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Electrochemical alkyl transfer reaction from trialkylboranes to polyhalo compounds

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Abstract

Reduction of decyl dichloro- and trichloroacetate, under mild electrolysis conditions by using the sacrificial anode process, affords α-chlorocarbanions which readily react with trialkylboranes to give alkylated products in a one step reaction.

Keywords: Electrochemistry; Alkylation; Trialkylborane; Polyhalo compound

1. Introduction

The ability of trialkylboranes to transfer alkyl groups is a useful feature and has been advantageously applied to C—C bond formation. The alkyl transfer from boron to electrophilic carbon is usually promoted by an attack of a nucleophile. If this reagent bears a leaving group, then in the next step, an intramolecular substitution would occur easily. Thus, the reaction is often conducted at low temperature, under basic conditions for the preparation and the stability of the α-halocarbanion. In the chemical route reported by Brown et al., reaction of R_3B with α-haloesters, nitriles, ketones [1] and haloforms [2] is induced by a sterically hindered strong base. The process involves (i) formation of the carbanion by removal of an acidic hydrogen (ii) formation of the organoborate (iii) 1,2-alkyl transfer (Scheme 1, path A). Electroreduction chemistry is also a convenient method to generate nucleophilic species in non-basic conditions. Some reactions involving electrochemical alkyl transfer reaction of trialkylborane have been reported in the literature. Suzuki et al. [3] reported the electrochemical alkylation of compounds containing acidic hydrogen by trialkyboranes. Involvement of radicals [4] in the mechanism has been suggested. Efficient reactions of trialkylboranes with α,β-unsaturated esters to give 1,4-addition products [5], as well as reactions with carbonyl compounds [6] to give alcohols or ketones, have also been described. Reduction of *gem*-dihalides under electrochemical conditions (Scheme 1, path B) is a straightforward alternative to obtain α-halocarbanions. We have reported that reactions of polyhalo compounds with electrophiles can be conducted in an undivided electrochemical cell by using the sacrificial anode procedure [7]. Under these conditions, selective reduction of easily electro-reducible compounds (CCl_4, $PhCCl_3$, CCl_3CO_2Me, $PhCHCl_2$, CH_2Br_2) affords stable α-halogenated anions which then react with ketones [7], aldehydes [8] and electron deficient olefins [9]. These reactions usually take place at room temperature. The use of the sacrificial anode process involves the release of metallic cations in the reaction mixture and thus affords formation of metalated species, which do not lead to carbene species. The reaction with trialkylboranes has not been investigated thus far.

Here, we report mainly our first results on the alkylation of decyl dichloro- and trichloroacetate with triethylborane (Scheme 2) under simple, mild electrolysis in *N*,*N*-dimethylacetamide (DMAc). A widening of the method to sterically different trialkylboranes and *gem*-polyhalo compounds is also introduced at the end of this paper.

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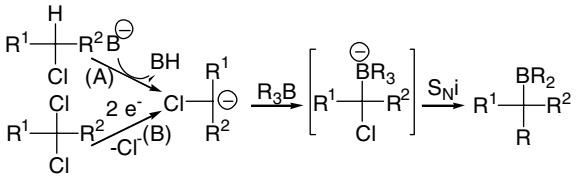
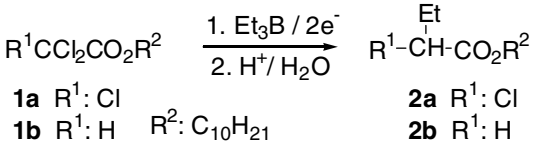


Scheme 1.

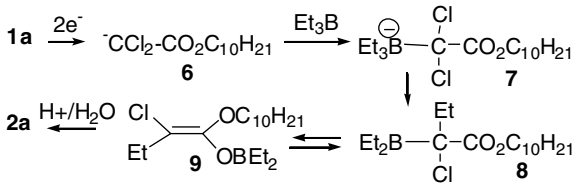


Scheme 2.

2. Results and discussion

Reactions are carried out in an undivided cell, equipped with a stainless steel grid (area 30 cm^2) as the cathode and a metal rod (see below) as the anode. Trialkylborane (4 mmol; 1 N in THF) is added at room temperature in one portion to a stirred solution of the organic halide (3.5 mmol) and NBu_4Br as supporting electrolyte in DMAc (20 mL). Electrolysis is run at constant current density (0.66 A dm^{-2}). The temperature is allowed to rise gradually to ca. 40 °C. The cathode potential throughout the electrolysis is at −1.2 V vs. SCE. The current is turned off after consumption of the polyhalo compound, as monitored by GC analysis. Then the reaction is stirred for 20 min and quenched by HCl. After the work up, the products are purified by column chromatography.

We first examined reaction of decyl trichloroacetate **1a** with triethylborane. The obtained results are summarized in Table 1 for each anode routinely used in electrosynthesis. With every tested anode, reduction of decyl trichloroacetate occurs and decyl 2-chlorobutanoate **2a** is formed as the main product along with, depending on the conditions, decyl 2-ethylbutanoate **3**, decyl 2,2-dichloroacetate **1b**, decanol **4** and succinate **5**. Formation of **2a** can be explained (Scheme 3) by bi-electronic reduction of **1a** to give carbanionic dihalide **6** which then reacts with Et_3B to form organoborate **7**. Then 1,2-migration occurs and the resulting α-chloroborylester **8** tautomerizes to alkenyloxyborane **9**.

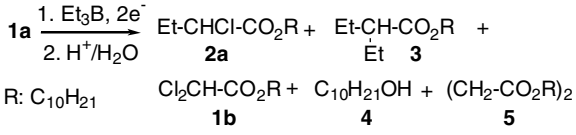


Scheme 3.

Under the above described electrochemical conditions, only a small excess of R_3B (1.14 equiv.) is required. Only one out of the three alkyl groups of the trialkylborane is transferred to the organic halide. Chemical yields are higher with zinc and magnesium (Table 1, entries 1 and 4) as the anode than with iron and aluminum. Also, taking into account that 1 h is required for full consumption of the starting halo compound, some chemical reduction should occur at the polarized zinc or magnesium anode accounting for the shorter reaction times. Reduction products are also formed in yields depending on the anode. Product **1b** is formed by simple protonation of **6** and this importantly occurs with iron and aluminum (Table 1, entries 2 and 3). The reduction of the ester group leading to **4** has already been observed with magnesium [10] as the anode. By holding the electrosynthesis for 2 extra hours, decyl butanoate **2b**, which is not observed otherwise, is obtained as the main product (isolated yield 59%). Two pathways (Scheme 4) can be proposed to account for the formation of **2b**. In the first pathway, reduction of α-chloroborylester **8** occurs and decyl propanoate **2b** is obtained after protonation. The second pathway is based on the in situ formation of **2a** by protonation of alkenyloxyborane **9** because of the presence of residual water in the solvent, followed by bi-electronic reduction to **12**.

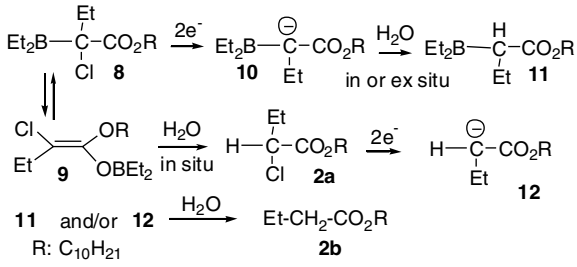
Attempts to trap intermediates **10** and **12**, under electrolysis conditions, have been conducted after full consumption of starting material **1a** by adding methyl vinyl ketone (MVK) as the electrophile. The expected coupling

Table 1
Anode effect in the reaction of decyl trichloroacetate **1a** with triethylborane



Entry[a]	Anode	Reaction time	Yield (%)[b] **2a**	Other products (%)[c]
1	Zn	30 min	59	**3**: 8
2	Fe	1 h 15 min	31	**1b**: 20; **5**: 20
3	Al	1 h	36	**1b**: 35
4	Mg	30 min	45	**1b**: 13; **4**: 6

[a] Decyl trichloroacetate 3.5 mmol; Et_3B 4 mmol (1 N in THF); solvent: DMAc 20 mL; supporting electrolyte NBu_4Br; temperature: 20 °C; stainless steel cathode; $I = 0.2$ A; anticipated reaction time 1 h.
[b] Isolated yield.
[c] Determinated by GC.



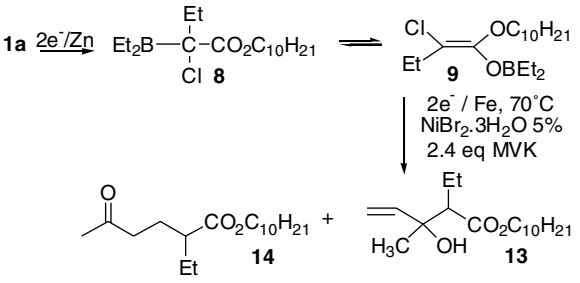
Scheme 4.

product did not occur and only **2b** was obtained. The sacrificial anode process associated with transition metal as the catalyst is also an efficient method to allow activation of organic halides, in mild reaction conditions, and further reaction with electrophile. We thus decided to perform a coupling reaction of α-chloroborylester **8** and/or alkenyloxyborane **9** and methyl vinyl ketone under the conditions described for the conjugate addition reaction [11] of organic halides with activated olefins mediated by nickel catalysis. $NiBr_2 \cdot 3H_2O$ as the catalyst precursor, pyridine as the cosolvent and MVK (2.5 equiv.) were successively added after full consumption of **1a** (Scheme 3). The zinc anode was replaced by iron which is the most appropriate for the conjugate addition reaction [11] and electrolysis was run at 70 °C. A mixture of 1,2- and 1,4-adducts (**13** and **14**) in a 34/66 ratio in GC was obtained and these were isolated in 13% and 34% yield, respectively (Scheme 5).

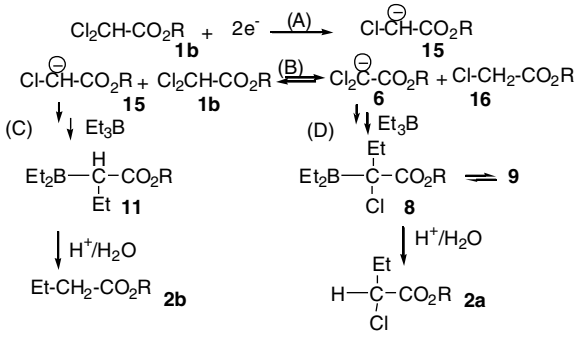
We also investigated the behaviour of decyl dichloroacetate **1b** with triethylborane in these reaction conditions. Results are reported in Table 2. Decyl butanoate **2b** is obtained as the main product along with decyl 2-chlorobutanoate **2a**, decyl chloroacetate **16**, decyl acetate **17** and decanol. Comparison of zinc and magnesium has been achieved. Full consumption of the starting material **1b** is reached after 1 h which means that no chemical reduction occurs at the surface of the anode (Table 2, entries 2 and 5). The best result is again obtained with zinc as the consumable anode. In the presence of a magnesium rod, reduction of **1b** occurs, **2b** is obtained but more decanol (Table 2, entry 5) is observed. An explanation to account for the uncontrolled formation of decanol, would be the chemical reduction of the ester group at the surface of the anode and this occurs more importantly when a magnesium rod is used. Formation of desired compound **2b** and side product **2a** is depicted in Scheme 6.

Reduction of decyl dichloroacetate **1b** affords formation of the carbanionic species **15** (A) which either reacts with triethylborane (C) to give expected compound **2b** or reacts (B) with its precursor **1b** to give carbanionic species **6**. This latter also undergoes reaction (D) with triethylborane to give compound **2a**. Compound **2a** coming from **8** and **9** can be transformed into **2b** by maintaining electron supply (Scheme 4) for 15 min (Table 2, entry 3). Then we have examined whether the reaction can be extended to other organoboranes and *gem*-dihalo compounds from commercial sources. Particularly the length and the steric hindrance of the transferable groups as well as steric and electronic effects on the electrophilic carbon of the organic halide have been considered.

In Table 3 are reported the results we have obtained until now. A noticeable disparity in chemical yields was found between sterically different trialkylboranes (entries 4, 5 vs. 1) as well as sterically hindered *gem*-polyhalide derivatives (entries 6–8 vs. 1). Also, yields decrease with

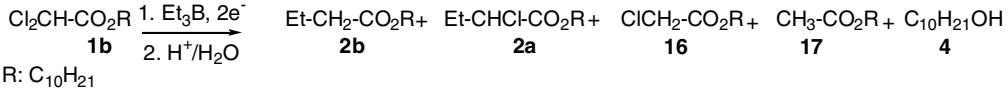


Scheme 5.



Scheme 6.

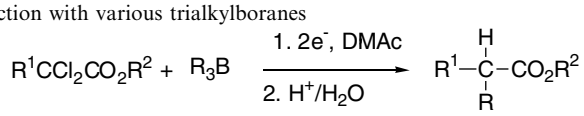
Table 2
Reaction of decyl dichloroacetate with triethylborane



Entry[a]	Anode	Time	% GC						Isolated yield%
			2b	**2a**	**16**	**17**	**4**	**1b**	**2b**
1	Zn	30 min	55.5	7	14	1.5	0	23	
2	Zn	1 h	76.5	7	14	1.5	0	1	
3	Zn	1 h 30 min	86.5	0	0	13.5	0	0	71
4	Mg	30 min	31	4.5	18	5	15	20	
5	Mg	1 h	44	1	10	9	32	2	23

[a] Decyl dichloroacetate 3.5 mmol (0.14 N); Et_3B 4 mmol (1 N in THF); solvent: DMAc; supporting electrolyte: NBu_4Br; 20 °C; stainless steel cathode; $I = 0.2$ A; anticipated reaction time: 1 h.

Table 3
Reaction with various trialkylboranes



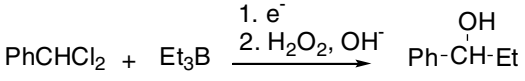
Entry	Chlorinated compounds[a]	R_3B	No.	Isolated yield (%)
1	$Cl_2CH–CO_2C_{10}H_{21}$	Et_3B	**2b**	71
2	$Cl_2CH–CO_2C_{10}H_{21}$	nBu_3B	**18**	62
3[b]	$Cl_2CH–CO_2C_{10}H_{21}$	$nOct_3B$	**19**	47
4[b]	$Cl_2CH–CO_2C_{10}H_{21}$	$(C_6H_{11})_3B$	**20**	30
5	$Cl_2CH–CO_2C_{10}H_{21}$	$secBu_3B$	**21**	32
6	$Cl_3C–CO_2C_{10}H_{21}$	Et_3B	**2a**	59
7	$Cl_2(CH_3)C–CO_2C_{10}H_{21}$	Et_3B	**22**	47
8	$Cl_2(OCH_3)C–CO_2CH_3$	nBu_3B	**23**	40[c]

[a] Chlorinated compound 3.5 mmol; R_3B 4 mmol (1 N in THF or Et_2O); solvent DMAc; supporting electrolyte NBu_4Br; zinc anode; temperature 20 °C; Intensity 0.1–0.2 A.
[b] Trialkylboranes prepared by hydroboration reaction from corresponding alkenes.
[c] Determinated by standard internal GC.

the lengthening of the transferable alkyl group (entries 1–3). However, the successful alkylation of decyl 2,2- dichloropropanoate (entry 7) and methyl dichloromethoxy-acetate (entry 8) indicates that the reaction is not confined to the alkylation of decyl trichloroacetate and dichloroacetate and allows envisaging further extension to various polyhalo compounds.

3. Conclusions

We have described, in this paper, the reaction of polyhalogenated derivatives with trialkylboranes to afford α-alkylated esters with moderate to good yields. The reactions are conducted in an undivided cell fitted with a consumable zinc rod under mild electrochemical conditions. Products of the reaction of decyl dichloroacetate and trichloroacetate with alkylboranes are indeed accessible under Brown reaction conditions from chloroacetates and dichloroacetates. However, under electrolysis, carbanions are smoothly obtained and the use of strong base is discarded. Alkylation of polyhalo compounds such as decyl 2,2-dichloropropanoate and methyl dichloromethoxyacetate illustrates the potential of this new method. Recently, we have found that the method can also be applied to the alkylation of benzal halides.



Study of the reaction conditions (temperature, solvent, concentration of starting material) and extension to other pohyhalide derivatives (haloform, $PhCHCl_2$, $RRCCl_2$) to define the scope and limitation of the method are underway.

4. Experimental

All solvents and reagents were purchased from commercial sources and used as received unless indicated. *N*,*N*-dimethylacetamide (DMAc) is of spectrophotometric grade 99+%- water content 0.05% and is stored under an argon atmosphere. Triethylborane (1 M in THF) tri-*n*butylborane (1 M in diethyl ether) and sec-butylborane (1 M in Et_2O) are the commercial products. Tricyclohexylborane and trioctylborane have been prepared by hydroboration [12] from the corresponding alkene and standardized by oxidation with alkaline-hydrogen peroxide. Decyl dichloroacetate and decyl trichloroacetate were prepared by esterification from dichloro- and trichloroacethyl chloride. GC analysis was carried out using a 25 m DB-1 capillary column. ^1H and ^{13}C spectra were recorded on a Bruker Avance 300 spectrometer unless indicated. Mass spectra were obtained on a GCQ Thermoquest spectrometer coupled to a chromatograph fitted with a 25 m CPSIL5 CB capillary column. The electrochemical cell has been previously described [7].

4.1. Electrochemical procedure for the preparation of decyl butanoate (*2b*)

The reaction was conducted in an undivided cell equipped with stainless steel grid (area 30 cm^2) as the cathode and zinc rod as the anode. Triethylborane (4 mL, 4 mmol) was added at 20 °C in one portion to a stirred solution of decyl dichloroacetate **1b** (0.94 g; 3.5 mmol) and tetrabutylammonium bromide (0.20 g; 0.62 mmol) as supporting electrolyte in DMAc (20 mL). Electrolysis was run at constant current density (0.66 A dm^{-2}). Temperature was allowed to rise gradually to 40 °C. The current was turned off after consumption of **1b** and after the reduction of side product, decyl 2-chlorobutanoate **2a**, controlled by GC monitoring. Then reaction was stirred for 15 min and quenched by HCl 1 N. The mixture was then extracted twice with diethylether (2 × 40 mL), dried over $MgSO_4$, evaporated to dryness. Purification by column chromatography over silica gel (eluting with 9.5:0.5 pentane/ethyl ether) gave **2b** (0.57 g; 71%) as a liquid.

4.1.1. Decyl butanoate (*2b*)

Colourless oil; ^1H NMR (300 MHz, $CDCl_3$) δ 4.01 (t, 2H, $J = 6.72$ Hz), 2.23 (t, 2H, $J = 7.40$ Hz), 1.60 (m, 4H), 1.36–1.12 (m, 14H), 0.90 (t, 3H, $J = 7.40$ Hz), 0.83 (t, 3H, $J = 6.63$ Hz). ^3C NMR (75.46 MHz, $CDCl_3$) δ 173.8, 64.4, 36.3, 31.9, 29.5 (2C), 29.3, 29.2, 28.7, 25.9, 22.7, 28.5, 14.1, 13.7. EIMS (*m*/*z*), 229, 213, 185, 157, 140, 126, 111, 97, 89, 83, 69, 55 (100%). IR (NaCl) *v* 2958, 2922, 2856, 1740, 1466, 1254, 1181, 723 cm^{-1}. Anal. Calc. for $C_{14}H_{28}O_2$: C, 73.63; H, 12.36. Found: C, 73.58; H, 12.32%.

4.1.2. Decyl 2-chlorobutanoate (*2a*)

Prepared according to the procedure described above. Colourless oil; ^1H NMR (200 MHz, $CDCl_3$) δ 4.15 (dd,

1H, $J = 7.41$ and 6.34 Hz), 4.10 (t, 2H, $J = 6.65$ Hz), 1.93 (m, 2H), 1.59 (m, 2H), 1.36–1.13 (m, 14H), 0.96 (t, 3H, $J = 7.32$ Hz), 0.81 (t, 3H, $J = 6.73$ Hz). ^{13}C NMR (50.32 MHz, $CDCl_3$) δ 169.4, 65.7, 58.7, 31.6, 29.2 (2C), 29.0, 28.9, 28.2, 28.1, 25.5, 22.4, 13.8, 10.2. EIMS (*m/z*), 265, 263, 228, 199, 185, 163, 141, 123, 97, 83, 69, 55 (100%). IR (NaCl) *v* 2958, 2927, 2855, 1742, 1461, 1175, 735 cm^{-1}. ^{1}H and ^{13}C spectra are consistent with authentic sample prepared from decyl dichloroacetate and triethylborane following procedure reported in literature [12].

4.1.3. Decyl 3-methylpentanoate (*21*)

Prepared according to the procedure described above. Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.09 (t, 2H, $J = 6.70$ Hz), 2.33 (dd, 1H, $J = 14.55$ and 6.15 Hz), 2.13 (dd, 1H, $J = 14.55$ and 8.10 Hz), 1.92 (m, 1H), 1.65 (m, 2H), 1.39–1.28 (m, 16H), 0.95 (t, 3H, $J = 7.20$ Hz), 0.90 (m, 6H). ^{13}C NMR (75.46 MHz, $CDCl_3$) δ 173.5, 64.3, 41.6, 32.0, 31.9, 29.5 (2C), 29.3 (2C), 29.2, 28.7, 26.0, 22.7, 19.3, 14.1, 11.3. EIMS (*m/z*), 257, 227, 185, 157, 141, 117 (100%), 97, 83, 69, 55; IR (NaCl) *v* 2958, 2927, 2855, 1737, 1464, 1181, 722 cm^{-1}.

4.1.4. Decyl 2-methylbutanoate (*22*)

Prepared according to the procedure described above. Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.09 (t, 2H, $J = 6.59$ Hz), 2.40 (sextuplet, 1H, $J = 6.98$ Hz), 1.78–1.60 (m, 3H), 1.50 (m, 1H), 1.40–1.25 (m, 14H), 1.17 (d, 3H, $J = 6.98$ Hz), 0.92 (m, 6H). ^{13}C NMR (75.46 MHz, $CDCl_3$) δ 176.8, 64.3, 41.2, 31.9, 29.5 (2), 29.3, 29.2, 28.7, 26.8, 25.9, 22.7, 16.7, 14.1, 11.6. EIMS (*m/z*), 243, 185, 158, 140, 111, 103 (100%), 97, 83, 75, 69, 57. IR (NaCl) *v* 2958, 2926, 2856, 1736, 1463, 1381, 1184, 1153, 734 cm^{-1}. Anal. Calc. for $C_{15}H_{30}O_2$: C, 74.32; H, 12.47. Found: C, 74.40; H, 12.50%.

4.1.5. Methyl 2-methoxyhexanoate (*23*)

Prepared according to the procedure described above. Colourless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.76 (m, 4H), 3.38 (s, 3H), 1.73–1.69 (m, 2H), 1.43–1.22 (m, 4H), 0.90 (m, 3H); ^{13}C NMR (75.46 MHz, $CDCl_3$) δ 173.4, 80.6, 58.1, 51.8, 32.5, 27.2, 22.4, 13.9. EIMS (*m/z*), 159, 131, 115, 101, 87 (100%), 72. IR (NaCl) *v* 2957, 2874, 1752, 1459, 1437, 1200, 1167, 735 cm^{-1}. ^{1}H and ^{13}C spectra are consistent with literature data [13].

4.2. Preparation of decyl 2-ethyl-3-hydroxy-3-methylpent-4-enoate (*13*) and decyl 2-ethyl-5-oxohexanoate (*14*)

The reaction was first conducted in an undivided cell equipped with stainless steel grid (area 30 cm^2) as the cathode and zinc rod as the anode. Triethylborane (1.14 equiv., 4 mL) was added at 20 °C in one portion to a stirred solution of decyl trichloroacetate **1a** (3.31 mmol; 1.00 g) and tetrabutylammonium bromide (0.20 g; 0.62 mmol) as supporting electrolyte in DMAc (20 mL). Electrolysis was run at constant current density (0.66 A dm^{-2}). Temperature was allowed to rise gradually to 40 °C. The current was turned off after consumption of **1a**. Zinc rod was then replaced by iron rod. Pyridine (2.50 mL) as co-solvent was added in the cell followed by introduction of $NiBr_2 \cdot 3H_2O$ (0.33 mmol; 72 mg) and methyl vinyl ketone (0.58 g; 8.27 mmol). The mixture was heated at 70 °C and electrosynthesis was run at constant current intensity (0.66 A dm^{-2}). The reaction was monitored by GC and current was turned off after consumption of decyl 2-chlorobutanoate which is the protonated form of α-chloroborylester **8** and alkenyloxyborane **9**. Then the reaction mixture was quenched by HCl 1 N, extracted twice with diethylether (2×40 mL), dried over $MgSO_4$ and evaporated to dryness. After purification by column chromatography on silica gel (230–400 Mesh, eluent: pentane/diethyl ether 9.5:0.5), 0.13 g (13%) of **13** and 0.34 g (34%) of **14** were obtained.

4.2.1. Decyl 2-ethyl-3-hydroxy-3-methylpent-4-enoate (*13*)

Compound **13** is a mixture of two diastereoisomers in 1/1 ratio:

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.93 (dd, 1H, *J trans*: 17.2 Hz, *J cis*: 10.65), 5.77 (dd, 1H, *J trans*: 17.2 Hz, *J cis*: 10.73), 5.32 (dd, 1H, *J trans*: 17.2 Hz, *J gem*: 1.55), 5.27 (dd, 1H, *J trans*: 17.2 Hz, *J cis*: 1.27 Hz), 5.12 (dd, 1H, *J trans*: 10.73 Hz, *J gem*: 1.55 Hz), 5.04 (dd, 1H, *J trans*: 10.65 Hz, *J gem*: 1.27 Hz), 4.08 (t, 2H, OCH_2, *J*: 6.66 Hz), 4.15 (t, 2H, OCH_2, *J*: 6.66 Hz), 3.20 (s, $2 \times OH$), 2.39 (dd, 1H, *J*: 10.58 Hz, *J*: 4.76 Hz), 2.34 (dd 1H, *J*: 10.72 Hz, *J*: 4.31 Hz), 1.8–1.6 (m, $2 \times 4H$), 1.29–1.26 (m, $2 \times 17H$), 0.90 (m, $2 \times 6H$). EIMS (*m/z*), 299 (M+1), 281 (M−18), 271, 251, 229, 185, 143, 131, 125, 111, 97, 89, 73 (100%), 55.

4.2.2. Decyl 2-ethyl-5-oxohexanoate (*14*)

^{1}H NMR (300 MHz, $CDCl_3$) δ 3.99 (2H, t, *J*: 6.67 Hz), 2.36 (2H, t, *J*: 6.63 Hz), 2.19 (1H, m), 2.04 (3H, s), 1.72 (2H, q, *J*: 7.34 Hz), 1.54 (2H, m), 1.45 (2H, m), 1.28–1.10 (14H, m), 0.80 (6H, m). ^{13}C NMR (75.46 MHz, $CDCl_3$) δ 175.6, 64.2, 46.3, 41.0, 31.8, 29.8, 29.4 (3C), 29.2, 29.1, 28.6, 25.9, 25.5, 25.4, 22.6, 14.0, 11.6. EIMS (*m/z*), 298, 241, 185, 159, 141, 112 (100%), 97, 81, 55. IR (NaCl) 1720 cm^{-1}. Anal. Calc. for $C_{18}H_{34}O_3$: C, 72.44; H, 11.48. Found: C, 72.41; H, 11.46%.

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