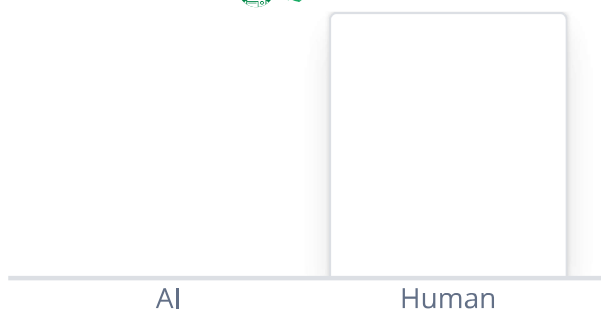




## Results

**0%**

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## CHAPTER SIX

## EXPERIMENTAL DETAILS

## 6.1 General Considerations

## 6.1.1 Reaction Conditions

All electrochemical reactions were performed in the fume hood. An inert atmosphere was used to assemble the electrochemical cell, which was maintained at a constant current density (11.2 mA, 2.1 F/cm<sup>2</sup>) during the electrolysis period. To prevent metal contamination, all chemical transfers were done using plastic syringes. Magnetic stirring bars were used for stirring, and the reaction progress was monitored by TLC and GC-MS analysis at regular intervals.

## 6.1.2 Solvents and Reagents

All chemicals, such as anhydrous solvents, were used as obtained from commercial sources and stored in Sureseal bottles under argon or nitrogen.

## 6.1.3 Chromatography

TLC was performed on aluminum-backed silica Gel 60A F254 plates from Fluorochem. Visualization was carried out using ultraviolet light (254 nm).

#### 6.1.4 Instrumentation

**NMR Spectroscopy:** NMR spectra were recorded at 400 MHz for  $^1\text{H}$  NMR and 100 MHz for  $^{13}\text{C}$  NMR or 500 MHz for  $^1\text{H}$  NMR and 125 MHz for  $^{13}\text{C}$  NMR on JEOL ECP and ECA spectrometers of 400 and 500 MHz, respectively. Chemical shifts are expressed as  $\delta$  values in parts per million (ppm). Samples were prepared in  $\text{CDCl}_3$ . Standardizations were set to:  $^1\text{H}$  NMR—tetramethylsilane (TMS) at 0 ppm;  $^{13}\text{C}$  NMR—residual peak in  $\text{CDCl}_3$  at 77.0 ppm.  $^1\text{H}$  NMR spectra were assigned as follows: chemical shift, number of hydrogens, multiplicity, coupling constant  $J$  (in Hz), assignment. Multiplicity is abbreviated as: s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet; combinations are also abbreviated (e.g., dd=doublet of doublets).

**Mass Spectrometry:** Gas chromatography-mass spectrometry (GC-MS) was performed using an Agilent 7890A gas chromatograph equipped with a 5975C MSD. The column used was HP-5MS (30 m  $\times$  0.25 mm  $\times$  0.25  $\mu\text{m}$ ). The temperature program started at 50°C (2 min hold), ramped at 10°C/min to 280°C, and was maintained at 280°C (5 min hold). Helium was used as the carrier gas at a flow rate of 1.0 mL/min. Samples were prepared in dichloromethane. Peaks are reported by retention time (RT) and mass/charge ( $m/z$ ) ratios.

**Electrochemical Equipment:** Electrochemical reactions were carried out using a constant current power supply (Keithley 2400 SourceMeter). The electrochemical cell consisted of a platinum electrode (2.0  $\text{cm}^2$  geometric surface area).

Current density was maintained at 11.2 mA total current throughout all experiments.

#### 6.2 Experimental Procedures

**Electrochemical Reaction and Radical Coupling (HO003-HO004)**

$\alpha$ -Quaternary amino acid precursor via Kolbe reaction

MW: 143.142g

Methyl 2 acetamidoacrylate (0.2 mmol) was transferred to a beaker, containing tetra-*n*-butylammonium tetrafluoroborate ( $\text{TBABF}_4$ , 100mg, 0.3mmol, ) as supporting electrolyte, acetonitrile (4 mL ),propionic acid (60microL 0.8mmol) and KOH (0.8mmol) was added . The resulting solution was transferred into the 5ml vial for electrolysis. Electrolysis was performed at constant current 11.2 mA, 2.1F/mol.

**Reaction monitoring:** The reaction progress was monitored by GC-MS analysis.

Workup: Diethyl ether was use for the extraction (3 × 20 mL). The combined organic layers were washed with water (5 x 25 mL), The organic phase was dried with magnesium sulphate and vacked down the rotary valve.

F: RF (hexane:ethyl acetate, 7:3) 0.45;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.35-7.18 (10H, m, 2 × Ph), 3.12 (4H, s, 2 ×  $\text{CH}_2\text{Ph}$ );  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  139.4 (C-Ar), 129.1 (C-Ar), 128.3 (C-Ar), 126.8 (C-Ar), 58.2 (quaternary C), 40.8 ( $\text{CH}_2\text{Ph}$ ); GC-MS: RT 8.19 min, m/z 142 ( $\text{M}^+-\text{CH}_2\text{Ph}$ ), 143 ( $\text{M}^+-\text{CH}_2\text{Ph}+\text{H}$ ), 206 ( $\text{M}^+$ ); IR  $\nu_{\text{max}}$  (neat)/ $\text{cm}^{-1}$ : 3028 (Ar C-H), 2924 (C-H), 1602 (Ar C=C), 1495 (Ar C=C), 1454 (C-H bend), 696 (C-H oop).

#### Synthesis of $\alpha$ -Quaternary Amino Acid (HO005)

Workup: The reaction was extracted with ethyl acetate (3 × 20 mL). The combined organic layers were washed with water (25 mL), dried over  $\text{MgSO}_4$ , and concentrated under reduced pressure.