

## Supplementary Information

### **Electrochemical Allylation of Carbonyl Compounds in Aqueous Electrolyte Catalyzed by Zinc**

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**Table of contents**

General methods.....	<b>S3</b>
Experimental procedure.....	<b>S3</b>
X-ray diffraction spectrum of the used Zn foil.....	<b>S4</b>
XPS spectra of the surface of the used Zn foil.....	<b>S5</b>
Cyclic voltammograms.....	<b>S6</b>
Table for yields for allylation of benzaldehyde in subsequent cycles with the resued zinc cathode.....	<b>S11</b>
Spectroscopic data of products.....	<b>S12</b>
NMR spectra of synthesized compounds.....	<b>S17</b>

## **General methods**

Commercial solvents and reagents were used without further purification with the following exceptions: benzaldehyde, furan-2-carbaldehyde, cinnamaldehyde and 3-phenylpropanal were distilled before use, and secondary distilled water was used for the reaction.

Crotyl bromide was purchased from Aldrich; heptanal, 3-methylbenzaldehyde, cinnamaldehyde, 2-bromobenzaldehyde, 3-phenylpropanal and allyl bromide were products from Alfa; furan-2-carbaldehyde, 4-methoxybenzaldehyde, 2-hydroxybenzaldehyde, 4-chlorobenzaldehyde and 2-oxoacetic acid were purchased from Aladian Corporation in China; Zn foils (98%), Pt foils and wires (99.9%) were purchased from domestic corporation.

Nafion<sup>®</sup>551 cation-exchange membrane was purchased from DuPont China R&D and Management Co., Ltd.

Analytical thin layer chromatography (TLC) plates and the silica gel for column chromatography were phased from Qingdao Haiyang Chemical and Special Silica Gel Co, Ltd.

pH Values were measured by pH Meter (PHS-25) which was purchased from SHANGHAI RIDAO CO., LTD.

Proton nuclear magnetic resonance (<sup>1</sup>H NMR) and carbon nuclear magnetic resonance (<sup>13</sup>C NMR) spectroscopy were performed on Bruker Advance 300 and 500 NMR spectrometers.

Divided cell was home-made from polypropylene sheet (SIMONA). The size of cathode and anode chambers is 2×2×4.5 ( cm<sup>3</sup> ) and the active area of the membrane is 4 cm<sup>2</sup>.

Cyclic voltammetry (CV) analysis was performed on Auto Lab<sup>®</sup> PGSTAT30 (product from Metrohm AG, Switzerland) and CHI600D. A platinum disc electrode (d= 1 mm) was used as working electrode and the auxiliary electrode was Pt wire. Cyclic voltammograms of the solutions was recorded at a scan rate of 50 mV s<sup>-1</sup> at rt.

The Zn surface composition was analyzed in the CISSY UHV chamber (base pressure:<1×10<sup>-9</sup> mbar) by X-ray photoelectron spectroscopy (XPS) using a Al K $\alpha$  X-ray source. The emitted photoelectrons were detected by a AXis Ultra ( DLD ) from Kratos.

Glancing angle X-ray diffraction (XRD) was done using Co K $\alpha$  radition with a PANalytical (Netherlands) X'Pert PRO X-ray diffractometer.

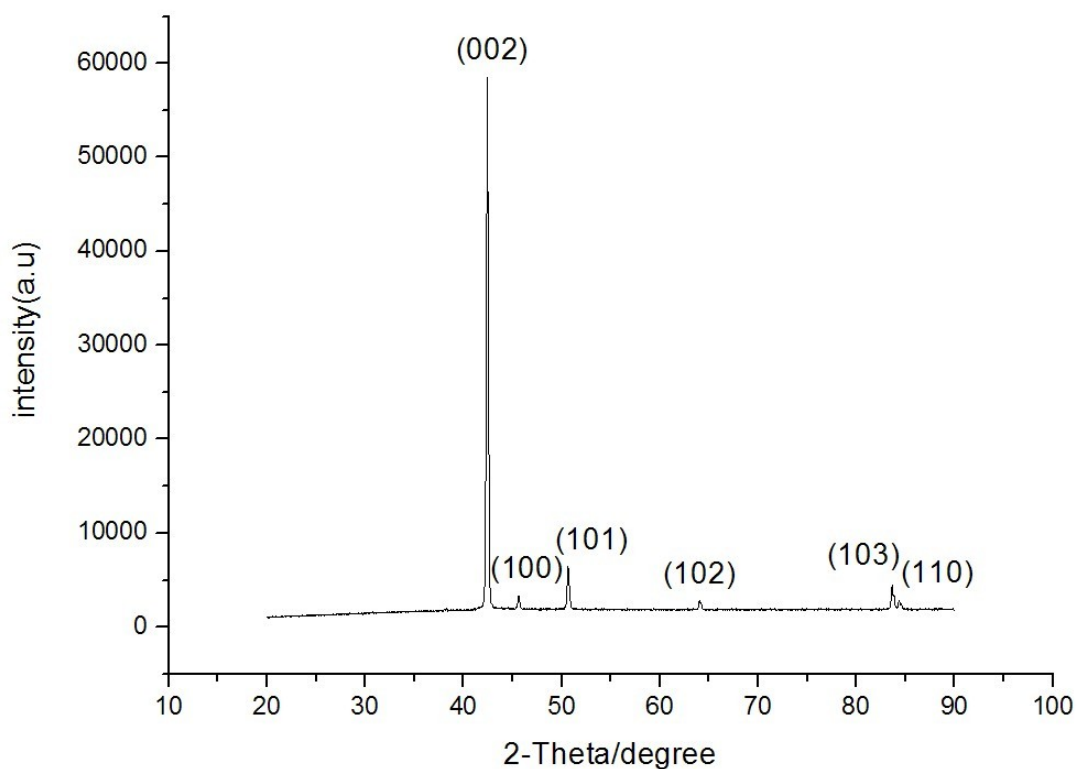
## **Experimental procedure**

**General procedure for the allylation of benzaldehyde:** Reactions were carried out in a two-compartment cell (Nafion<sup>®</sup>551 cation-exchange membrane) equipped with a zinc foil cathode (3 cm<sup>2</sup>) and a platinum foil (1.5 cm<sup>2</sup>) anode. The anodic compartment contained 10 mL LiClO<sub>4</sub> (0.275 M) solution. The cathodic one contained 5 mL THF and 5 mL NH<sub>4</sub>Cl (0.1 M), to which 1 mmol of benzaldehyde and 2 mmol of allyl bromide were added. The solution was stirred and

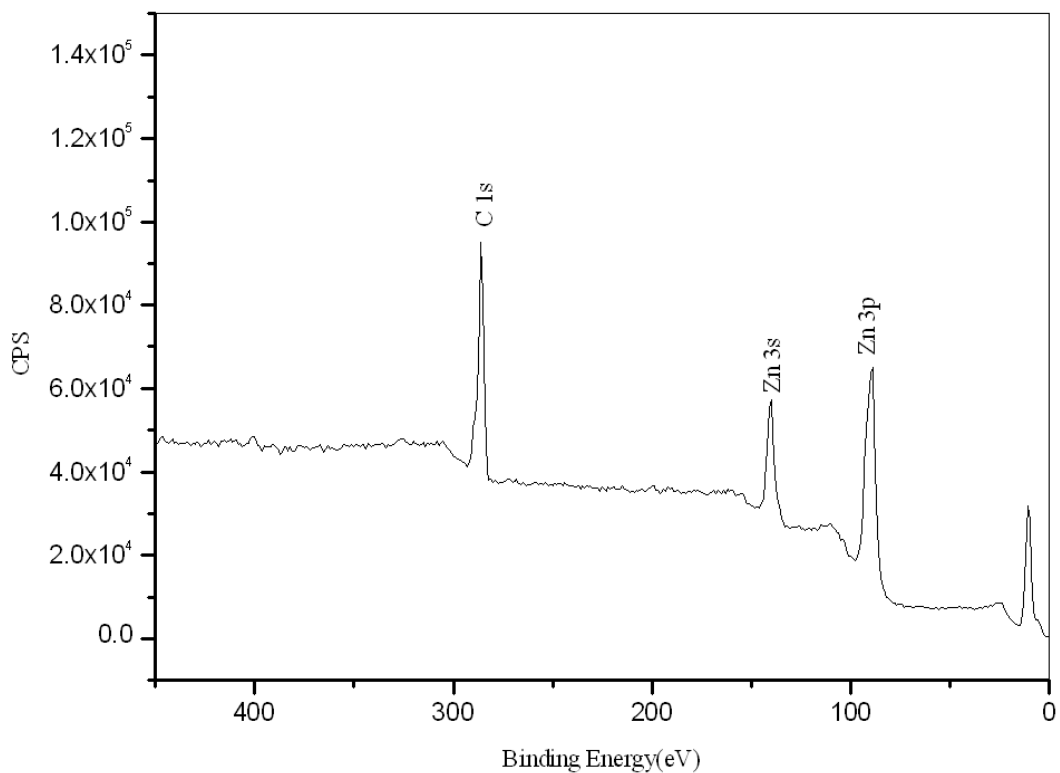
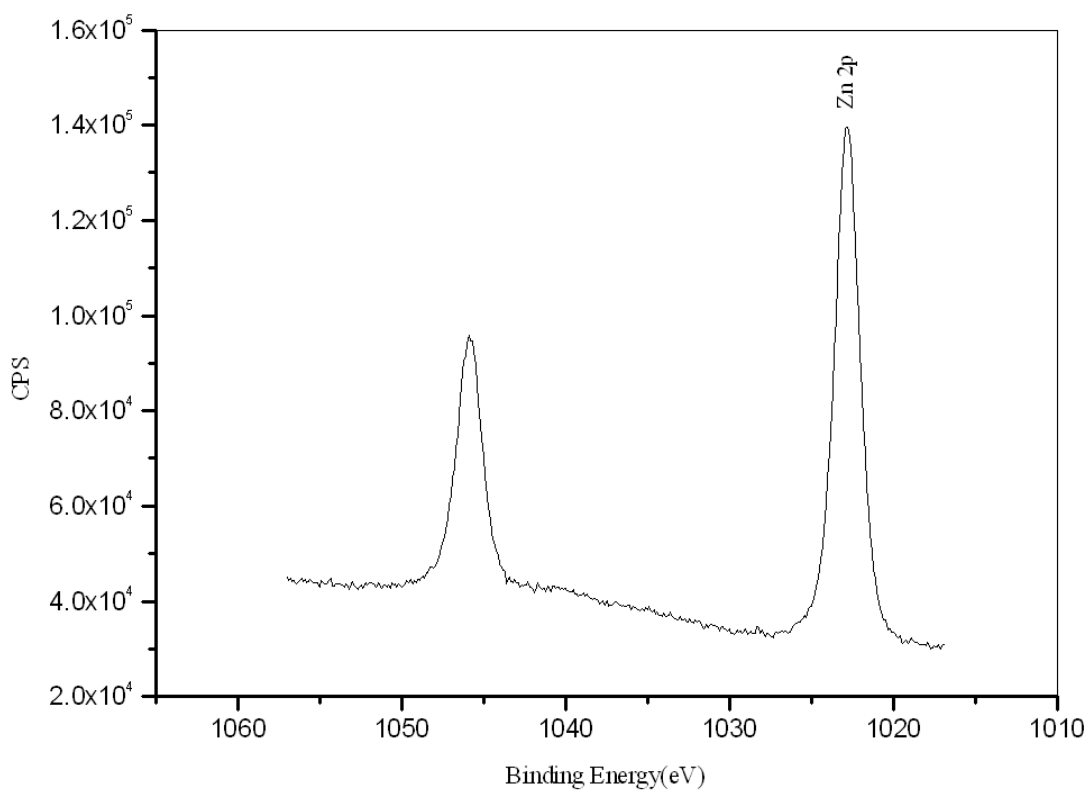
electrolyzed at a constant current (30 mA) until  $3.5 \text{ F mol}^{-1}$  was consumed. After the electrolysis, THF was removed on a rotary evaporator. The residue was extracted three times with methylene dichloride. The organic phase was dried over sodium sulfate and concentrated. The crude product was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 7:1) and the product was obtained as a colorless oil (**3a**, 138 mg, 93%). The totally consumed Zn on the cathode was 16 mg, 0.25 mmol. The authenticities of the products are verified by comparing their  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectral with reported data.

The zinc foil was reused for a repeat reaction after simply washing with water and ethyl acetate.

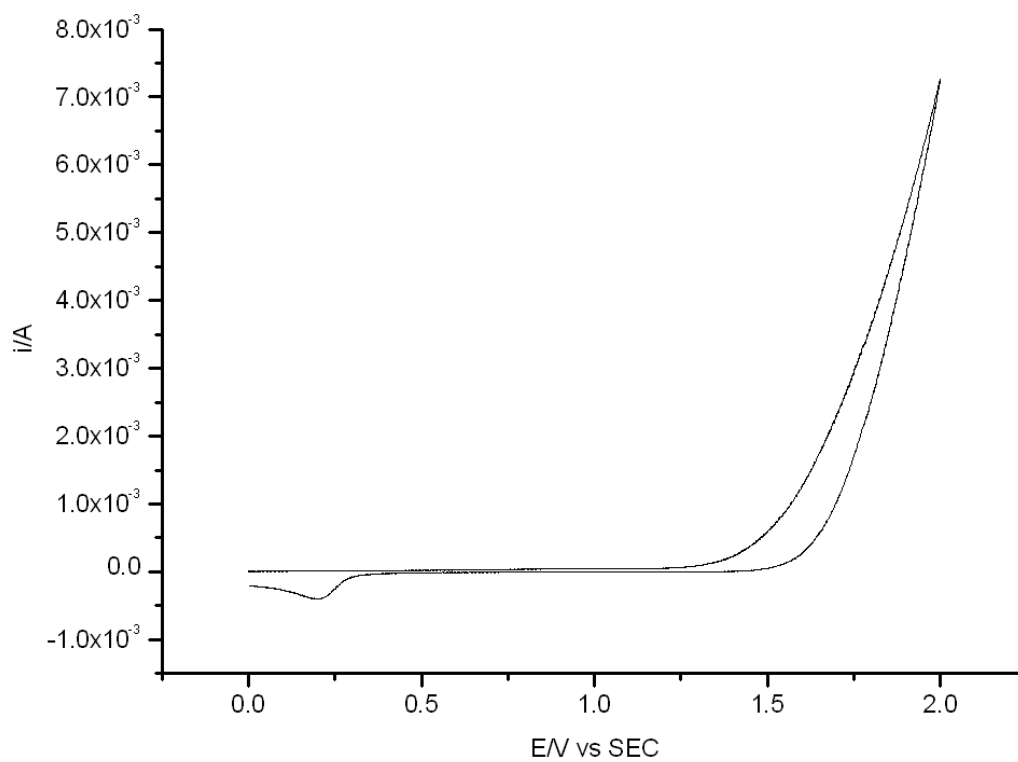
### X-ray diffraction spectrum of the used Zn foil



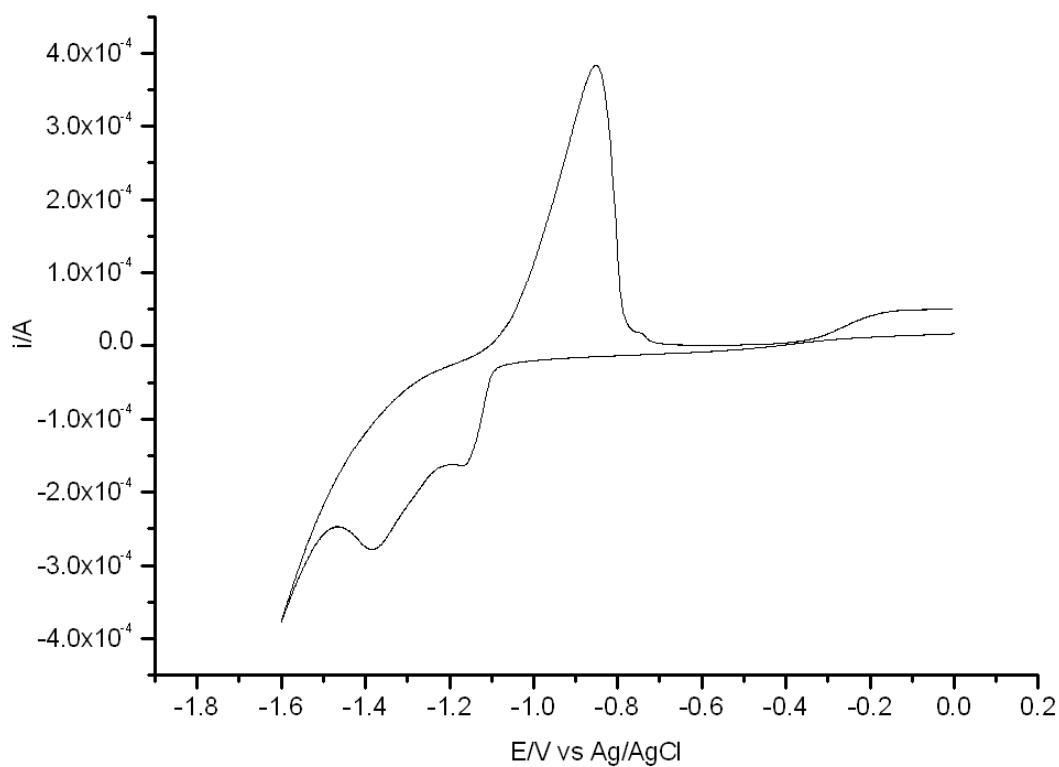
**XPS spectra of the surface of the used Zn foil**



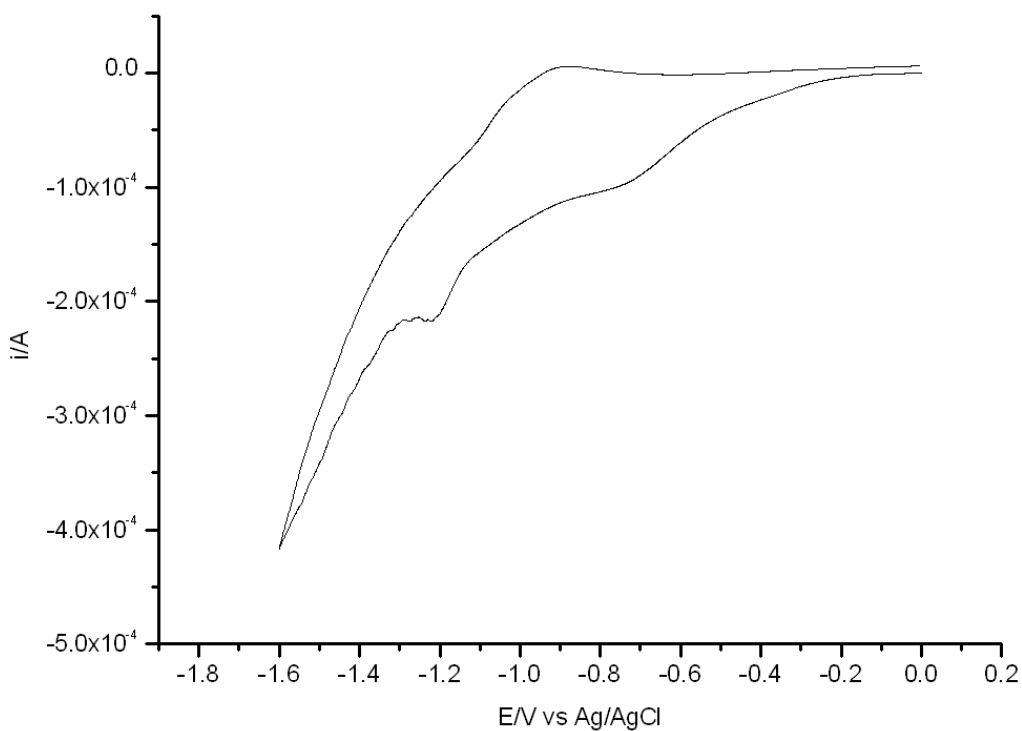
**Cyclic voltammograms:**



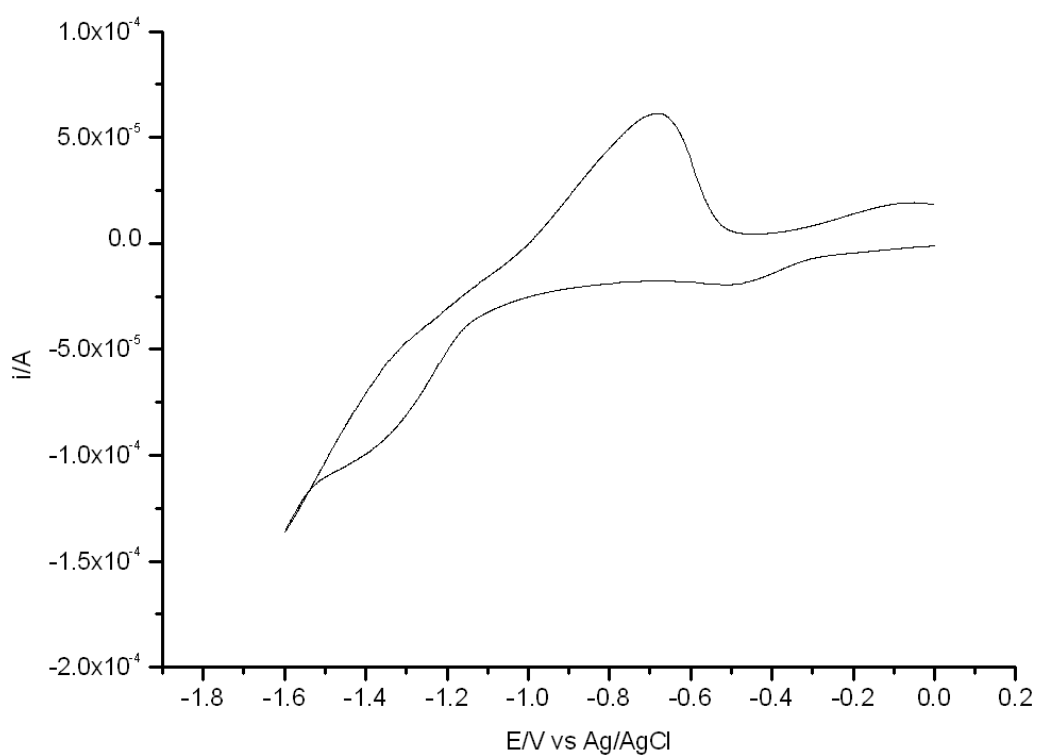
10 mL of 0.275 M LiClO<sub>4</sub> solution (anolyte)



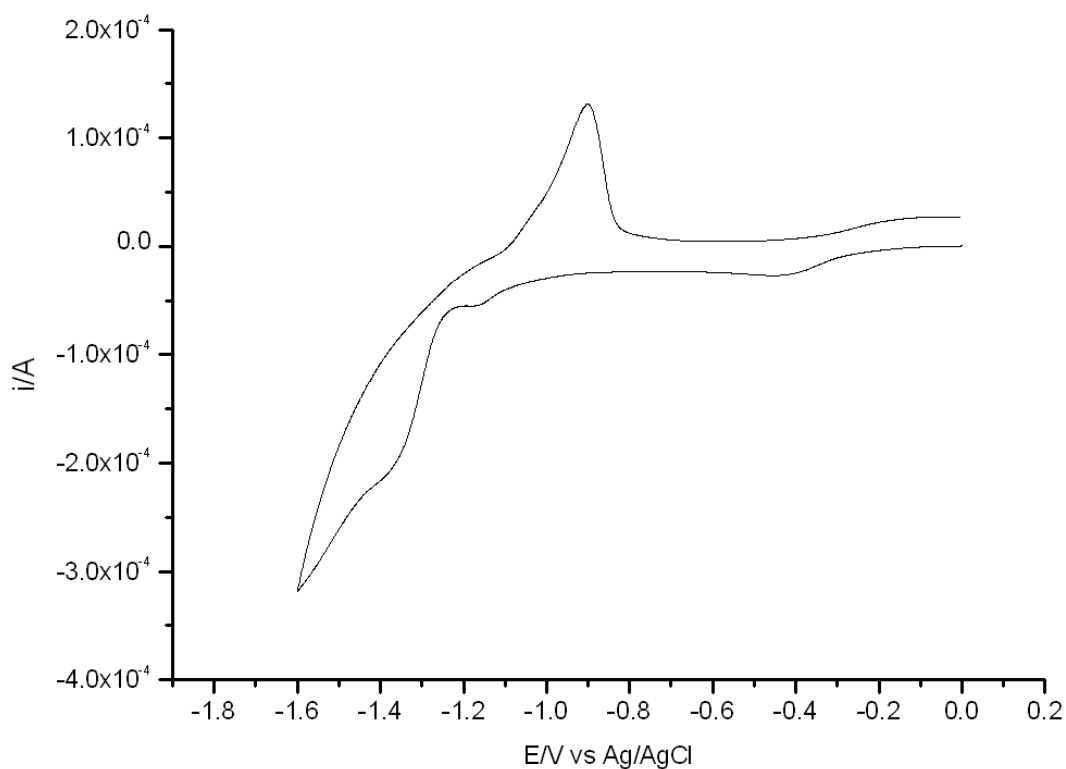
0.3 mmol  $\text{ZnSO}_4$  in a solution of  $\text{NH}_4\text{Cl}$  (0.1 M) and THF (5 mL:5 mL)



Solution in cathode chamber after reaction had proceeded for 1 h

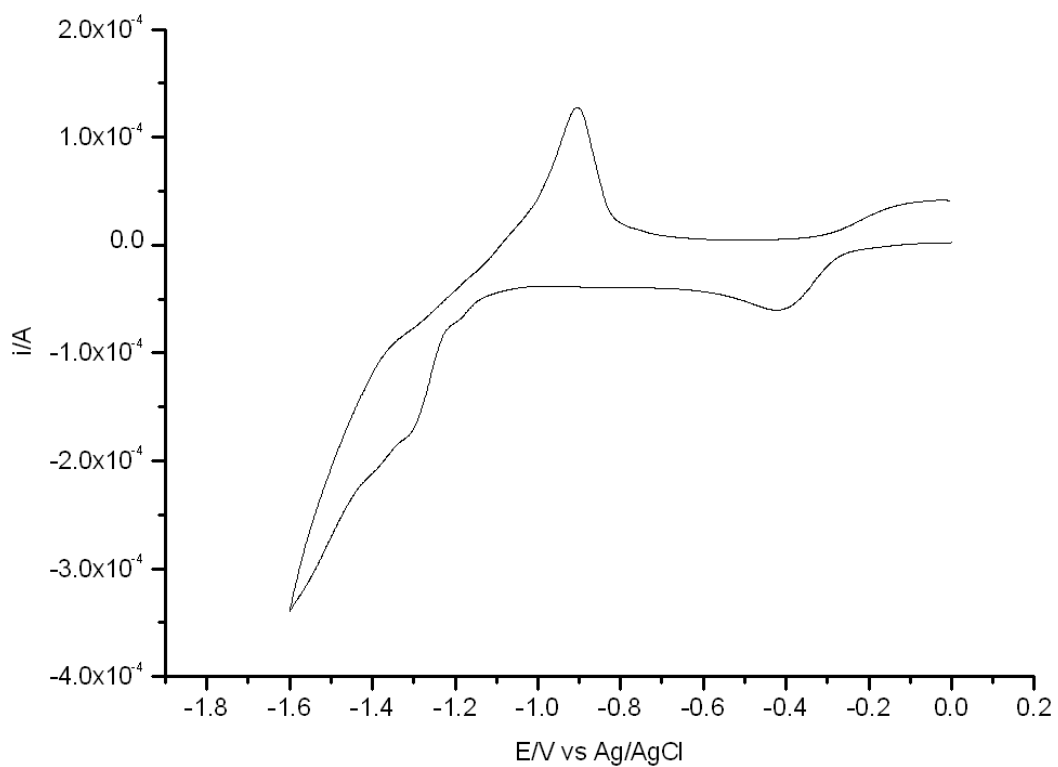


Solution in cathode chamber after reaction had proceeded for 1.5 h

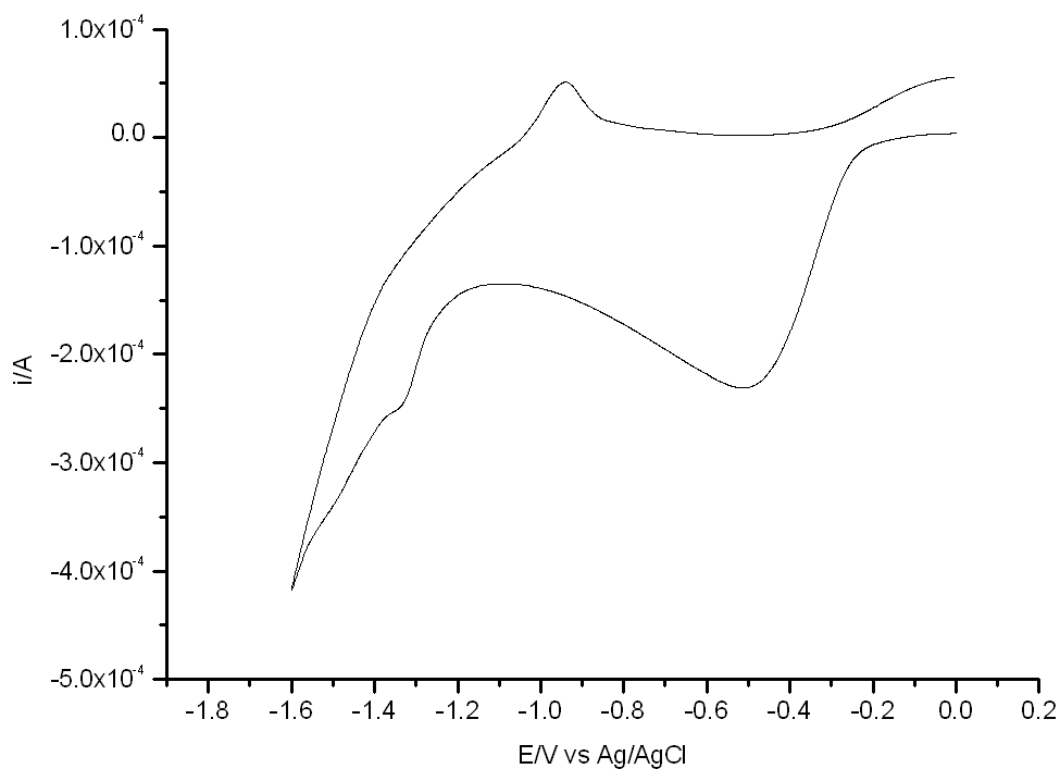


Solution in cathode chamber after the reaction had proceeded for 2 hrs

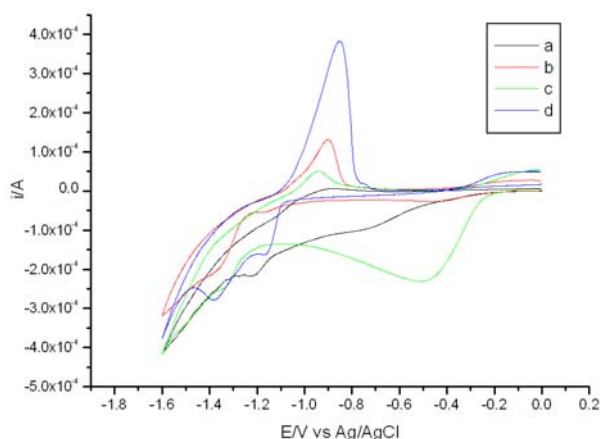




Solution in cathode chamber after the reaction had proceeded for 2.5 hrs



Solution in cathode chamber after the reaction had proceeded for 3 hrs

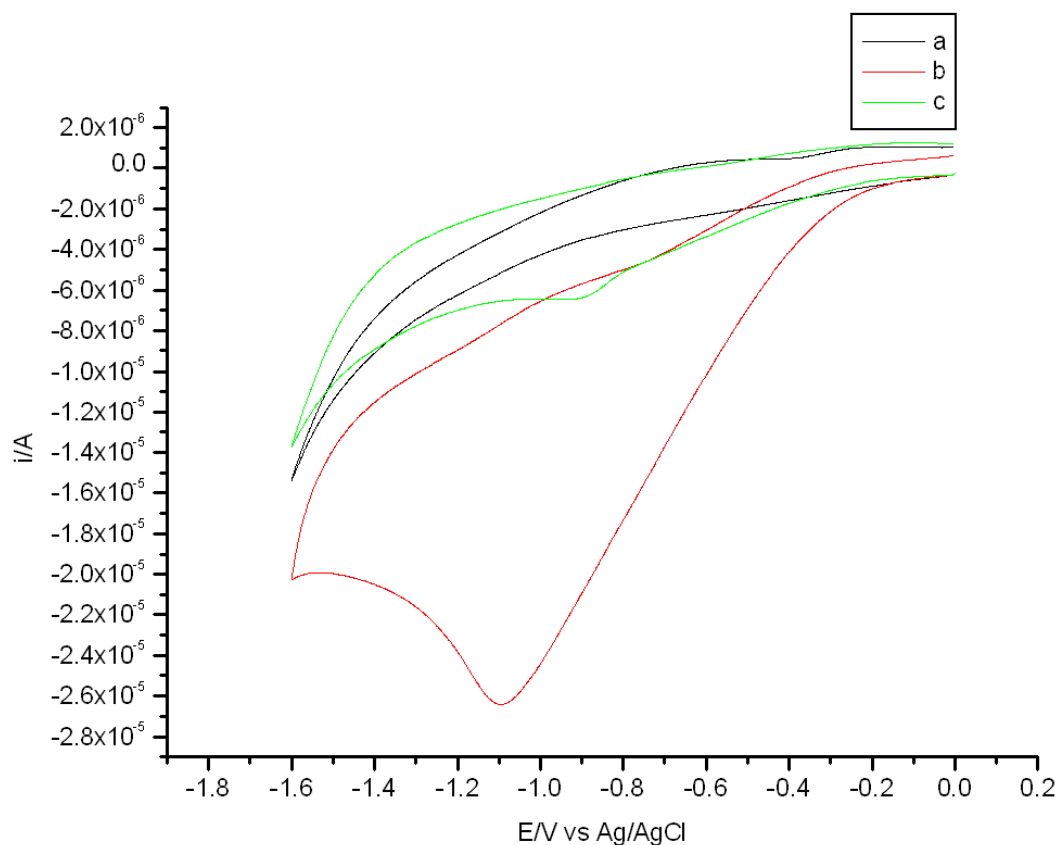


Cyclic voltammograms of the solutions recorded at a scan rate of  $50 \text{ mV s}^{-1}$  at rt with two platinum electrodes, Scan started from 0 to  $-1.6 \text{ V}$ . (a) solution in cathode chamber after reaction proceeded for 1 h (b) 2 hrs (c) 3 hrs (d)  $0.3 \text{ mmol ZnSO}_4$  in a solution of  $\text{NH}_4\text{Cl}$  ( $0.1 \text{ M}$ ) and THF ( $5 \text{ mL}:5 \text{ mL}$ ).

Both of the two reduction peaks (about  $-1.2 \text{ V}$  and  $-1.4 \text{ V}$ ) in solution 'd' can be attributed to the reduction of  $\text{Zn}^{2+}$ . This reduction process might be associated with the surface-confined to surface-confined species reduction conversions. Along with the redox reactions, different species like ' $\text{Zn}^+$ ' and different local pH are expected, hence the reductive process can be very complicated. Similar behavior of the  $\text{Zn}^{2+}$  reduction, please refer to: W. Miao, I. S. Cole, A. K. Neufeld, S. Furman, *J. Electrochem. Soc.*, 2007, **154**, C7-C15.

The value of pH in cathode chamber decreases during the process of the reaction, hence the  $\text{H}^+$  concentrations for solution 'a', 'b' and 'c' are different, which resulting different reduction potentials for  $\text{Zn}^{2+}$ .

As the  $\text{H}^+$  concentration for solution 'c' is very high, the CV curve for solution 'c' showed a strong reductive peak of  $\text{H}^+$  at potential of about  $-0.5 \text{ V}$ .



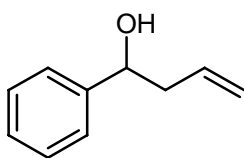
- a. 10ml THF+0.1M Bu<sub>4</sub>BF<sub>4</sub>  
b. 10ml THF+0.1M Bu<sub>4</sub>BF<sub>4</sub>+2.6 mmol PhCHO  
c. 10ml THF+0.1M Bu<sub>4</sub>BF<sub>4</sub>+3mmol allyl bromide

**Table Yields for allylation of benzaldehyde in subsequent cycles with the resued zinc cathode.<sup>a</sup>**

Entry	Zn cathode	Yield (%) <sup>b</sup>	Zinc Consumption (mol%)
1	1st cycle	93	25
2	2nd cycle	93	26
3	3rd cycle	92	26
4	4th cycle	91	24
5	5th cycle	90	28
6	6th cycle	90	27

<sup>a</sup> Conditions: Performed with 1 mmol of benzaldehyde and 2 mmol of allyl bromide in each cycle. <sup>b</sup> Isolated yield.

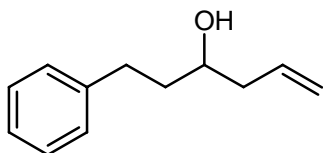
**Spectroscopic data of products**



**1-phenylbut-3-en-1-ol :**

**3a** (138 mg, 93%) colourless oil  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ )  $\delta$  7.17-7.27 (5 H, m,  $5 \times Ph$ ), 5.68-5.76 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.04-5.09 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.64 (1 H, dd,  $J$  5.4 and 7.6 Hz,  $\text{CHOH}$ ), 2.38-2.47 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 2.07 (1 H, br s,  $\text{CHOH}$ );  $^{13}\text{C}$  NMR (125 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ )  $\delta$  143.9, 134.4, 128.4, 127.5, 125.8, 118.3, 73.3, 43.8.

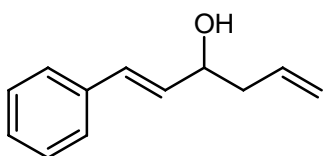
Reference: Y. Yamamoto, H. Yatagi, K. Maruyama, *J. Am. Chem. Soc.* **1981**, *103*, 1969.



**1-phenylhex-5-en-3-ol :**

**3b** (161 mg, 91%) colourless oil  $^1\text{H}$  NMR (300 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 7.14-7.20 (5 H, m,  $5 \times \text{Ph}$ ), 5.69-5.78 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.04-5.09 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 3.67-3.69 (1 H, m,  $\text{CHOH}$ ), 2.60-2.73 (2 H, m,  $2 \times \text{PhCH}_2\text{CH}_2$ ), 2.23-2.24 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 2.10-2.12 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 1.69-1.73 (2 H, m,  $2 \times \text{PhCH}_2\text{CH}_2$ ).  $^{13}\text{C}$  NMR(75 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 142.0, 134.6, 128.4, 128.3, 125.8, 118.3, 69.9, 42.0, 38.4, 32.0.

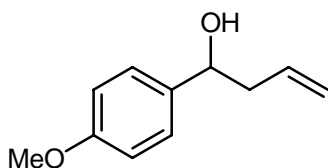
Reference: B. Schmidt, *J. Org. Chem.* **2004**, 69, 7772.



**(E)-1-phenylhexa-1,5-dien-3-ol :**

**3c** (89 mg, 51%) colourless oil  $^1\text{H}$  NMR (300 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 7.16-7.32 (5 H, m,  $5 \times \text{Ph}$ ), 6.53 (1 H, d,  $J$  15.9 Hz,  $\text{CH}=\text{CH}$ ), 6.17 (1 H, dd,  $J$  6.3 and 15.9 Hz,  $\text{CH}=\text{CH}$ ), 5.71-5.85 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.08-5.14 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.30 (1 H, dd,  $J$  6.0 and 12.3 Hz,  $\text{CHOH}$ ), 2.32-2.35 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ );  $^{13}\text{C}$  NMR(75 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 136.6, 134.0, 131.5, 130.3, 128.6, 127.6, 126.4, 118.5, 71.7, 42.0.

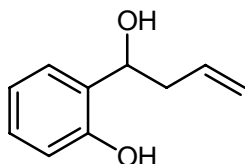
Reference: S. Kobayashi, S. Nagayama, *J. Org. Chem.* **1996**, 61, 2256.



**1-(4-methoxyphenyl)but-3-en-1-ol:**

**3d** (169 mg, 95%) colourless oil  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 6.80-7.21 (4 H, m,  $4 \times \text{Ph}$ ) 5.68-5.76 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.03-5.08 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.60 (1 H, t,  $J$  6.3 Hz,  $\text{CHOH}$ ), 3.72 (3 H, s,  $3 \times \text{OCH}_3$ ), 2.40-2.43 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 1.94 (1 H, br s,  $\text{CHOH}$ );  $^{13}\text{C}$  NMR(125 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 159.1, 136.1, 134.6, 127.1, 118.1, 112.9, 73.0, 55.3, 43.7.

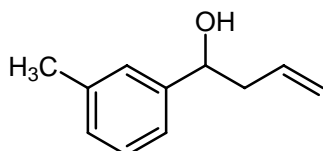
Reference: J. H. Dam, P. Fristrup, R. Madsen, *J. Org. Chem.* **2008**, 73, 3228.



**2-(1-hydroxybut-3-enyl)phenol:**

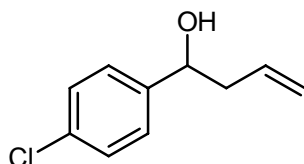
**3e** (134 mg, 82%) colourless oil  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 6.75-7.11 (4 H, m, 4  $\times$  Ph), 5.72-5.80 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.11-5.14 (2 H, m, 2  $\times$   $\text{CH}_2\text{CH}=\text{CH}_2$ ), 4.79 (1 H, dd,  $J$  5.0 and 8.2 Hz,  $\text{CHOH}$ ), 2.93 (1 H, br s,  $\text{CHOH}$ ), 2.48-2.58 (2 H, m, 2  $\times$   $\text{CH}_2\text{CH}=\text{CH}_2$ );  $^{13}\text{C}$  NMR(125 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 155.4, 133.9, 128.9, 127.1, 126.5, 119.8, 119.1, 117.1, 74.5, 42.0.

Reference: T. Zhang, M. Shi, M. Zhao, *Tetrahedron* **2008**, *64*, 2412.



**1-m-tolylbut-3-en-1-ol:**

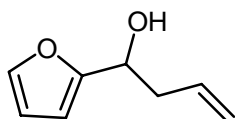
**3f** (159 mg, 98%) colourless oil  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 7.04-7.20 (4 H, m, 4  $\times$  Ph), 5.72-5.80 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ) 5.08-5.13 (2 H, m, 2  $\times$   $\text{CH}_2\text{CH}=\text{CH}_2$ ), 4.63 (1 H, dd,  $J$  5.4 and 7.3 Hz,  $\text{CHOH}$ ), 2.42-2.49 (2 H, m, 2  $\times$   $\text{CH}_2\text{CH}=\text{CH}_2$ ), 2.32 (3 H, s,  $\text{CH}_3$ ), 2.11 (1 H, br s,  $\text{CHOH}$ );  $^{13}\text{C}$  NMR(125 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 143.8, 138.0, 134.6, 128.2, 128.1, 126.5, 122.8, 118.1, 73.3, 43.7, 21.4.



**1-(4-chlorophenyl)but-3-en-1-ol :**

**3g** (150 mg, 82%) colourless oil  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 7.19-7.25 (4 H, m, 4  $\times$  Ph), 5.66-5.74 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.07-5.10 (2 H, m, 2  $\times$   $\text{CH}_2\text{CH}=\text{CH}_2$ ), 4.63 (1 H, dd,  $J$  5.0 and 7.6 Hz,  $\text{CHOH}$ ), 2.35-2.45 (2 H, m, 2  $\times$   $\text{CH}_2\text{CH}=\text{CH}_2$ );  $^{13}\text{C}$  NMR(125 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 142.3, 133.9, 133.1, 128.5, 127.2, 118.7, 72.6, 43.8.

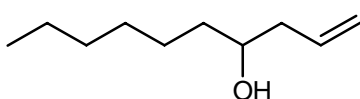
Reference: W. Makoto, O. Hidenori, A. Kinya, *Bull. Chem. Soc. Jpn.* **1990**, *63*, 1738.



**1-(furan-2-yl)but-3-en-1-ol :**

**3h** (80mg, 58%) colourless oil  $^1\text{H}$  NMR (500 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 7.30-7.31 (1 H, m, *furyl*), 6.26 (1 H, dd,  $J$  1.9 and 3.1 Hz, *furyl*), 6.18 (1 H, d,  $J$  3.1 Hz, *furyl*), 5.68-5.82 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.06-5.13 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.68 (1 H, t,  $J$  6.3 Hz,  $\text{CHOH}$ ), 2.52-2.58 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ );  $^{13}\text{C}$  NMR (75 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 156.0, 141.9, 133.7, 118.5, 110.1, 106.1, 66.9, 40.0.

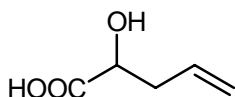
Reference: Z. Wang, Z. Zha, C. Zhou, *Org. Lett.* **2002**, *4*, 1683.



**dec-1-en-4-ol:**

**3i** (131 mg, 86%) colourless oil  $^1\text{H}$  NMR (300 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 5.74-5.92 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.06-5.18 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 3.61 (1 H, m,  $\text{CHOH}$ ), 2.23-2.39 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 2.05-2.18 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 1.15-1.50 (10 H, m,  $10 \times \text{CH}_2$ ), 0.87 (3 H, m,  $\text{CH}_3$ ).

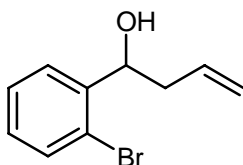
Reference: S. Jiang, G. E. Agoston, T. Chen, M-P. Cabal, E. Turos, *Organometallics*, **1995**, *14*, 4697.



**1-hydroxypent-4-enoic acid:**

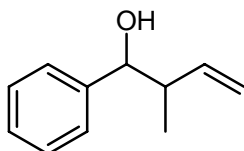
**3j** (63 mg, 54%) colourless oil  $^1\text{H}$  NMR (300 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 5.72-5.81 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.11-5.16 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.28 (1 H, dd,  $J$  4.4 and 6.6 Hz,  $\text{CHOH}$ ), 2.54-2.62 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 2.40-2.47 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ );  $^{13}\text{C}$  NMR (75 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 178.3, 132.1, 119.3, 69.8, 38.3.

Reference: P. Kaur, P. Singh, S. Kumar, *Tetrahedron* **2005**, *61*, 8231.



**1-(2-Bromophenyl)-3-buten-1-ol:**

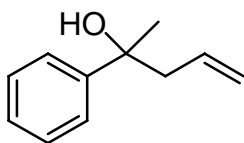
**3k** (218mg, 96%) colourless oil  $^1\text{H}$  NMR (300 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) 7.13-7.56 (4 H, m,  $4 \times Ph$ ), 5.90 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.17 (2H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 2.37-2.66 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 2.14 (1 H, d,  $J$  3.3 Hz);  
Reference: N. Kurono, E. Honda, F. Komatsu, K. Orito, M. Tokuda, *Tetrahedron* **2004**, *60*, 1796.



**3-methyl-1-phenylbut-3-en-1-ol:**

**3l** (156 mg, 96%) colourless oil  $^1\text{H}$  NMR (300 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) (*syn* isomer) 7.20-7.37 (5 H, m,  $5 \times Ph$ ), 5.66-5.86 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 4.98-5.07 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.59 (1 H, d,  $J$  5.5 Hz,  $\text{CHOH}$ ), 2.41-2.60 (1 H, m,  $\text{CHCH}_3$ ), 0.99 (3 H, d,  $J$  6.8 Hz,  $\text{CH}_3$ ). (*anti* isomer)  $\delta$  7.20-7.37 (5 H, m,  $5 \times Ph$ ), 5.66-5.86 (1 H, m,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.12-5.22 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 4.34 (1 H, d,  $J$  7.8 Hz,  $\text{CHOH}$ ), 2.41-2.60 (1 H, m,  $\text{CHCH}_3$ ), 0.85 (3 H, d,  $J$  6.8 Hz,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (75 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ) (*syn* isomer) 142.5, 140.3, 128.0, 127.3, 126.5, 115.6, 77.2, 44.6, 14.0;  $^{13}\text{C}$  NMR (*anti* isomer) 142.4, 140.6, 128.2, 127.6, 126.8, 116.9, 77.8, 46.3, 16.5.

Reference: Z. Wang, Z. Zha, C. Zhou, *Org. Lett.* **2002**, *4*, 1683.



**1-methyl-1-phenyl-3-buten-1-ol:**

**3m** (148 mg, 91%) colourless oil  $^1\text{H}$  NMR (400 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ )  $\delta$  7.25-7.46 (5 H, m,  $5 \times Ph$ ), 5.65 (m, 1H,  $\text{CH}_2\text{CH}=\text{CH}_2$ ), 5.16-5.12 (m, 2H,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 2.50-2.69 (2 H, m,  $2 \times \text{CH}_2\text{CH}=\text{CH}_2$ ), 2.04 (s, 1H,  $\text{CHOH}$ ), 1.54 (s, 3H,  $\text{CH}_3$ );

Reference: N. T. Barczak, E. R. Jarvo, *Eur. J. Org. Chem.* **2008**, 5507



**NMR spectra of synthesized compounds**

