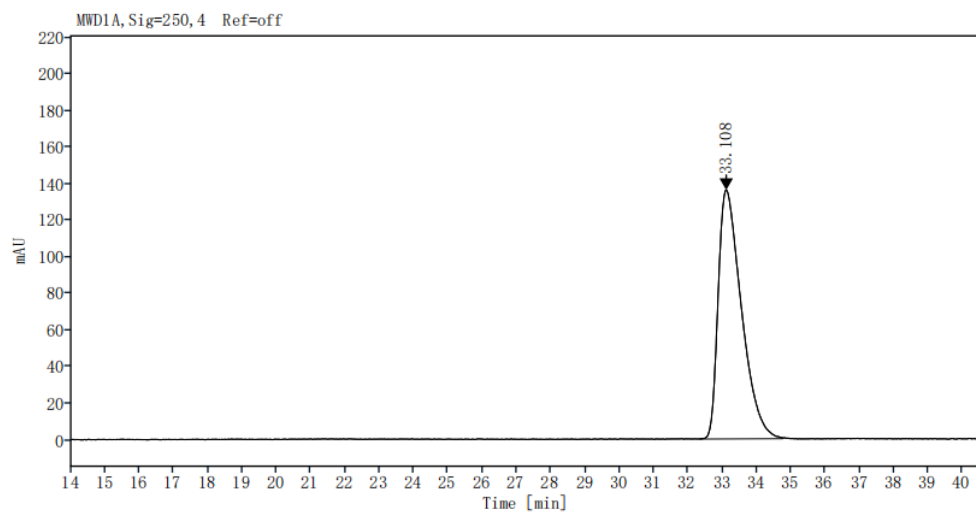
HPLC spectra of 2a- chiral



MWD1A, Sig=250, 4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
21.153	2714.66	77.05	50.69	57.17
33.632	2640.71	57.72	49.31	42.83

HPLC spectra of 3a-rac

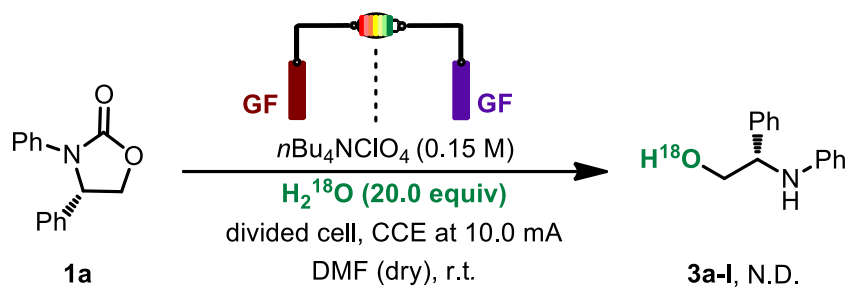


MWD1A, Sig=250, 4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
33.108	6396.75	136.00	100.00	100.00

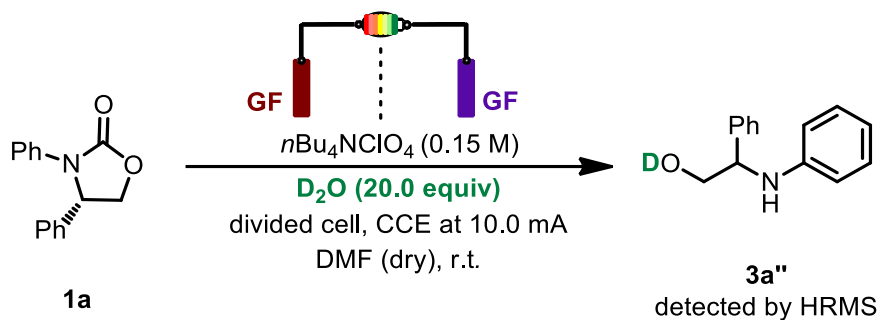
HPLC spectra of 3a-chiral

c) H₂¹⁸O isotope labeling experiment:



Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with (S)-**1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), H₂¹⁸O (20 equiv), DMF (4.0 mL each). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. No any ¹⁸O labeled product **3a-I** was detected under the reaction condition.

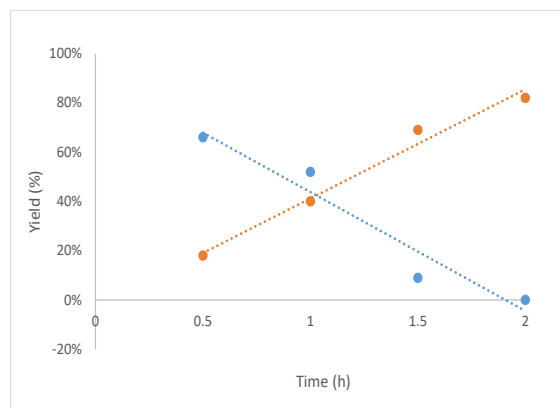
d) Deuterium substitution experiment:



Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with (*S*)-**1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), D₂O (20.0 equiv), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. The Deuterated product **3a''** was detected by HRMS under the reaction condition.

e) Kinetic Profiles Experiments:

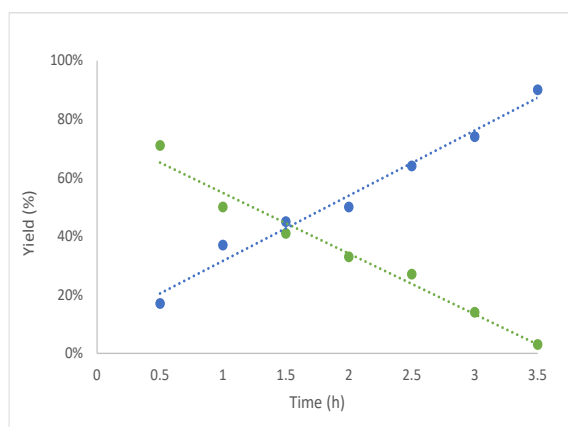
Hydrogenation procedure: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for indicated time. Upon the reduction, the reaction mixture was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3×15 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. The NMR yield of **1a** and **2a** were determined by analysis of ¹H NMR with C₂H₂Cl₄ as internal standard.



Hydrogenation

Reaction time (h)	Yield of 2a (%)	Yield of 1a (%)
0.5	18	66
1.0	40	52
1.5	69	9
2.0	82	0

Hydrolysis procedure: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), H₂O (20.0 equiv), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for indicated time. Upon the reduction, the reaction mixture was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3×15 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. The NMR yield of **1a** and **3a** were determined by analysis of ¹H NMR with C₂H₂Cl₄ as internal standard.

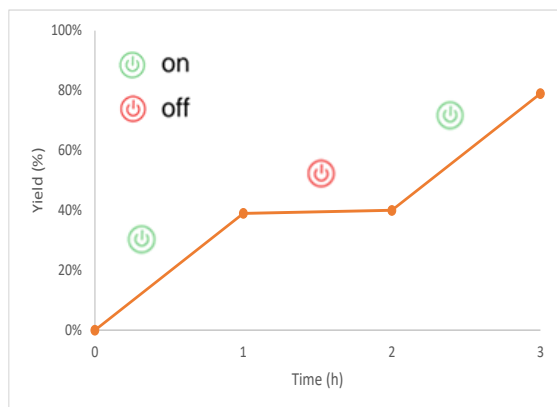


Hydrolysis

Reaction time (h)	Yield of 3a (%)	Yield of 1a (%)
0.5	17	71
1.0	37	50
1.5	45	41
2.0	50	33
2.5	64	27
3.0	74	14
3.5	90	3

f) Electricity on/off Experiments

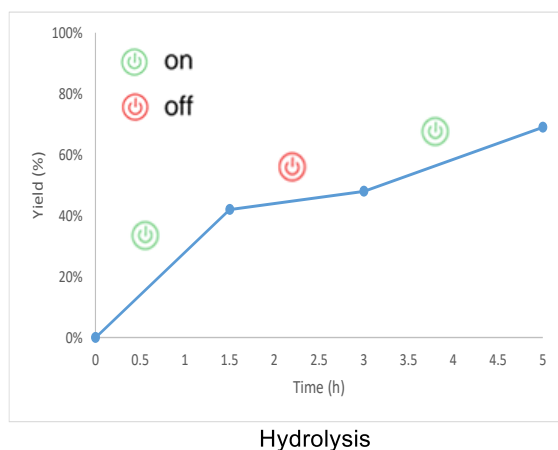
Hydrogenation procedure: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA or without electricity for indicated time. Upon the reduction, the reaction mixture was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3×10 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. The NMR yield of **2a** was determined by analysis of ¹H NMR with C₂H₂Cl₄ as internal standard.



Hydrogenation

Reaction time (h)	Yield of 2a (%)
0	0
1.0	39
1.5	40
2.0	79

Hydrolysis procedure: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), H₂O (20.0 equiv) and DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA or without electricity for indicated time. Upon the reduction, the reaction mixture was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3×10 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. The NMR yield of **3a** was determined by analysis of ¹H NMR with C₂H₂Cl₄ as internal standard.

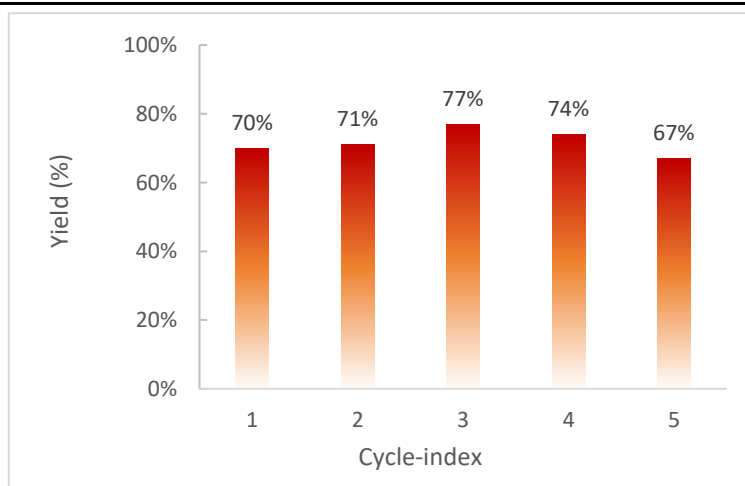


Reaction time (h)	Yield of 2a (%)
0	0
1.5	42
3.0	48
5.0	69

g) Anodic chamber recycling experiment:

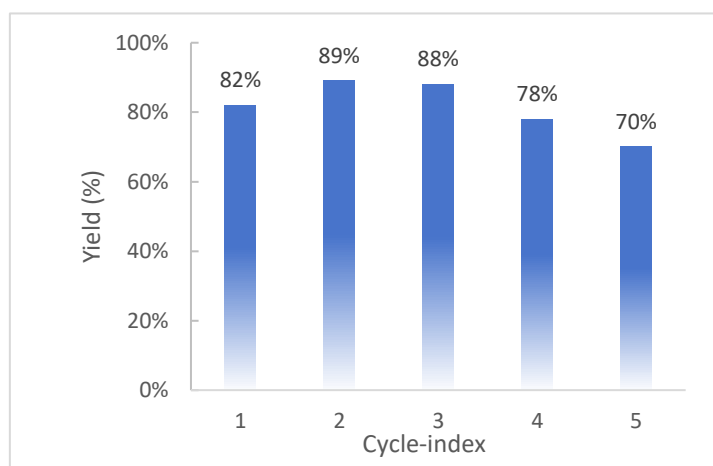
Hydrogenation procedure: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. Upon the reduction, the reaction mixture of cathodic chamber was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3×15 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. While the solution in the anodic chamber was retained without change and reused for subsequent electrolytic reactions.

Entry	Cycle-index	Yield of 2a (%)
1	1	70
2	2	71
3	3	77
4	4	74
5	5	67

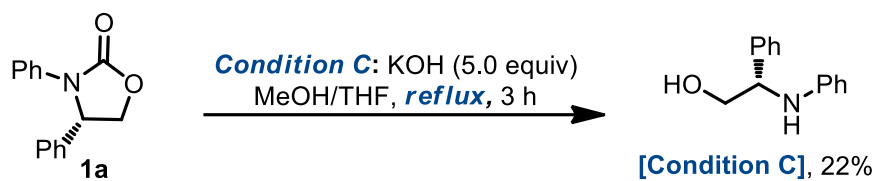


Hydrolysis procedure: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with **1a** (0.3 mmol, 1.0 equiv), and both chambers were charged with *n*Bu₄NClO₄ (205 mg, 0.15 M), H₂O (20.0 equiv), DMF (4.0 mL). Graphite felts (15 mm×10 mm×3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. Upon the reduction, the reaction mixture of cathodic chamber was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3×15 mL). The organic fractions were dried by Na₂SO₄, filtered and concentrated in vacuo. While the solution in the anodic chamber was retained without change and reused for subsequent electrolytic reactions.

Entry	Cycle-index	Yield of 3a (%)
1	1	82
2	2	89
3	3	88
4	4	78
5	5	70

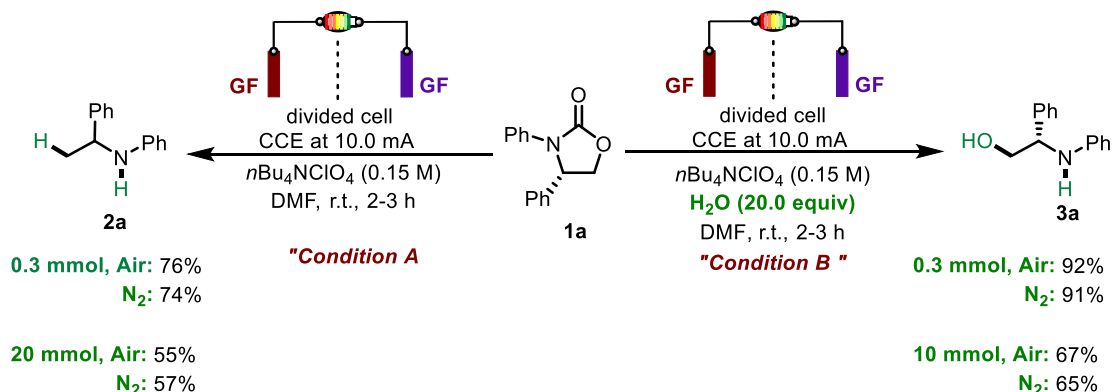


h) Contrast experiment with strong base:



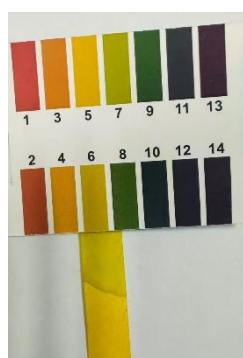
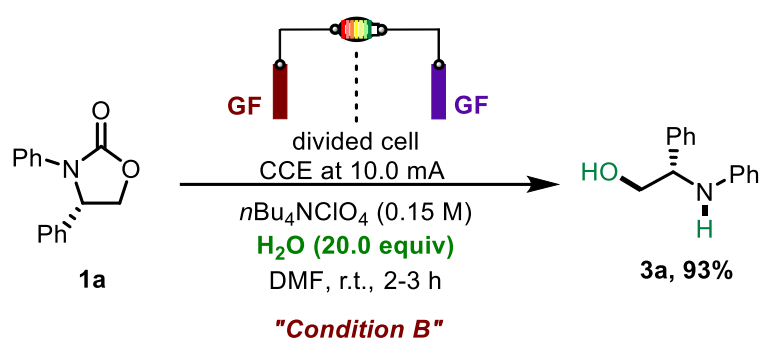
An oven-dried 5 mL Schlenk tube equipped with a magnetic stir bar was charged with **1a** (0.3 mmol), KOH (5.0 equiv), MeOH/DMF (1.0/3.0 mL), then the reaction mixture was refluxed for 3.0 h. Only 22% yield of hydrolysis product **3a** obtained under traditional condition.

i) Control experiments under N₂ and air:

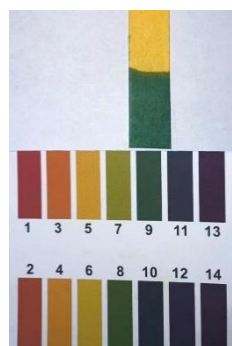


The reaction was repeated under General Procedure: Electroreduction of 2-Oxazolidinones and Scalability of Electrochemical Reduction. Control experiments were conducted under a rigorous N₂ atmosphere to evaluate the oxygen sensitivity of the transformation. For the standard hydrogenation and hydrolysis of **1a**, the isolated yields of **2a** (77%) and **3a** (91%) were nearly identical to those obtained under ambient air (76% and 92%, respectively). Additionally, gram-scale synthesis performed under N₂ afforded **2a** and **3a** in yields consistent with those observed under air. These results unambiguously demonstrate that the electroreductive hydrogenation and hydrolysis processes exhibit excellent tolerance to molecular oxygen.

j) pH Measurement of the Catholyte before and after Electrolysis under hydrolysis pathway:



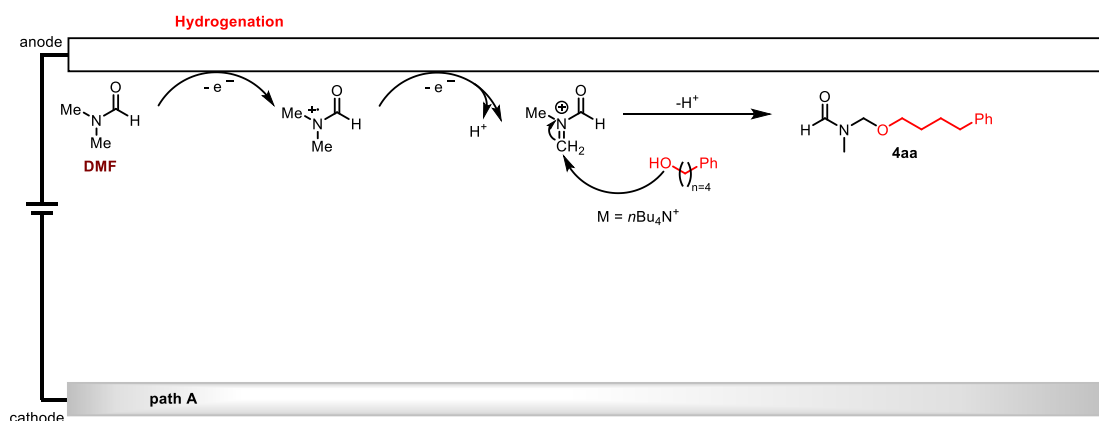
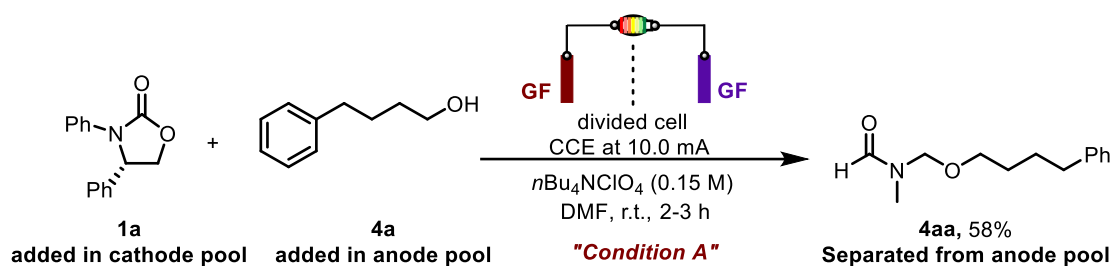
Before the reaction pH= 6



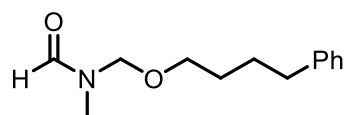
After reacted pH= 9

Hydrolysis reaction: An oven-dried H-type divided cell was equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with 2-Oxazolidinones (0.3 mmol, 1.0 equiv), and both chambers were charged with $n\text{Bu}_4\text{NClO}_4$ (205 mg each, 0.15 M), DMF (4.0 mL each) and deionized H_2O (108 μL each, 20.0 equiv). Graphite felts (15 mm \times 10 mm \times 3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3-4 hours. The pH of the catholyte was measured before and after electrolysis using pH test paper. Prior to electrolysis, the catholyte exhibited a pH = 6, whereas after the reaction, the pH increased to 9. This marked increase in alkalinity confirms the in-situ generation of hydroxide ions at the cathode. These results provide direct experimental support for the proposal that ring-opening is induced by the electrogenerated base, rather than by direct cathodic reduction of the substrate.

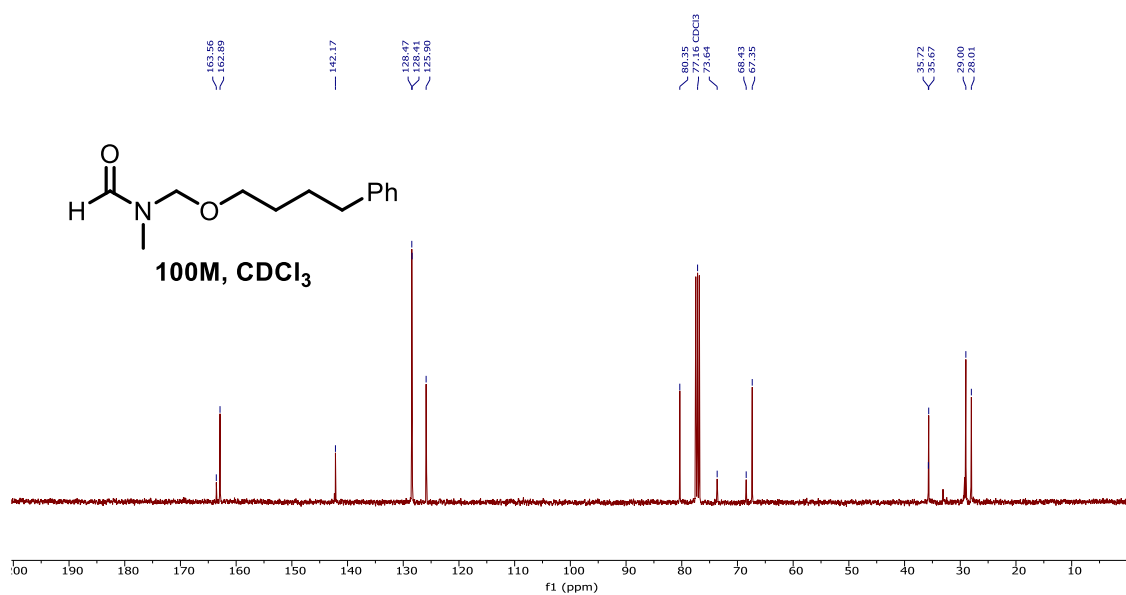
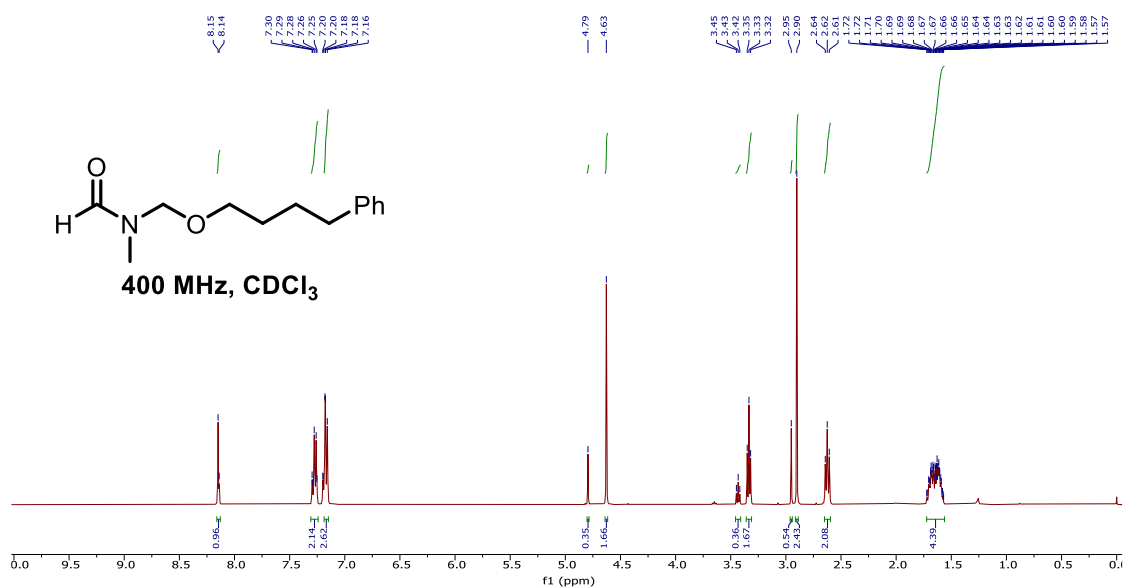
k) Iminium Ion Trapping Experiment:



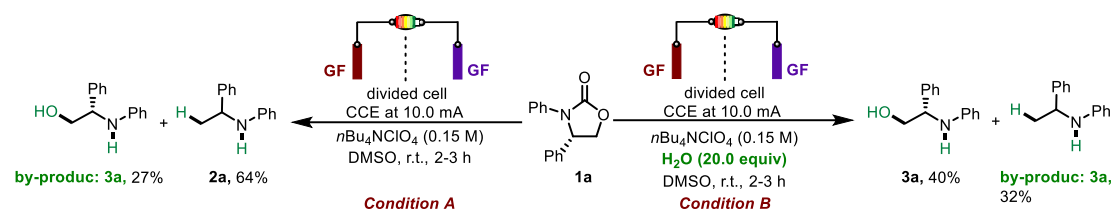
Procedure: the electrolysis was carried out in an oven-dried H-type divided cell equipped with a magnetic stir bar in each chamber. The cathodic chamber was charged with (*S*)-**1a** (0.3 mmol, 1.0 equiv), and the cathodic chamber was charged with **4a** (916 μL , 20.0 equiv). Both chambers were charged with $n\text{Bu}_4\text{NClO}_4$ (205 mg, 0.15 M), DMF (4.0 mL). Graphite felts (15 mm \times 10 mm \times 3 mm) were installed as the cathode and anode. Electrolysis was performed at room temperature with a constant current of 10.0 mA for 3 hours. Upon the reduction, the reaction mixture was transferred to 50 mL of ethyl acetate and subjected to washing with saturated brine (3 \times 15 mL). The organic fractions were dried by Na_2SO_4 , filtered and concentrated in vacuo. The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate: 40/1 to 20/1) yielded **4aa** (39 mg, 58%) as a yellow oil. The product **4aa** was detected by ^1H NMR and ^{13}C NMR under the reaction condition.



N-methyl-N-((4-phenylbutoxy)methyl)formamide (4aa): trans: cis = 83: 17. ¹H-NMR (400 MHz, CDCl₃) δ 8.14 (d, J = 3.9 Hz, 1H), 7.28 (t, J = 7.3 Hz, 2H), 7.22 – 7.14 (m, 3H), 4.79 (s, 0.35H), 4.63 (s, 1.66H), 3.43 (t, J = 6.2 Hz, 0.36H), 3.34 (t, J = 6.1 Hz, 1.67H), 2.95 (s, 1.54H), 2.90 (s, 2.43H), 2.62 (t, J = 7.3 Hz, 2H), 1.73 – 1.56 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 162.89, 142.17, 128.47, 128.41, 125.90, 80.35, 73.64, 67.89 (d, J = 109.1 Hz), 35.69 (d, J = 5.6 Hz), 29.00, 28.01.

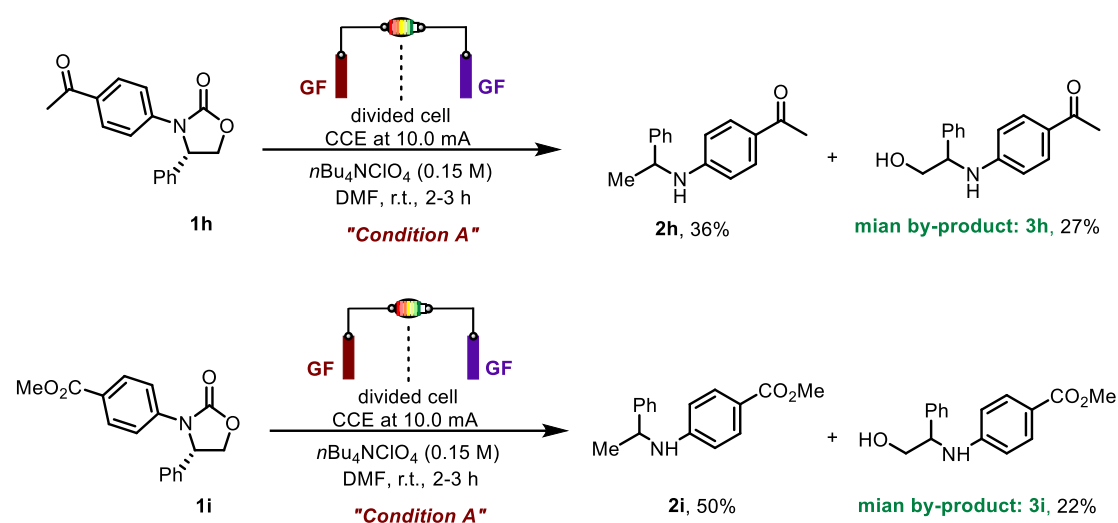


l) : By-product identification experiment



Under the fixed electrolysis time and charge input, most entries in Table 1 achieved conversion of **1a**, with conversions ranging from approximately 55% to 90%. Notably, the optimal conditions (entry 1) afforded the highest conversion efficiency. Based on previous experimental records and repeated experiments, the lower conversions observed for other entries are primarily attributed to the formation of by-products from the starting material. Under Condition A (hydrogenation), the major by-product was **3a**, whereas under Condition B (hydrolysis), the major by-product was **2a**. In addition, entry 4 was independently repeated, and the yield of the corresponding by-product was confirmed to be consistent with the reported trend.

m) : Low-yield research of 1h and 1l.



Substance **2h** and **2i** were reacted in General Procedure (Hydrogenation Condition). The crude product was purified by column chromatography on silica gel. After re-performing the reactions of **2h** and **2i**, the lower isolated yields of **2h** and **2i** are primarily due to selectivity issues rather than incomplete conversion. TLC monitoring of the crude reaction mixtures indicated that substrates **1h** and **1i** were fully consumed under the standard hydrogenation conditions. Further analysis of the reaction mixtures under Condition A revealed that the major by-products were the corresponding amino alcohols **3h** and **3i**, formed in 27% and 22% yields, respectively. These hydrolysis products likely arise from competing nucleophilic attack by trace water or cathodically generated hydroxide at the oxazolidinone carbonyl, thereby diverting the reaction from the desired hydrogenation pathway.

Cyclic Voltammetry Studies

The cyclic voltammetry was carried out with a Shanghai Chenhua CHI700E workstation and following analysis was performed with Originpro software. A glassy carbon electrode was used as the working electrode, a Pt wire was used as the auxiliary electrode and an Ag/AgCl electrode was used as a reference electrode. The measurements were carried out at a scan rate of 100 mVs^{-1} .

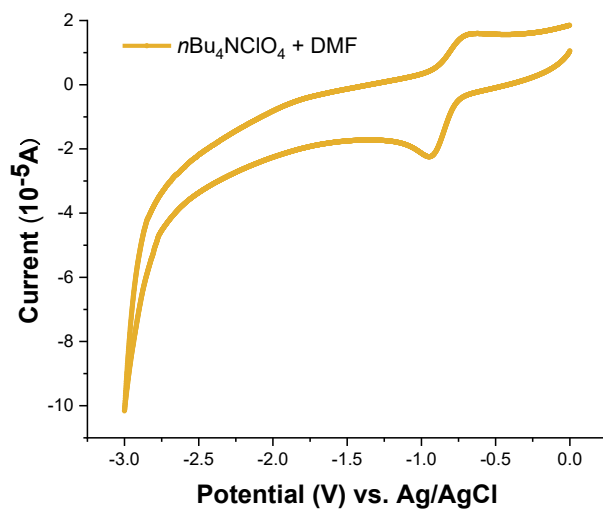


Figure S3. Cyclic voltammogram of *n*Bu₄NClO₄ (0.1 M) in DMF (5 mL) vs. Ag/AgCl.

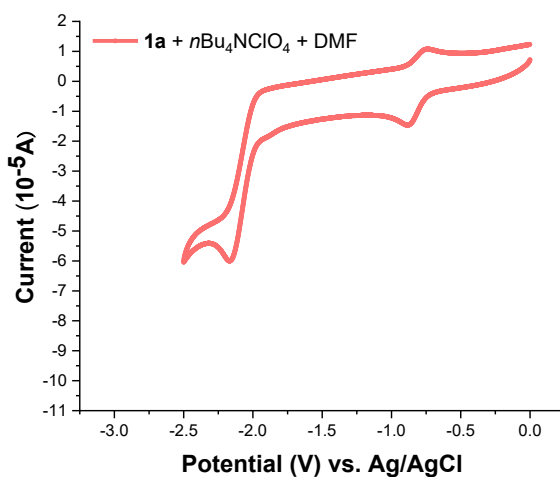
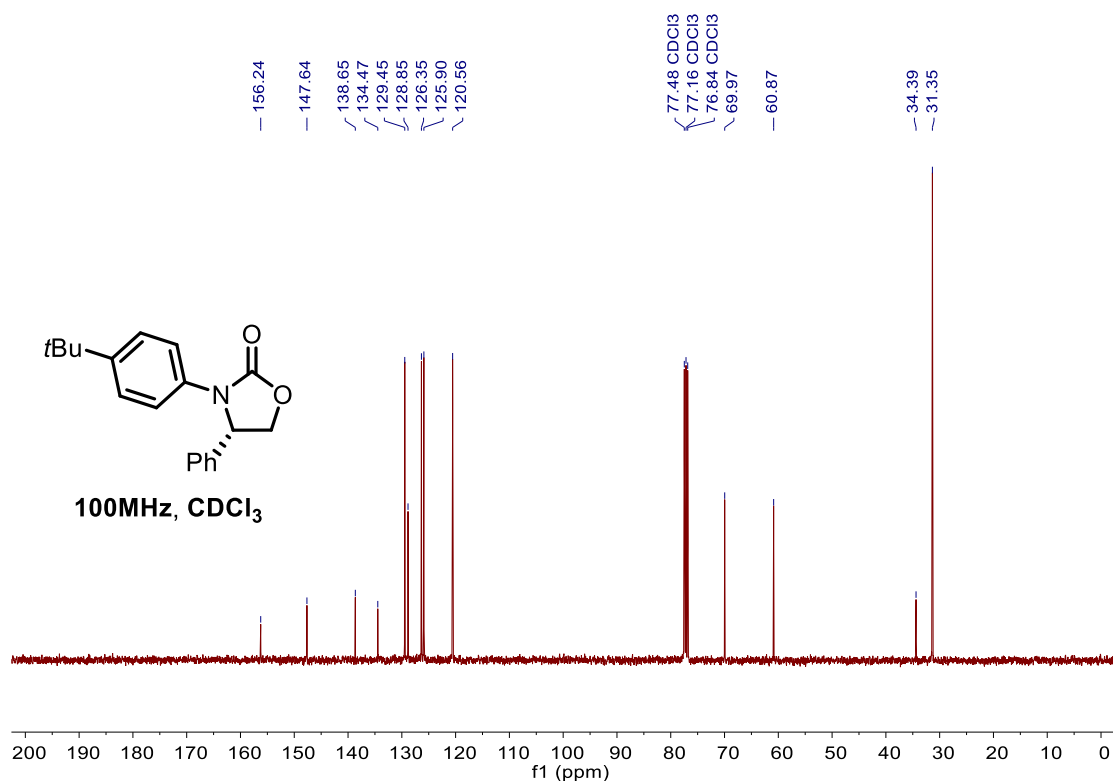
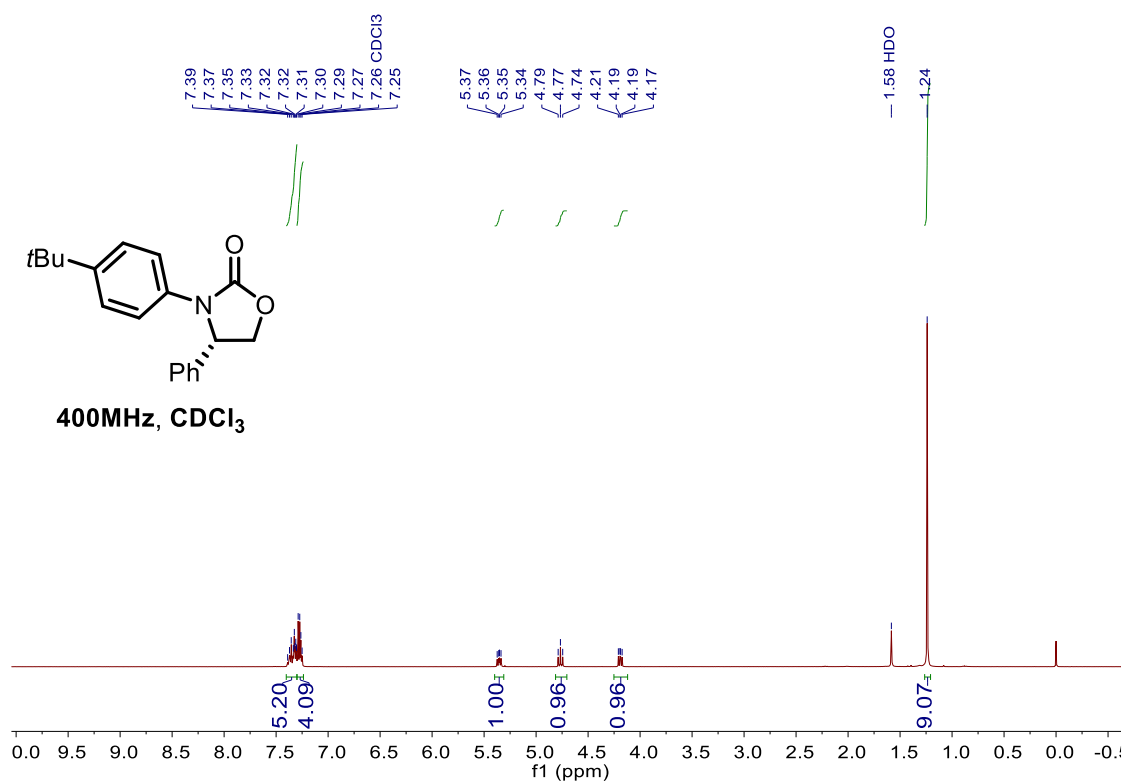
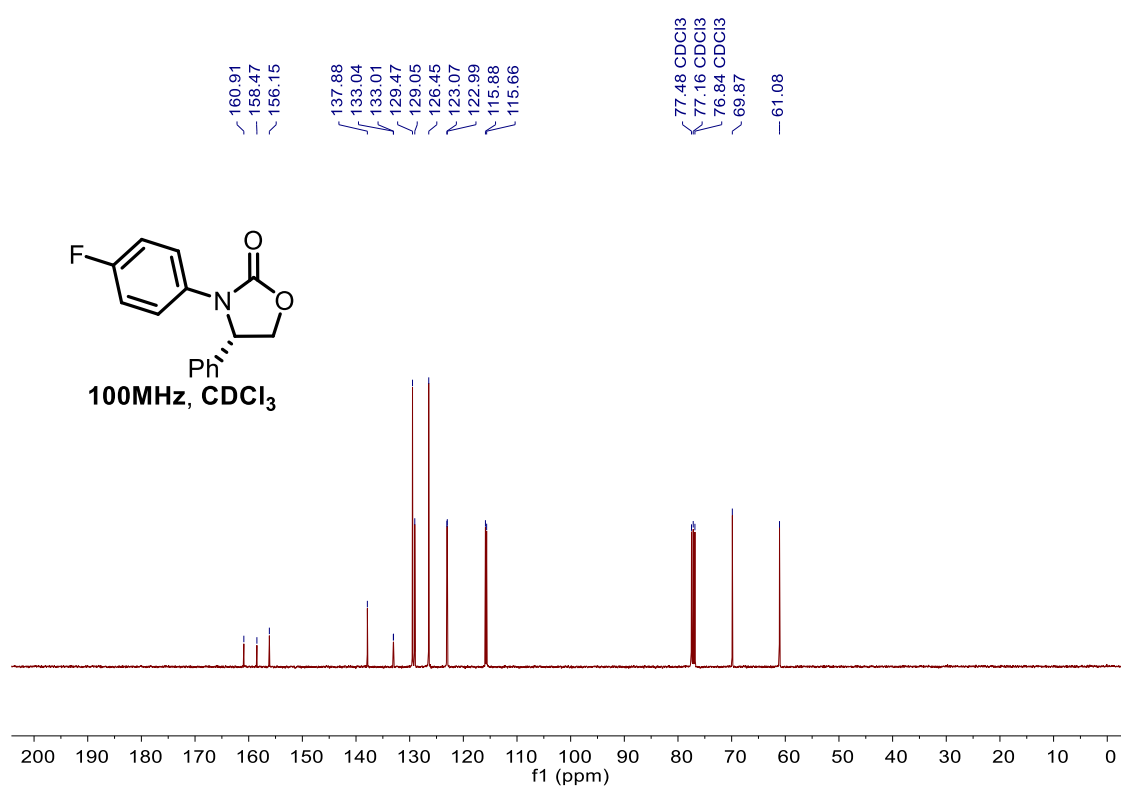
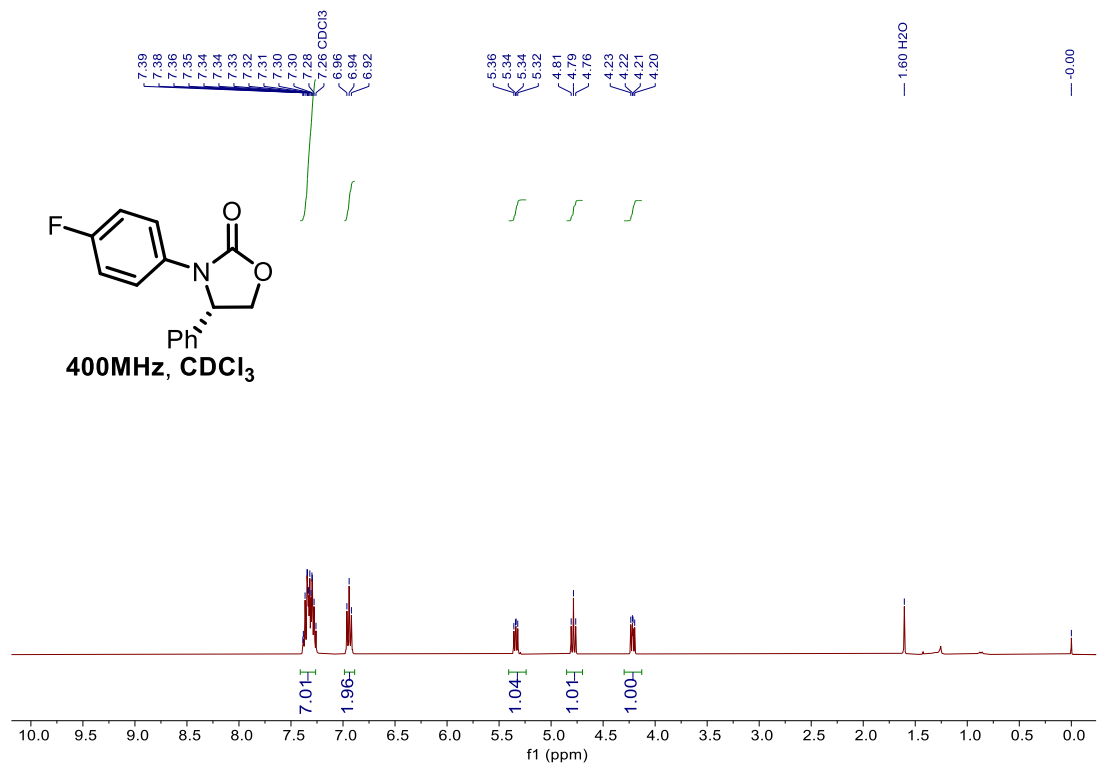
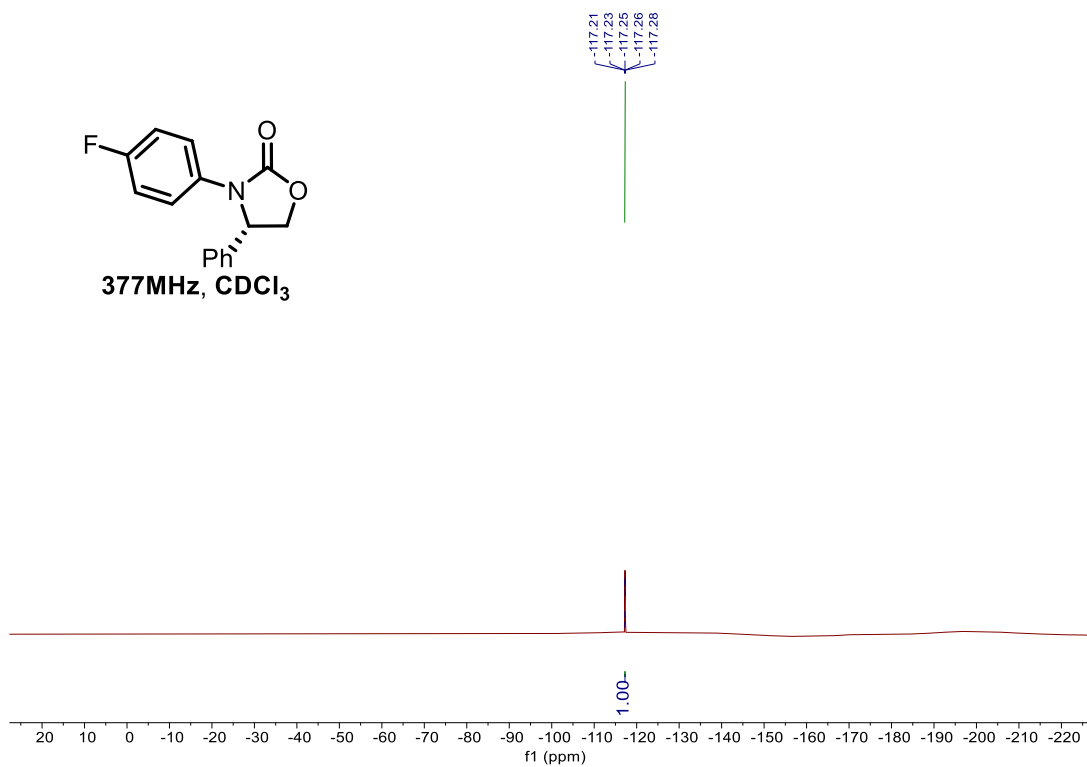
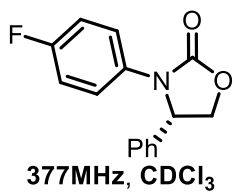


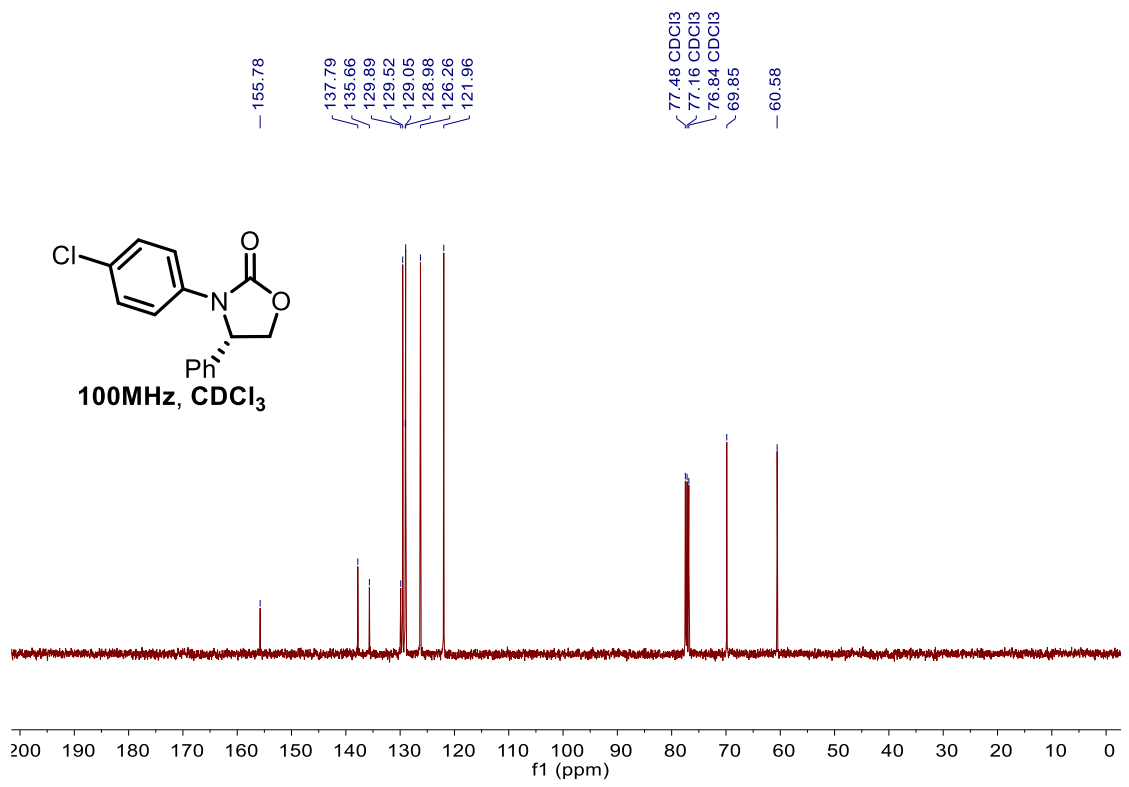
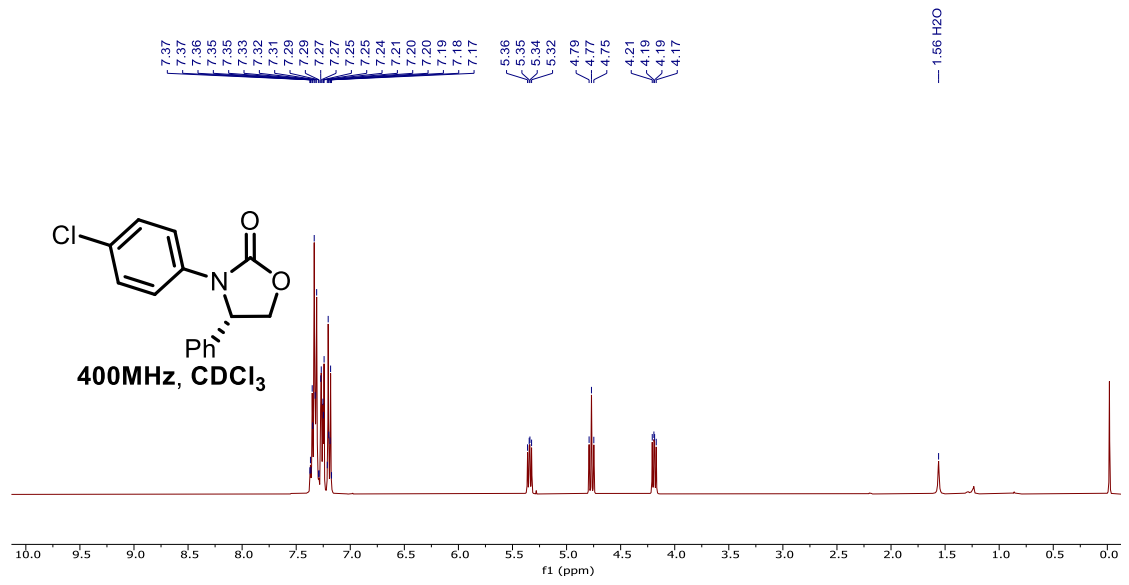
Figure S4. Cyclic voltammogram of **1a** (0.1 M) in an electrolyte of *n*Bu₄NClO₄ (0.1 M) in DMF (5 mL) vs. Ag/AgCl. $E_{p/2}(\mathbf{1a}) = -2.14 \text{ V vs. Ag/AgCl}$.

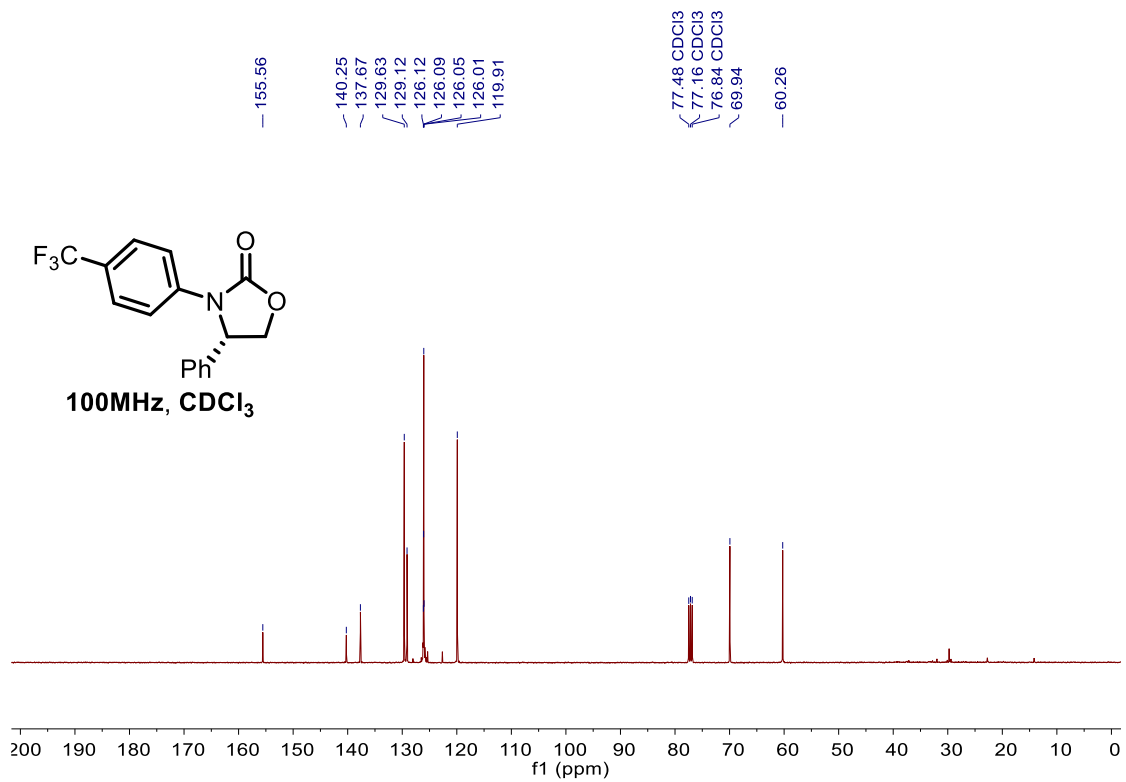
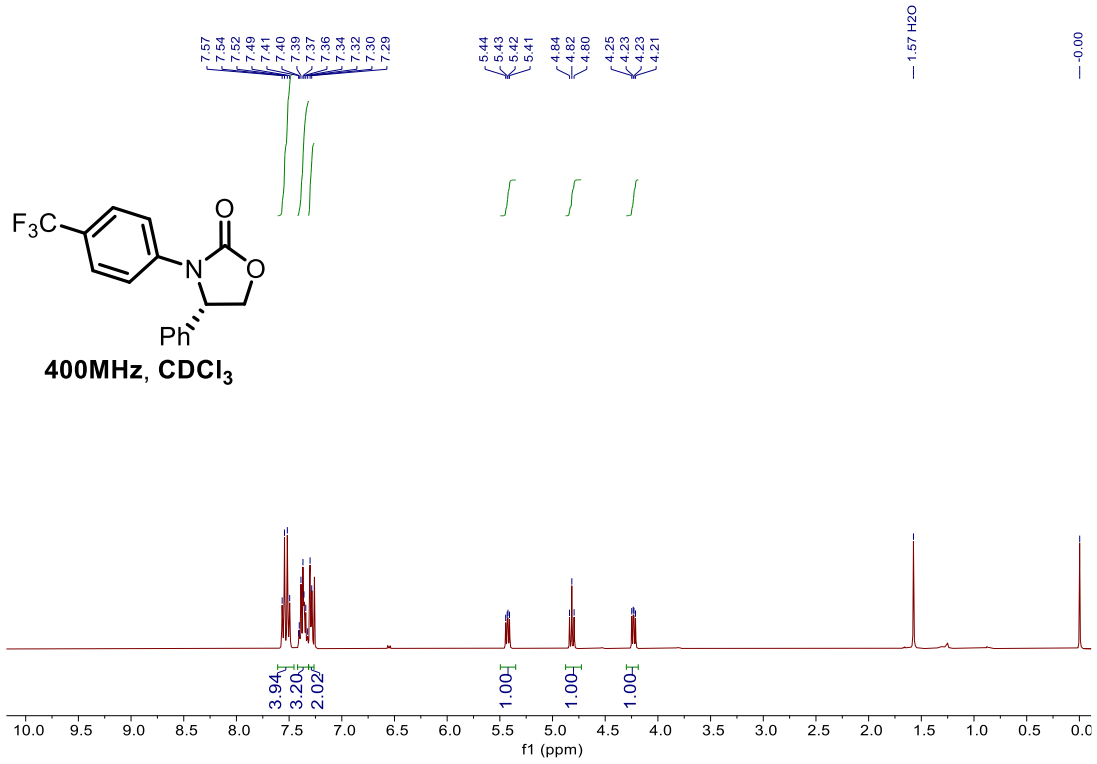
^1H and ^{13}C NMR Spectra of Unreported Starting Materials



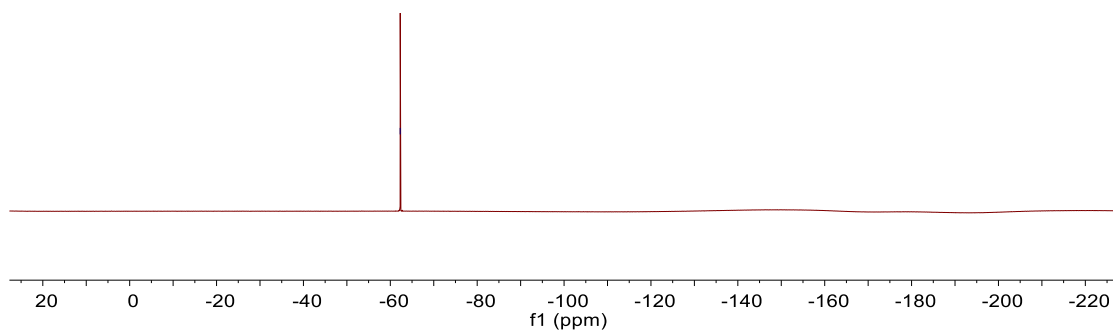
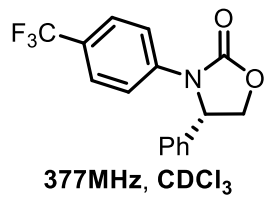


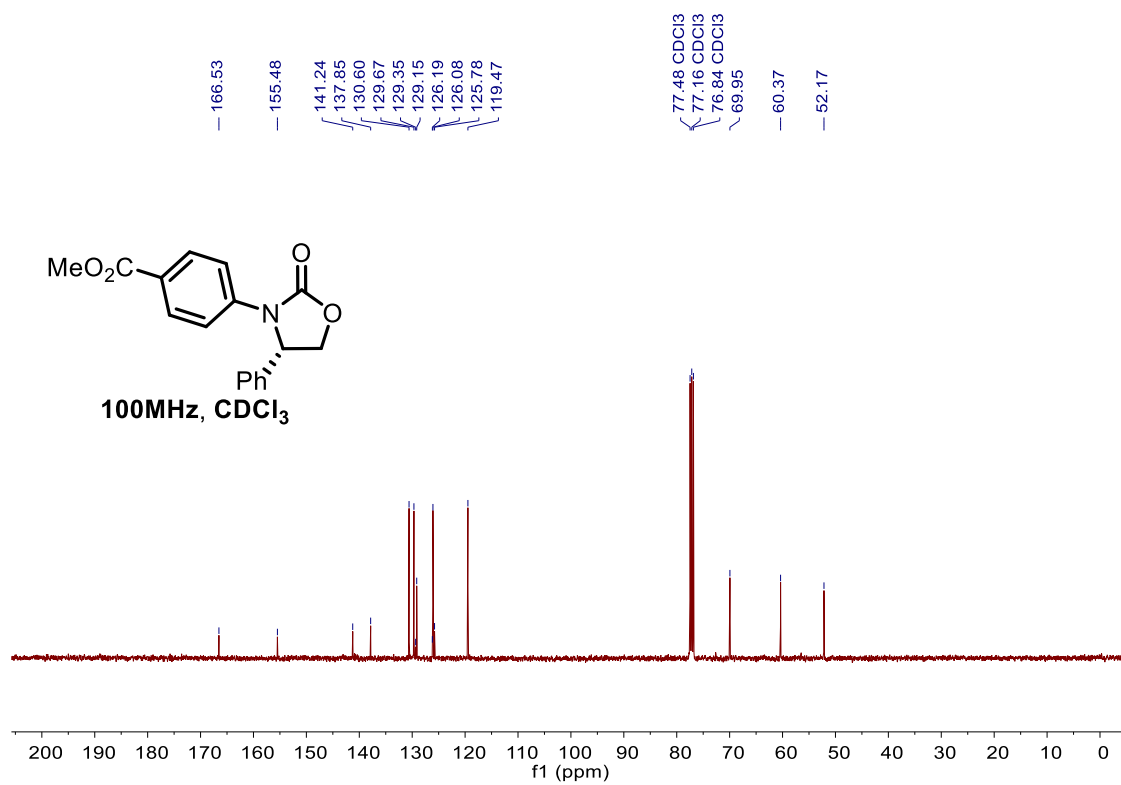
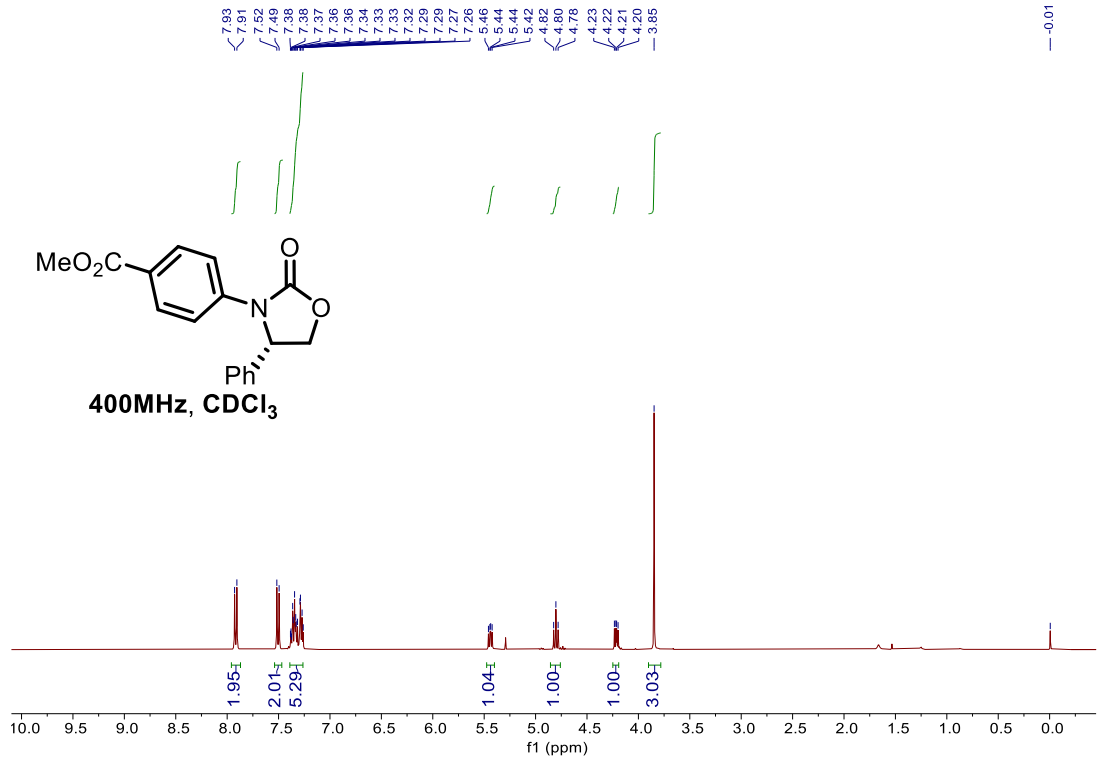


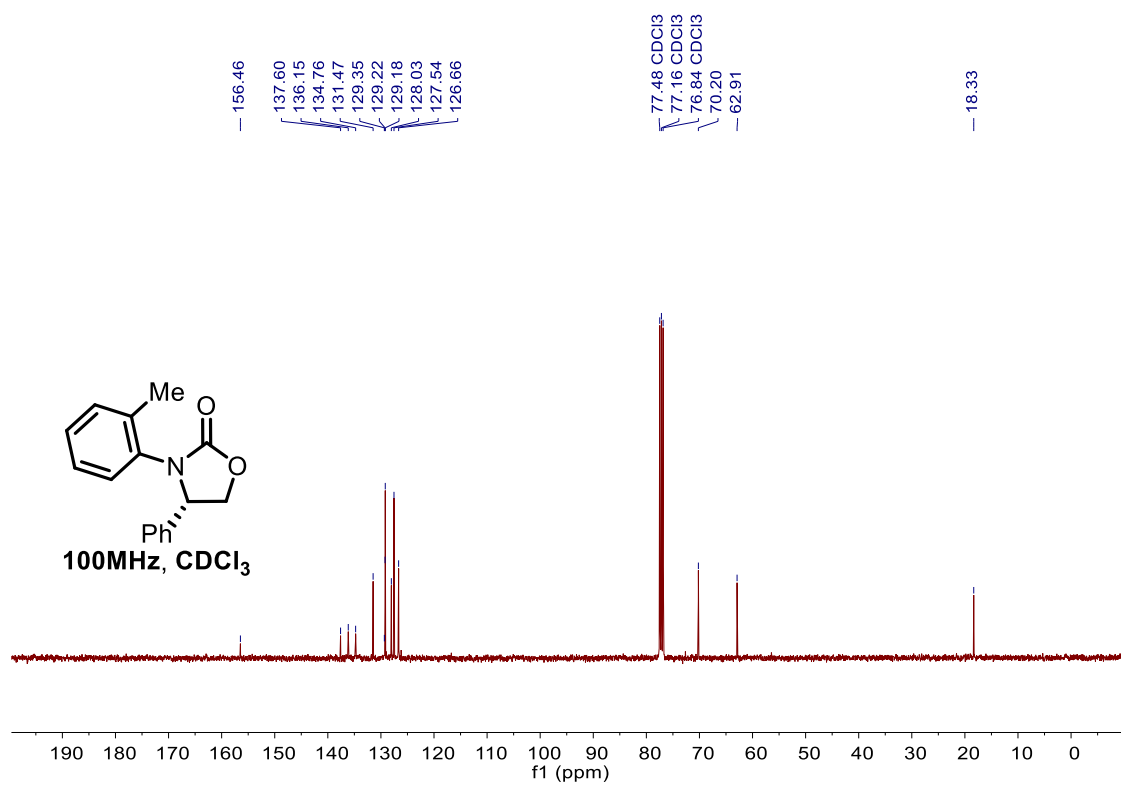
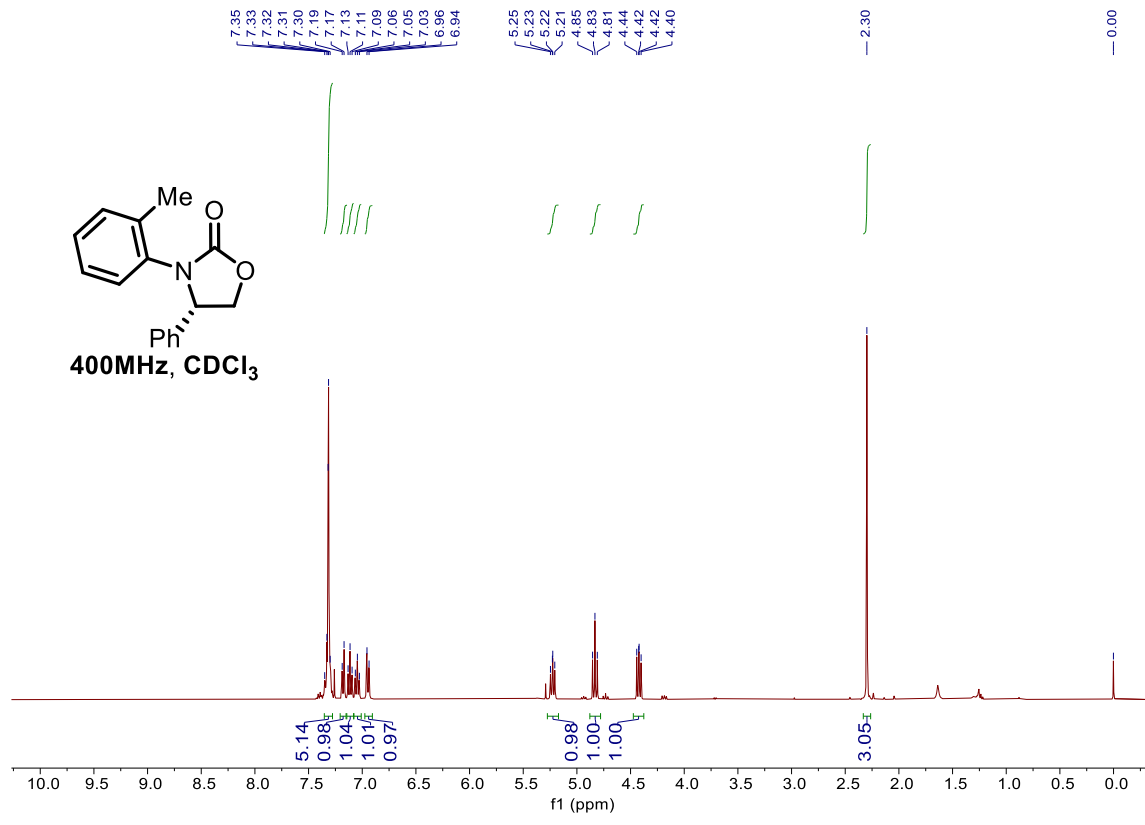


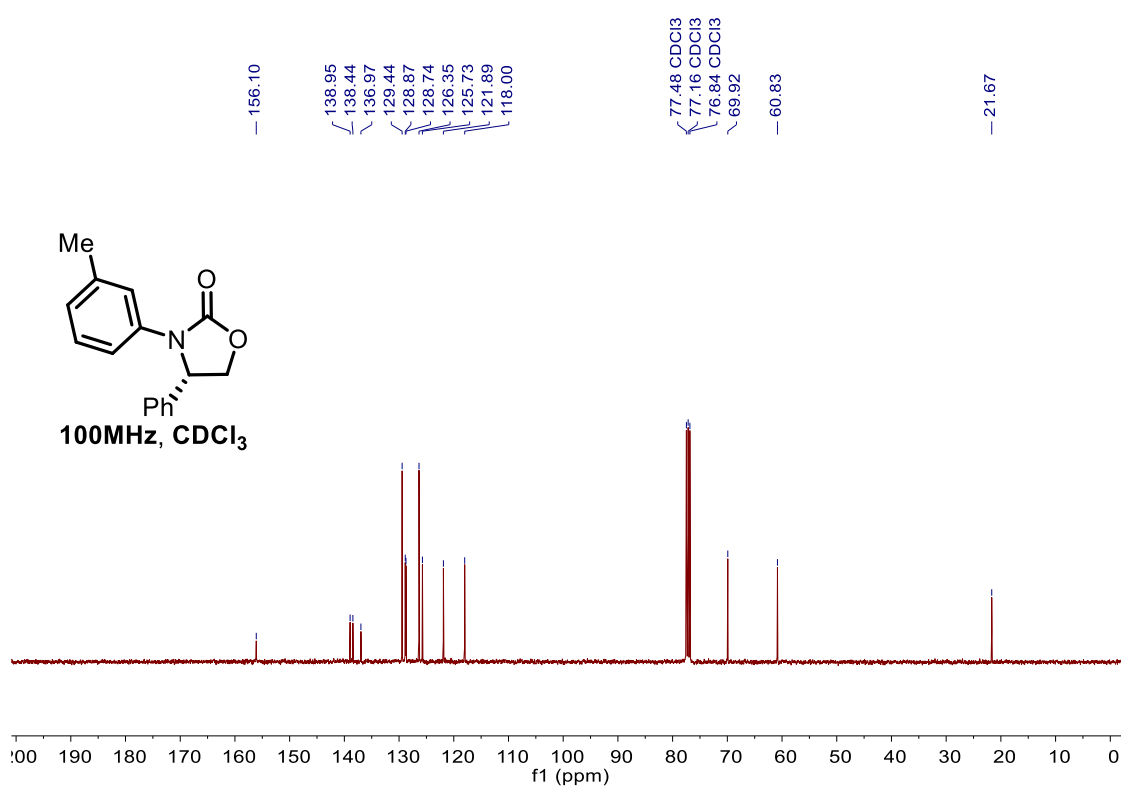
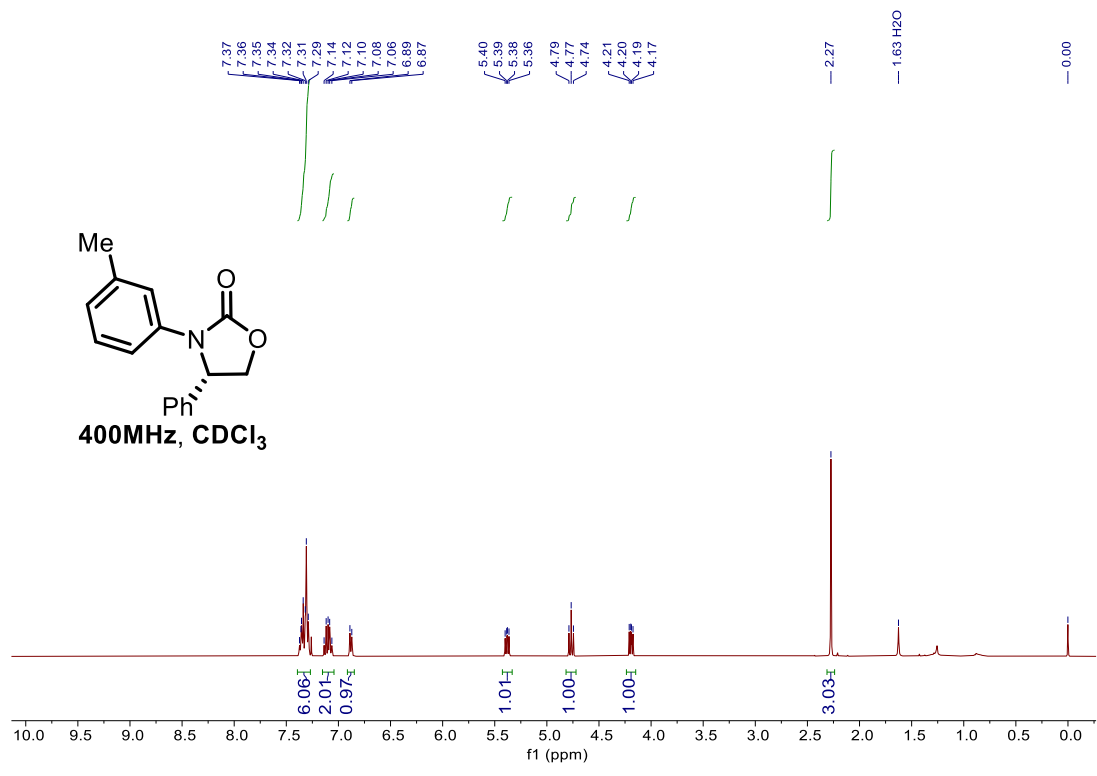


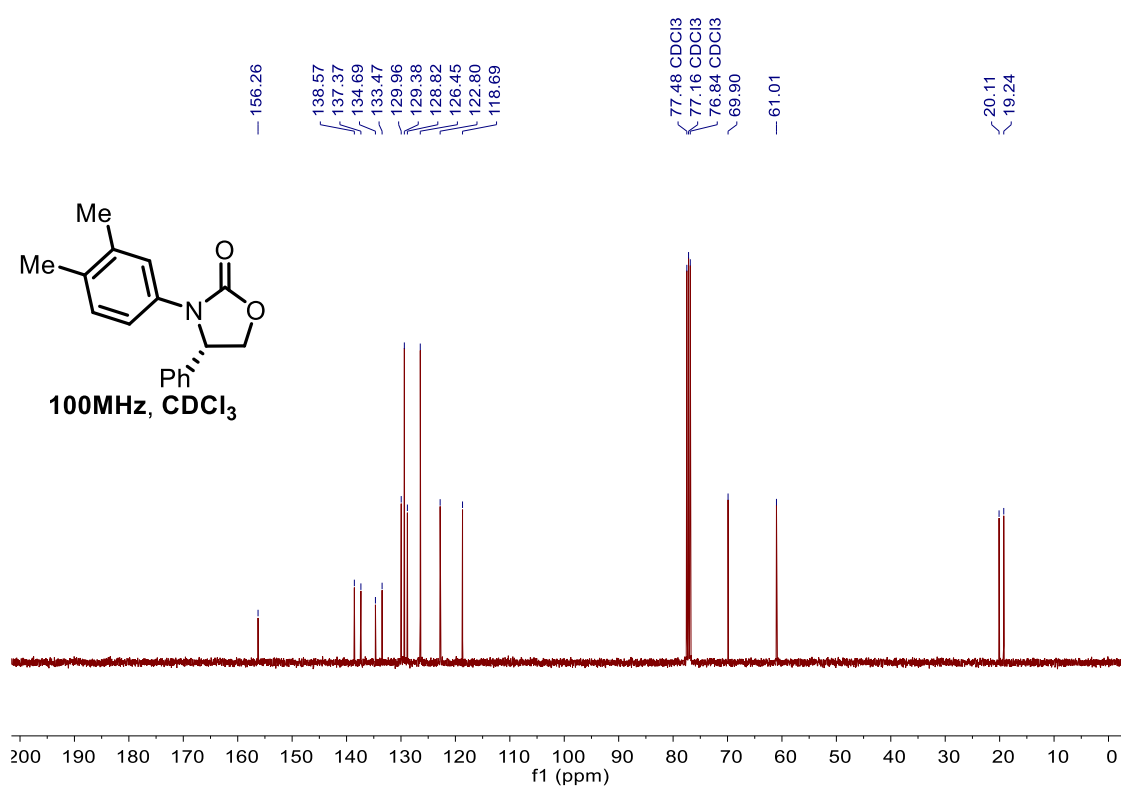
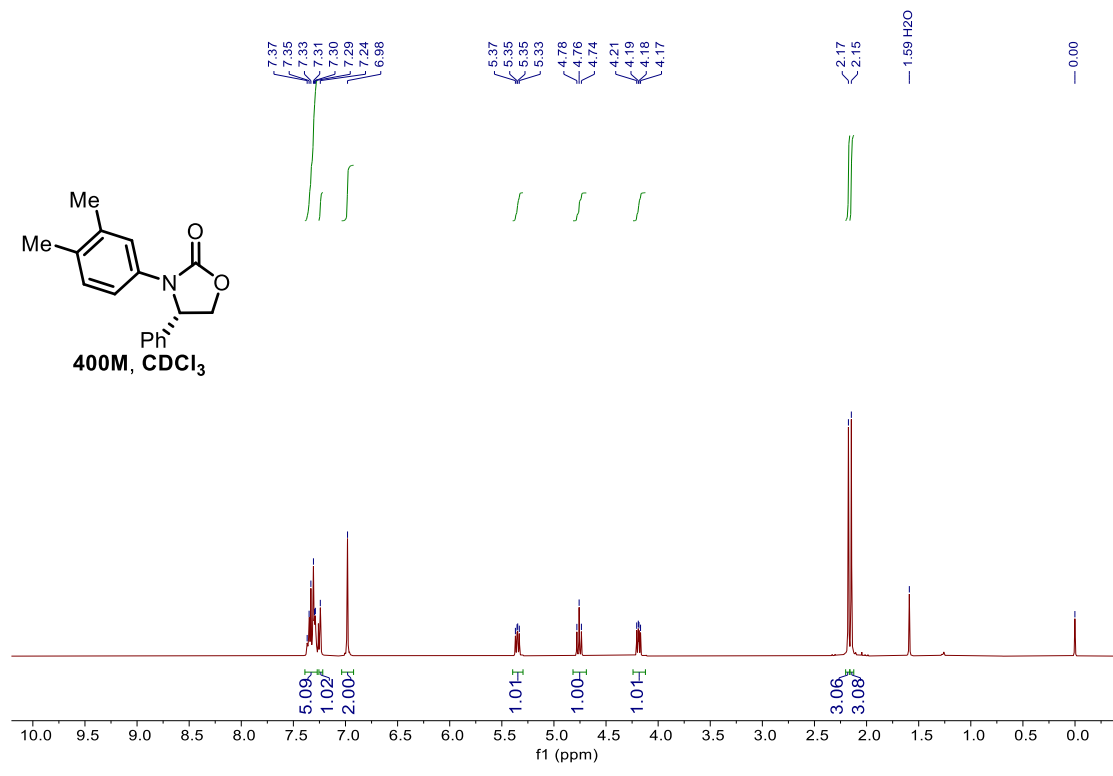
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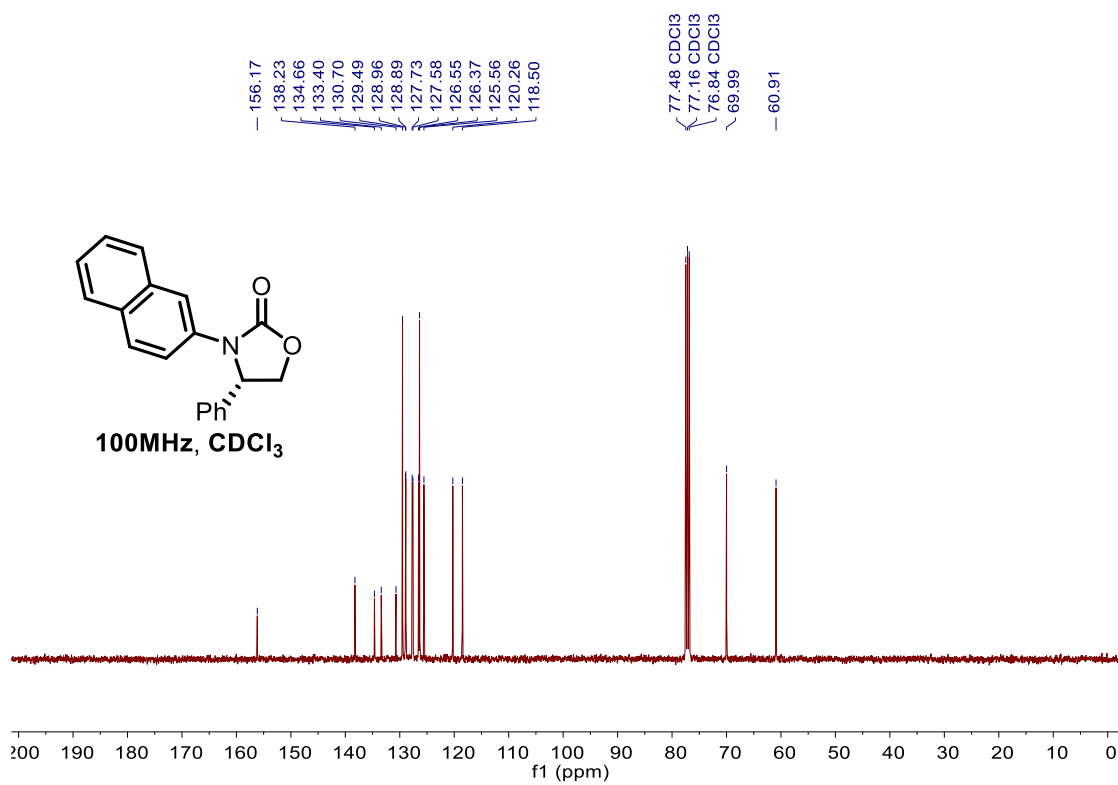
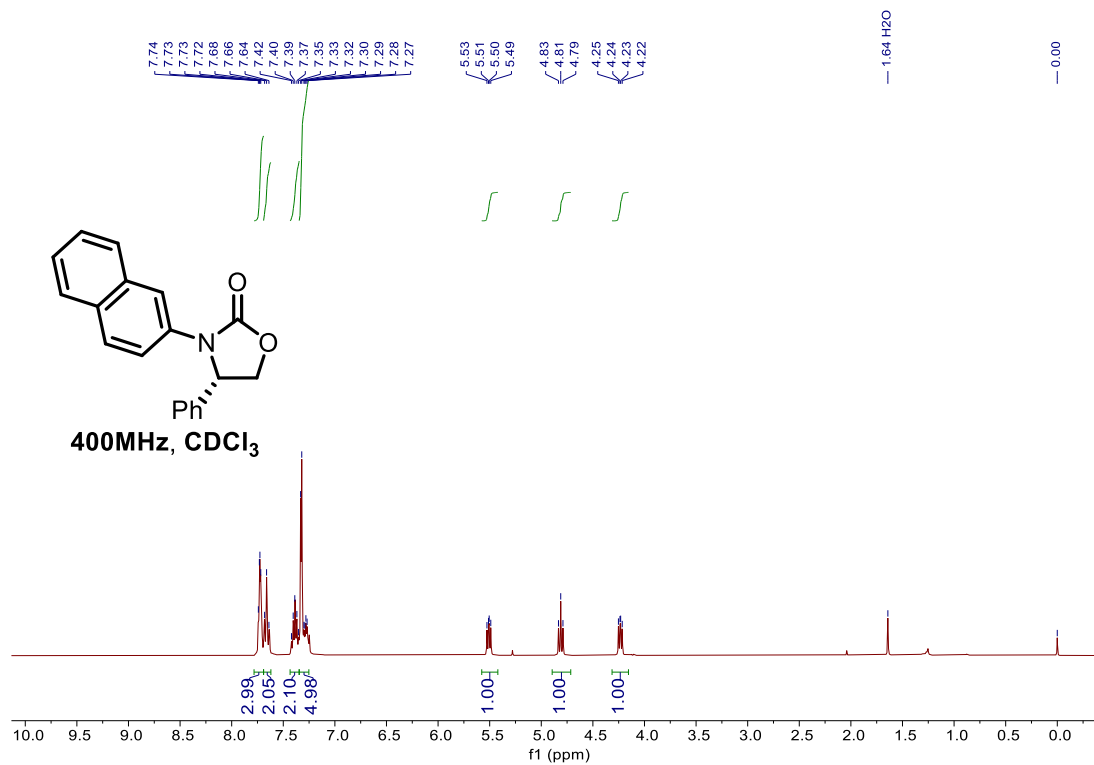


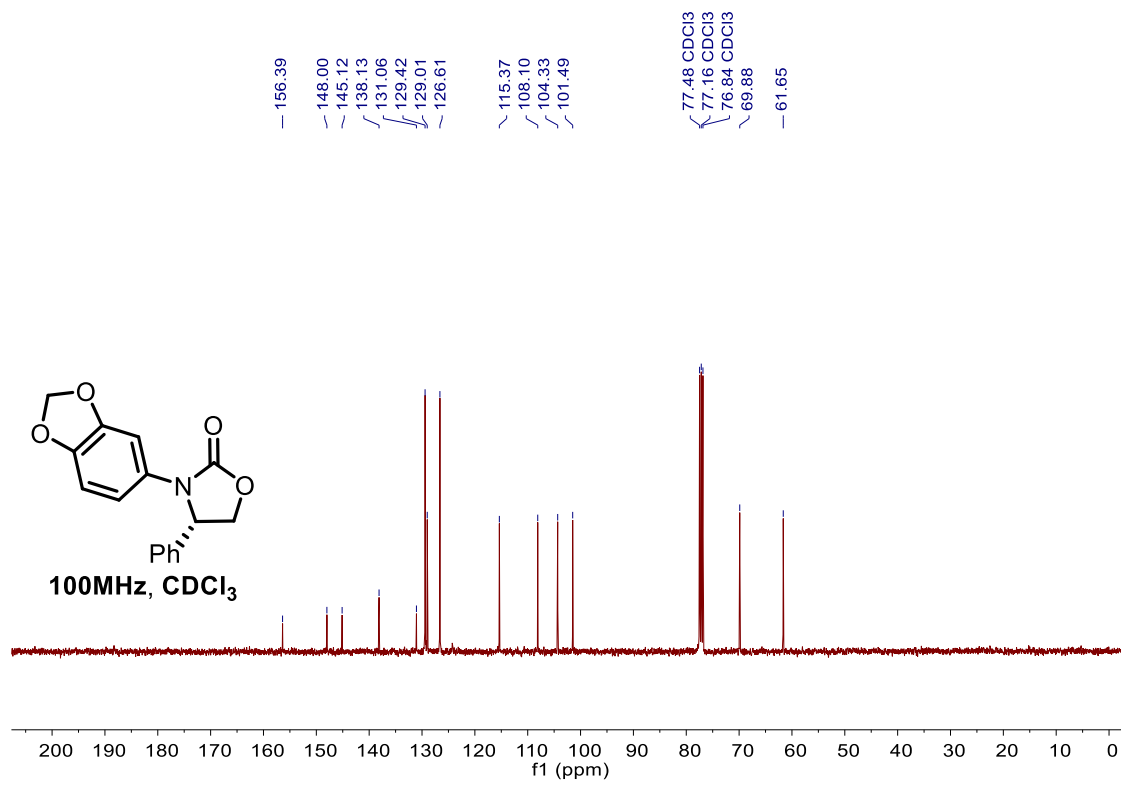
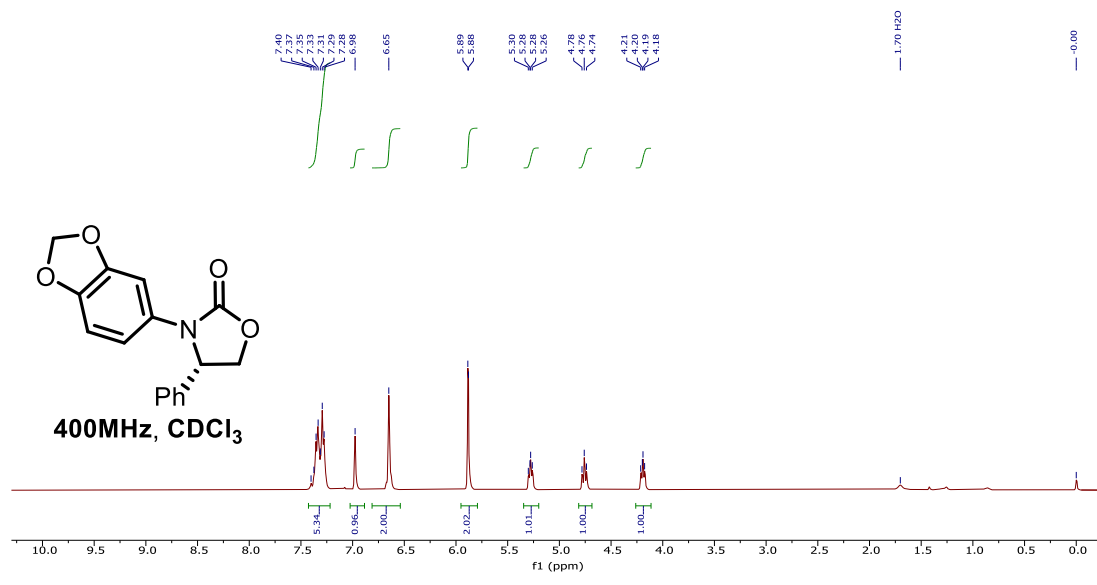


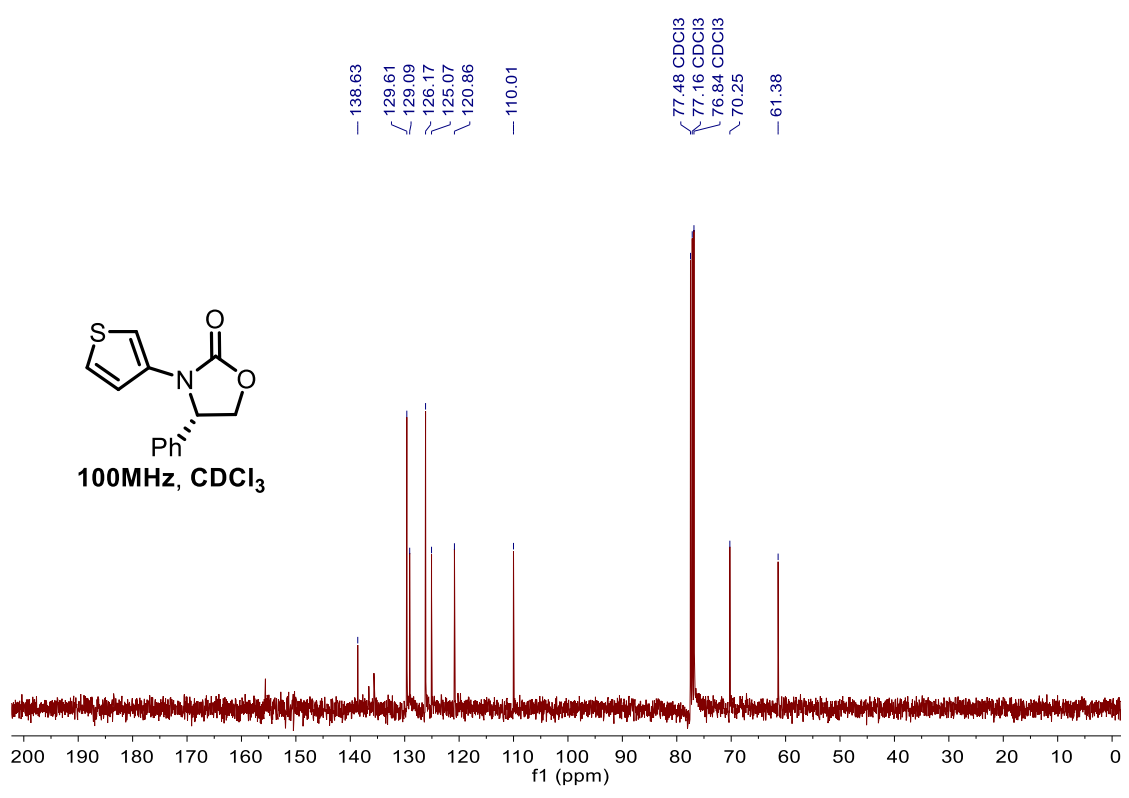
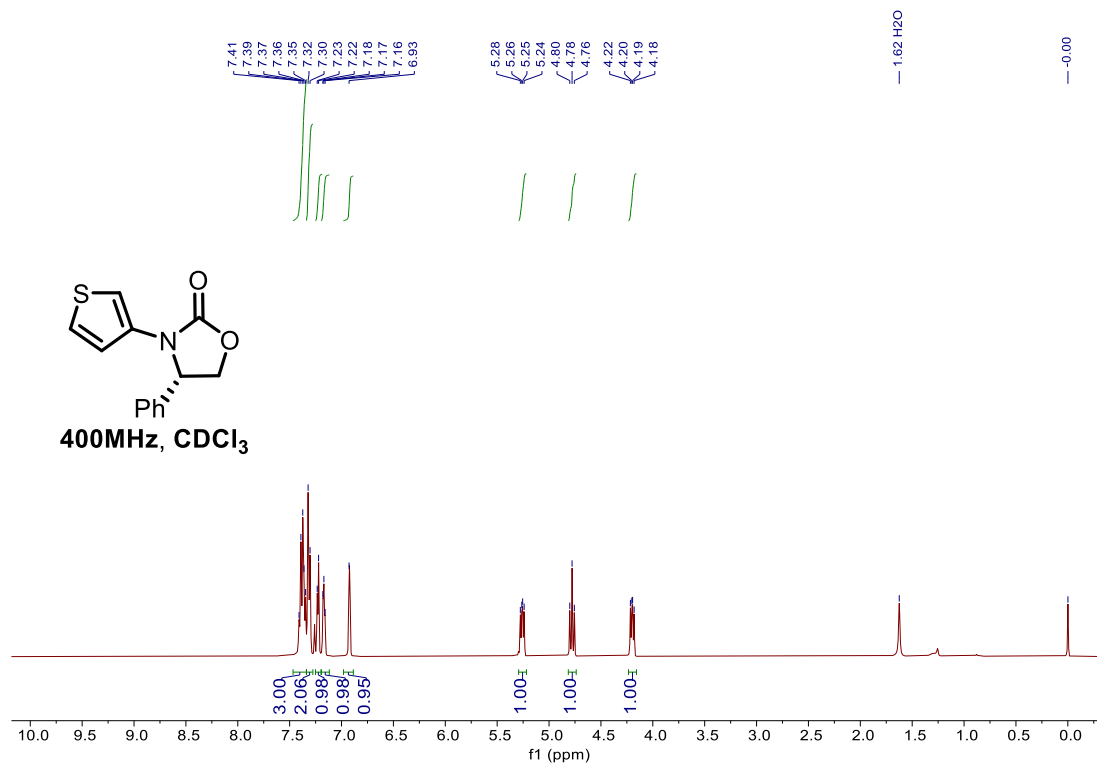


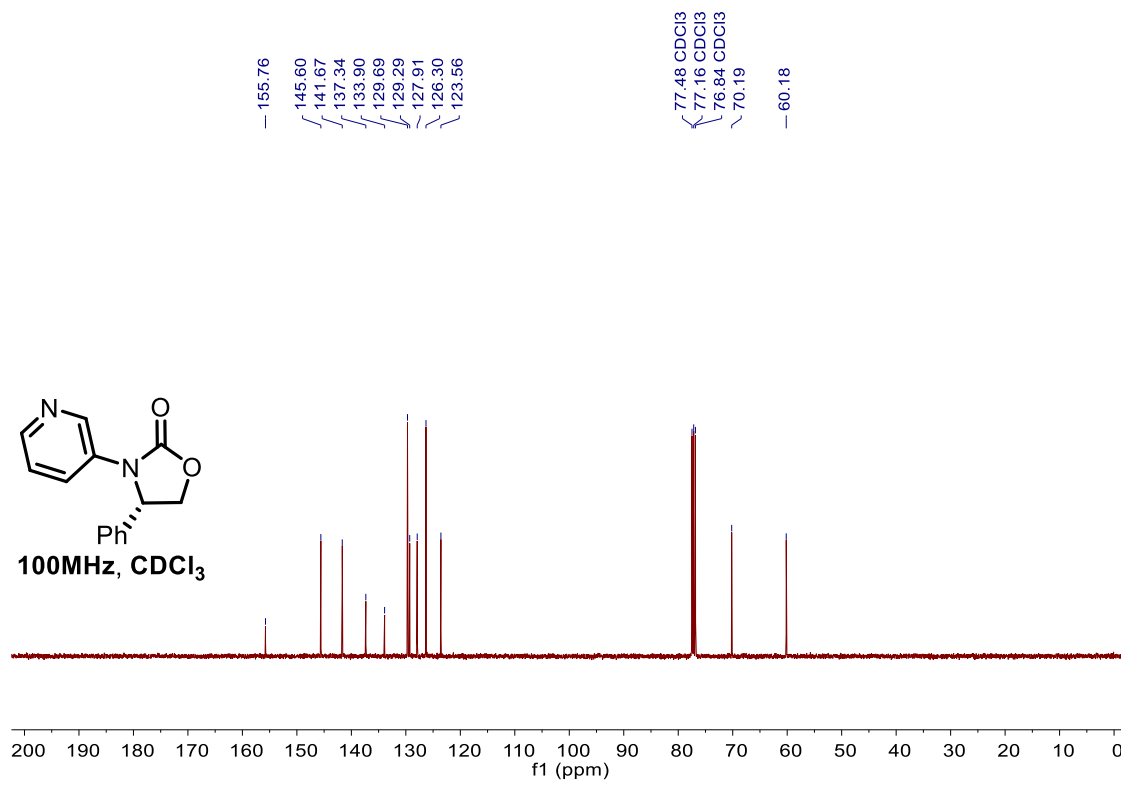
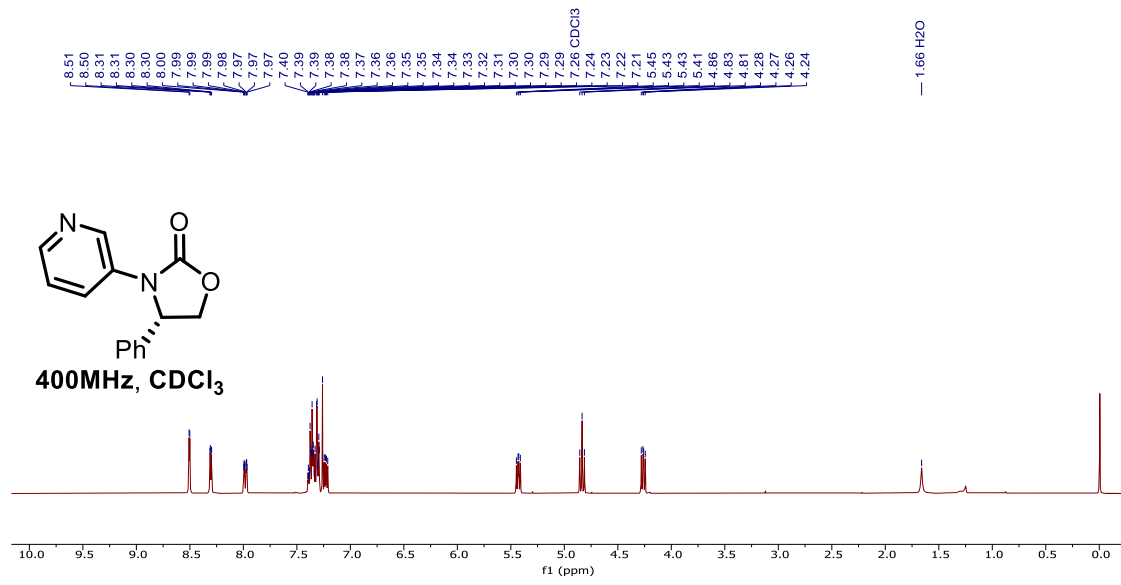


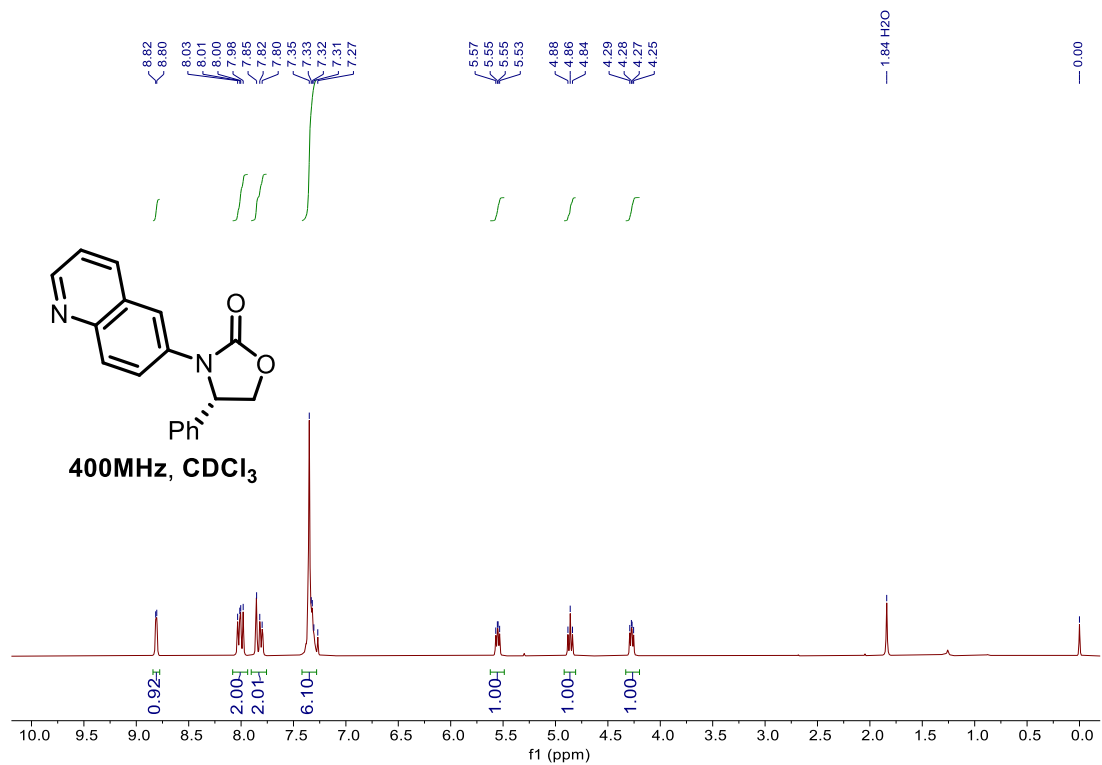




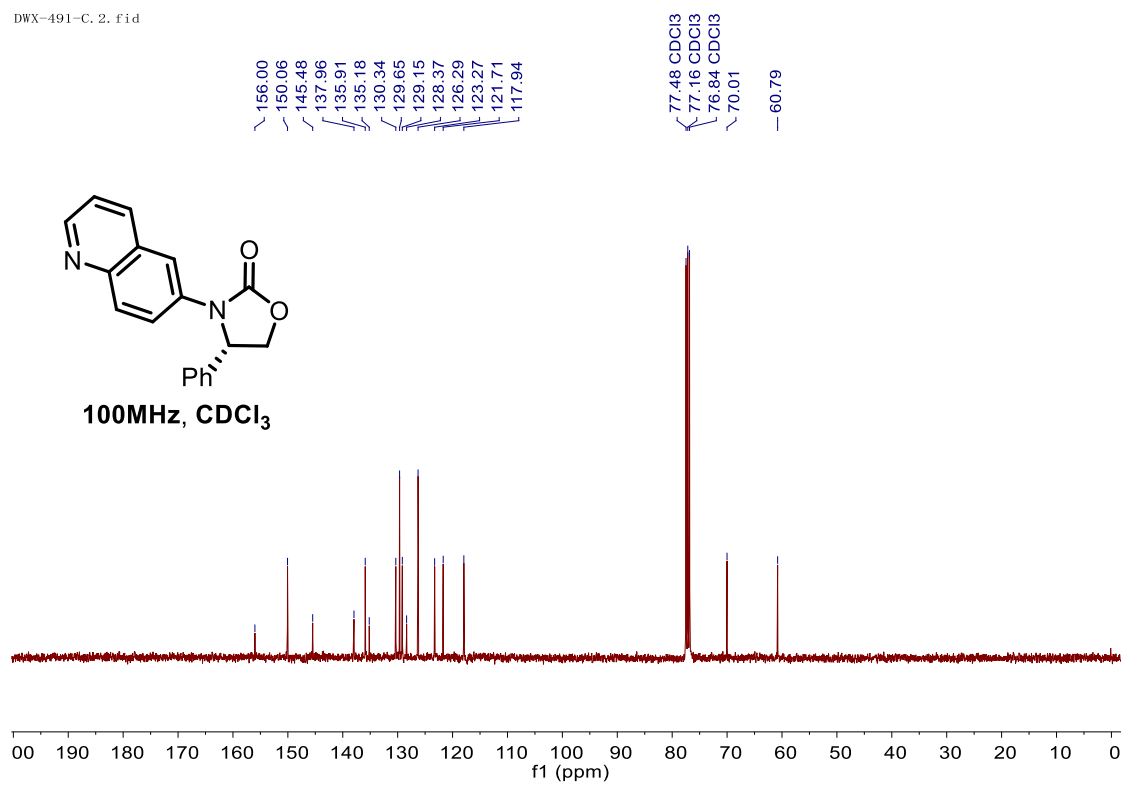


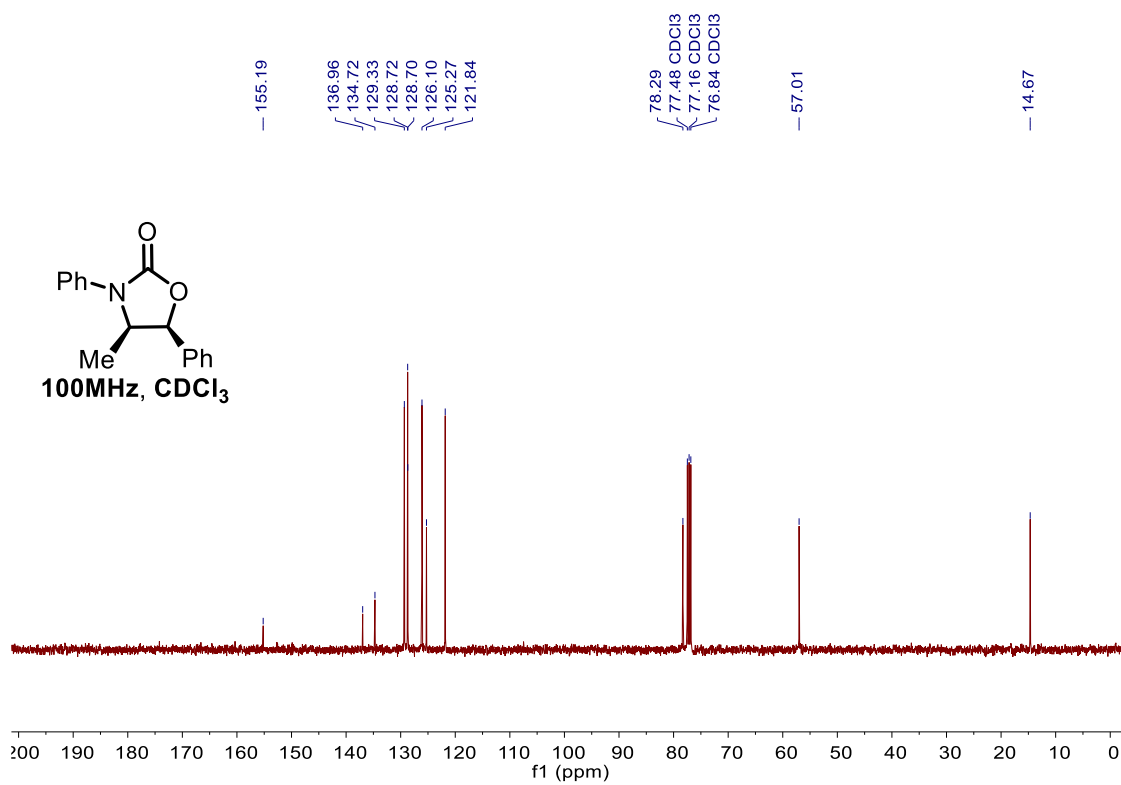
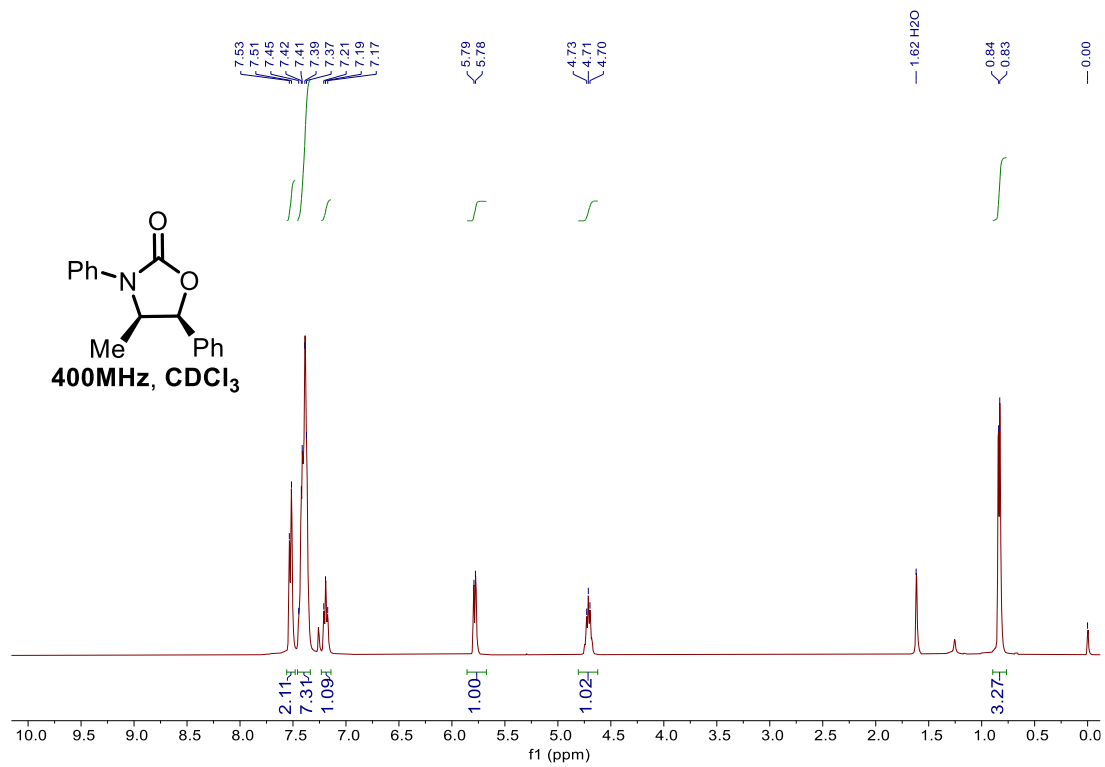




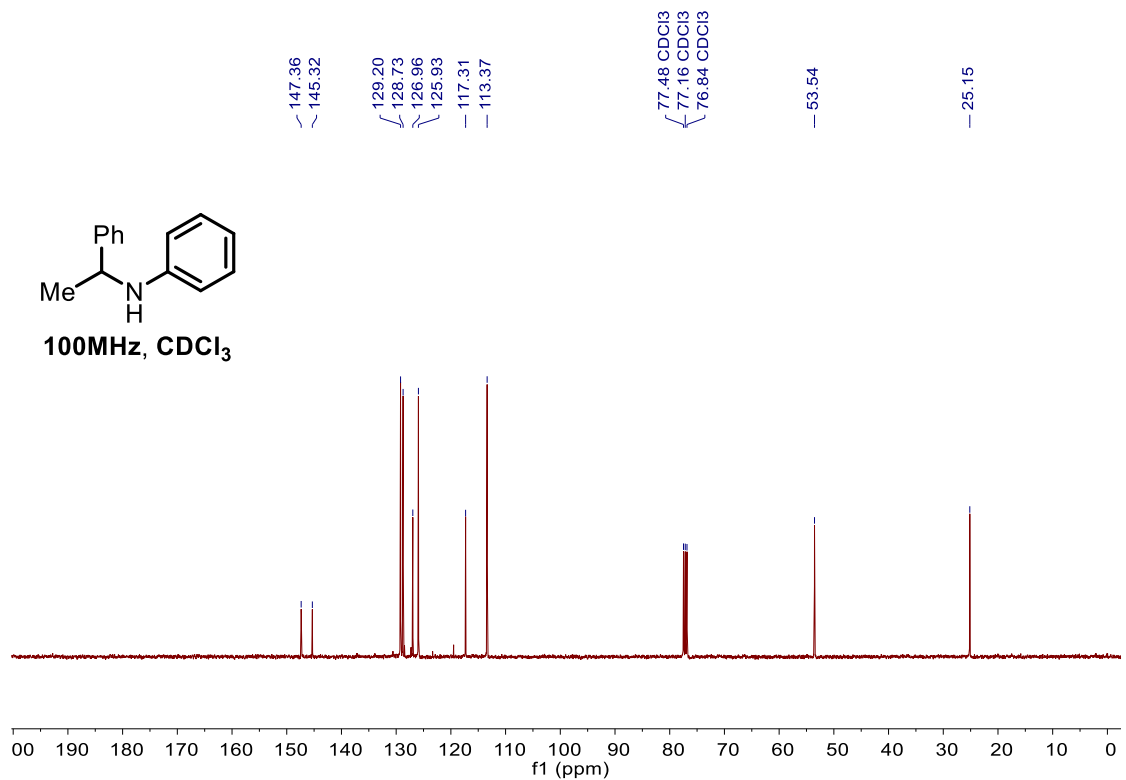
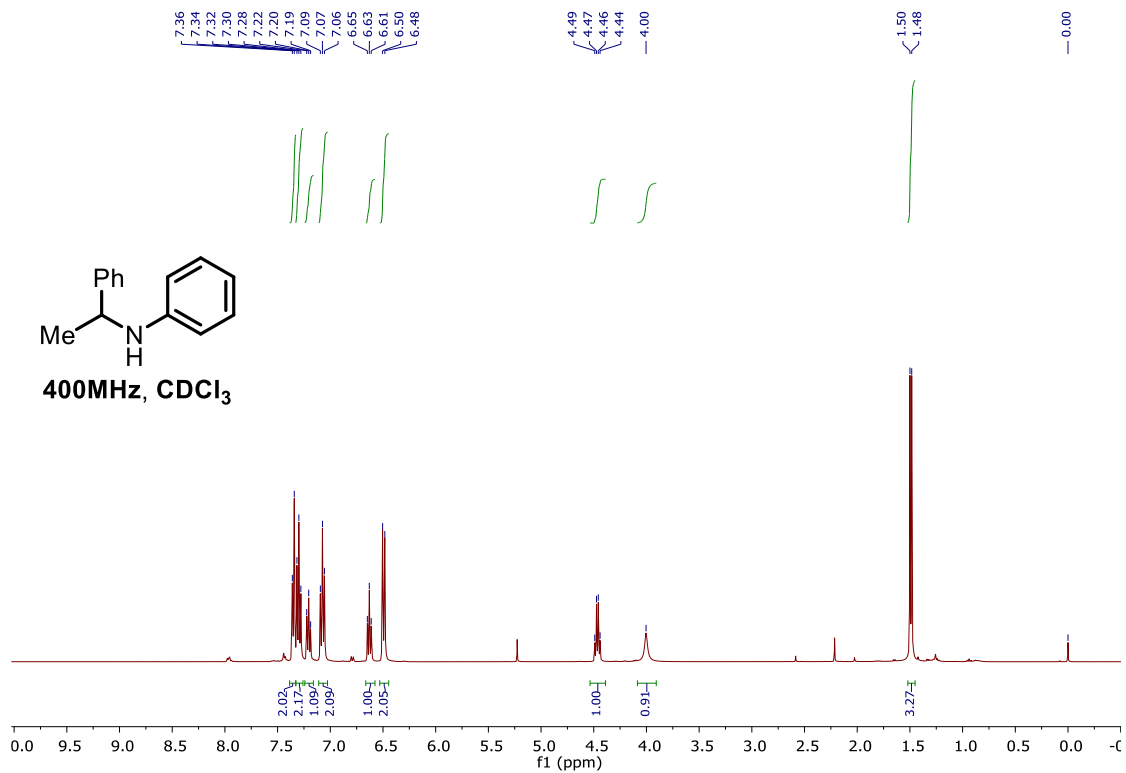


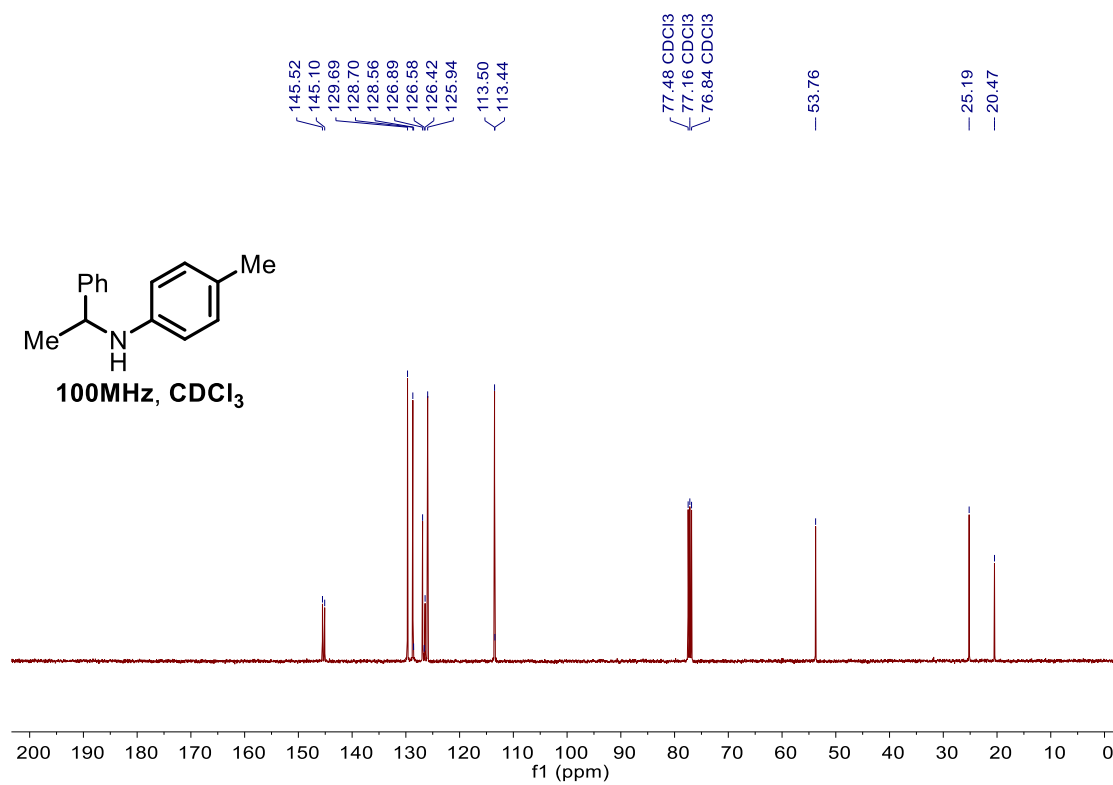
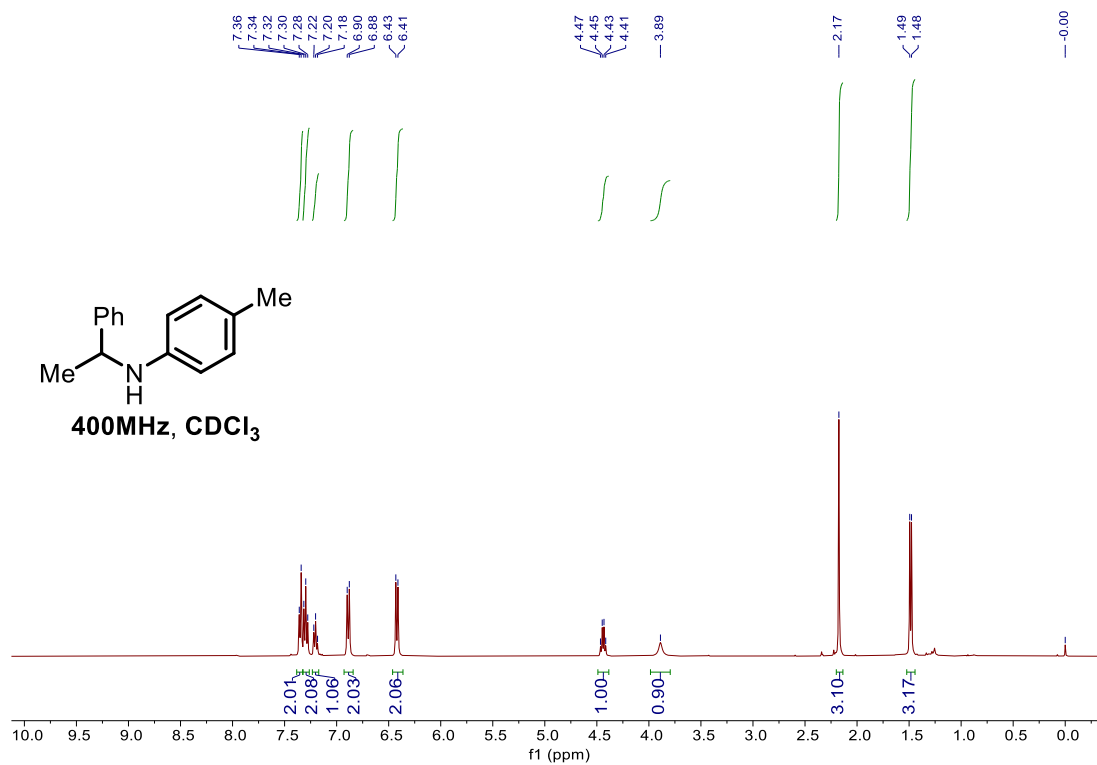
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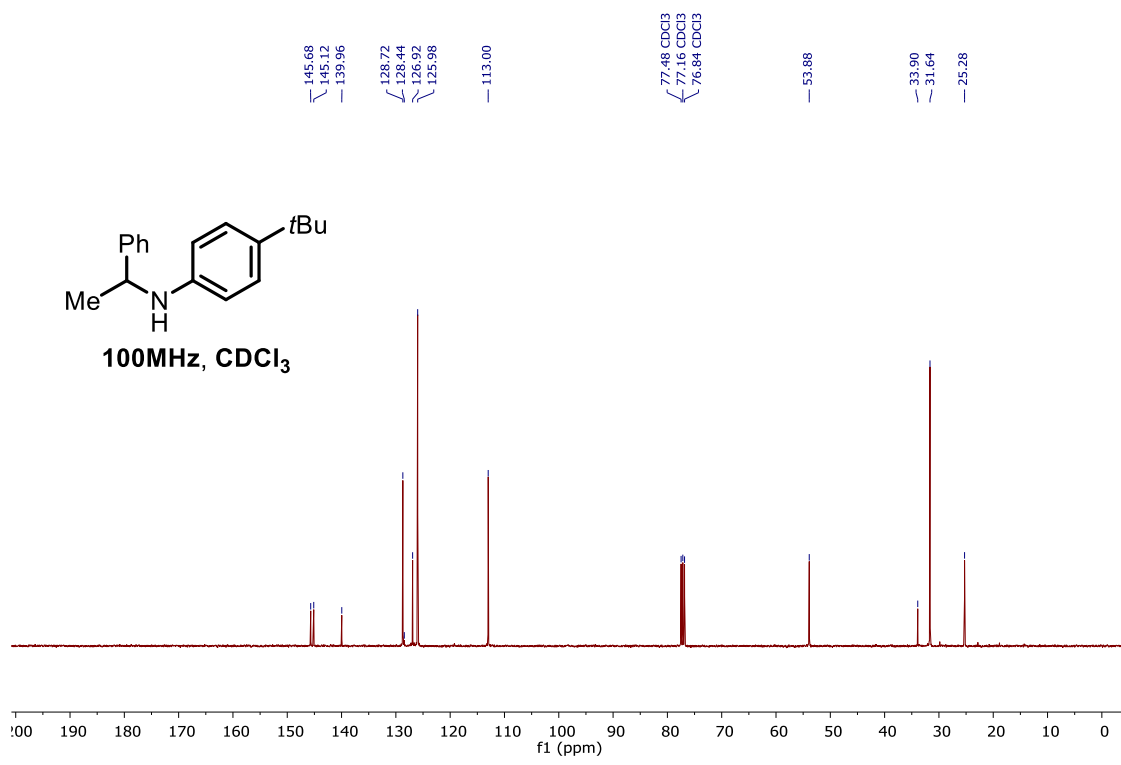
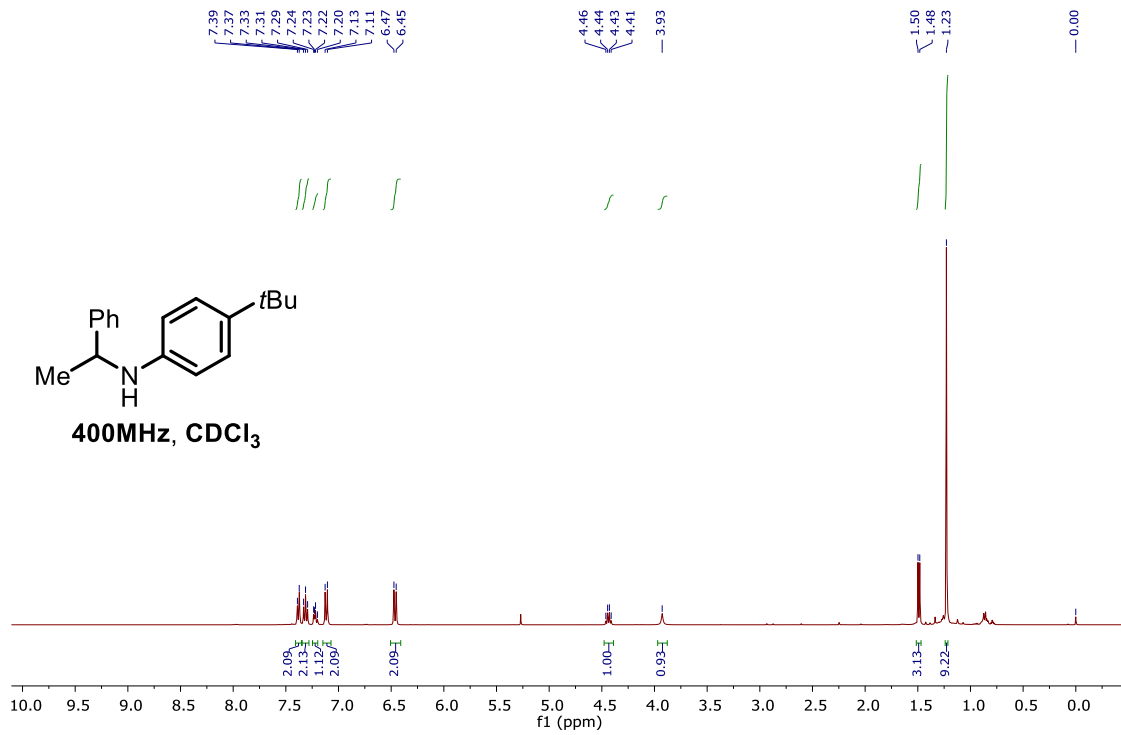


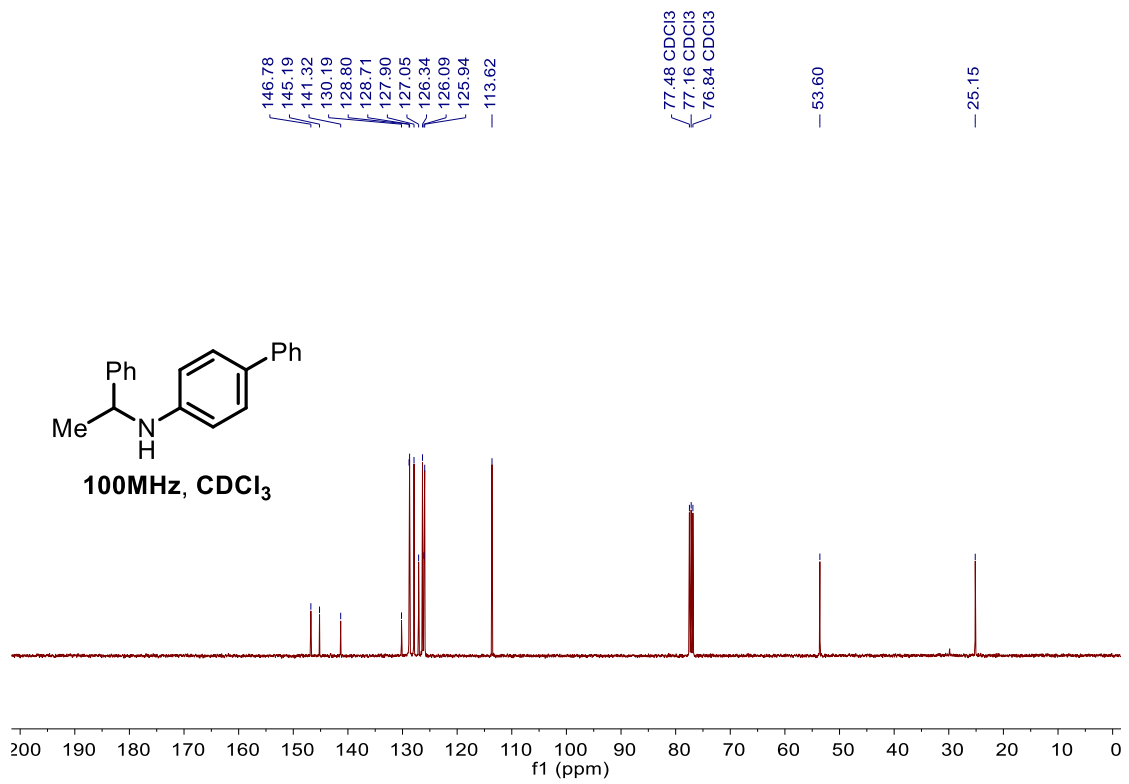
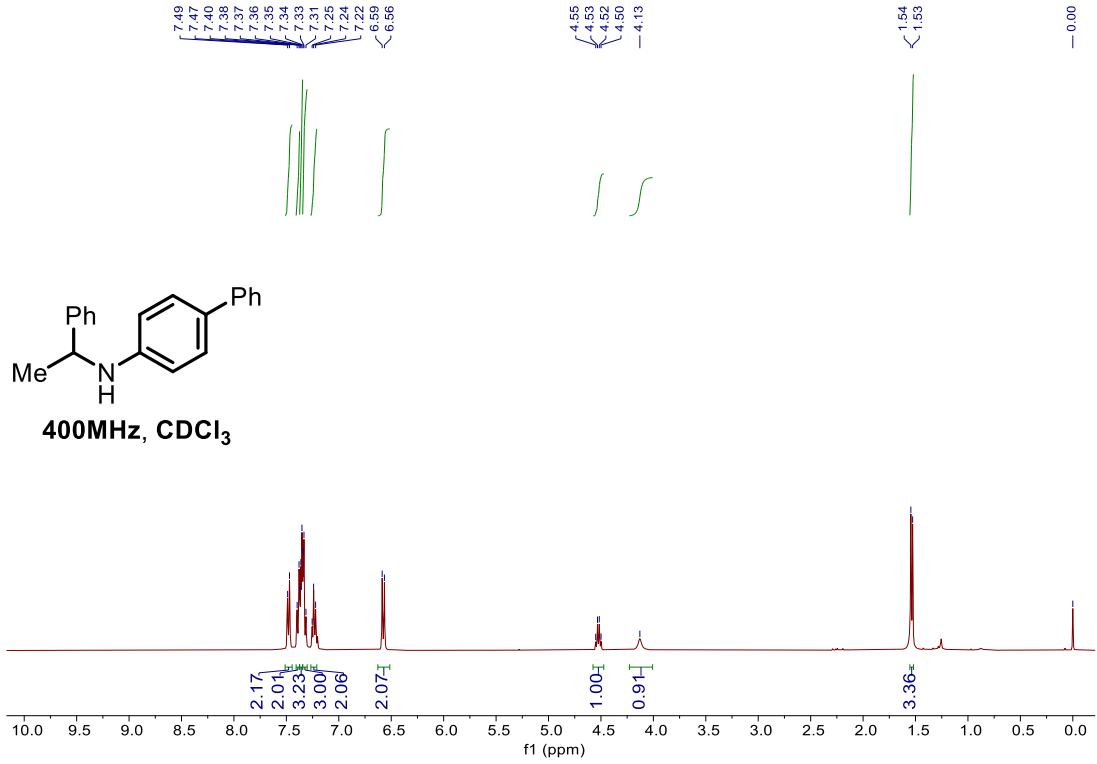


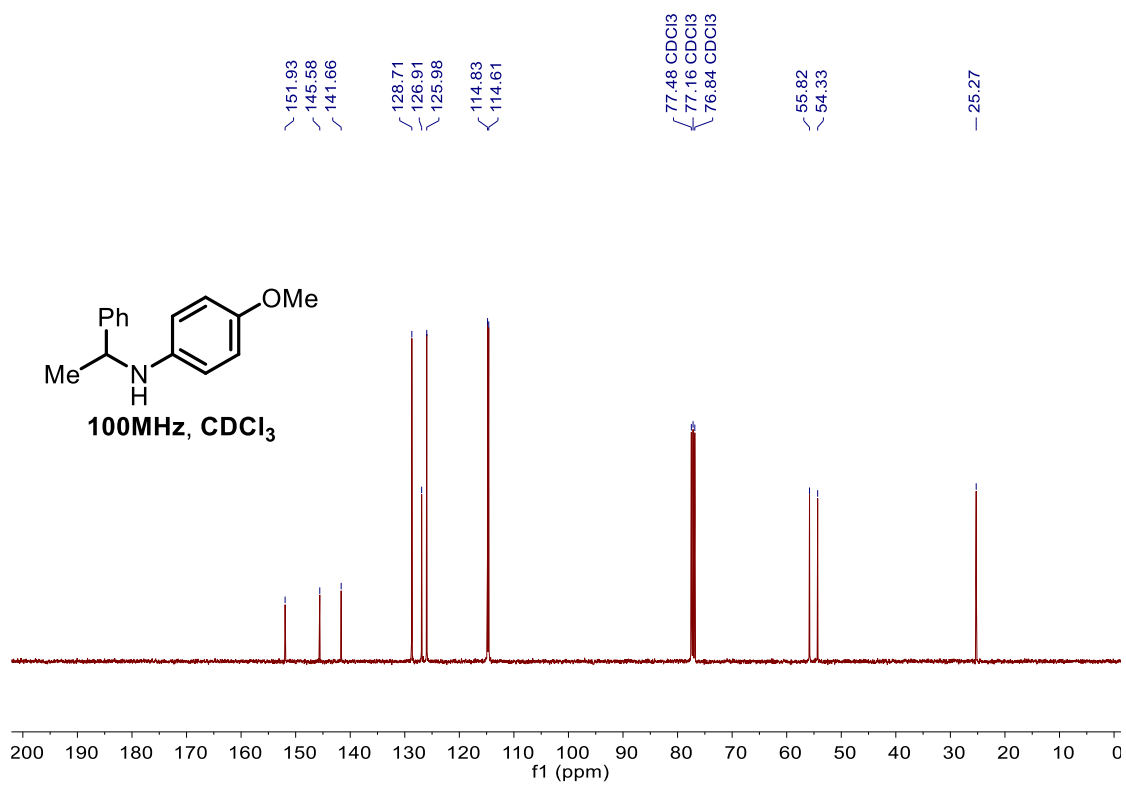
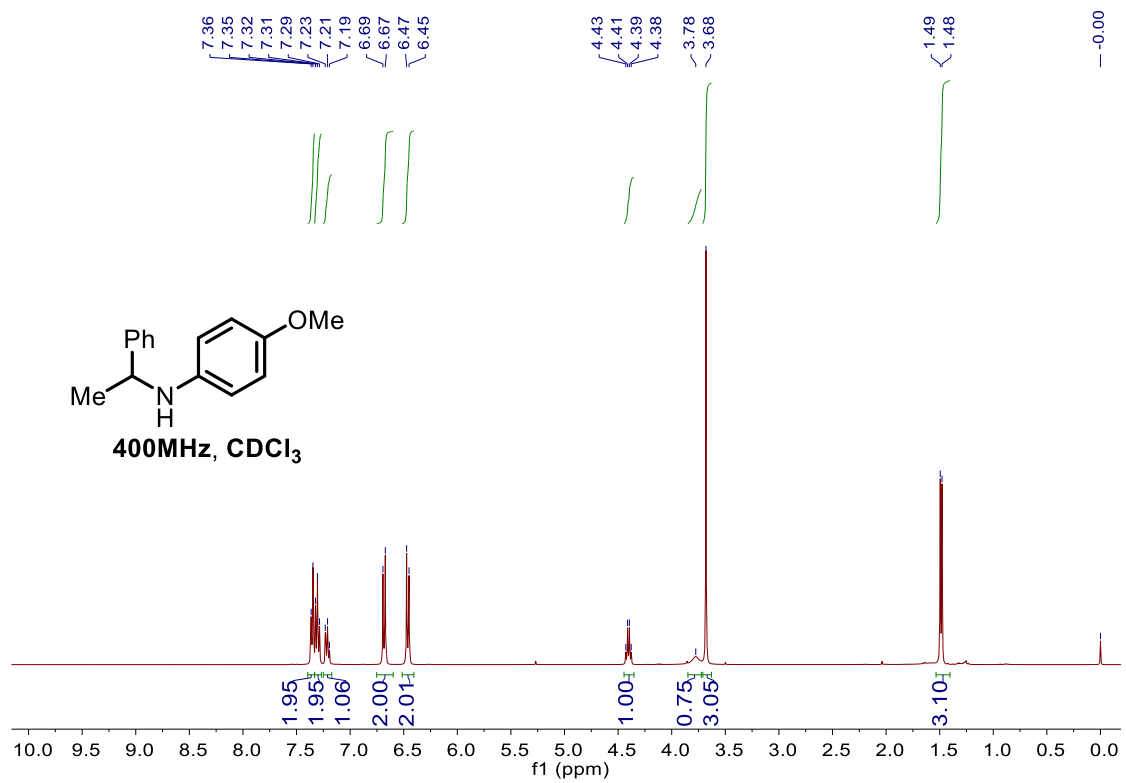
10. NMR Spectra of Products

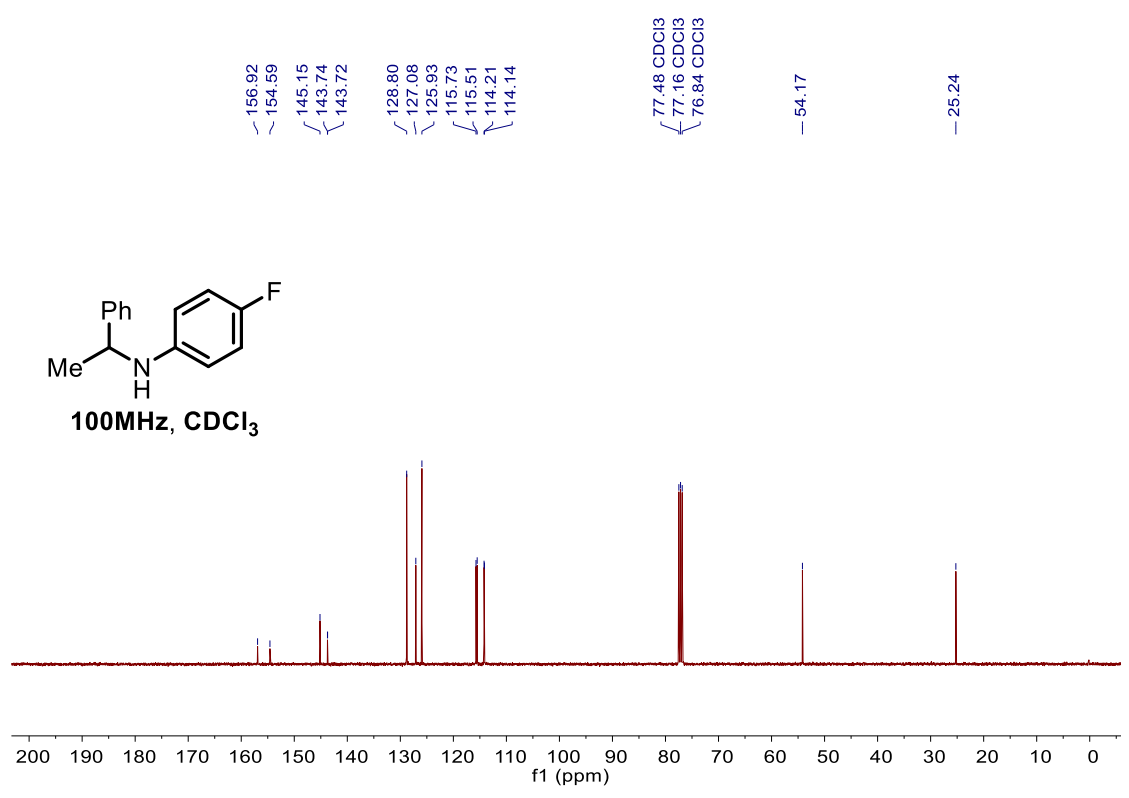
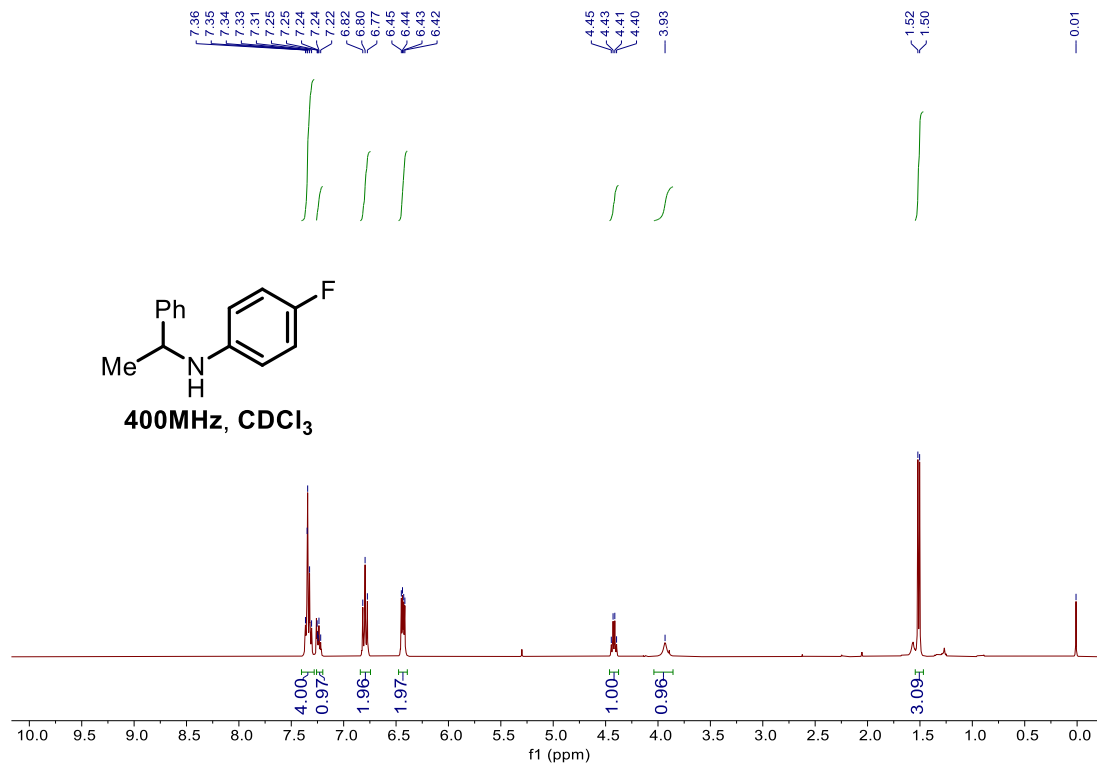


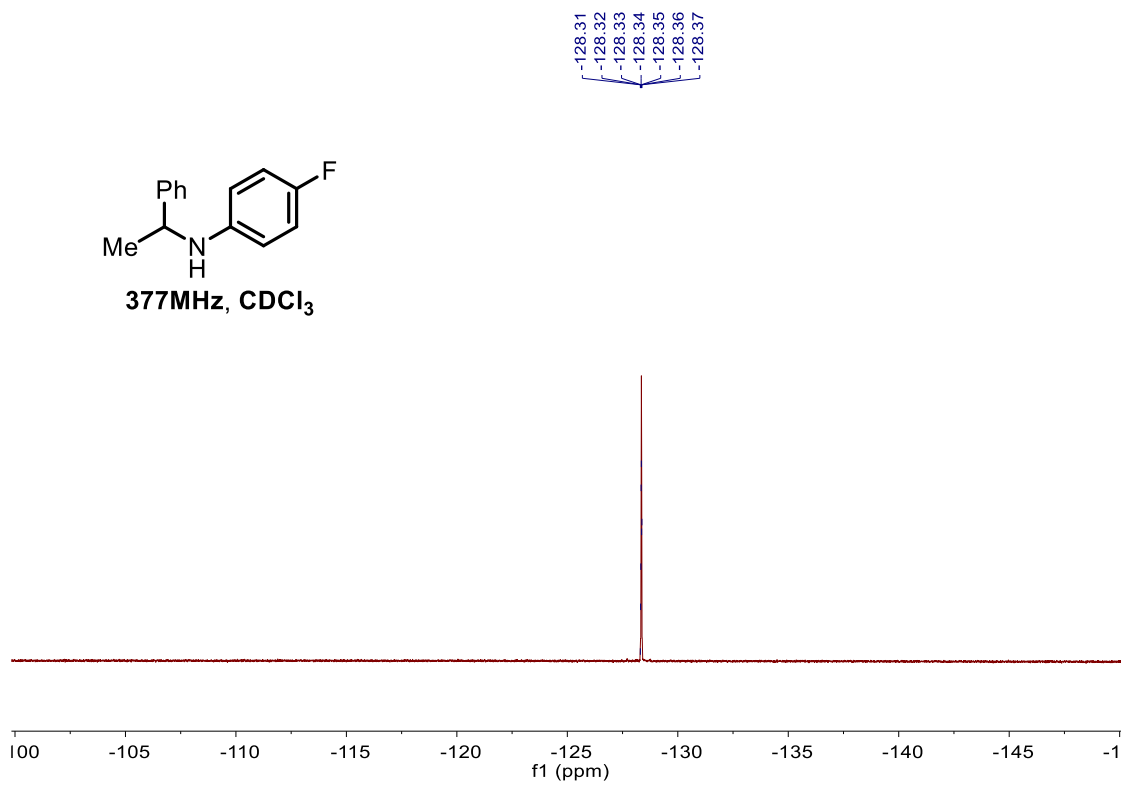
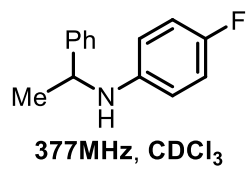


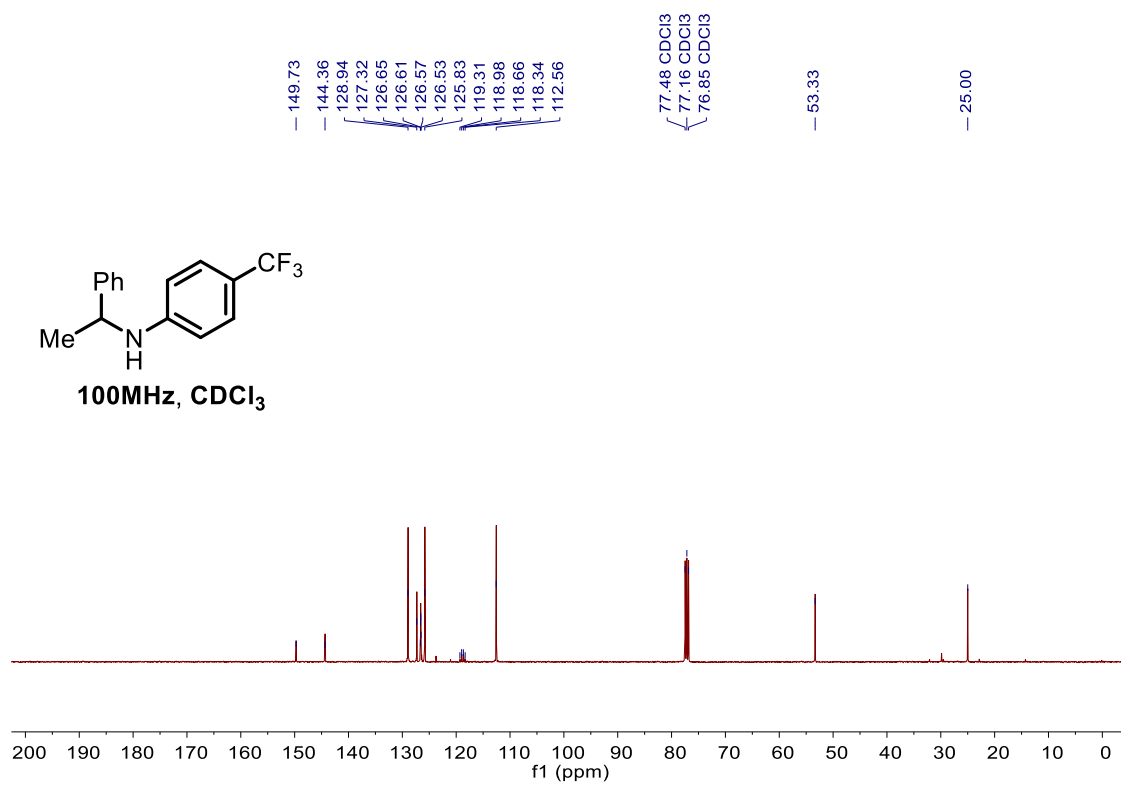
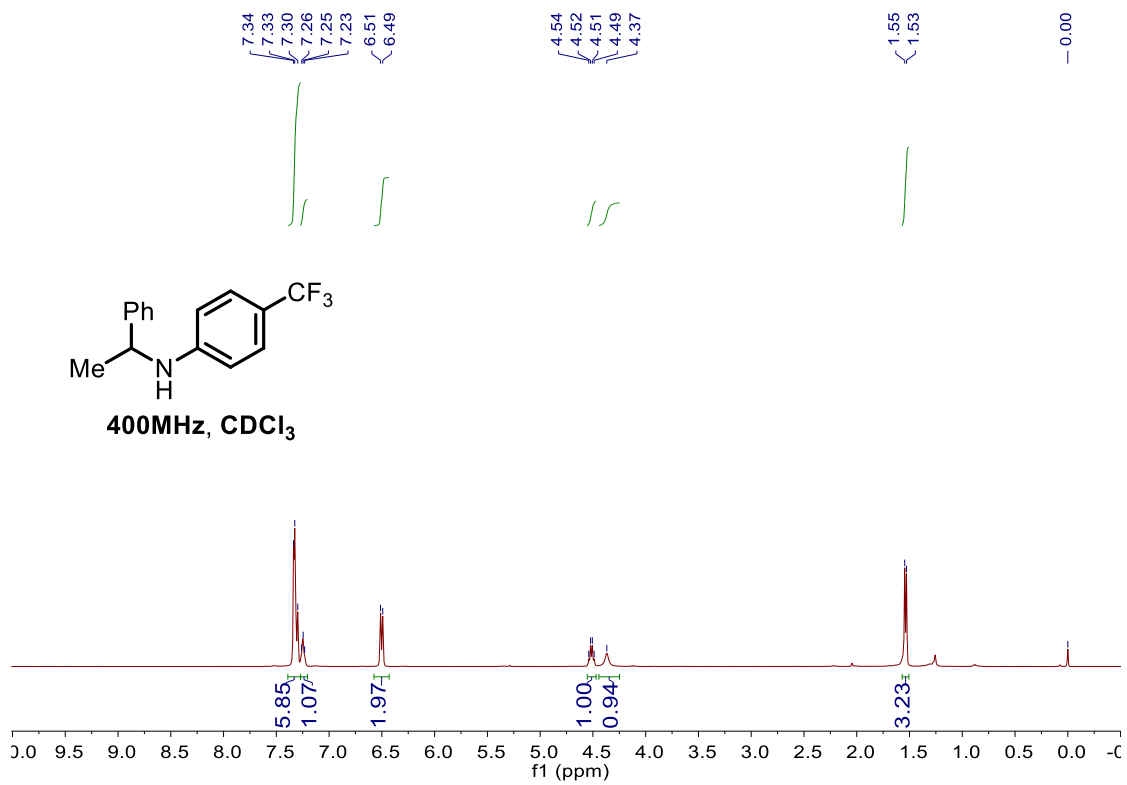


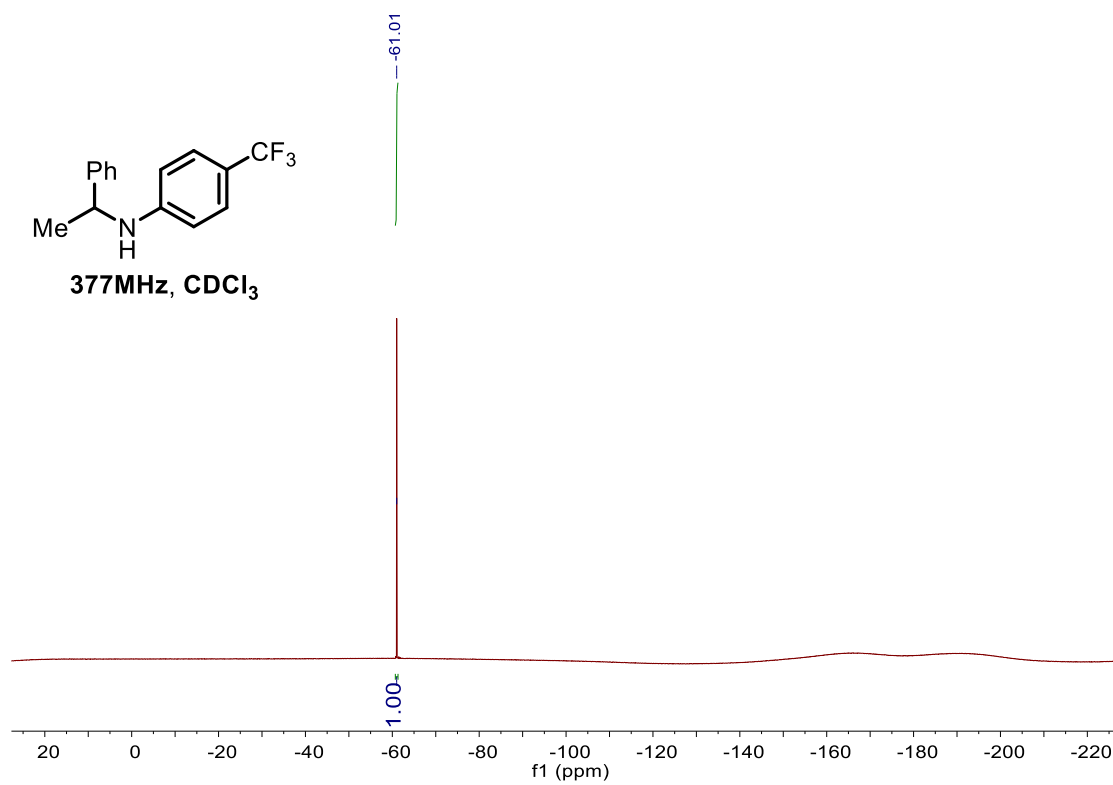
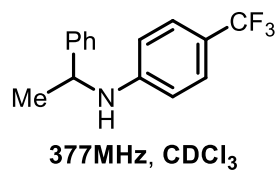


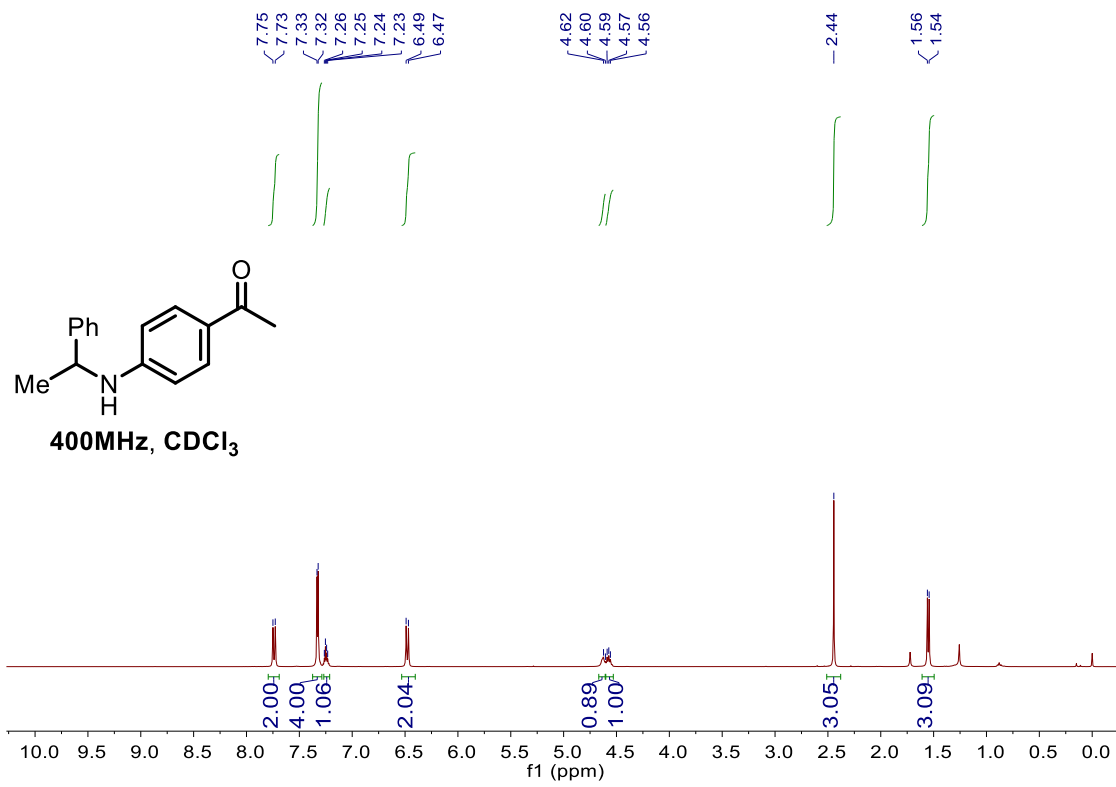


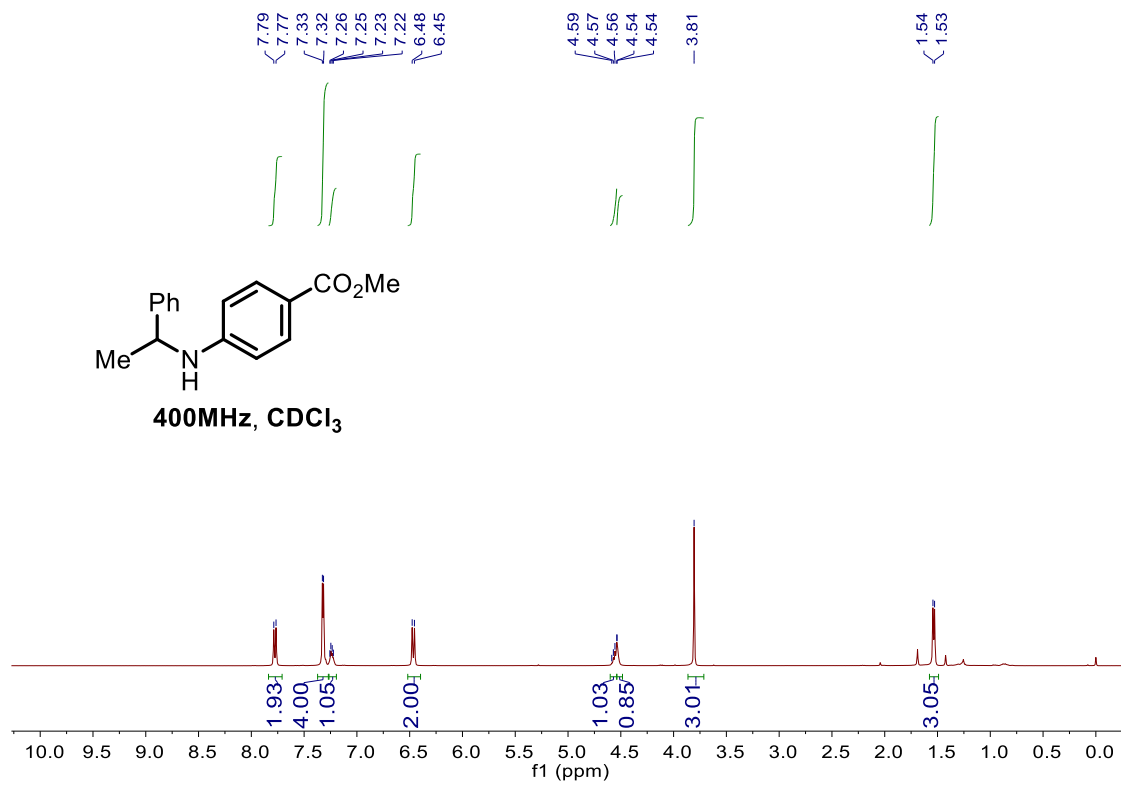


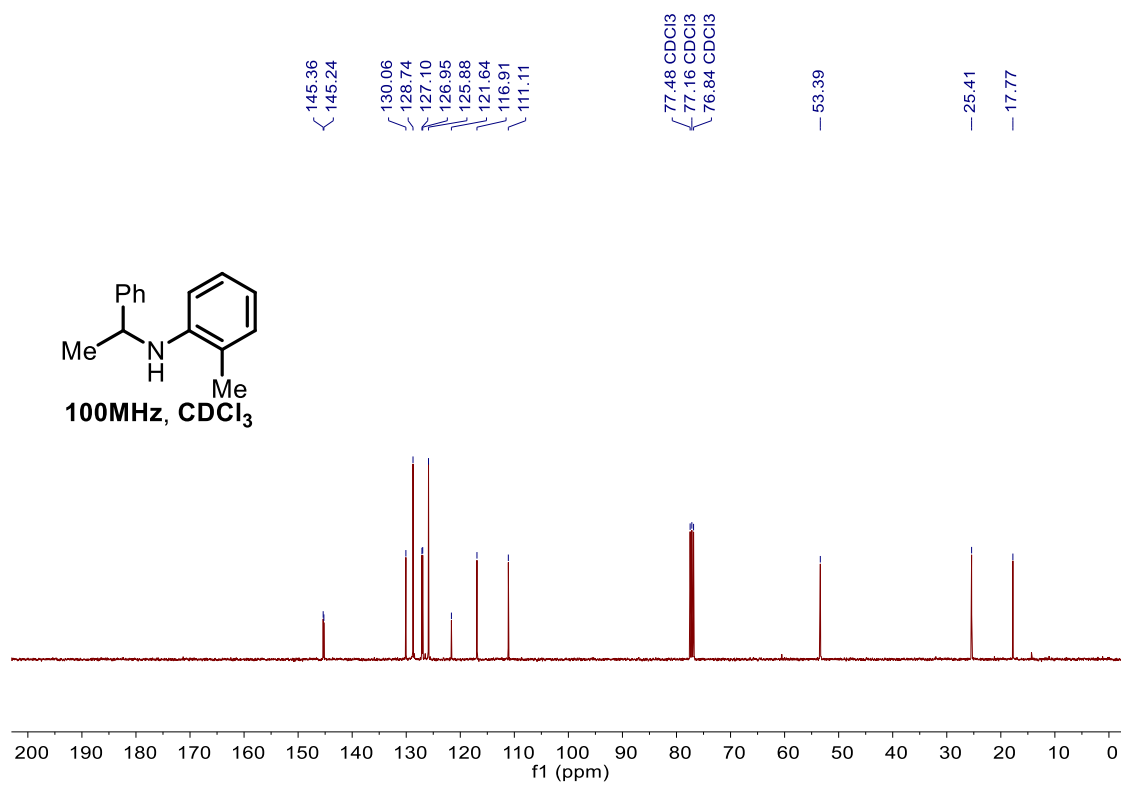
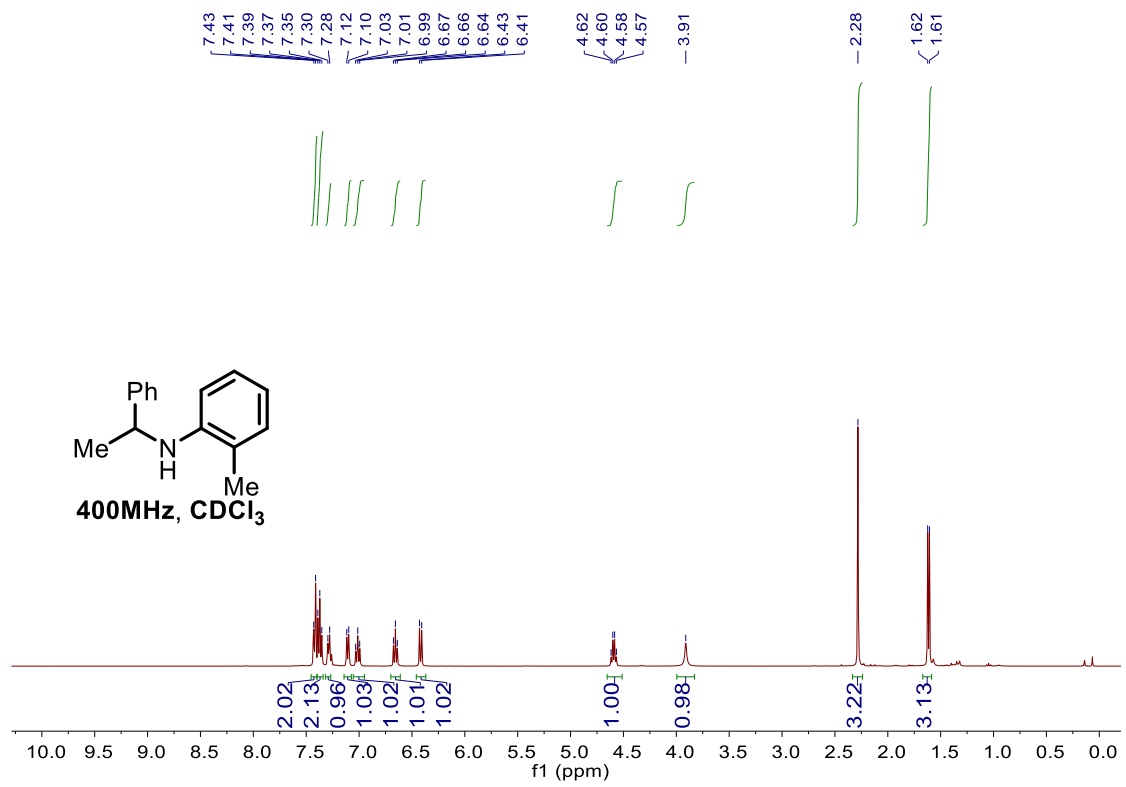


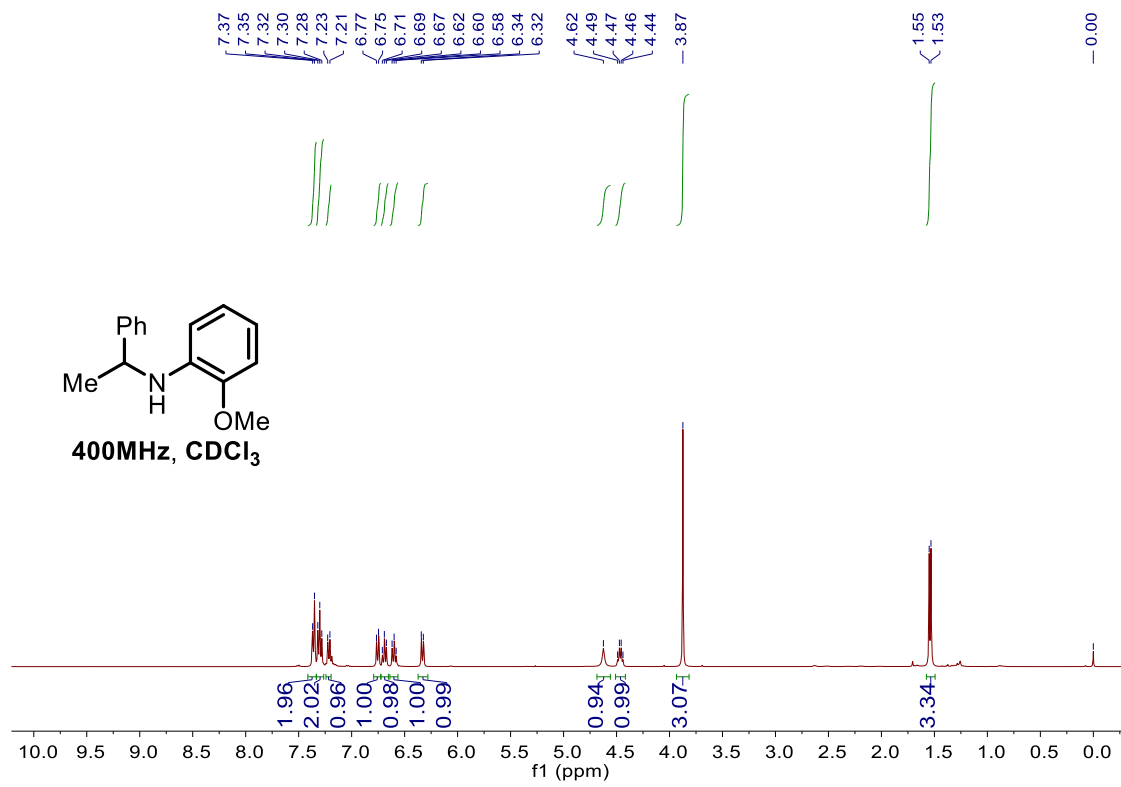


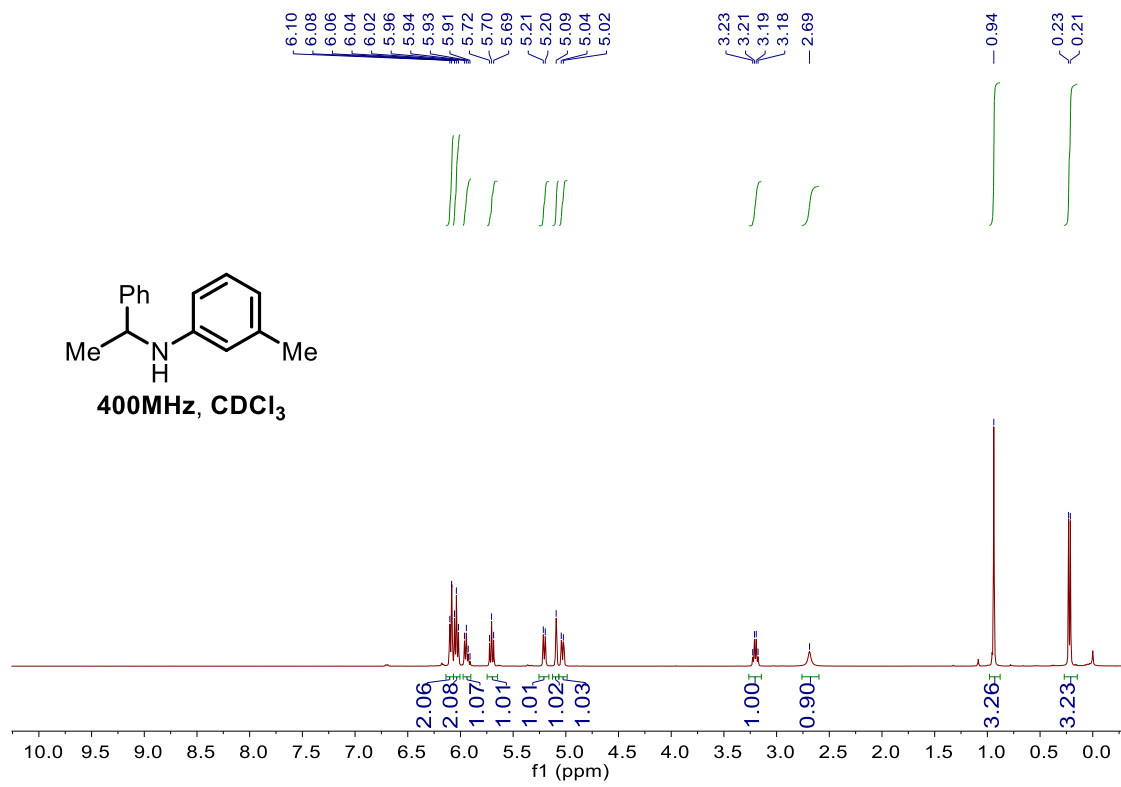


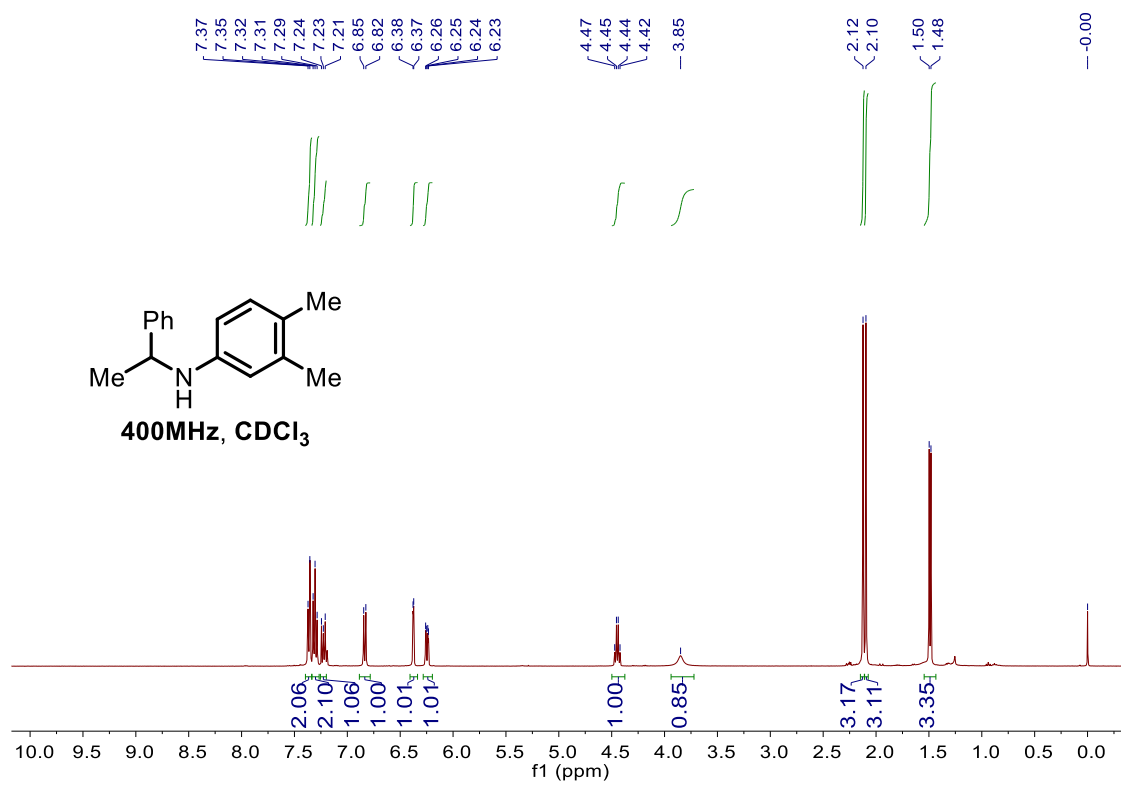


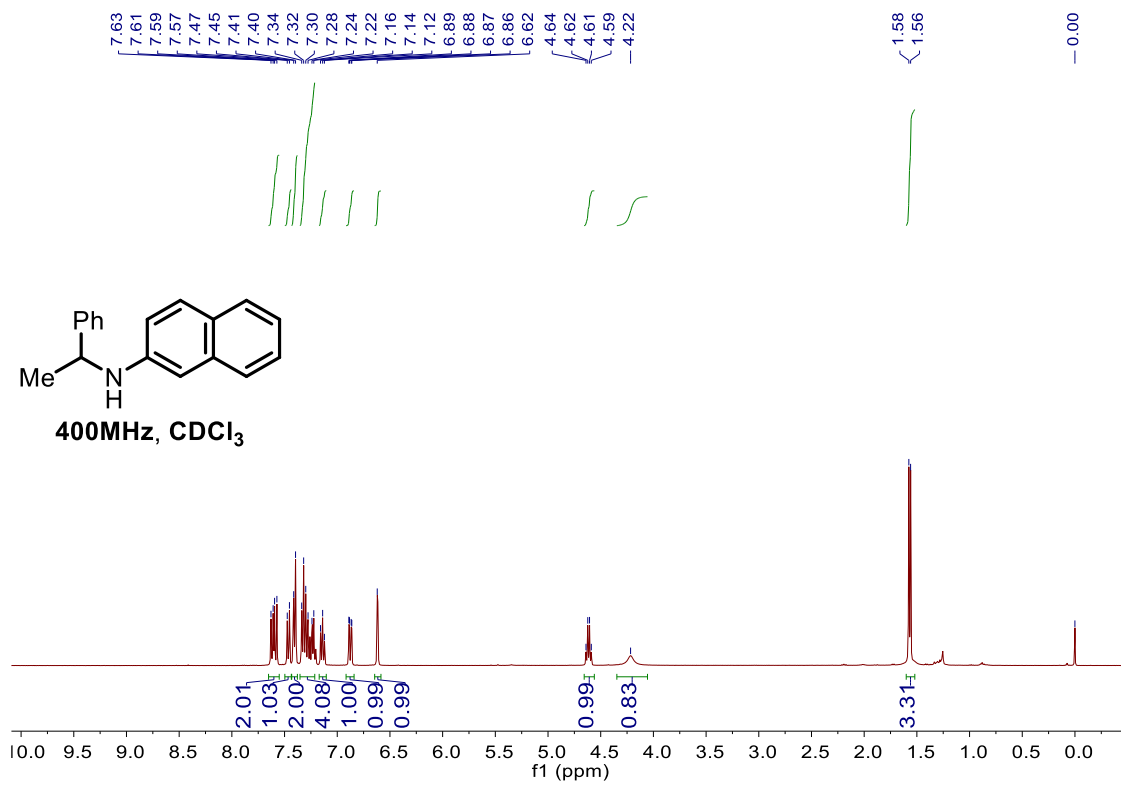


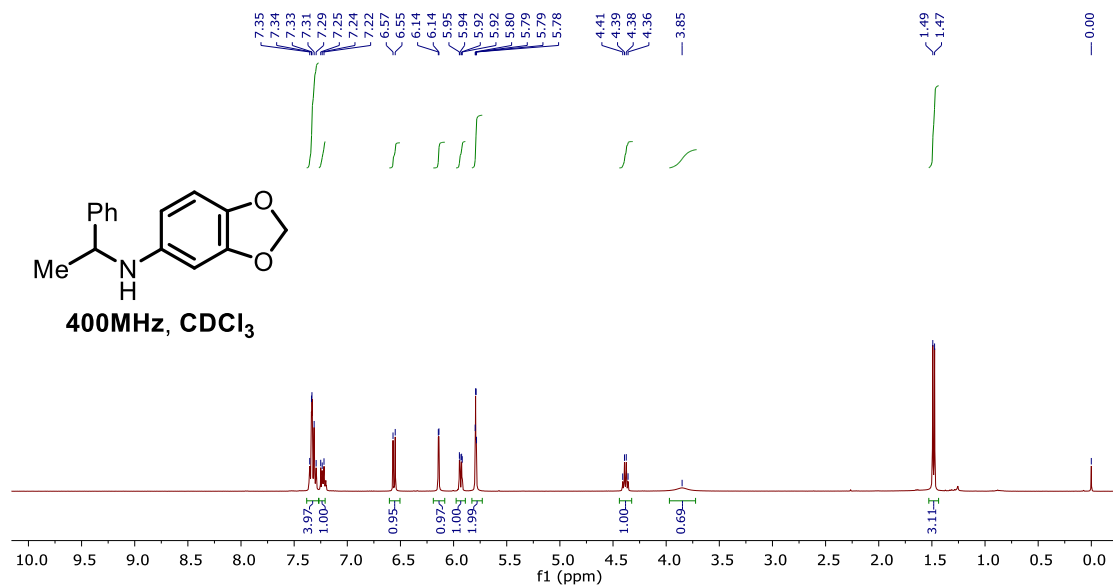


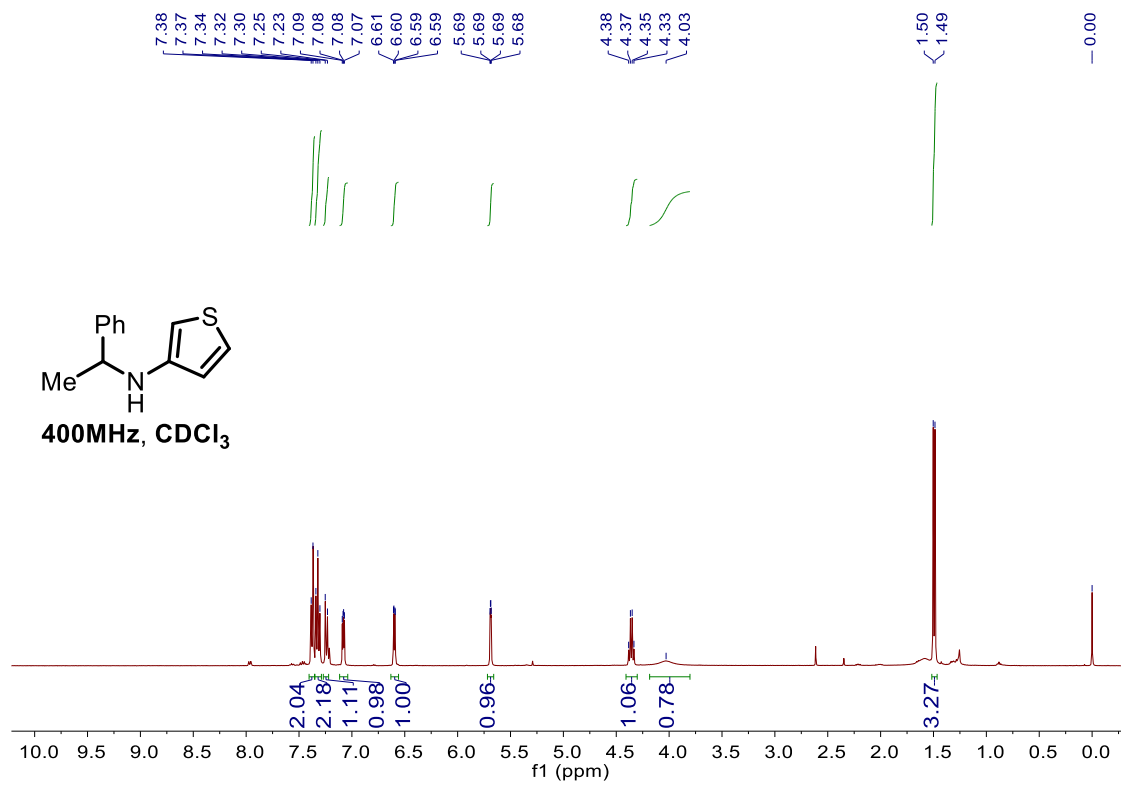


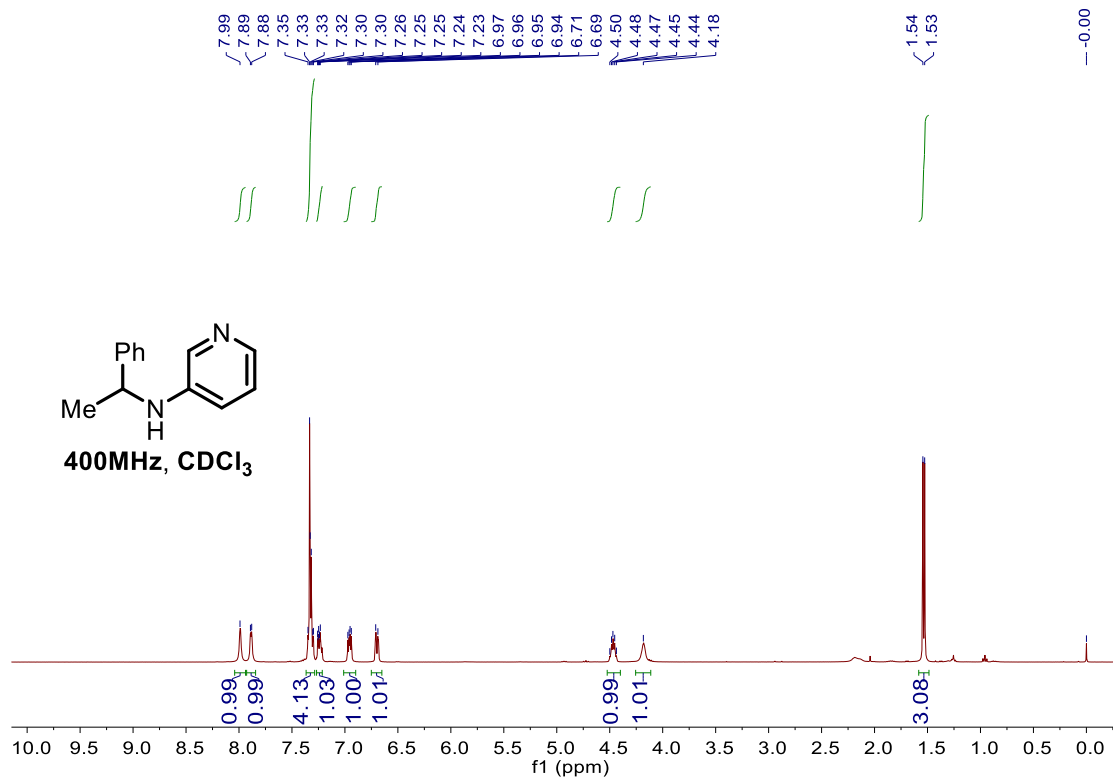


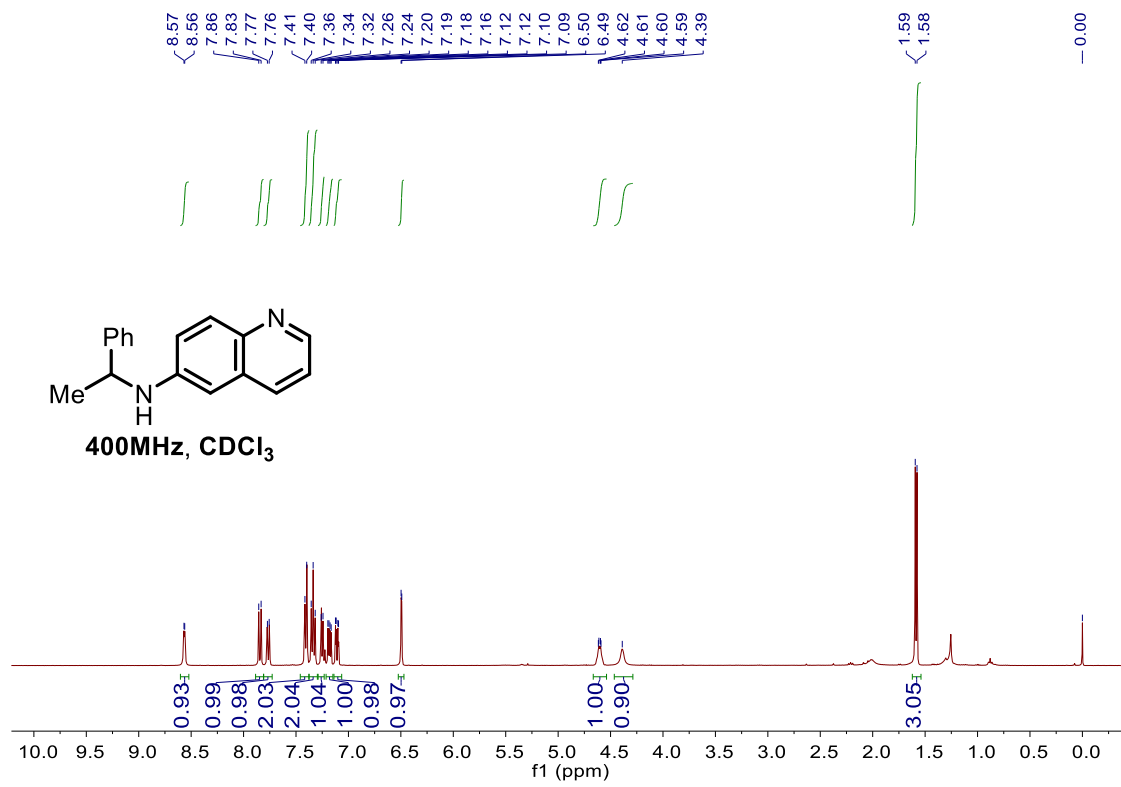


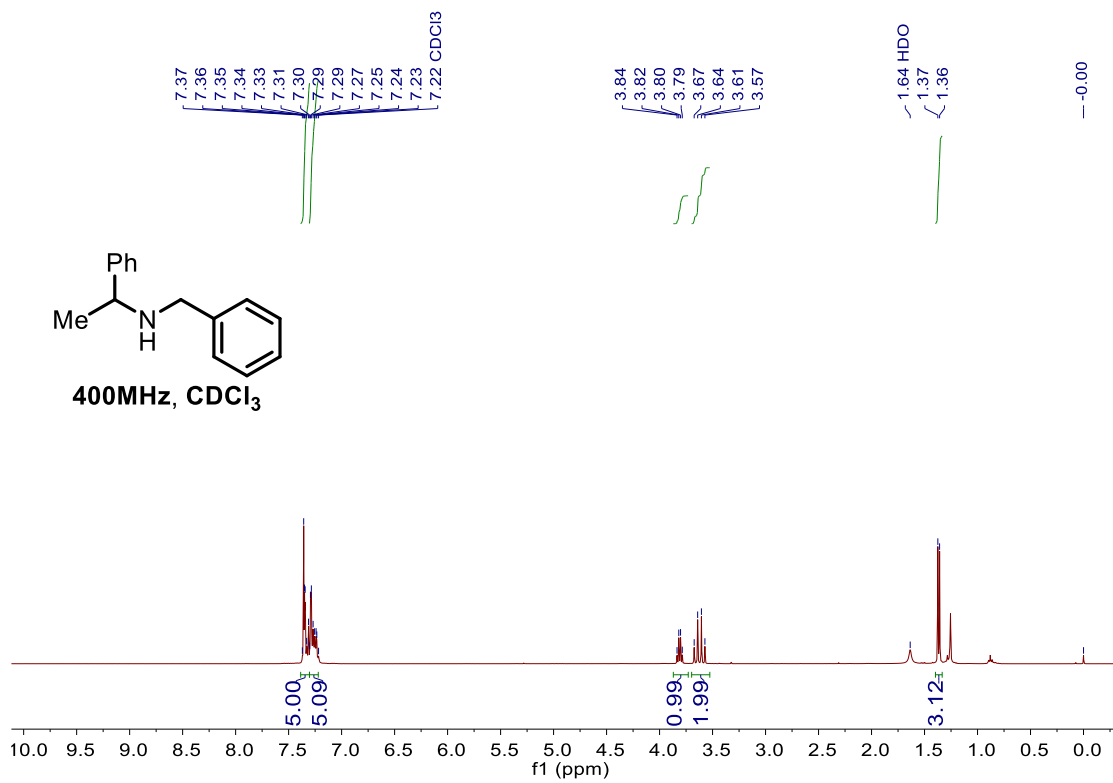
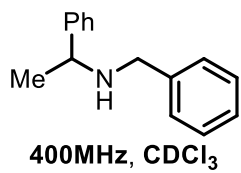


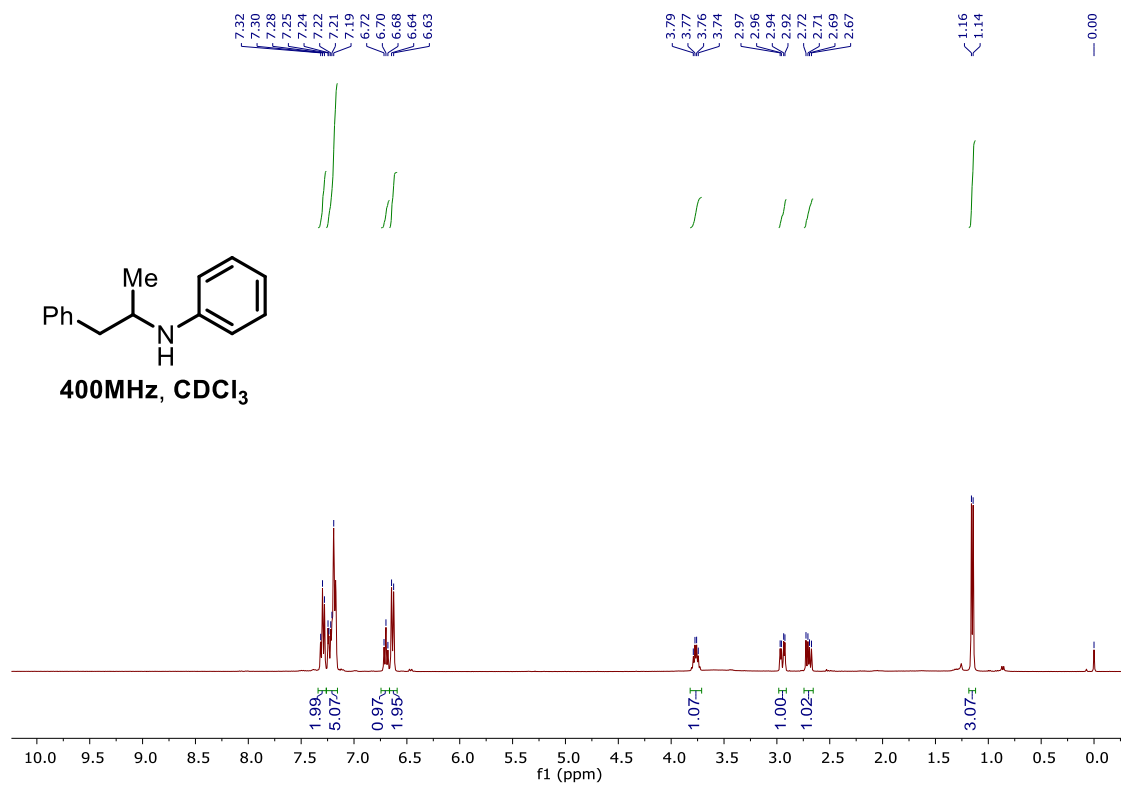


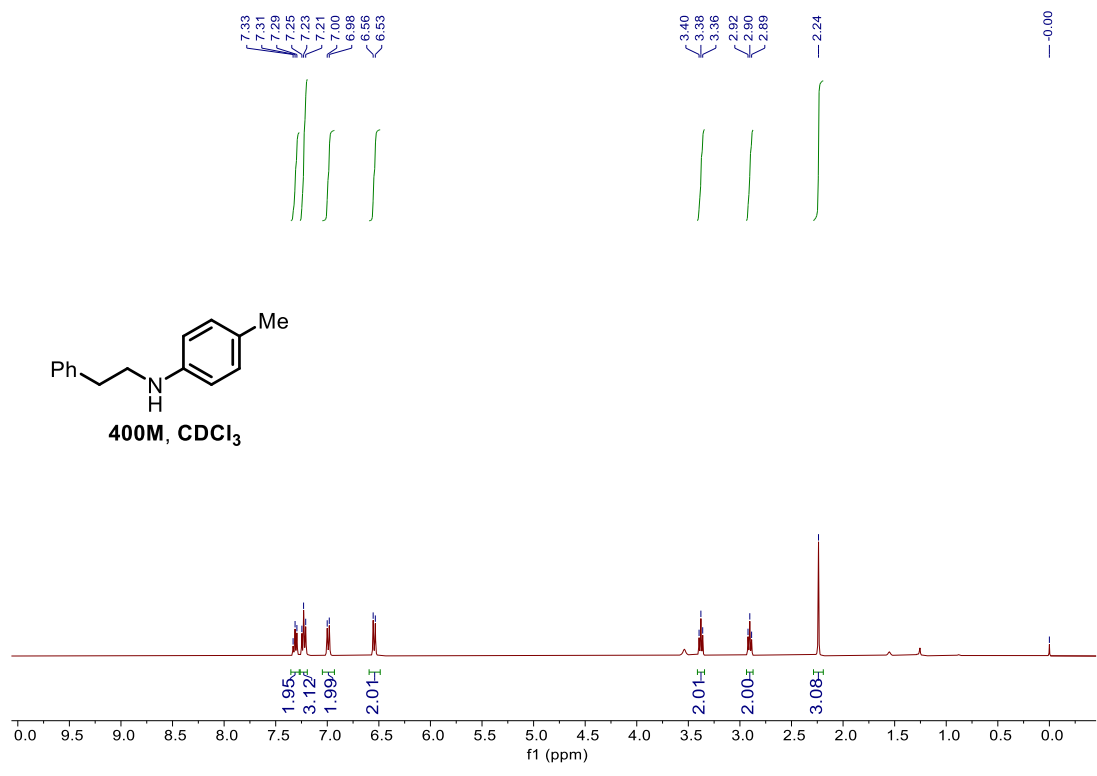


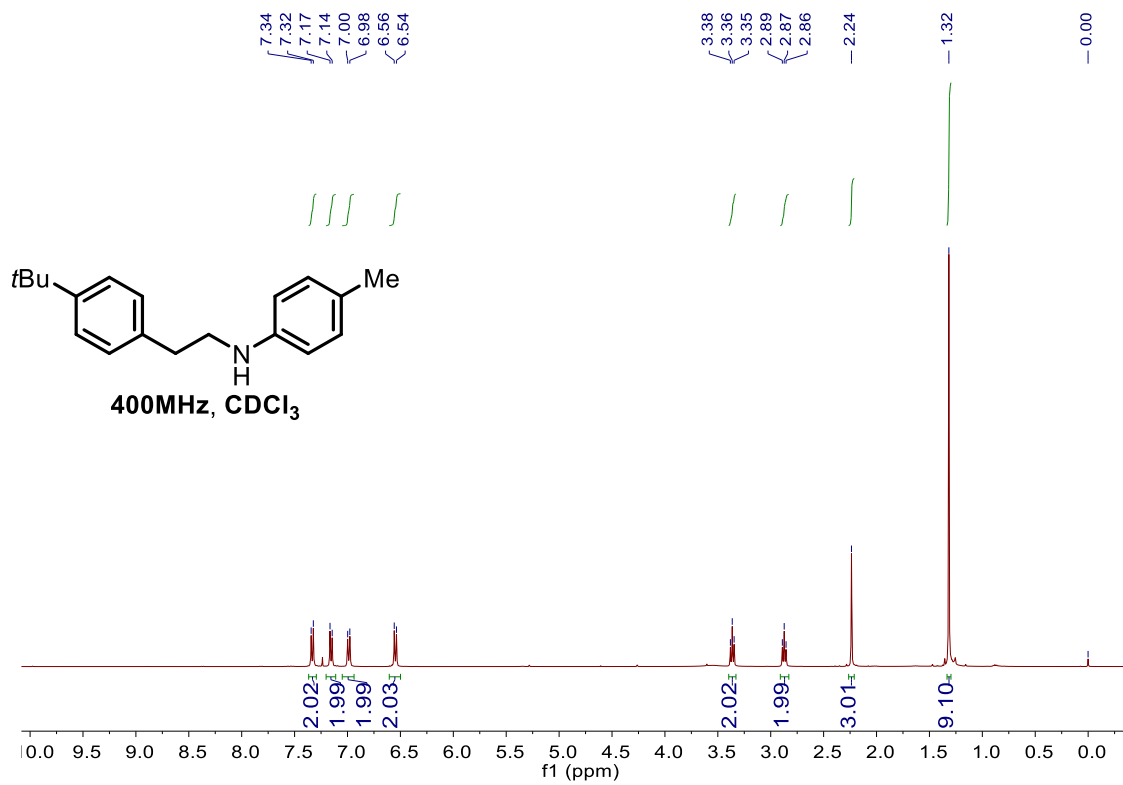


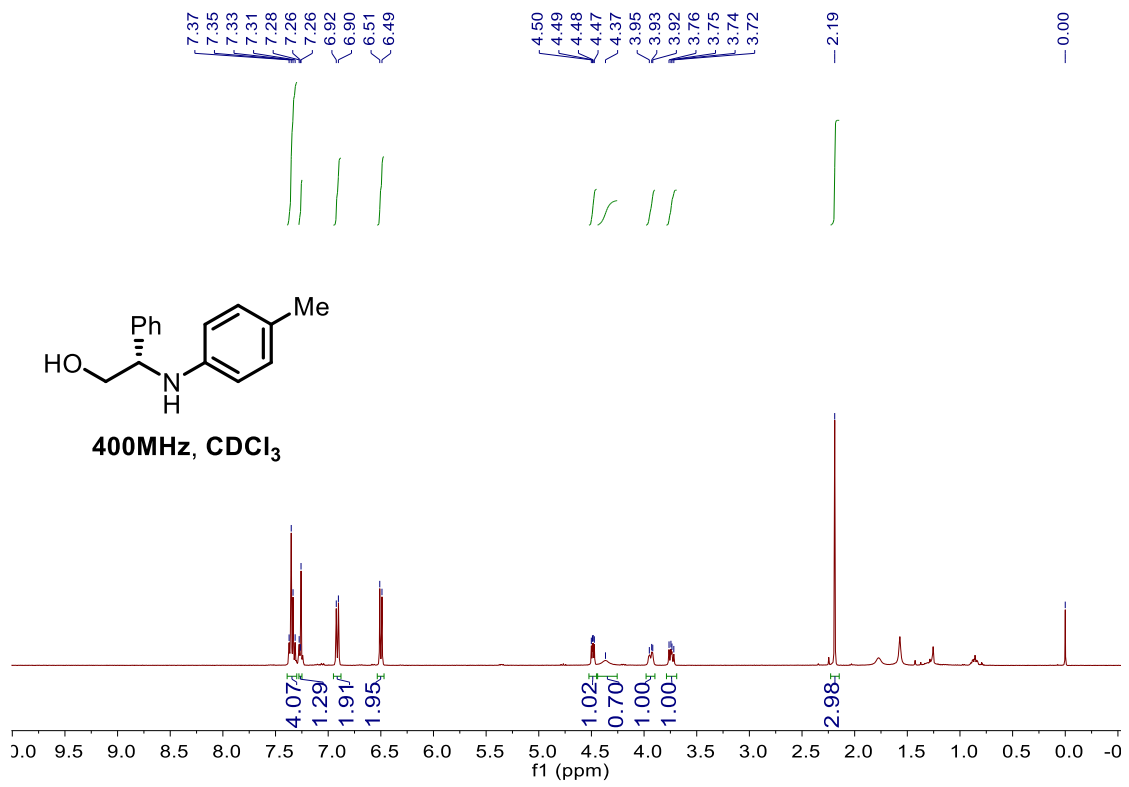
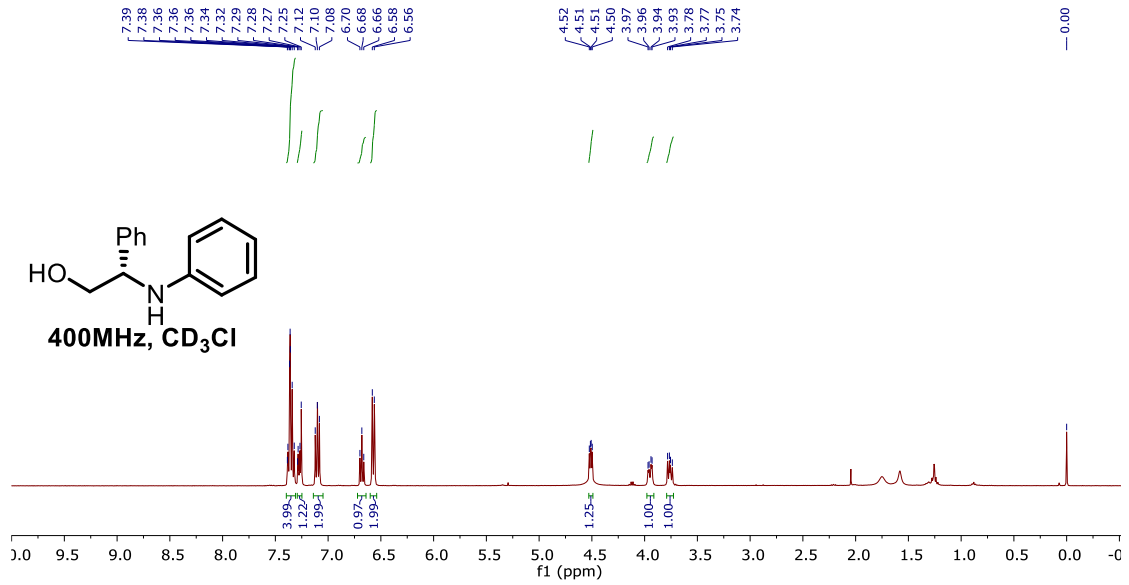


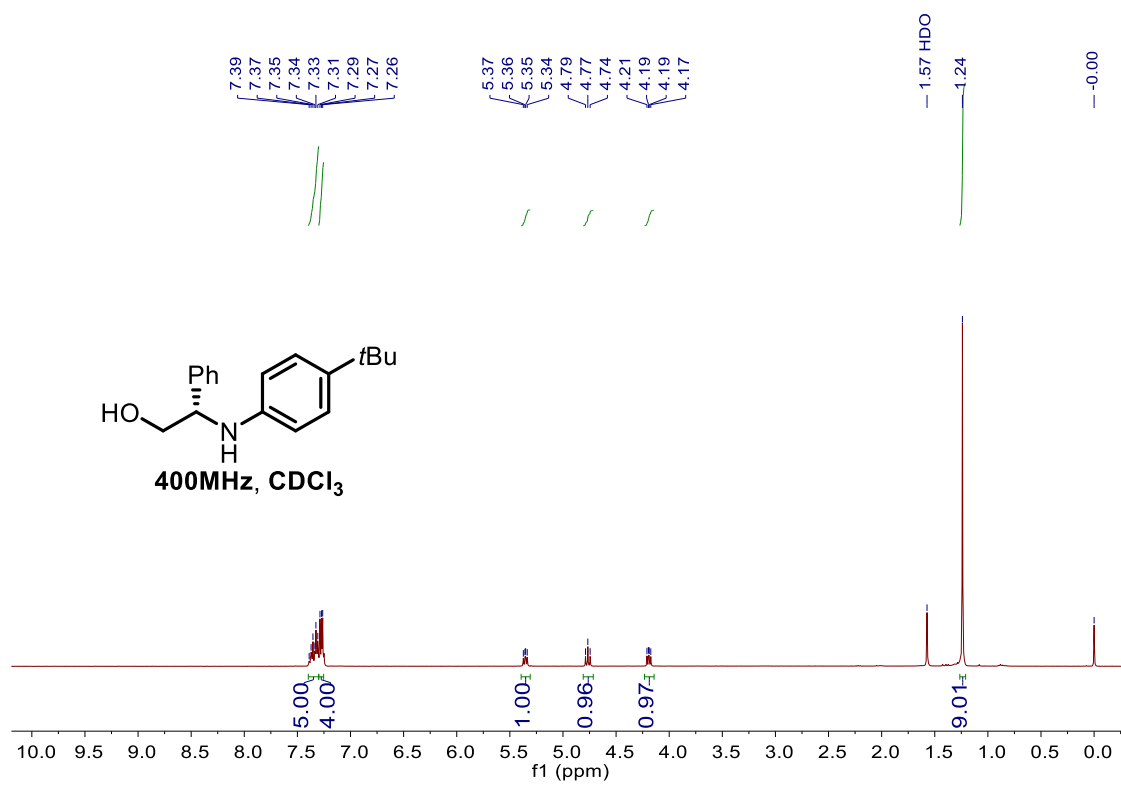


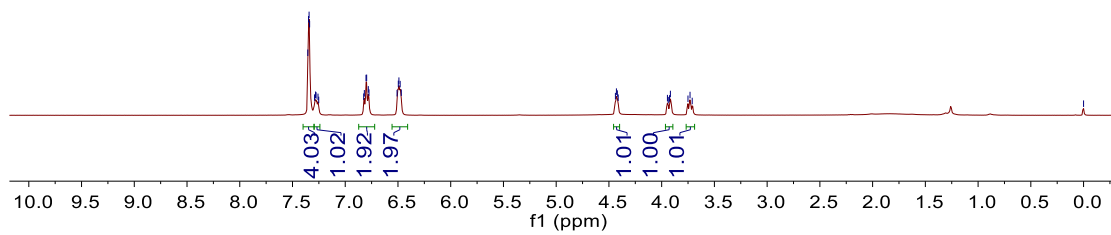
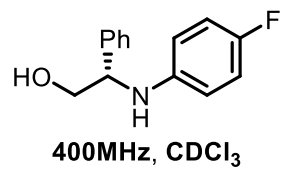


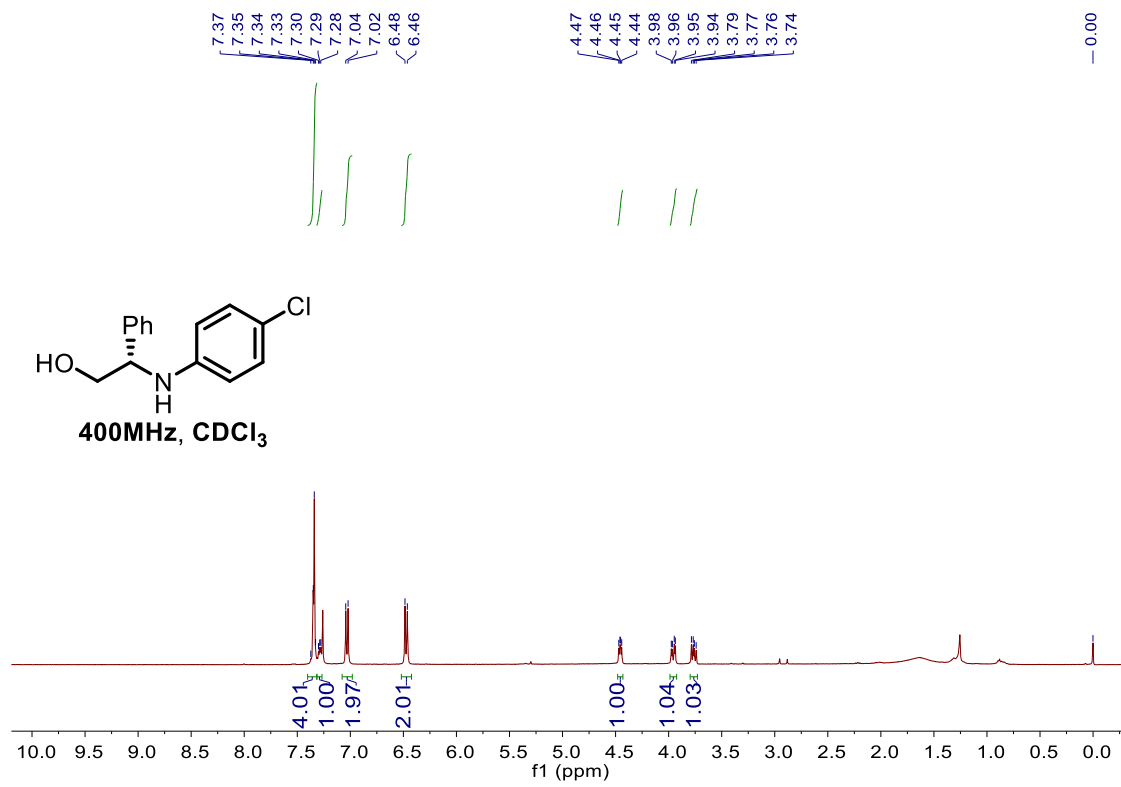


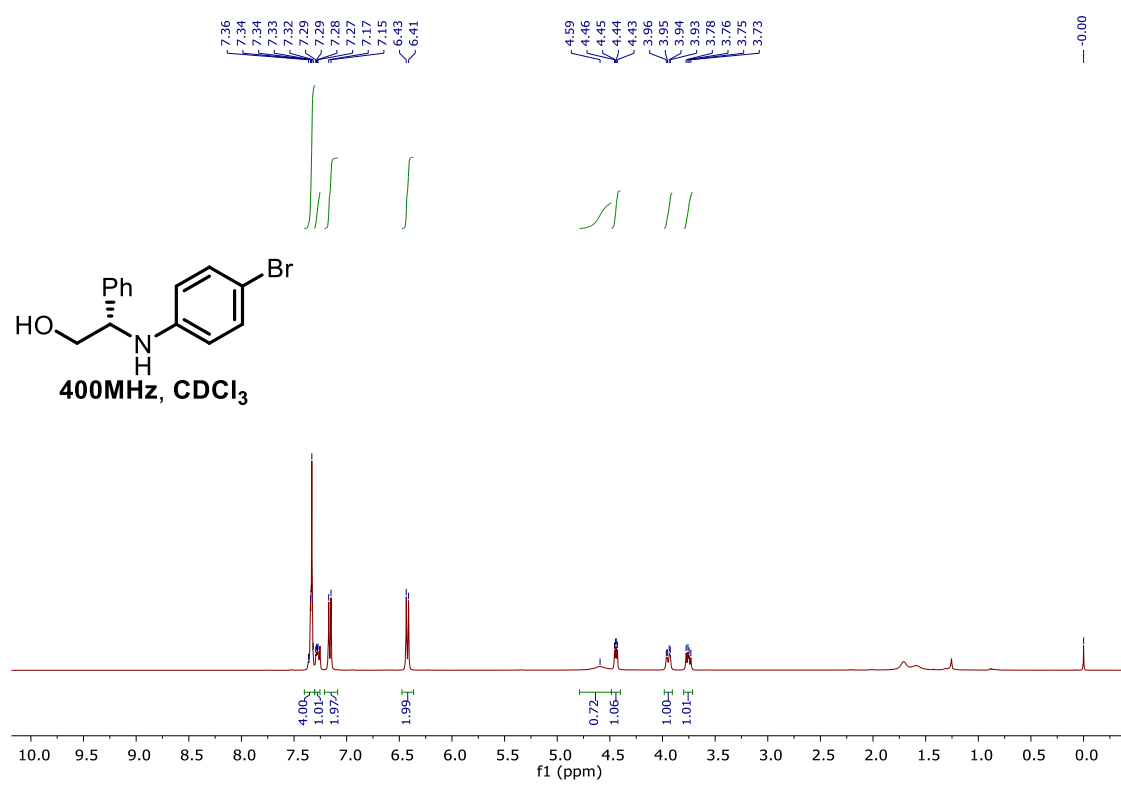


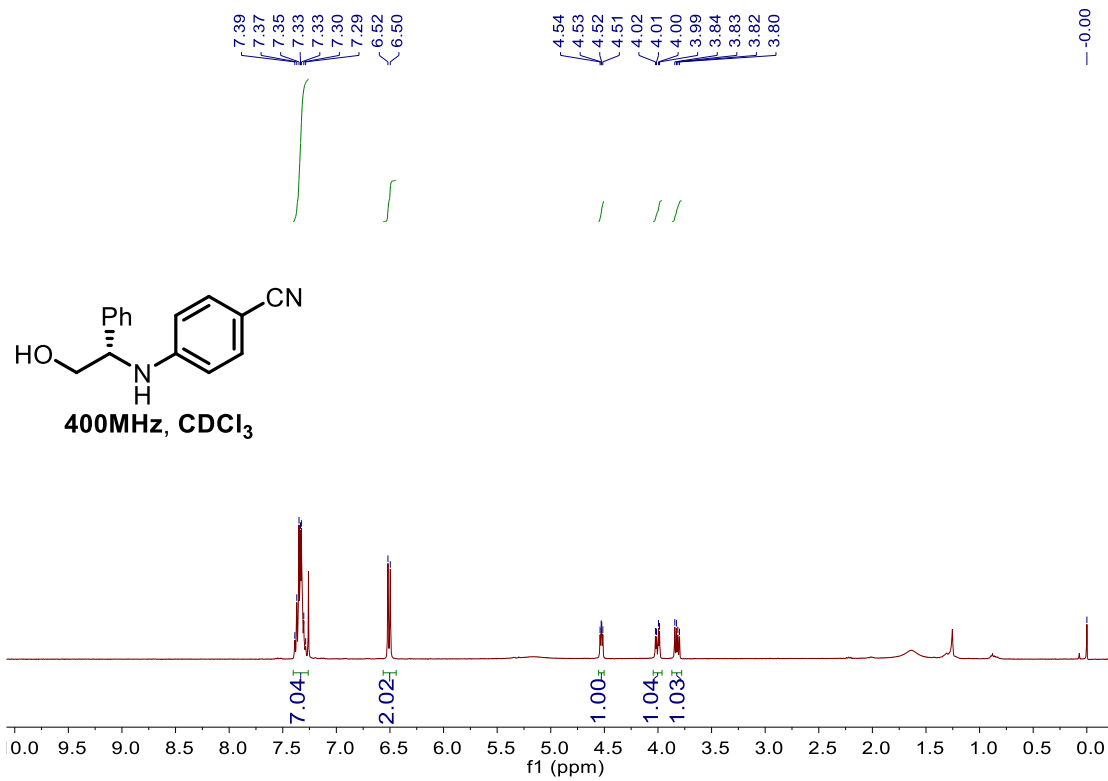


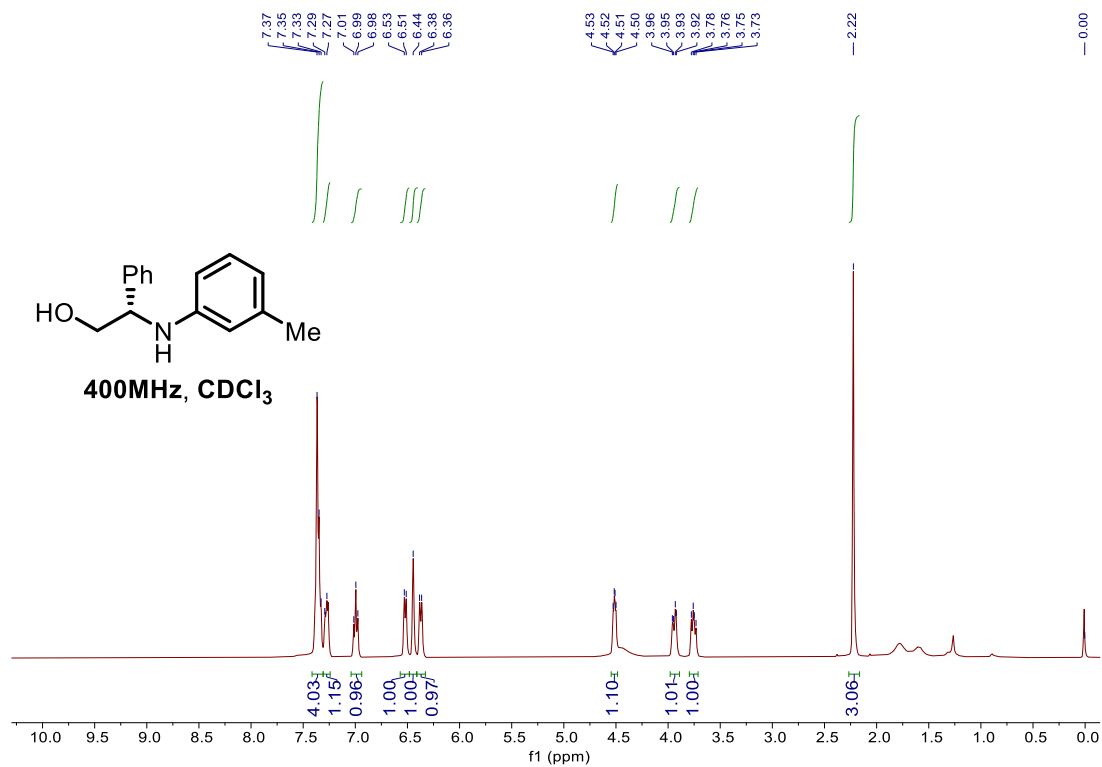


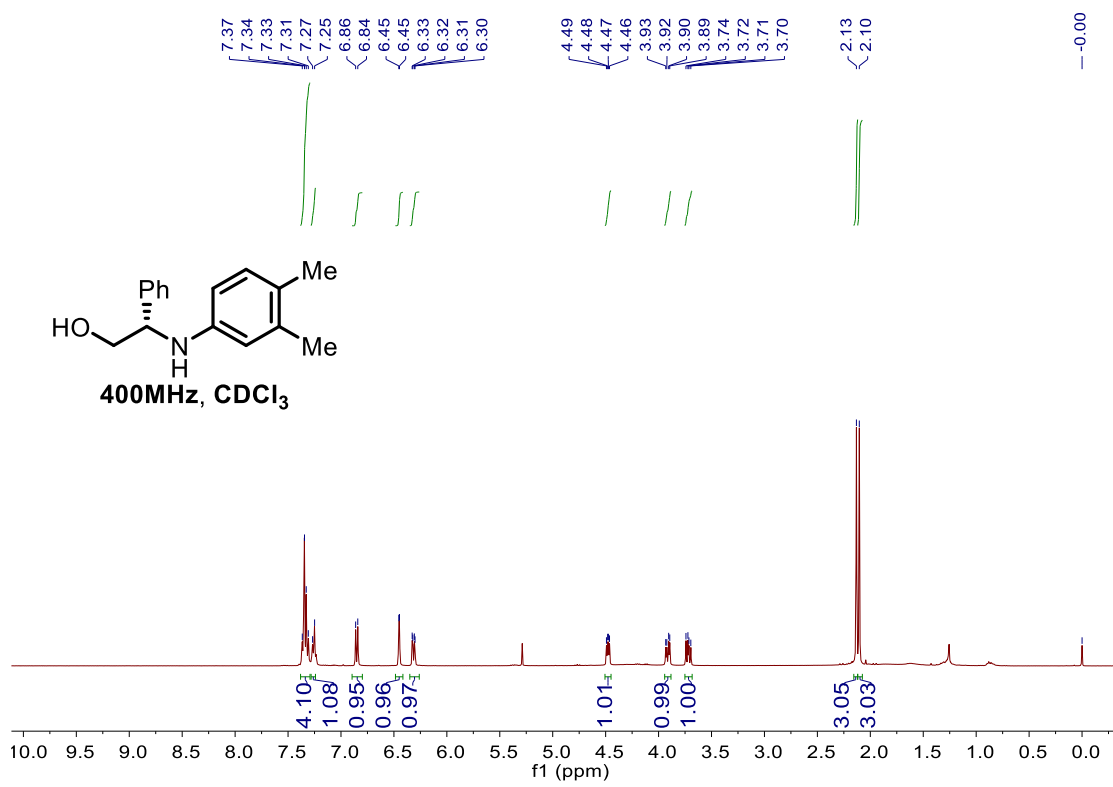


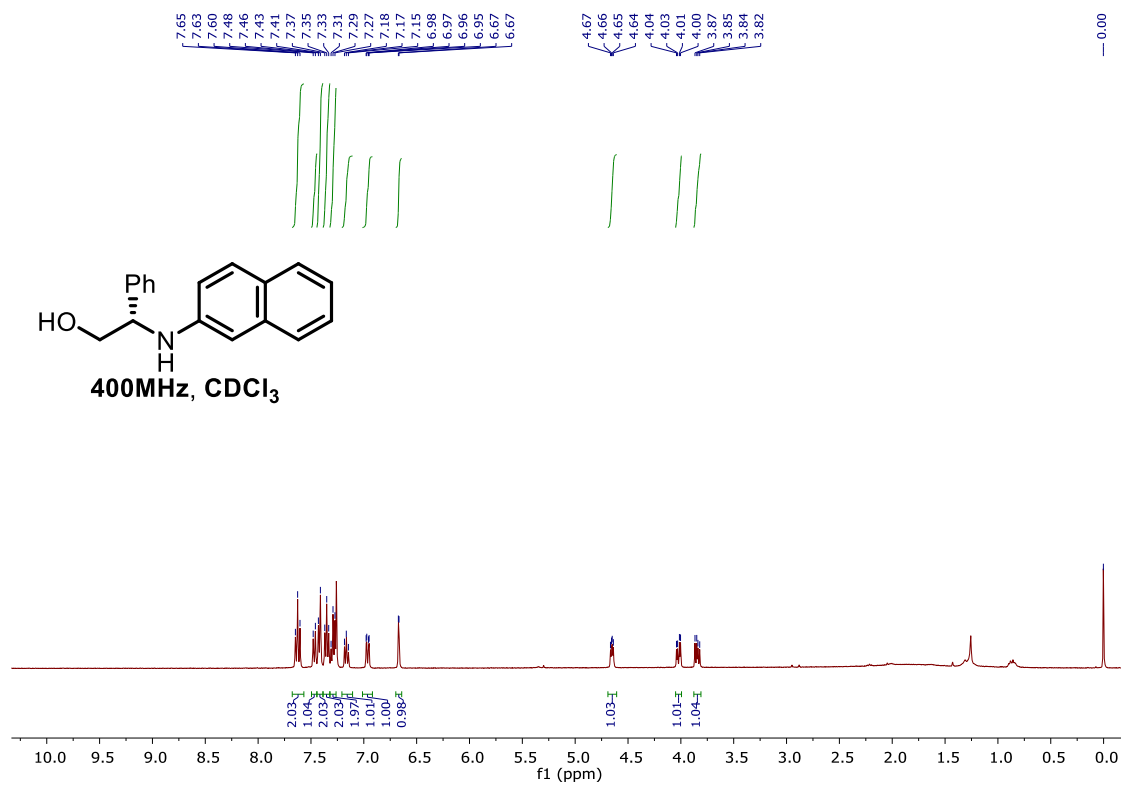


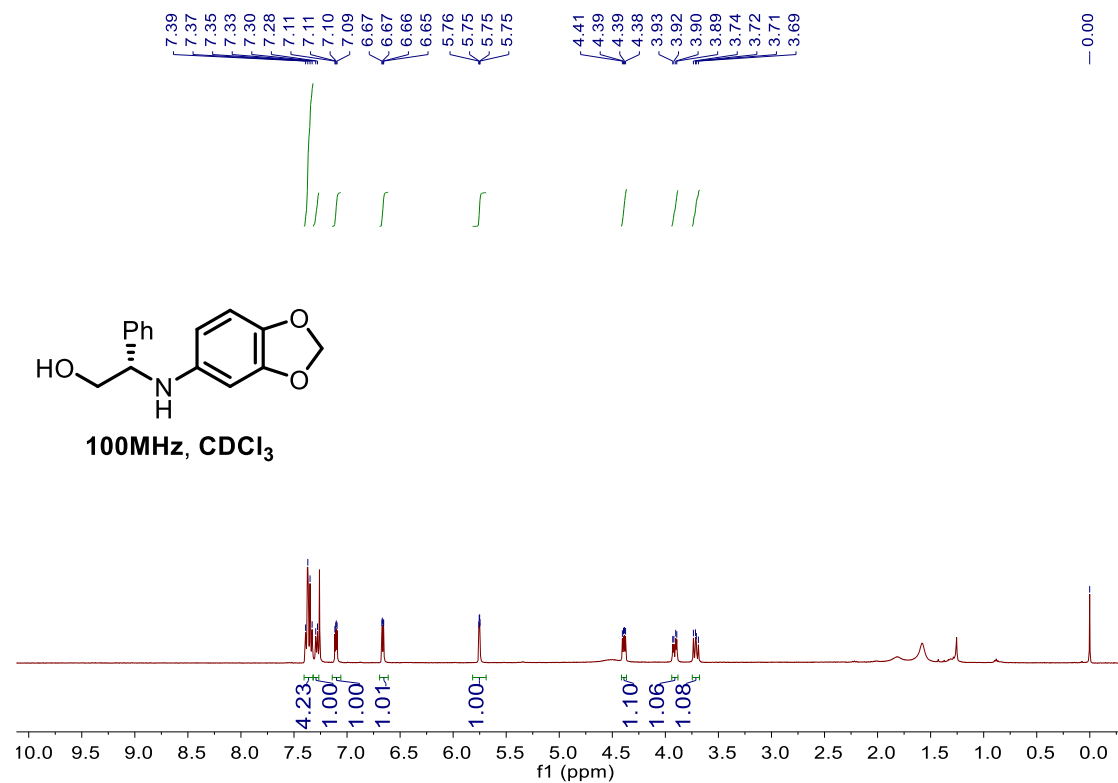
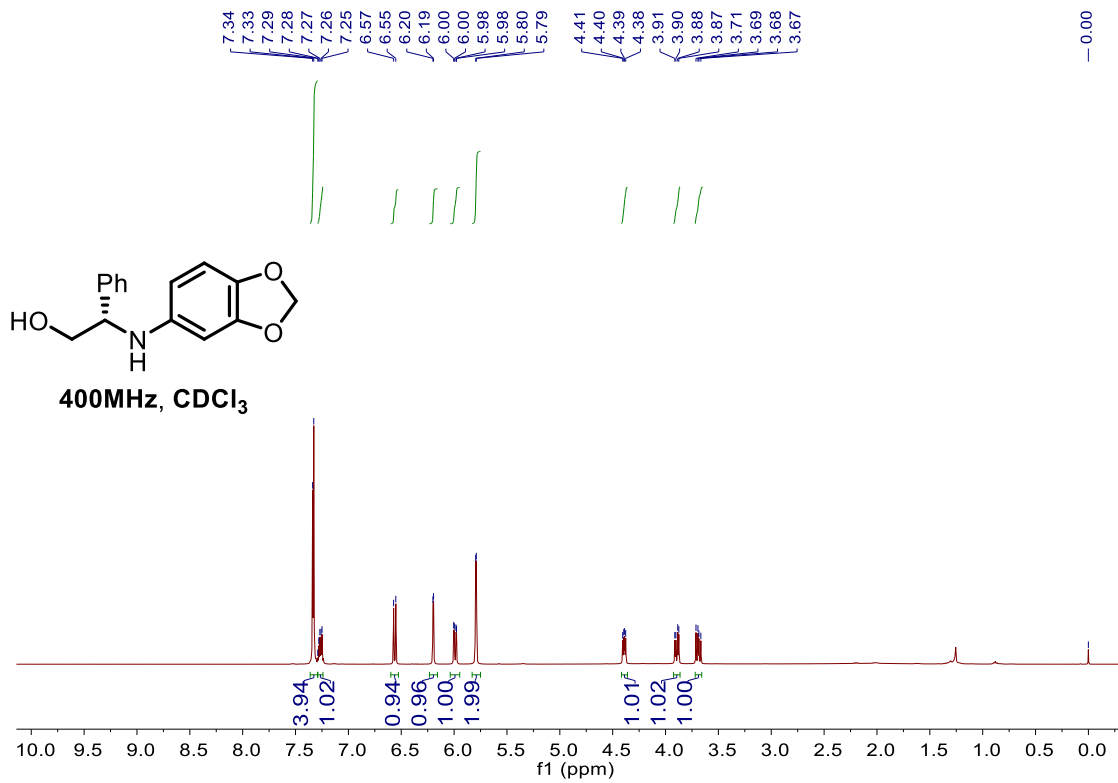


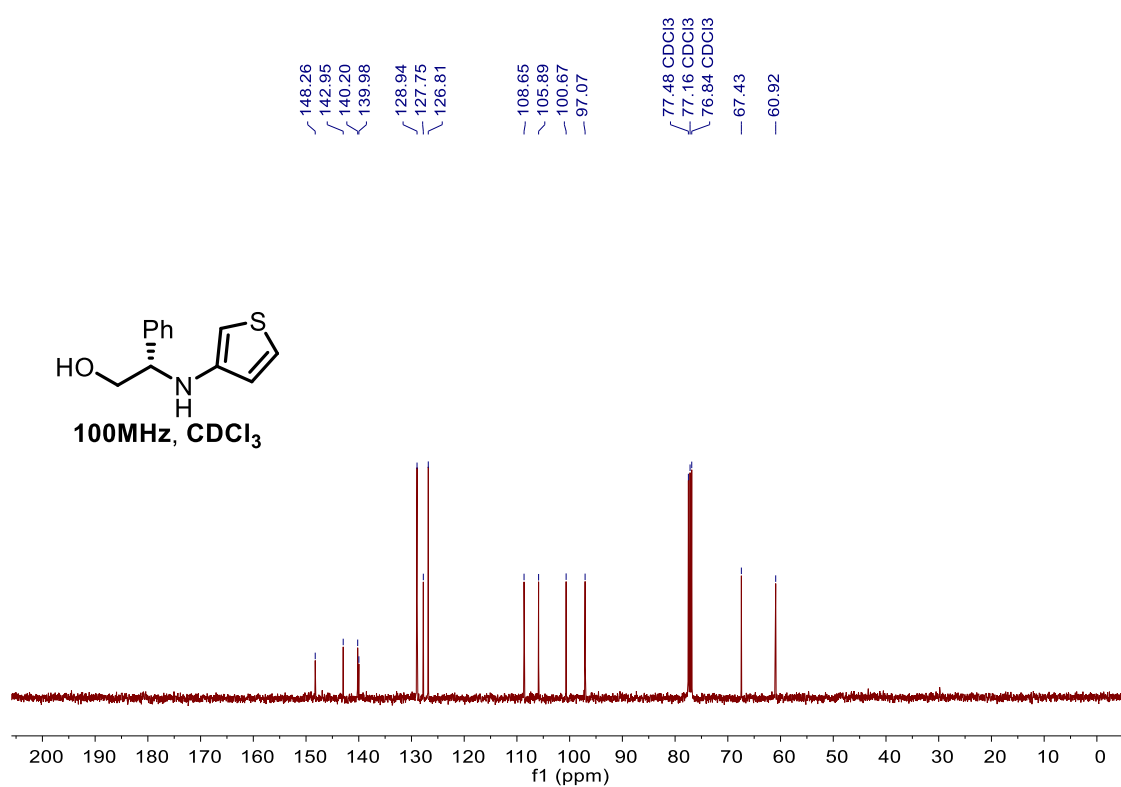
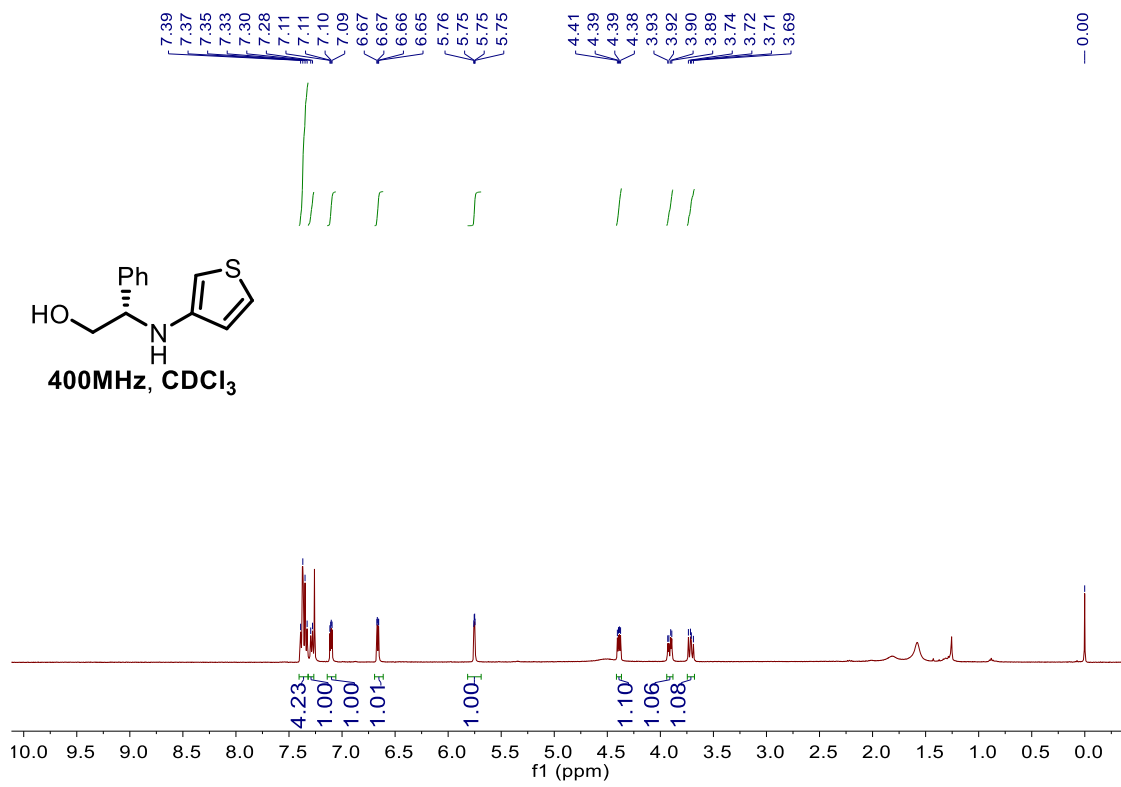


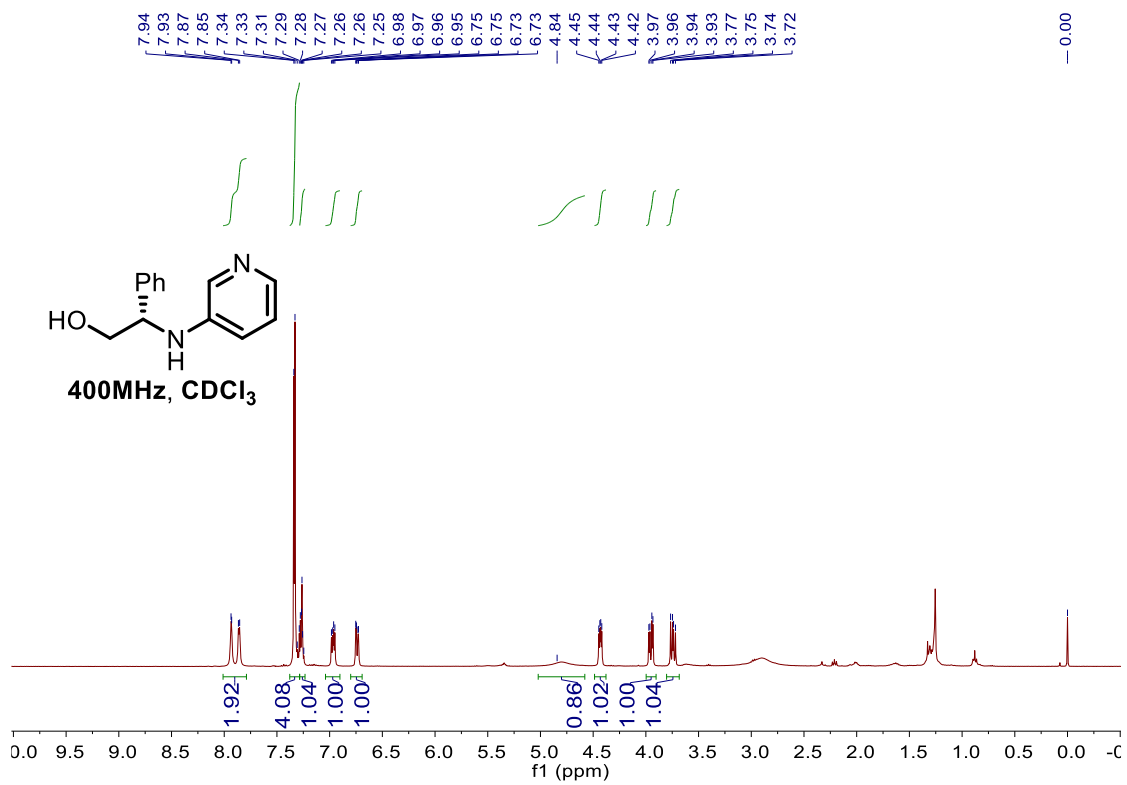


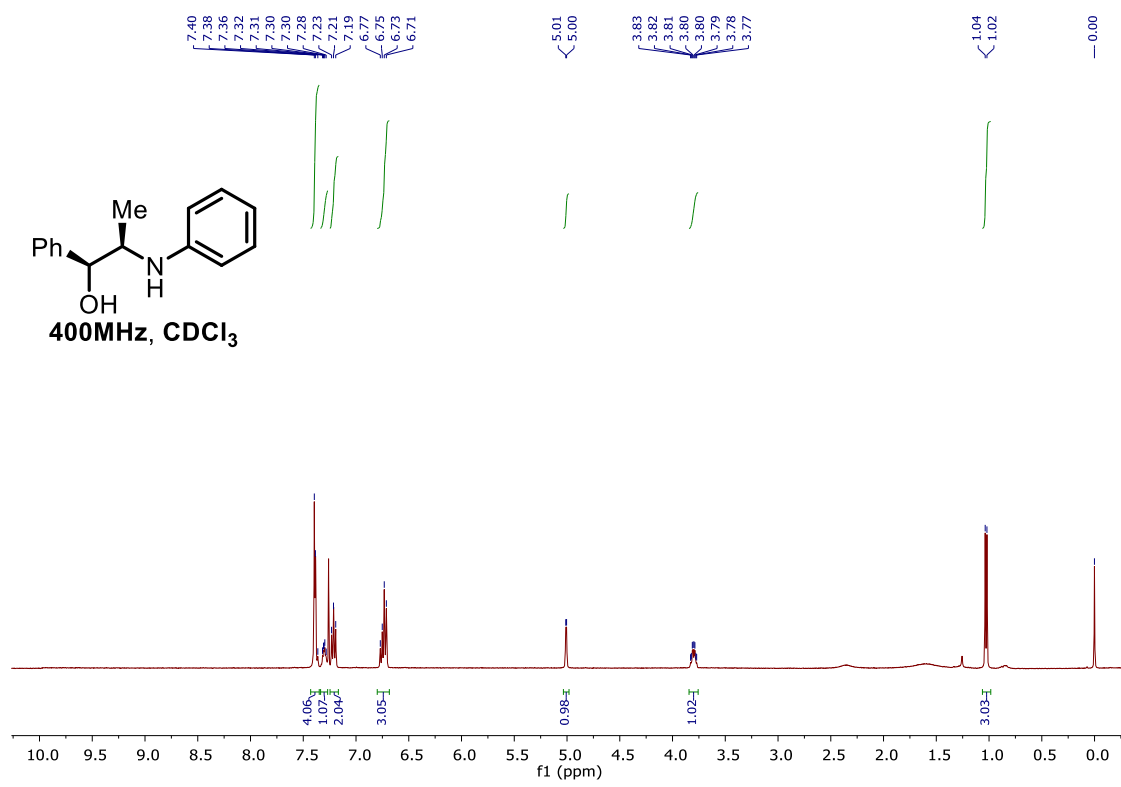


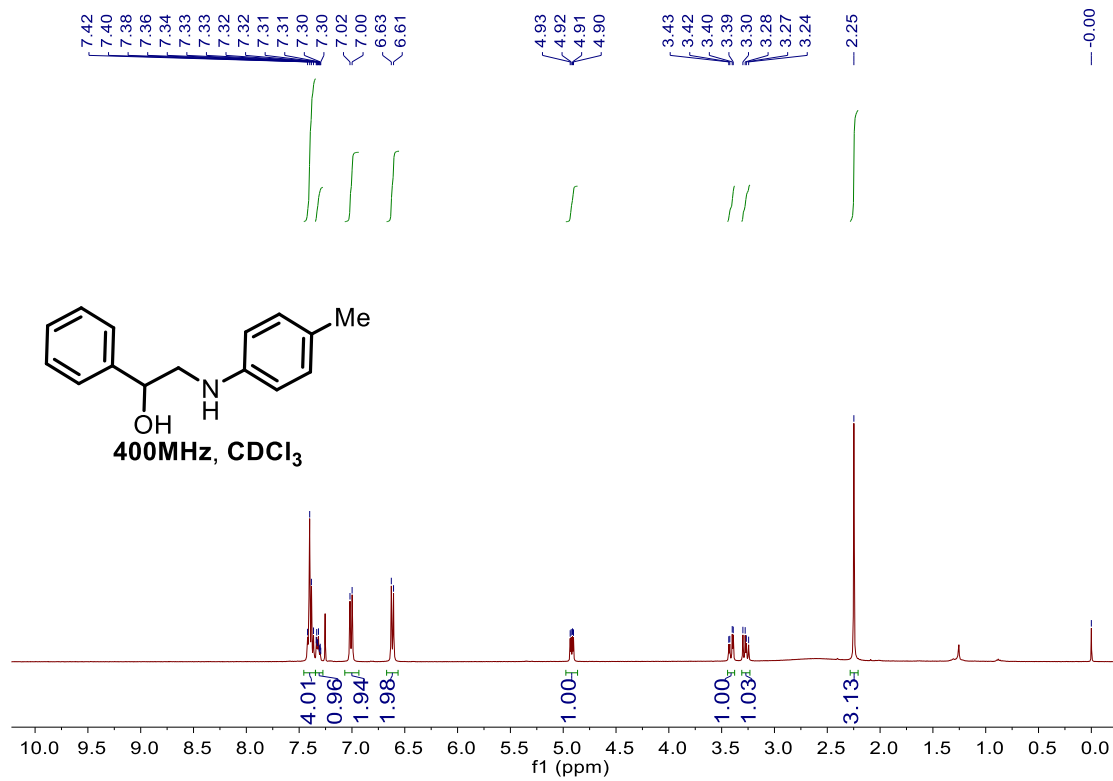


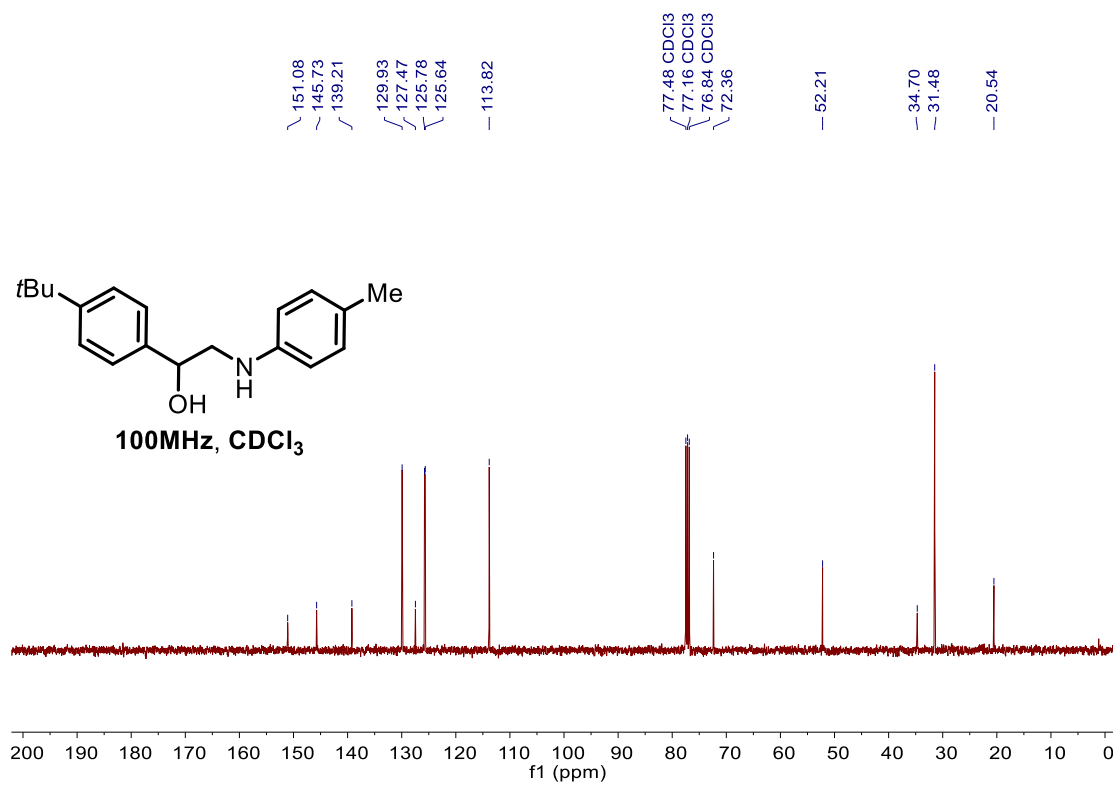
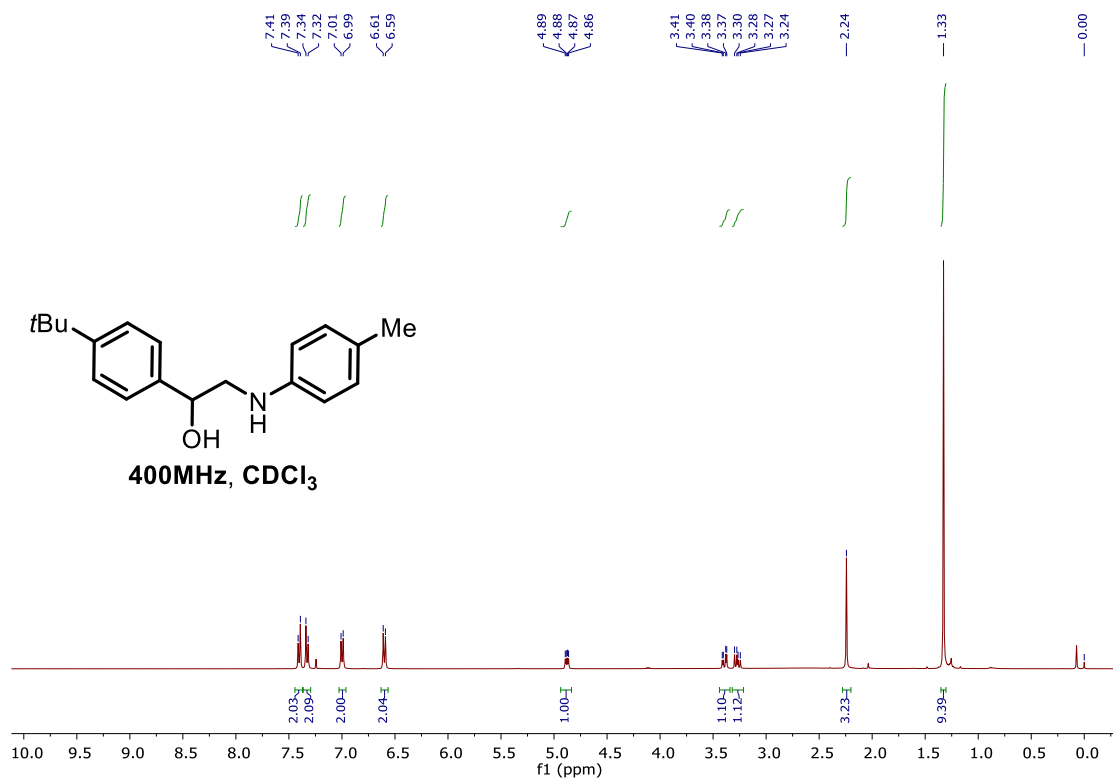


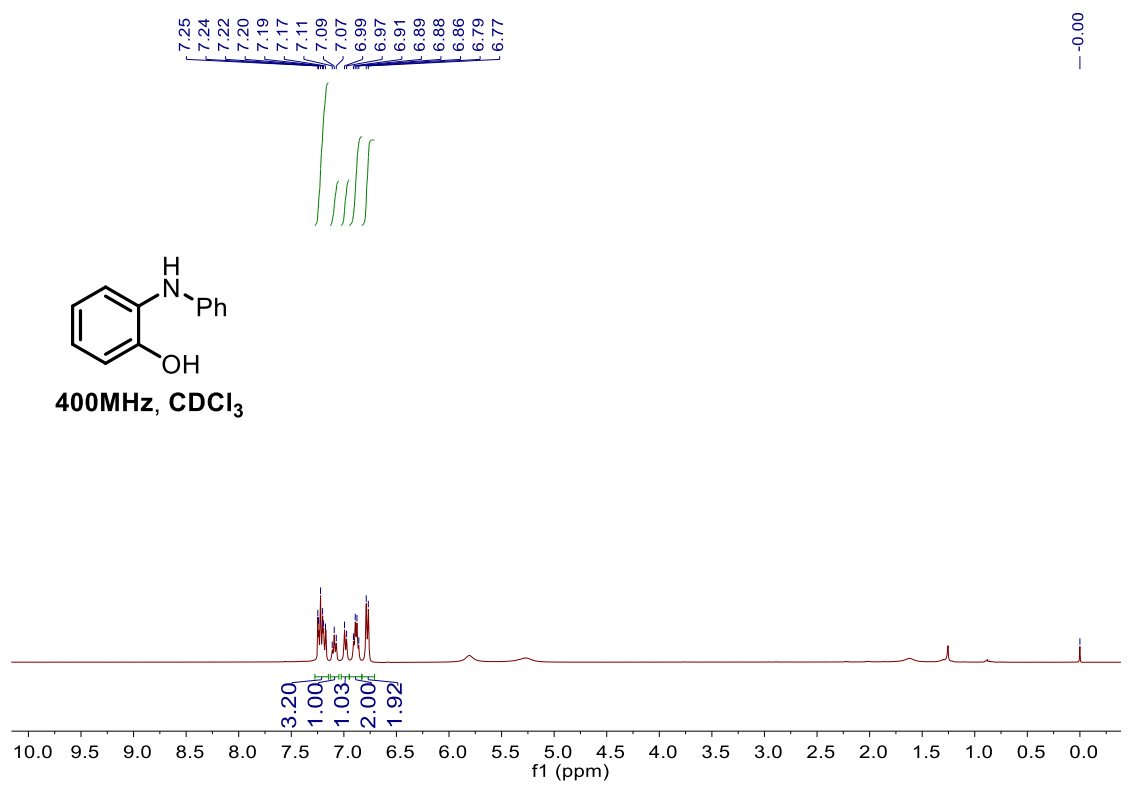
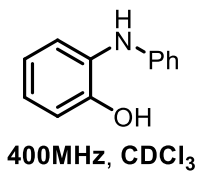


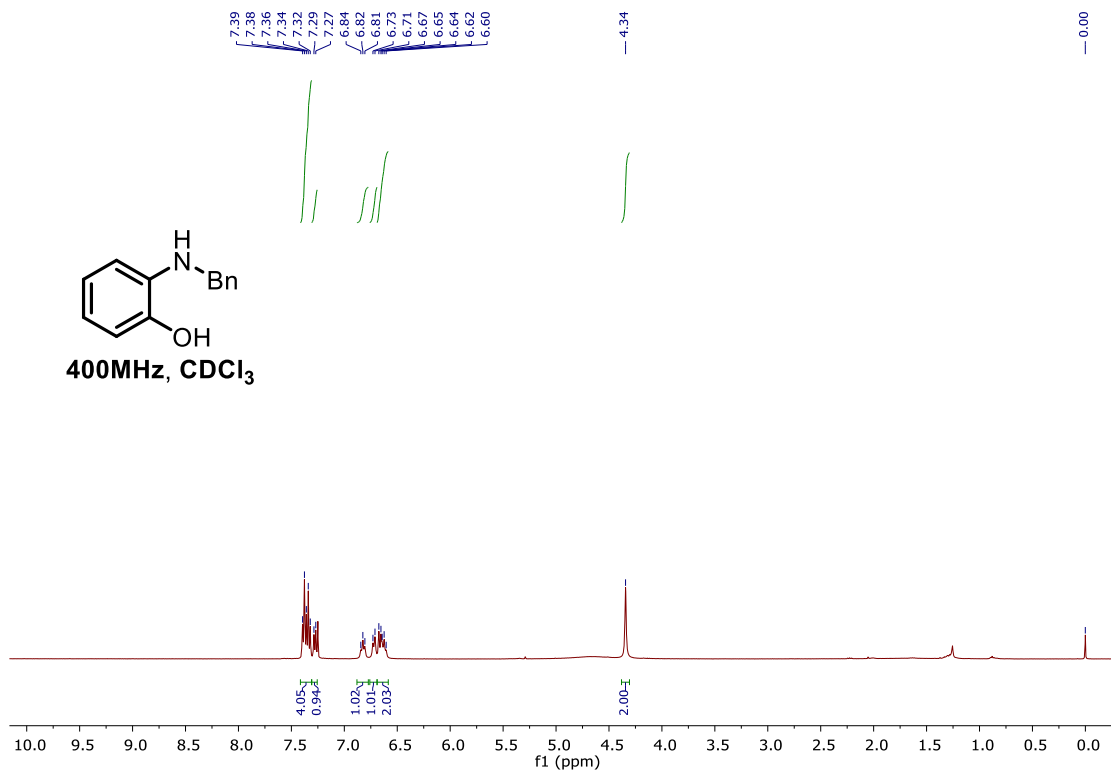
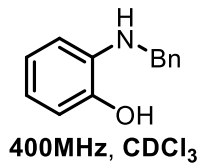


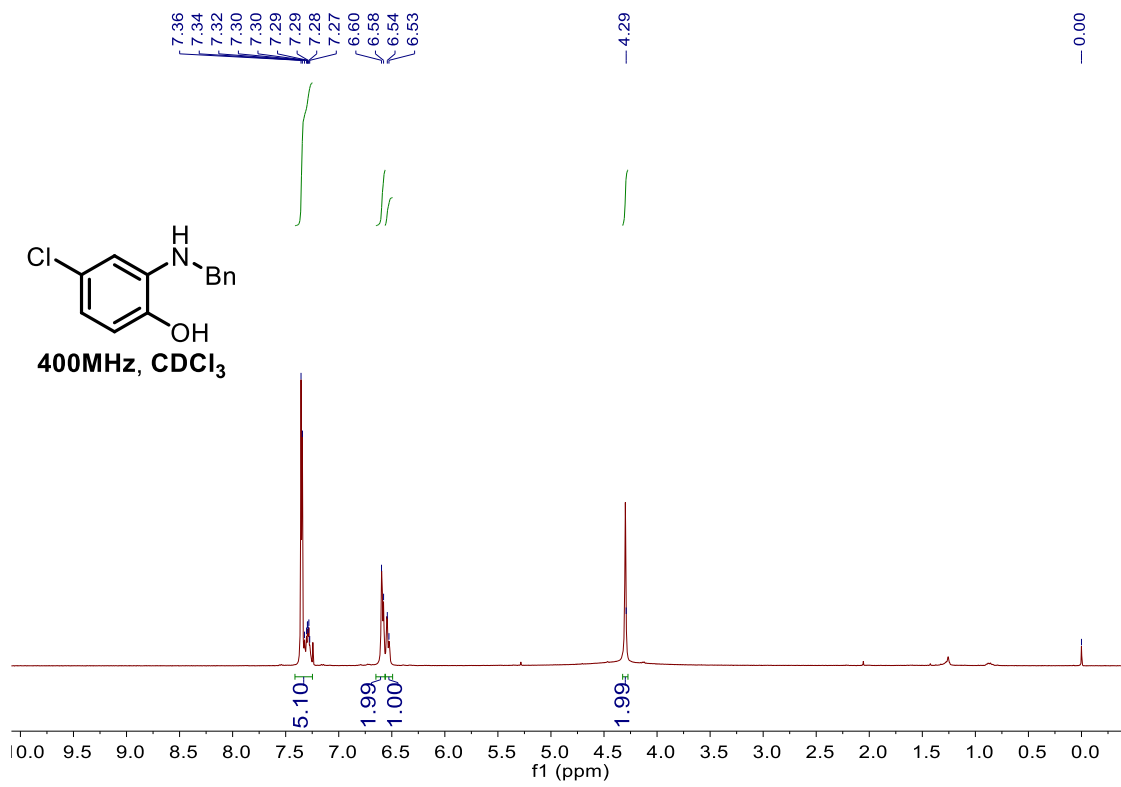




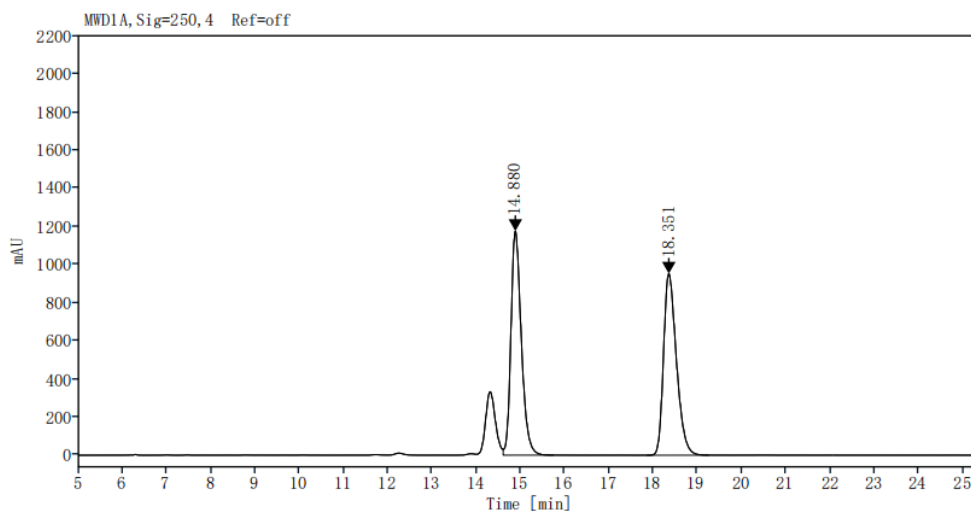








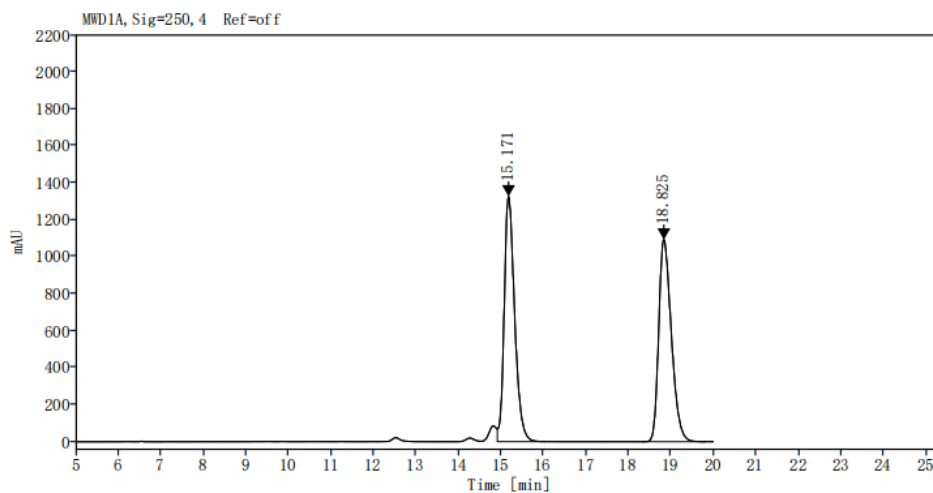
HPLC Spectra of Products



MWD1A, Sig=250,4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
14.880	19188.08	1176.38	49.88	55.24
18.351	19276.74	953.05	50.12	44.76

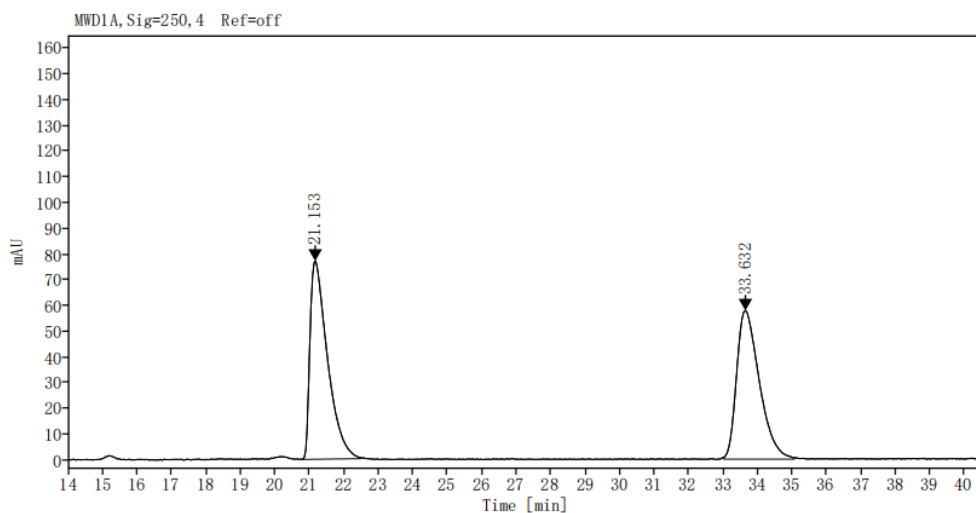
HPLC spectra of **2a-rac**



MWD1A, Sig=250,4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
15.171	22678.36	1326.23	50.10	54.82
18.825	22585.31	1093.01	49.90	45.18

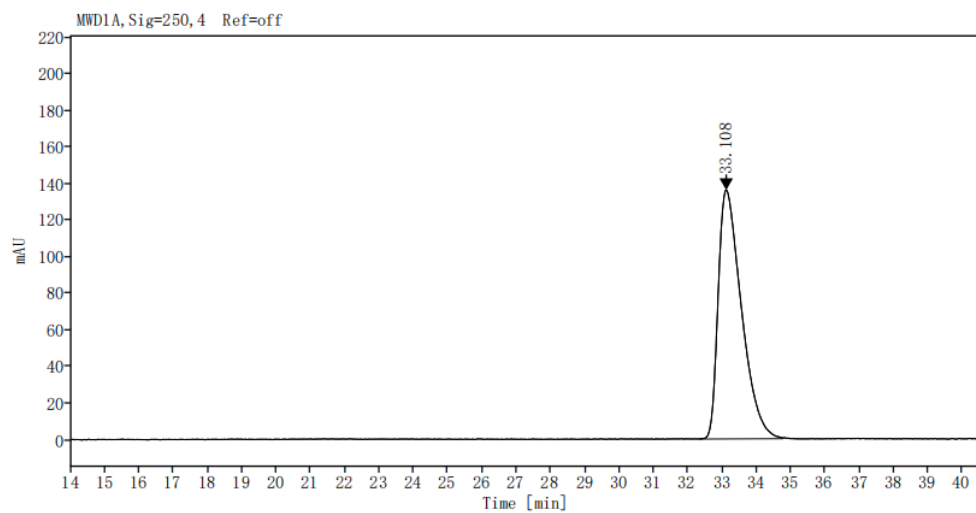
HPLC spectra of **2a- chiral**



MWD1A, Sig=250, 4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
21.153	2714.66	77.05	50.69	57.17
33.632	2640.71	57.72	49.31	42.83

HPLC spectra of **3a-rac**



MWD1A, Sig=250, 4 Ref=off

retention_time	peak.area [mAU*min]	peak.height [mAU]	peak.rel_area %	peak.rel_height %
33.108	6396.75	136.00	100.00	100.00

HPLC spectra of **3a-chiral**

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