

## Supporting Information

### **Convergent Paired Electrosynthesis of $\beta$ -Nitroalcohols Combining of Anodic Generation of Benzaldehydes and Cathodic Formation of Nitromethyl Anion via an Electrogenerated Base**

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## 1. General

$^1\text{H}$  and  $^{19}\text{F}$  NMR spectra were recorded on JEOL JNM-ECS400 ( $^1\text{H}$ : 400 MHz,  $^{19}\text{F}$ : 376.5 MHz) spectrometer in  $\text{CDCl}_3$ . The chemical shifts for  $^1\text{H}$  and  $^{19}\text{F}$  NMR spectra were given in  $\delta$  (ppm) from internal TMS and monofluorobenzene, respectively. EI mass spectra were measured with a Shimadzu PARVUM2 gas chromatograph-mass spectrometer. Cyclic voltammetry was performed by using a computer-controlled electrochemical analyzer (ALS/CH Instruments 610B), and preparative electrolyses were carried out with a DC power supply (Matsusada Precision P4K-80H).

## 2. Materials

All reagents and solvents were purchased from commercial suppliers and used without further purification.

## 3. Cyclic voltammetry

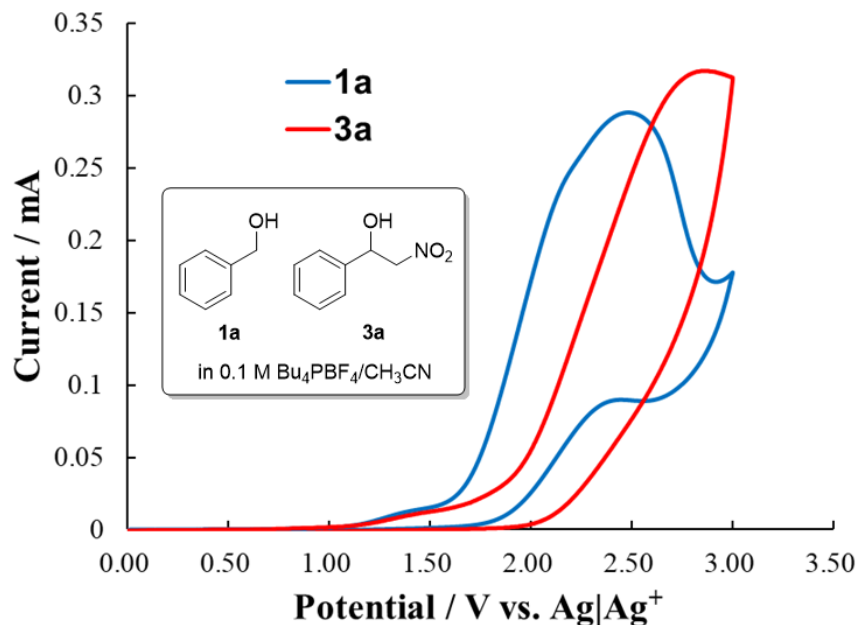
Cyclic voltammetry was carried out with a three-electrode system using a platinum disk ( $\phi = 1\text{ mm}$ ) working electrode, a platinum wire counter electrode, and a silver/silver ion reference electrode ( $\text{Ag}|\text{Ag}^+$ ).

## 4. General procedure for paired electrolysis

Paired electrolysis of a substrate (1 mmol) was carried out with platinum plate electrodes ( $2 \times 2\text{ cm}^2$ ) in 0.4 M  $\text{Bu}_4\text{PBF}_4/\text{CH}_3\text{NO}_2$  (10 mL) using an undivided cell. Constant current electrolysis ( $10\text{ mA cm}^{-2}$ ) was conducted. After the charge ( $3.5\text{ F mol}^{-1}$ ) was passed, the electrolytic solution was concentrated under vacuum and the residue was passed through a short column of silica gel eluting with  $\text{CHCl}_3$  to remove  $\text{Bu}_4\text{PBF}_4$ . The eluent was evaporated under vacuum to provide the reaction mixture containing benzyl alcohol, benzaldehyde, and  $\beta$ -nitroalcohol without  $\text{Bu}_4\text{PBF}_4$ . The yields of the products in the reaction mixture were determined by  $^1\text{H}$  NMR using toluene as an internal standard. The  $\beta$ -nitroalcohol was identified by comparison with literature data (**3a**<sup>1-3</sup>, **3b**<sup>1,3,4</sup>, **3c**<sup>2,3</sup>, **3d**<sup>5</sup>, and **3e**<sup>1-3</sup>) using  $^1\text{H}$ ,  $^{19}\text{F}$  NMR and mass spectroscopy.

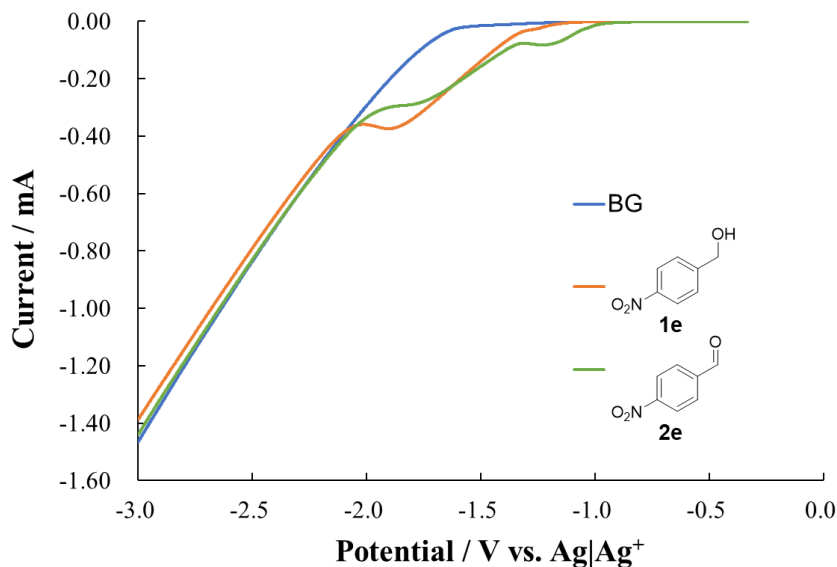
## 5. Supplementary data

### 5-1. Cyclic voltammograms of **1a** and **3a**



**Fig. S1.** Cyclic voltammograms of **1a** and **3a** (1 mmol) in 0.1 M Bu<sub>4</sub>PBF<sub>4</sub>/CH<sub>3</sub>NO<sub>2</sub>, recorded at a Pt disk electrode ( $\phi = 1$  mm). Scan rate was 100 mV s<sup>-1</sup>.

### 5-2. Linear sweep voltammograms of **1e** and **2e**



**Fig. S2.** Linear sweep voltammograms of **1e** and **2e** (1 mmol) in 0.1 M Bu<sub>4</sub>PBF<sub>4</sub>/CH<sub>3</sub>NO<sub>2</sub>, recorded at a Pt disk electrode ( $\phi = 1$  mm). Scan rate was 100 mV s<sup>-1</sup>.

## 6. Supporting references

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