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Nickel-catalysed electrochemical carboxylation of allylic acetates and carbonates

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ABSTRACT

The electrochemical carboxylation of a series of allylic acetates and carbonates was carried out in the presence of CO_2 under atmospheric pressure, with a catalytic amount of nickel-bipyridine complex, to afford the corresponding β,γ-unsaturated carboxylic acids. In the absence of nickel catalyst, alcohols were obtained.

1. Introduction

In the context of carbon–carbon bond formation by electrochemical methods, carboxylation reactions using carbon dioxide are particularly interesting. Carbon dioxide is an easily available, cheap and abundant starting material which is difficult to activate by conventional organometallic chemistry [1].

The electrochemical reduction of organic halides in the presence of CO_2 has been shown to afford the corresponding carboxylic acids under mild conditions [2]. This reaction has been reported to occur directly (without catalyst) in the case of aryl [3], benzyl [4–6] and allyl [7,8] halides in the presence of consumable magnesium or aluminum anodes. Electrochemical carboxylation of halides in the presence of metal complexes has also been reported, for example in the Ni(II)/phosphine catalysed carboxylation of bromobenzene [9,10], or in the Pd(II)/phosphine catalysed carboxylation of aryl triflates [11,12]. CO_2 incorporation into carbonyl compounds to afford α-hydroxyacids [13,14] or to alkynes and olefins has also been reported [15,16]. Although, the electrochemical carboxylation of organic halides has been thoroughly examined [2], that of allylic acetates or carbonates had not been previously reported.

2. Experimental

2.1. Preparation of starting compounds

2.1.1. Allylic acetates (1a–d)

Acetic anhydride (10 mL) is slowly added during 15 min to the alcohol (30 mmol) in pyridine (10 mL). After addition, the solution is stirred at room temperature during 15 h. The solution is hydrolysed with ice-water and extracted with petroleum ether. The organic layer is washed with water, with a 5% solution of sulfuric acid and with a saturated sodium carbonate solution, dried over $MgSO_4$ and evaporated under vacuum. The acetates (**1a–e**) are obtained in yields of about 90–95%.

2.1.2. Allylic carbonates (5a–d)

The alcohol (4 mmol), pyridine (8 mmol) and ethyl chloroformate (6 mmol) are added in a flask with a cooler. The solution is stirred at room temperature during 30 mn. The solution is hydrolysed with water and acidic water (HCl 0.1 M) and then extracted with petroleum ether. The organic layer is washed with a saturated sodium carbonate solution, with water, dried over $MgSO_4$ and evaporated under vacuum. The carbonates **(5a–d)** are obtained in yields of about 90–95%.

2.1.3. Catalyst

Ni(bipy)Br_2 is prepared following the procedure described in Ref [17]. A mixture of Ni(DME)Br_2 (30 mmol) (DME = 1,2

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dimethoxyethane) and 2,2′-bipyridine (30 mmol) in absolute ethanol is stirred overnight at room temperature. Ni(bipy)Br$_2$ is formed by precipitation. The complex is filtered on a Büchner, washed with acetone and dried at 70 °C under vacuum. The complex is obtained with 95% yield.

2.2. *Electrodes*

Electrodes for cyclic voltammetry were fabricated from 3 mm-diameter glassy carbon rods (Tokai Electrode Manufacturing Company, Tokyo, Japan, Grade GC-20) press-fitted into Teflon shrouds to provide planar, circular working electrodes with areas of 0.077 cm^2. Before use, the electrodes were cleaned with an aqueous suspension of 0.05 μm alumina (Buehler) on a Master-Tex (Buehler) polishing pad. All potentials are quoted with respect to an Ag/AgCl/3 M KCl in water reference electrode (0.036 V *vs* SCE).

2.3. *Cells and instrumentation*

Cells for cyclic voltammetry were identical to those described in publication [18]. Cyclic voltammograms were obtained with the aid of an AUTOLAB model PGSTAT12 potentiostat–galvanostat. The data from the above experiments were acquired and stored by locally written software, which controlled a data acquisition board installed in a personal computer.

2.4. *General electrolysis procedure*

The constant-current electrolyses were carried out in a single compartment cell (capacity 50 mL), such as described in Ref. [18], generally with Mg rod as the sacrificial anode (diameter 1 cm) and a carbon fibre cathode (apparent surface, 20 cm^2) around the counter electrode. Freshly distilled DMF (50 mL), *n*-Bu$_4$NBF$_4$ (6×10^{-3} M), Ni(bipy)Br$_2$ (3.0×10^{-3} M) and allylic acetate or carbonate **1** or **5** (1.5×10^{-2} M) were introduced into the cell under CO$_2$ flow at atmospheric pressure. The solution was stirred and electrolysed at room temperature, at a constant current of 30 mA (current density of 0.15 A dm^{-2} and 2–5 V between the rod anode and the carbon fibre cathode) until disappearance of the starting material (checked by GC analysis of aliquots). Generally, 2–7 F mol^{-1} of starting material were necessary to achieve a complete conversion. Controlled current electrolyses were carried out with the aid of a stabilized constant current supply (Sodilec, EDL 36.07).

2.5. *Identification and quantification of products*

After electrolysis, the crude solution is introduced in a round-bottom flask with K$_2$CO$_3$ and heated at 60 °C during 1 h. Methyl iodide is introduced and heated during 5 h. The mixture is hydrolysed with 0.1 M HCl saturated with NaCl, up to pH 1–2, extracted with CH$_2$Cl$_2$ and washed with H$_2$O. The dried (MgSO$_4$) organic layer is evaporated and the residue analysed by GC, GC-MS and ^{1}H NMR, and the compounds characterised and compared with spectra of authentic samples.

^{1}H NMR data were recorded on a Bruker 200 MHz spectrometer in CDCl$_3$; δ ppm was measured *vs* residual peak of the solvent. Identities of the electrolysis products were confirmed by means of a Hewlett-Packard 5890 Series II gas chromatograph coupled to a Hewlett-Packard 5971 mass-selective detector.

2.5.1. ***1e**, commercial compound*

For other compounds:*(E)*-2,6-Octadien-1-ol 3,7-dimethyl 1-acetate (**1a**): ^{1}H NMR 5.36 (1H, T, 7.3 Hz); 5.09 (1H, m); 4.56 (2H, D, 7.2 Hz); 2.08 (4H, m); 1.77 (3H, s); 1.68 (3H, s); 1.61 (3H, s)

(E)-2-Octen-1-ol 1-acetate (**1b**): ^{1}H NMR 5.74 (1H, m); 5.59 (1H, m); 4.5 (2H, Dd, 5.7 Hz, 0.76 Hz); 2.06 (5H, m); 1.29 (6H, m); 0.89 (3H, m). MS: 170 (M$^+$); 128; 110; 81; 67; 54; 43 (100%).

(*E*)-2,6-Heptadien-1-ol 2,4-dimethyl 1-acetate (**1c**): ^{1}H NMR 5.74 (1H, m); 5.26 (1H, Dd, 8.3 Hz, 1.14 Hz); 5.01 (2H, m); 4.44 (2H, s); 2.48 (1H, m); 2.08 (5H, m); 1.65 (3H, D, 1.4 Hz); 0.96 (3H, D, 6.7 Hz). ^{13}C NMR: 171.28, 137.24, 135.59, 129.2, 116.15, 70.59, 41.84, 32.58, 21.37, 14.49. MS: 182 (M$^+$); 141; 99; 81; 43 (100%).

(*E*,*E*)-2,6,10-Dodecatrien-1-ol 3,7,11-trimethyl 1-acetate (**1d**): ^{1}H NMR 5.33 (1H, m); 5.1 (1H, m); 4.58 (2H, D, 6.8 Hz); 2.06, (10H, m); 1.76 (3H, s); 1.68 (3H, s); 1.65 (3H, s); 1.60 (3H, s).

(E)-4,8-Dimethyl-3,7-nonadienoic acid (**2a**): ^{1}H NMR 5.24 (1H, Td, 5.95 Hz, 1.3 Hz); 5.03 (1H, m); 3.02 (2H, Dd, 6.2 Hz, 1.3 Hz); 1.69 (3H, D, 1.3 Hz); 1.61 (3H, s); 1.53 (3H, s). ^{13}C NMR: 178.14, 140.25, 132.52, 124.11, 116.04, 33.53, 32.51, 26.64, 26.08, 23.79, 18.02. MS: 182 (M$^+$), 139, 69 (100%), 41.

(E)-3-Nonenoic acid (**2b**): ^{1}H NMR 5.48 (2H, m); 3.01 (2H, d, 5.5 Hz); 1.96 (2H, m); 1.22 (6H, m); 0.81 (3H, t, 6.5 Hz). ^{13}C NMR 178.03, 135.97, 121.08, 38.11, 32.83, 31.75, 29.2, 22.9, 14.43. MS: 156 (M$^+$), 138, 96, 67, 55, 41 (100%).

(E)-3,5-Dimethyl-3,7-octadienoic acid (**2c**): ^{1}H NMR: 5.7 (1H, m); 4.8 (3H, m); 3.02 (2H, d, 6.5 Hz); 2.3 (1H, m); 1.96 (2H, t, 6.5 Hz); 1.6 (3H, m); 0.88 (3H, d, 6.5 Hz). ^{13}C NMR: MS: 168 (M$^+$), 127, 85, 85 (100%), 67

(E,E)-4,8,12-Trimethyl-3,7,11-tridecatrienoic acid (**2d**): ^{1}H NMR: 5.2 (1H, m); 5.03 (1H, m); 3.02 (2H, d, 7.2 Hz); 1.98 (8H, m); 1.69 (3H, d, 1.1 Hz); 1.61 (3H, s); 1.57 (3H, s); 1.57 (3H, s); 1.53 (3H, s). MS: +250 (M$^+$), 207, 121, 81, 69, 41 (100%).

(E)-3-Heptenoic acid (**2e**): ^{1}H NMR 5.49 (2H, m); 3.01 (2H, d, 5.75 Hz); 1.95 (2H, m); 1.35 (2H, m); 0.83 (3H, t, 7.3 Hz). MS: 128 (M$^+$), 110, 68, 55, 45, 39 (100%).

(*E*)-3,7-Dimethyl-2,6-octadien-1-yl carbonic acid ethyl ester (**5a**): ^{1}H NMR 5.38 (1H, m); 5.09 (1H, m); 4.63 (2H, Dd, 6.6 Hz, 0.76); 1.2 (2H, q, 7.2 Hz); 2.11 (4H, m); 1.76 (3H, s); 1.68 (3H, s); 1.6 (3H, s); 1.3 (3H, t, 7.2 Hz). 155.85, 150.14, 143.78, 136.82, 132.82, 124.1, 119.31, 32.77, 27.19, 26.24, 18.22, 14.86. MS: 226 (M$^+$), 121, 107, 93, 69 (100%), 43, 41.

(E)-2-Octenyl carbonic acid ethyl ester (**5b**): ^{1}H NMR 5.7 (1H, m); 5.54 (1H, m); 4.48 (2H, d, 5.5 Hz); 4.11 (2H, q, 7.2 Hz); 1.97 (5H, m); 1.23 (9H, m); 0.8 (3H, m). ^{13}C NMR: 154.07, 136.35, 122.28, 67.42, 62.79, 31.19, 30.34, 27.5, 21.48, 13.25. MS: 200 (M$^+$), 110, 81, 67, 54, 41.

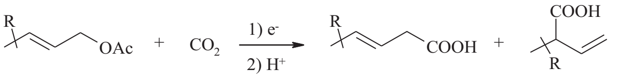
(E)-2,6-Heptadien 2,4-dimethyl carbonic acid ethyl ester (**5c**): ^{1}H NMR 5.72 (1H, m); 5.30 (1H, Dd, 8.3 Hz, 1.14 Hz); 5.01 (2H, m); 4.5 (2H, s); 4.2 (2H, q, 7.15 Hz); 2.48 (1H, m); 2.03 (2H, m); 1.67 (3H, s); 1.31 (3H, t, 7.2 Hz); 0.96 (3H, D, 6.7 Hz). ^{13}C NMR: 155.45, 137.1, 136.19, 128.75, 116.1, 73.82, 64.08, 41.72, 32.56, 20.44, 14.57, 14.33. MS: 212 (M$^+$), 171, 99, 81, 43 (100%).

(E,E)-3,7,11-Trimethyl 2,6,10-dodecatrien 1-yl carbonic acid ethyl ester (**5d**): ^{1}H NMR 5.31 (1H, m); 5.02 (1H, m); 4.57 (2H, d, 6 Hz), 4.1 (2H, Td, 6 Hz, 1.3 Hz); 1.95 (10H, m); 1.69 (3H, s); 1.65 (3H, s); 1.61 (3H, s); 1.52 (3H, s); 1.23 (3H, Td, 6 Hz, 1.3 Hz). MS: 294 (M$^+$), 204, 161, 133, 107, 93, 69, 41 (100%).

3. Results and discussion

3.1. *Electrocarboxylation of allylic acetates*

We were interested in the electrochemical reactivity of allylic acetates **1**, easily available from the corresponding alcohols, in the presence of CO$_2$, in order to obtain the corresponding β,γ-unsaturated carboxylic acids **2** and/or **2′**, according to Eq. (1).

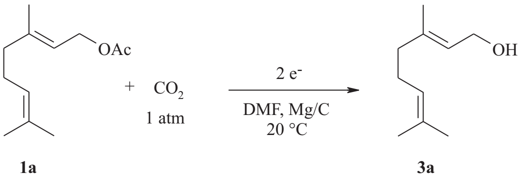


1 **2** **2′** (1)

Table 1
Preliminary electrochemical carboxylation tests of allylic acetate **1a** with CO_2 (1 atm) at constant current density 0.15 A dm^{-2}.

Entry	Catalyst (10 mol%)	Anode/cathode	F mol^{-1}	Consumption of **1a**	Selectivity		
					Carboxylic acids **2a** + **2′a**	Alcohol **3a**	Alkene by-product
1	–	Mg/C	3	63%	29%	47%	24%
2	–	Mg/stainless steel	3	66%	–	68%	32%
3	–	Mg/nickel foam	3	94%	7%	31%	62%
4	Ni(bipy)Br$_2$	Mg/C	7	100%	41%	–	59%
5	Ni(bipy)Br$_2$	Mg/C	4.5	80%	45%	–	55%
6	Ni(bipy)Br$_2$	Mg/C	2	42%	60%	–	40%

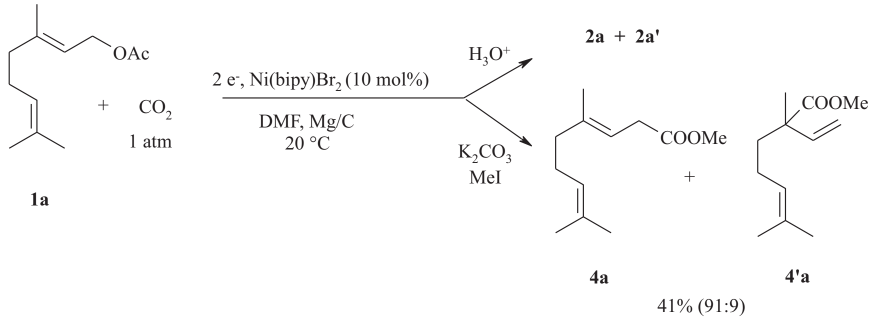
When the model geranyl acetate **1a** was reacted in the presence of carbon dioxide at atmospheric pressure in a one-compartment cell fitted with a Mg anode and a carbon fibre cathode in DMF at 20 °C, the direct electrolysis led to 63% consumption of **1a** after 3 F mol^{-1}, as summarized in Table 1, entry 1. However, the main product obtained was not the expected carboxylic acids **2a** and/or **2a′** (selectivity 29%) but the alcohol **3a** formed in 47% selectivity, in an unprecedented electrochemical process (Eq. (2)).



(2)

Alcohol **3a** was obtained in up to 68% selectivity when the electrolysis was run with stainless steel cathode and a Mg anode. This alcohol was issued from a hydrolysis process from **1a**. In these processes, the reaction involved also the electrochemical reduction of CO_2, mainly to oxalate as well as the formation of alkene reduction by-products. To obtain carboxylic acids **2** and/or **2′** without any catalyst, a Mg/C couple gave the best results, with a combined selectivity of 29%. The temperature was changed from 20 to 0 °C but no significant change occurred.

Several organometallic complexes were used as the catalysts, to get a more selective cleavage of the carbon-acetate bond. Among the complexes tested, Co(bipy)Cl$_2$ (bipy = 2,2′-bipyridine), Co(cyclam)Br$_2$ (cyclam = 1,4,8,11-tetraazacyclotetradecane), Co(OTf)$_2$ (OTf = CF_3SO_3), Ni(cyclam)(BF$_4$)$_2$ or Ni(OTf)$_2$ afforded low yields (<10%) of expected acids **2** and/or **2′**. The best conditions were obtained with Ni(bipy)Br$_2$ as the catalyst, used in 10 mol% with respect to **1a**. Under these conditions, the acetate **1a** could be carboxylated with an overall yield of 41%, as a 91:9 mixture of regioisomers (**2a**:**2′a**) (Eq. (3) and Table 1, entry 4). The carboxylation reaction reached 100% conversion of **1a** after 7 F mol^{-1}. After 2 or 4.5 F mol^{-1}, the conversions of **1a** were of 42% and 80% respectively (Table 1, entries 5, 6). Interestingly, in the presence of Ni(bipy)Br$_2$ as the catalyst, no formation of alcohol **3a** was observed. For ease of treatment and analysis, the carboxylic acids were isolated in the form of their corresponding methyl esters **4a** and **4a′** after *in situ* esterification of the carboxylates in the presence of K_2CO_3 and methyl iodide.



(3)

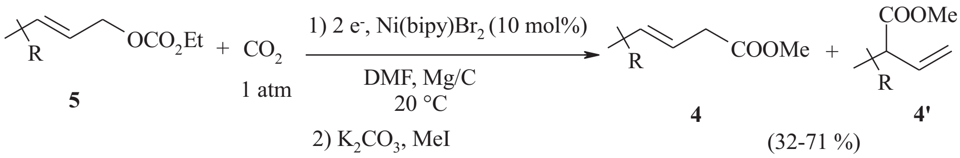
The Ni(II)-catalysed carboxylation was then extended to several other allylic acetates, as presented in Table 2.

The electrocarboxylation of several allylic acetates **1**, gave the corresponding carboxylic acids or esters in moderate to good yields.

(*E*)-2-Octen-1-ol acetate, **1b** (entry 2), afforded 3-nonene carboxylic acid in 54% yield as two regioisomers in 86:14 ratio. Allylic acetate **1c** (entry 3) afforded exclusively the ester **4c** in 55% yield. The highly hindered regioisomer **4′c** was not observed in this case. The farnesyl acetate **1d** (entry 4) gave the corresponding ester in 30% yield and a regioisomeric ratio **4d**:**4′d** of 84:16. The carboxylic esters **4e** and **4′e** were obtained in 63% yield and a ratio of 89:11 was observed when the allylic acetate **1e** (entry 5) was electrolysed in the presence of carbon dioxide and a catalytic amount of Ni(bipy)Br$_2$. The faradaic consumption was of 4–7 F mol^{-1}, indicating a concomitant CO_2 electroreduction under these conditions.

3.2. Electrocarboxylation of allylic carbonates

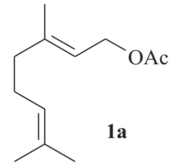
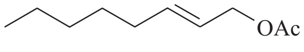
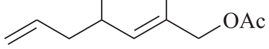
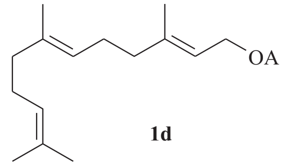
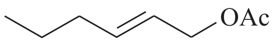
The electrochemical carboxylation was also examined with a series of allylic carbonates **5**. As indicated in Eq. (4), and in Table 3, the corresponding homoallylic carboxylic esters **4** and **4′** were obtained in 32–71% yields in the presence of the Ni(bipy)Br$_2$ as the catalyst. In the absence of the metal catalyst, the formation of the alcohol **3** was again observed as the main reaction product.



(4)

When the allylic ethyl carbonates **5** were electrolysed in the presence of a catalytic amount of Ni(bipy)Br$_2$ at 1 atm of carbon

Table 2
Electrochemical carboxylation of allylic acetates **1** with CO_2 (1 atm) in the presence of Ni(bipy)Br_2 as the catalyst (10 mol%) in DMF at 20 °C with Mg/C electrodes, at constant current density of 0.15 A dm^{-2}.

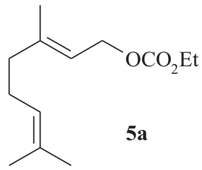
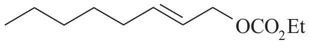
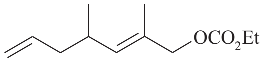
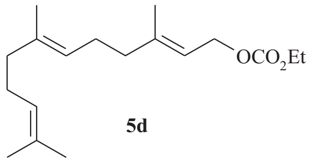
Entry	Substrate	F mol^{-1}	Consumption of **1**	Yield of esters **4** + **4′**	Regioisomer ratio **4**:**4′**
1	 **1a**	7	100%	41%	91:9
2	 **1b**	4	96%	54%	86:14
3	 **1c**	7	85%	55%	100:–
4	 **1d**	7	78%	30%	86:14
5	 **1e**	4	100%	63%	89:11

dioxide, the corresponding carboxylic acids were formed. Geranyl carbonate **5a** (entry 1) was carboxylated and the corresponding carboxylic esters were obtained in 71% yield of with a regioisomer ratio **4a**:**4′a** of 91:1. Allylic carbonate **5b** (entry 2) afforded carboxylic esters **4b** and **4′b** in 32% yield in a ratio of 57:43. The carbonate **5c** gave 66% yield of carboxylic ester **4c**, the regioisomer **4′c** not being observed (entry 3). The farnesyl carbonate **5d** (entry 4) afforded 46% yield of carboxylic acids with a ratio of regioisomers **4d**:**4′d** of 84:16. The isomeric ratio of carboxylic acids issued from starting acetates or carbonates is sensibly the same.

3.3. Cyclic voltammetry studies

The cyclic voltammetry of this electrochemical system was carried out in the particular case of geranyl acetate, **1a**. Fig. 1 indicates the electrochemical behaviour of geranyl acetate **1a** alone (curve

Table 3
Electrochemical carboxylation of allylic carbonates **5** with CO_2 (1 atm) in the presence of Ni(bipy)Br_2 as the catalyst (10 mol%) in DMF at 20 °C with Mg/C electrodes at constant current density of 0.15 A dm^{-2}.

Entry	Substrate	F mol^{-1}	Consumption of **5**	Yield of esters 4 and **4′**	Regioisomer ratio **4**:**4′**
1	 **5a**	4	100%	71%	91:9
2	 **5b**	5	88%	32%	57:43
3	 **5c**	7	100%	66%	100:–
4	 **5d**	4	90%	46%	84:16

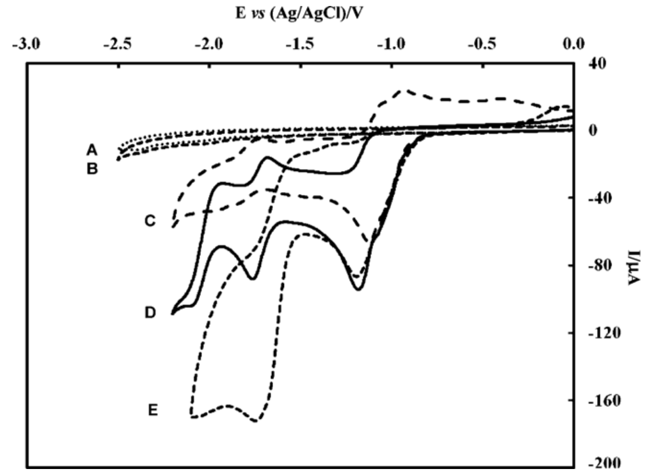
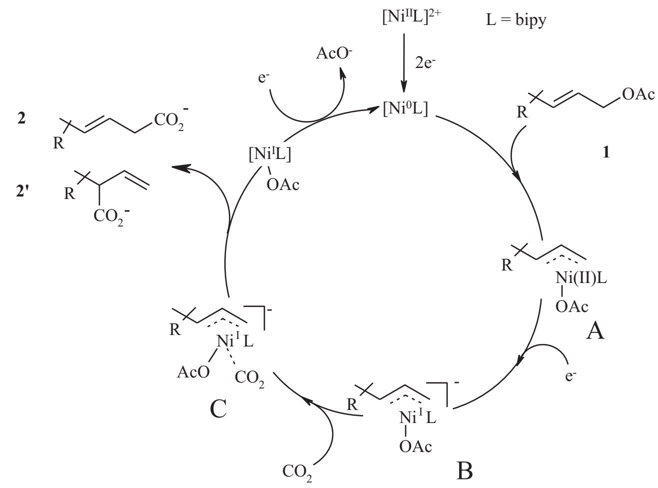


Fig. 1. Cyclic voltammograms recorded on a glassy carbon electrode (area = 0.07 cm^2) at 100 mV s^{-1} in: (A) DMF containing 0.1 M of tetrabutylammonium bromide; (B) solution (A) after addition of 10 mM of geranyl acetate; (C) solution (A) after addition of 4 mM of Ni(bipy)Br_2; (D) solution (C) with 20 mM of geranyl acetate; (E) solution (D) saturated with CO_2.



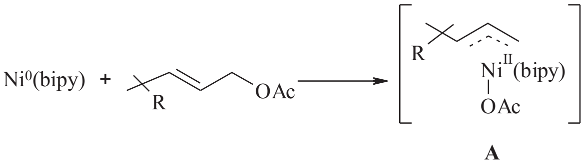
Scheme 1.

B). No reduction occured between 0 and −2.5 V *vs* Ag/AgCl. Curves C and D of the Fig. 1 show the behaviour of Ni(bipy)Br_2 alone and in the presence of **1a**, respectively. Ni^{II}(bipy)Br_2 shows a reversible two-electron reduction at −1.1 V *vs* Ag/AgCl, which corresponds to the formation of Ni^0(bipy) (curve C). Upon addition of 1–5 equivalents of **1a** (curve D, 5 equiv.), the reduction occurs at −1.2 V *vs* Ag/AgCl and becomes irreversible. Its intensity corresponds to a three-electron wave independently of the concentration of **1a** (1 equiv. or more). A reversible reduction is also observed in curve D at −1.75 V *vs* Ag/AgCl, for the further reduction of the Ni-bipy complex [19]. Curve E corresponds to curve D in the presence of CO_2 and indicates a first three-electron reduction wave at −1.2 V and a further irreversible catalytic wave at −1.75 V.

3.4. Mechanistic proposal

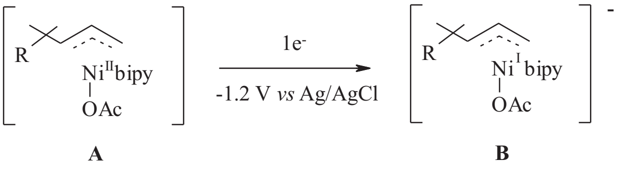
According to these electrochemical data and to the redox behaviour of Ni(bipy)Br_2, we propose that the first step of the carboxylation reaction is the reduction of the Ni^{II} complex to Ni^0 at −1.1 V *vs* Ag/AgCl (Eq. (5)), in agreement with literature data [19]. In the presence of the eletrogenerated Ni^0 complex, an oxidative addition on the acetate group of **1a** takes place, generating a π-allyl Ni^{II} intermediate complex, **A** (Eq. (6)).

$$\mathrm{Ni^{II}(bipy)Br_2} \xrightarrow[-1.1\ \mathrm{V}\ vs\ \mathrm{Ag/AgCl}]{2e^-} \mathrm{Ni^{0}(bipy)} + 2\mathrm{Br^-} \qquad (5)$$



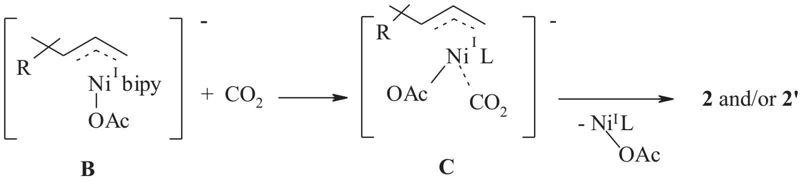
(6)

A one-electron reduction of **A** may occur at −1.2 V *vs* Ag/AgCl to give intermediate **B** (Eq. (7)). This step explains the three-electron wave observed at this potentiel in curve (D).



(7)

The coordination of CO_2 to the Ni-complex **B** forms an intermediate **C** that can evolve to the carboxylates **2** (and/or **2′**) (Eq. (8)), releasing a Ni^I-complex which can be further reduced to Ni^0 to restart the catalytic cycle.

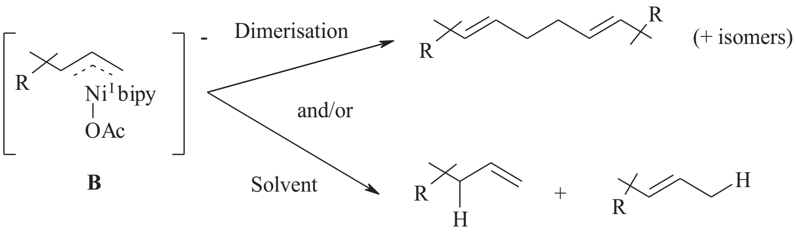


(8)

The proposed catalytic cycle is presented in Scheme 1.

After hydrolysis, the carboxylic acids **2** and **2′** are formed. The ratio of carboxylated regioisomers **2** and **2′** mainly depends on the steric hindrance of the intermediates **A**, **B** and **C**.

In the absence of CO_2, intermediate **B** may also undergo dimerisation or protonation by the solvent (Eq. (9)). These dimers or reduced compounds (according to the GC-MS data) constitute the by-products of the reaction.



(9)

It is to note that carbon dioxide can also be reduced, and its reduction can also be catalysed by the Ni^{II} complex [20,21]. The Ni-catalysed reduction of CO_2 affords mainly oxalate dianion, and may also form carbon monoxide and carbonate [22,23].

4. Conclusions

In conclusion, the electrochemical reductive carbon dioxide incorporation into a series of allylic acetates and carbonates proceeded in moderate to good yields under mild conditions in the presence of $Ni(bipy)Br_2$ as the catalyst. In the absence of the catalyst, the main reaction products were the corresponding alcohols.

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