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Short communication

Asymmetric electrocarboxylation of 1-phenylethyl chloride catalyzed by electrogenerated chiral [Co^I(salen)]⁻ complex



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1. Introduction

The conversion of CO_2 to desirable, economically competitive chemicals has recently received much attention [1]. One interesting method for the chemical fixation of CO_2 is the electrocarboxylation of organic halides with CO_2 to carboxylic acids [2,3]. In recent decades, many efforts have been devoted to the electrocarboxylation of aliphatic halides [4] and aromatic halides [5] into the corresponding racemic carboxylic acid. Catalytic systems based on transition-metal complexes, such as cobalt [6], nickel [7], and palladium complexes [8], have been proposed to improve the yield of carboxylation.

Last few decades have also seen several advances in asymmetric electrochemical reduction [9]. However, only a few studies concerning the asymmetric electrocarboxylation have been reported due to the difficulty in enantioselective fixation of CO₂, owing to the fact that the electron is not chiral and enantioselective electron transfer is not possible in principle [10]. In the last few years, Feroci and coworkers [11,12] reported the asymmetric electrocarboxylation of chiral organic halides to afford chiral methylmalonic ester derivatives. Similarly, asymmetric electrocarboxylation of chiral organic halides to afford chiral methylmalonic. However, the need of stoichiometric quantities of optically active substrates can be prohibitively expensive. Therefore, efficient synthetic methodologies for the production of a chiral carboxylic acid from a low-cost achiral substrate are needed.

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ABSTRACT

The feasibility of asymmetric electrocarboxylation of 1-phenylethyl chloride catalyzed by the electrogenerated chiral $[Co^{I}(salen)]^{-}$ complex has been investigated for the first time. Using this system, optically active 2-phenylpropionic acid in 37% yield and 83% ee is synthesized from 1-phenylethyl chloride and CO₂. The electrochemical behavior of the catalyst and the optimization of synthesis conditions are discussed. This study provides a new procedure for the asymmetric synthesis of a chiral compound and expands the applications of chiral Co^{II}(salen) in the electrochemical asymmetric fixation of CO₂.

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Recently, we reported the asymmetric electrocarboxylation of achiral aromatic ketones in the presence of cinchona alkaloids to give chiral aromatic hydroxyl carboxylates [14–16].

Herein, we wish to report a possible electrochemical route for the asymmetric carboxylation of achiral 1-phenylethyl chloride with CO_2 to form optically active 2-phenylpropionic acid in the presence of the chiral $Co^{II}(salen)$ complex (Scheme 1).

2. Experimental sections

2.1. Reagents and apparatus

All the Co(salen) complexes were purchased from J&K. Cyclic voltammograms were recorded with a CHI 600c electrochemical station at 25 °C in an undivided glass cell, with a glassy carbon (GC) cathode (d = 2 mm), a platinum (Pt) spiral anode, and a Ag/AgI/0.1 M TBAI in DMF reference electrode, calibrated after each experiment against the ferrocene/ferricenium (Fc/Fc⁺) couple. All potentials are reported versus the KCl saturated calomel electrode (SCE), using $E_{Fc/Fc}^+ = 0.450$ V versus SCE [7]. The product yields and enantiomeric excesses (ee) were determined by a high-performance liquid chromatography (HPLC) (DIONEX U-3000 pump) equipped with a UV detector and a chiralcel OD-H column [DAICEL Chiral technologies CO., Ltd.]. Gas chromatography-mass spectrometry (GC-MS) spectrum was obtained on a 5973 N spectrometer connected with an HP 6890 gas chromatograph.

2.2. Typical electrocarboxylation experiment

A typical potentiostatic electrolysis was carried out in a mixture of 1-phenylethyl chloride (PhCH(CH₃)Cl), tetrabutylammonium iodide

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Scheme 1. Asymmetric carboxylation of achiral 1-phenylethyl chloride.

(TEAI), and chiral Co^{II}(salen) complex in 10 mL of CO₂-saturated DMF in an undivided glass cell equipped with a magnesium (Mg) anode and a GC ($1.5 \times 2 \text{ cm}^2$) cathode. At the end of the electrolysis, the solvent was distilled off in vacuo. The residue was hydrolyzed and extracted with Et₂O, and the organic layers was dried over anhydrous MgSO₄, filtered and concentrated. The product yield and ee were both determined by HPLC.

3. Results and discussion

3.1. Asymmetric electrocarboxylation of 1-phenylethyl chloride

Asymmetric electrocarboxylation of PhCH(CH₃)Cl was carried out in 10 mL DMF-0.1 M TEAI containing Co^{II} -(R,R)(salen) as catalyst with a Mg anode and a GC cathode in the presence of CO₂ under potentiostatic

| Table 1 |
|--------------------------------------------------------------------------|
| Asymmetric electrocarboxylation of 1-phenylethyl chloride ^a . |

| Entry | $E_{\rm app}^{\rm b}(V)$ | Catalyst/substrate (mol%) | C _{substrate} (mM) | Charge (F mol^{-1}) | T (°C) | Yield ^c (%) | ee ^c (R-%) |
|-------|--------------------------|---------------------------|-----------------------------|------------------------|--------|------------------------|-----------------------|
| 1 | -1.54 | 10 | 10 | 2.0 | 50 | 16 ^d | 83 |
| 2 | -1.54 | 0 | 10 | 2.0 | 50 | _e | - |
| 3 | -1.54 | 10 ^f | 10 | 2.0 | 50 | 15 | - |
| 4 | -1.54 | 10 ^g | 10 | 2.0 | 50 | 15 | 82 ^h |
| 5 | -1.49 | 10 | 10 | 2.0 | 50 | 9 | 83 |
| 6 | -1.59 | 10 | 10 | 2.0 | 50 | 23 | 83 |
| 7 | -1.64 | 10 | 10 | 2.0 | 50 | 24 | 76 |
| 8 | -1.74 | 10 | 10 | 2.0 | 50 | 28 | 69 |
| 9 | -1.59 | 1 | 10 | 2.0 | 50 | 9 | 82 |
| 10 | -1.59 | 5 | 10 | 2.0 | 50 | 17 | 82 |
| 11 | -1.59 | 15 | 10 | 2.0 | 50 | 27 | 83 |
| 12 | -1.59 | 20 | 10 | 2.0 | 50 | 27 | 82 |
| 13 | -1.59 | 25 | 10 | 2.0 | 50 | 27 | 83 |
| 14 | -1.59 | 15 | 20 | 2.0 | 50 | 28 | 82 |
| 15 | -1.59 | 15 | 40 | 2.0 | 50 | 30 | 82 |
| 16 | -1.59 | 15 | 50 | 2.0 | 50 | 31 | 83 |
| 17 | -1.59 | 15 | 60 | 2.0 | 50 | 32 | 80 |
| 18 | -1.59 | 15 | 80 | 2.0 | 50 | 33 | 77 |
| 19 | -1.59 | 15 | 100 | 2.0 | 50 | 34 | 72 |
| 20 | -1.59 | 15 | 50 | 1.5 | 50 | 23 | 83 |
| 21 | -1.59 | 15 | 50 | 2.5 | 50 | 35 | 83 |
| 22 | -1.59 | 15 | 50 | 3.0 | 50 | 37 | 83 |
| 23 | -1.59 | 15 | 50 | 3.5 | 50 | 38 | 82 |
| 24 | -1.59 | 15 | 50 | 3.0 | 0 | 66 | 3 |
| 25 | -1.59 | 15 | 50 | 3.0 | 25 | 55 | 33 |
| 26 | -1.59 | 15 | 50 | 3.0 | 40 | 45 | 63 |
| 27 | -1.59 | 15 | 50 | 3.0 | 60 | 23 | 72 |
| 28 | -1.59 | 15 | 50 | 3.0 | 70 | 17 | 58 |
| 29 | - 1.59 | 15 | 50 | 3.0 | 80 | 13 | 40 |

^a Solvent: DMF; cathode: GC; anode: Mg; substrate: 1-phenylethyl chloride; supporting electrolyte: 0.1 M TEAI; catalyst: Co^{II}-(R,R)(salen). $P_{CO2} = 1 \times 10^5$ Pa.

^b Applied potential (V vs. SCE).

^c The yield, based on the quantity of the starting substrate, and ee were determined by HPLC analysis using a chiralcel OD-H.

^d 2,3-Diphenylbutane (39%), phenylethane (9%), and styrene (3%) were also obtained with the 1-phenylethyl chloride conversion of 68%.

^e Almost no 1-phenylethyl chloride is converted.

^f Catalyst: Co^{ll}(salen).

^g Catalyst: Co^{II}-(S,S)(salen).

^h The ee of product is (S-%).

condition. As shown in Table 1 (entry 1), the corresponding chiral product 2-phenylpropionic acid is obtained in 16% yield and 83% ee. No reaction occurs in the absence of Co^{II} -(R,R)(salen) (entry 2, Table 1). In addition, racemic 2-phenylpropionic acid is obtained when achiral Co^{II} (salen) is used (entry 3, Table 1). Furthermore, when the catalyst Co^{II} -(R,R)(salen) is replaced by Co^{II} -(S,S)(salen) (entry 4, Table 1), similar yield and ee values are observed, except that the stereochemistry is inverted. The data from entries 1–4 indicate that the chiral Co^{II} (salen) complex plays an important role in the reaction. To investigate the probable catalytic role of the Co^{II} (salen) complex, cyclic voltammetric studies of Co^{II} -(R,R)(salen) were carried out (Fig. 1).

Cyclic voltammogram (CV) of Co^{II} -(R,R)(salen) (2 mM) in DMF shows a reversible couple, designated as A/B in Fig. 1a, corresponding to the reduction of Co^{II} to Co^{I} with E° =-1.50 V vs. SCE (Eq. (1)). The addition of PhCH(CH₃)Cl (19 mM) causes an increase in the peak current and a positive shift in the peak potential of peak A while its anodic partner B disappears completely, and a new reduction peak C appears at a more negative potential of -2.21 V (Fig. 1b). The absence of the peak B on the inclusion of PhCH(CH₃)Cl indicates the existence of a chemical reaction following the electron transfer associated with peak A. For comparison, the CV of PhCH(CH₃)Cl (19 mM) was investigated subsequently; as shown in curve c, an irreversible reduction peak at -2.19 V corresponding to the two-electron reductive cleavage of the C-X bond of the aromatic halide [7] was observed. The above investigations indicate that the new reduction peak C in curve b is attributed to the two-electron reductive cleavage of the C-Cl bond of PhCH(CH₃)Cl (Eq. (2)).

Deutsch reported that Co^I complexes are strong nucleophiles that react with organic halides to give organocobalt complexes [17]. In addition, the CV of Co^{II}-(R,R)(salen) in the presence of PhCH(CH₃)Cl is similar to that of Co^{II}(salen) in the presence of RCI [18]. Accordingly, we propose the reactions shown in Eqs. (1), (3), and (4) to be associated with the peak A of curve b. Nucleophilic [Co^I-(R,R)(salen)]⁻ generated from the one-electron transfer to Co^{II}-(R,R)(salen) can react with PhCH(CH₃)Cl to give Co^{III}-(R,R)(salen)(PhCH(CH₃)). The increment in current of peak A ($i_{b-pcA}/i_{a-pcA} > 2$) implies the occurrence of another electron transfer. Based on a previous report [18], we presume that Co^{III}-(R,R)(salen)(PhCH(CH₃)) is one-electron reduced at the working potential.

It is reported that the $[Co^{II}(L)R]^-$ species are unstable and undergo rapid homolytic (Eq. (5)) or heterolytic (Eq. (6)) Co–R bond cleavage



Fig. 1. Cyclic voltammograms of Co^{II} -(R,R)(salen) and PhCH(CH₃)Cl in DMF-0.1 M TEABF₄ at a 2 mM diameter GC electrode at a scan rate of 0.1 V s⁻¹: (a) 2 mM Co^{II}-(R,R)(salen), (b) as (a) + 19 mM PhCH(CH₃)Cl, (c) 19 mM PhCH(CH₃)Cl, (d) as (b) in the presence of 0.2 M CO₂, (e) as (c) in the presence of 0.2 M CO₂. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.)

[19]. In our case, the homolytic Co–R bond cleavage is more likely to occur, as styrene (3%) resulting from disproportionation of $[PhCH(CH_3)]^*$ (Eq. (7)) is detected by GC–MS (entry 1, Table 1).

$$Co^{II} - (R, R)(salen) + e^{-} \approx [Co^{I} - (R, R)(salen)]^{-}$$
(1)

$$PhCH(CH_3)CI + 2e^{-} \rightarrow [PhCH(CH_3)]^{-} + CI^{-}$$
(2)

$$[\text{Co}^{I} - (\text{R}, \text{R})(\text{salen})]^{-} + PhCH(\text{CH}_{3})\text{CI} \rightarrow \text{Co}^{III} - (\text{R}, \text{R})(\text{salen})(PhCH(\text{CH}_{3}) + \text{CI}^{-} (3))$$

$$Co^{III} - (R, R)(salen)(PhCH(CH_3) + e^{-} \neq [Co^{II} - (R, R)(salen)(PhCH(CH_3))]^{-}$$
(4)

$$[\text{Co}^{\text{II}} - (\text{R}, \text{R})(\text{salen})(\text{PhCH}(\text{CH}_3))]^{-} \rightarrow [\text{PhCH}(\text{CH}_3)]^{\bullet} + [\text{Co}^{\text{I}} - (\text{R}, \text{R})(\text{salen})]^{-} (5)$$

$$[\mathrm{Co}^{\mathrm{II}} - (\mathrm{R}, \mathrm{R})(\mathrm{salen})(\mathrm{PhCH}(\mathrm{CH}_3))]^{-} \rightarrow [\mathrm{PhCH}(\mathrm{CH}_3)]^{-} + \mathrm{Co}^{\mathrm{II}} - (\mathrm{R}, \mathrm{R})(\mathrm{salen}) \qquad (6)$$

$$2[PhCH(CH_3)] \rightarrow PhCH_2CH_3 + PhCHCH_3$$
(7)

$$[PhCH(CH_3)]^{\bullet} + e^{-} \rightarrow [PhCH(CH_3)]^{-}$$
(8)

$$[Co^{II}-(R,R)(salen)(PhCH(CH_3))]^{-} + CO_2 \longrightarrow OOC + Co^{II}-(R,R)(salen)$$

$$R$$
(9)

$$[PhCH(CH_3)]^- + CO_2 \longrightarrow R + S$$
(10)

Generally, the reduction potential of R[•] is more positive than that of RX [5], and the $E^{\circ}_{R,R}$ value of [PhCH(CH₃)][•] is -1.57 V vs. SCE in DMF [20]. Thus, the resulting radical [PhCH(CH₃)][•] formed in Eq. (5) may be further reduced to PhCH(CH₃)⁻ (Eq. (8)).

When CO_2 is bubbled into the DMF solution used to obtained curve d or e of Fig. 1, an increase in the peak current is observed, respectively (peak A of curve d and peak C of curve e). Vianello and coworkers [18] reported that the Co^{II} alkyl complexes can directly attack CO_2 . It is likely that the resulting organometallic product of Eq. (4) can initiate a nucleophilic attack on CO_2 , resulting in the optically active 2-phenylpropionic acid (R configuration) (Eq. (9)). Successful stereoselective nucleophilic attack on CO_2 is attributed to the specific three-dimensional configuration of $[Co^{II}-(R,R)(salen)(PhCH(CH_3))]^-$ (Eq. (4)). Besides, the intermediate $[PhCH(CH_3)]^-$ can react with CO_2 to give the racemic 2-phenylpropionic acid (Eq. (10)).

Eqs. (1), (3), (4), and (9) reveal that chiral 2-phenylpropionic acid can be obtained in the presence of Co^{II} -(R,R)(salen). However, the occurrence of reaction represented by Eq. (10) indicates that the product will not reach 100% ee (entry 1, Table 1). Moreover, curve c is

consistent with the result that no product is obtained in the absence of catalyst at a potential of -1.54 V (entry 2, Table 1).

However, the yields of PhCH(CH₃)COOH are relatively low (Table 1). The likely reason for this is the reductions of the intermediates involved in the process leading to the decrease of PhCH(CH₃)COOH yield. For example, in the electrocarboxylation of PhCH(CH₃)Cl in the presence of [Co^{II}-(R,R)(salen)] (entry 1, Table 1) along with unreacted PhCH(CH₃)Cl (32%), 2,3-diphenylbutane (39%), phenylethane (9%), and styrene (3%) are also detected by GC-MS. In theory, the intermediate [PhCH(CH₃)][•] (Eq. (5)) may undergo further reactions, such as disproportionation (Eq. (7)), radical-radical coupling (Eq. (11)), hydrogenatom abstraction (Eq. (12)), and reduction to $[PhCH(CH_3)]^-$ (Eq. (8)), which is then protonated by any available proton source in the reaction medium (Eqs. (13) and (14)). Coupling of alkyl radical and protonation of the alkyl carbanion are well documented in the literature [21]. In this report, only the yields of PhCH(CH₃)COOH is reported; other products stemming from subsequent reaction of [PhCH(CH₃)][•] or [PhCH(CH₃)]⁻ with reactants other than CO₂, although quite often observed in relatively high yields, are omitted since they are not relevant to the discussion on asymmetric electrocarboxylation.

$$2[PhCH(CH_3)] \rightarrow PhCH(CH_3)CH(CH_3)Ph$$
(11)

 $[PhCH(CH_3)]^{\bullet} + SH \rightarrow PhCH_2CH_3 + S^{\bullet}$ (12)

$$[PhCH(CH_3)]^{-} + SH \rightarrow PhCH_2CH_3 + S^{-}$$
(13)

$$[PhCH(CH_3)]^- + H_2O \rightarrow PhCH_2CH_3 + OH^-$$
(14)

3.2. Influence of various experimental parameters

With the aim of optimizing the yield and ee of the PhCH(CH₃)COOH, we turned our attention on the various experimental parameters influencing the reaction. These include applied potential, catalyst-to-substrate ratio, substrate concentration, charge passed, and temperature. The results of these experiments are reported in Table 1.

Initially, the influence of variation in applied potential is examined. As is evident from Table 1 (entries 1, 5–8), a more negative potential favors an increase in PhCH(CH₃)COOH yield. No substantial change in ee is observed when the potential is changed from -1.49 to -1.59 V, whereas on further increase in the applied potential from -1.59 to -1.74 V, the ee of PhCH(CH₃)COOH decreases. Considering the yield and ee, -1.59 V is the optimal working potential.

Next, as the catalyst strongly influences the reaction (entries 1–4, Table 1), the effect of the catalyst-to-substrate ratio is evaluated. As shown in Table 1 (entries 6, 9–13), the ee of PhCH(CH₃)COOH is independent of the catalyst-to-substrate ratio. On the other hand, the yield increases from 9% to 27% as the catalyst-to-substrate ratio increases from 1 to 15 mol%, whereas subsequent increase in the catalyst quantity to 25 mol% has no significant effect on the product yield (entries 11–13, Table 1). A lower catalyst-to-substrate ratio generates lower quantities of [Co^{III}-(R,R)(salen)(PhCH(CH₃))], hence lowering the probability of contact between CO₂ and [Co^{III}-(R,R)(salen)(PhCH(CH₃))]⁻ (Eq. (9)), which results in lower yield. Once the catalyst-to-substrate ratio reaches 15%, it is likely that the reaction rate is limited, which results in a nearly constant yield of the desired product.

Entries 11 and 14–19 (Table 1) present the results obtained with different initial substrate concentrations. The yield of desired product increases from 27% to 34% as substrate concentration increases from 10 to 100 mM. On the other hand, the ee remains unchanged, except when the electrolysis is carried out in a more concentrated solution (>50 mM, Table 1, entries 16–19). Considering the ee and yield of PhCH(CH₃) COOH, the optimal substrate concentration is 50 mM. Besides, due to the presence of the non-faradaic current, the faradaic efficiency cannot attain 100%. However, increasing the number of faradays per mole of substrate above 3.0 does not improve the CO_2 fixation significantly.

It is known that an increase in temperature leads to a decrease in the solubility of reactant CO₂ in DMF [22]; however, high temperatures also affect both the thermodynamics and the kinetics of the reaction. Increasing the temperature from 0 °C to 50 °C reduces the yield, whereas the ee increases from 3% to 83%. Further increase in temperature from 50 °C to 80 °C decreases the ee to 13% (Table 1, entries 22, 24-29). At a lower temperature, it is likely that the reaction rate of Eq. (9) is slower than that of Eq. 5, which can account for the observed low ee at 0 °C (Table 1, entry 24). Besides, a faster rise in the rate of Eq. (9) when compared to that of Eq. (5) with the increase in temperature can account for the gradual increase in the ee with the increase in temperature from 0 °C to 50 °C (Table 1, entries 22, 24–26). By contrast, at higher temperatures (>50 °C), the concentration of CO₂ becomes so low that, together with the instability of the Co–R bond, $[Co^{II}-(R,R)(salen)(PhCH(CH_3))]^{-}$ can readily decompose to [PhCH(CH₃)][•] (Eq. (5)), thereby decreasing the ee (Table 1, entries 22, 27–29). Therefore, the optimum temperature is 50 °C (Table 1, entry 22). Efforts are currently underway to investigate the thermodynamic and the kinetic aspects of the asymmetric electrocarboxylation.

4. Conclusion

In summary, optically active 2-phenylpropionic acid has been successfully synthesized through asymmetric catalytic carboxylation of 1-phenylethyl chloride via electrogenerated [Co^I-(R,R)(salen)]⁻. The electrochemical behavior of Co^{II}-(R,R)(salen) shows that the asymmetric carboxylation reaction is an electrocatalytic process. Further advances in this electrocarboxylation system can provide a new method for the synthesis of optically active carboxylic acids from racemic substrates and CO₂.

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