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Electrochemical vicinal dichlorination and dibromination of unactivated alkenes using TMSX as the halogen source

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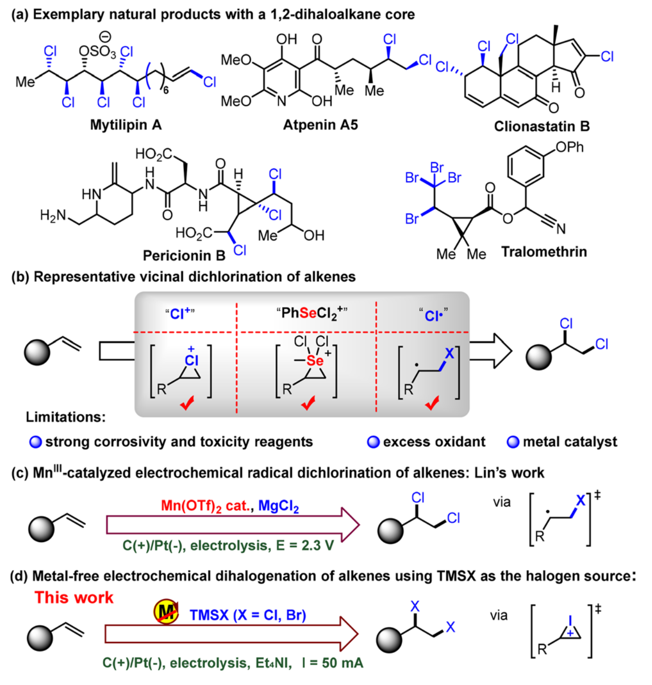
An environmentally friendly electrochemical method for vicinal dichlorination of unactivated alkenes has been developed using TMSCl as a chlorine source, facilitated by Et_4NI-mediated electrophilic addition under undivided electrolytic conditions. This sustainable and clean electrochemical system provides an efficient route to a series of dichloroalkanes. Replacing TMSCl with TMSBr enables the dibromination of unactivated alkenes. Mechanistic experiments indicate that this electrochemical strategy proceeds *via* an ionic process, where *in situ* generation of iodide ions and subsequent chlorination reactions drive the formation of dichloroalkanes.

Organochlorides, particularly dichlorinated compounds, are highly valuable substances in both industrial and academic contexts. They not only represent key structural motifs in many pharmaceutical molecules and natural products (Scheme 1(a)), but also serve as versatile intermediates in organic transformations.[1,2] Alkenes, which can be sourced in bulk from renewable resources and petrochemical feedstocks, are considered one of the most cost-effective and widely used organic building blocks in synthetic chemistry. Consequently, the dichlorination of alkenes has long been recognized as an effective strategy and remains one of the most anticipated reactions in organic synthesis.[3] Initially, Cl_2 was employed as a chlorine source for alkene dichlorination, but its strong corrosivity and toxicity pose significant safety concerns.[4] To address these issues, milder and more practical chlorinating reagents have been developed, facilitating electrophilic addition reactions for alkene dichlorination (Scheme 1(b)).[5,6] Examples of such reagents include NCS-PPh_3, SO_2Cl_2, Oxone-NaCl, $KMnO_4$-Me_3SiCl-$BnEt_3NCl$, and Et_4NCl. However, the strong oxidative and electrophilic nature of these chlorinating agents limits their compatibility with certain functional groups, often resulting in *trans*-selectivity. By incorporating organocatalysis, Denmark and Gilmour achieved dichlorination of alkenes to access elusive vicinal *syn*-configured adducts *via* PhSeSePh catalysis and $I^{(I)}$/$I^{(III)}$ catalysis, respectively.[7] Nevertheless, these methods still require stoichiometric oxidants, reducing substrate compatibility and generating environmentally harmful waste.

Electrochemical methods have emerged as a promising alternative, offering a greener and more efficient pathway for



Scheme 1 Representative natural products containing vicinal dihalogen unit and dihalogenation of alkenes.

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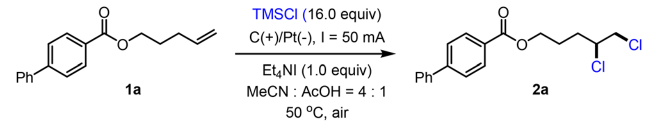
traditional organic redox reactions.[8] Through electrochemical redox, high-energy intermediates can be generated directly, enabling reactions that were previously challenging to achieve, such as Umpolung process. Furthermore, this approach significantly reduces the dependence on conventional chemical oxidants. In 2017, Lin pioneered the Mn-catalyzed electrochemical dichlorination of alkenes using $MgCl_2$ as the chlorine source *via* a Mn-mediated Cl atom transfer strategy (Scheme 1(c)).[9] In 2021, Morandi reported the Mn-catalyzed electrochemically-assisted shuttle paradigm for the facile and scalable interconversion of alkenes and vicinal dihalides, enabling the synthesis of dihalogenated molecules.[10] Despite these important advancements, the development of a metal-free, mild, selective, and sustainable approach to alkene dichlorination remains a significant and challenging frontier in organic synthesis.

Driven by our interest in electrochemical halogenation,[11] we herein report an Et_4NI-mediated electrochemical strategy that employs readily available TMSCl as a chlorine source for the regioselective synthesis of 1,2-dichloroalkanes (Scheme 1(d)). Under analogous conditions, substitution of TMSCl with TMSBr enables the corresponding dibromination of unactivated alkenes.

Pent-4-en-1-yl [1,1′-biphenyl]-4-carboxylate **1a** was selected as the model substrate, with TMSCl (16 equiv.) as the chloride source and Et_4NI (1.0 equiv.) as the supporting electrolyte in MeCN/AcOH (4 : 1, 5.0 mL). Using a graphite anode and a Pt cathode under constant current electrolysis (50 mA) at 50 °C in air, the desired vicinal dichloride **2a** was obtained in 77% yield (Table 1, entry 1). Evaluation of the electrode materials revealed that a C(+)/C(−) electrode pair was almost ineffective, whereas Pt(+)/Pt(−) and C(+)/Pb(−) gave the product **2a** in 62% and 51% yield, respectively (Table 1, entries 2–4). Screening of chlorine sources showed that HCl and NaCl were inferior to TMSCl (Table 1, entries 5 and 6). The amount and nature of the supporting electrolyte were also crucial: when the loading of Et_4NI was significantly reduced, or when Et_4NI was replaced with nBu_4NBF_4 or nBu_4NPF_6, diminished yields were observed (Table 1, entries 7–9). Solvent effects were pronounced. Replacing AcOH with acetone or HFIP led to decreased yields (Table 1, entries 10 and 11). Using either MeCN or AcOH alone gave only 21% yield or no reaction, respectively, and DMF afforded only trace amounts of product (Table 1, entries 12–14). Performing the reaction under N_2 resulted in a lower yield, and no product formation was observed in the absence of an electric current (Table 1, entries 15 and 16).

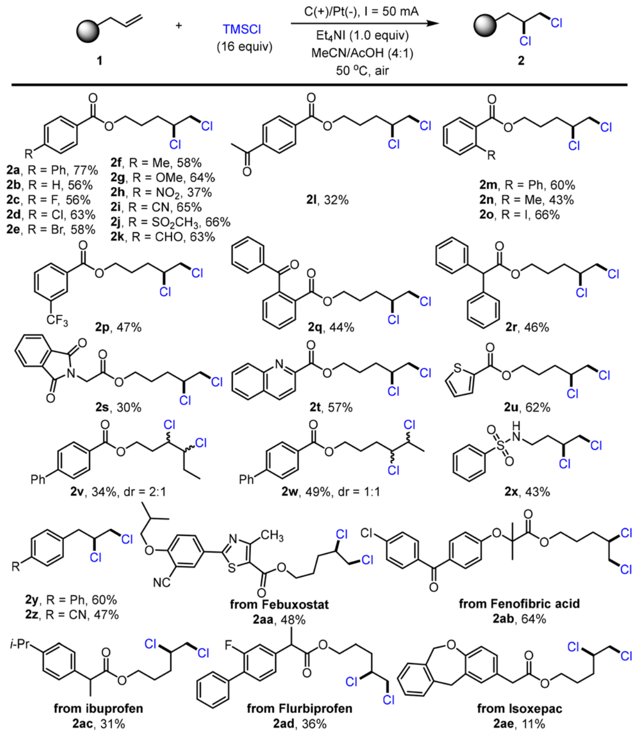
Under the optimized conditions, the substrate scope of unactivated alkenes **1** was examined (Scheme 2). A broad range of substituents, including electron-withdrawing groups (F, Cl, Br, I, NO_2, CN, SO_2Me, CF_3) and electron-donating groups (Me, OMe), were well tolerated, providing the corresponding vicinal dichlorides (**2a–2r**) in generally good yields. Redox-sensitive functionalities such as aldehydes (**2k**) and carbonyl groups (**2l**, **2q**), as well as acid/base-sensitive groups such as sulfonamides (**2x**), also remained intact under the electrolysis conditions. Both *Z*- and *E*-configured alkenes delivered the desired

Table 1 Optimization of reaction conditions[a]



Entry	Variation from the standard conditions	Yield[b] (%)
1	None	77
2	C(+)/C(−) instead of C(+)/Pt(−)	N.R.
3	Pt(+)/Pt(−) instead of C(+)/Pt(−)	62
4	C(+)/Pb(−)instead of C(+)/Pt(−)	51
5	HCl instead of TMSCl	67
6	NaCl (4.0 equiv.) instead of TMSCl	13
7	Et_4NI (0.1 equiv.), Et_4NCl (0.9 equiv.) instead of Et_4NI	34
8	nBu_4NBF_4 instead of Et_4NI	26
9	nBu_4NPF_6 instead of Et_4NI	31
10	MeCN : acetone = 4 : 1 as solvent	Trace
11	MeCN : HFIP = 4 : 1 as solvent	66
12	MeCN (5.0 mL) as solvent	21
13	AcOH (5.0 mL) as solvent	N.R.
14	DMF (5.0 mL) as solvent	13
15	Under N_2	62
16	No electricity	N.R.

[a] Standard conditions: a platinum plate as cathode (1.0 cm × 1.0 cm × 0.1 mm) and a graphite plate as anode (1.0 cm × 1.0 cm × 2.0 mm), constant current = 50 mA, **1a** (0.5 mmol), TMSCl (16 equiv.), Et_4NI (0.5 mmol), MeCN/AcOH (4/1, 5.0 mL), undivided cell, open to air, 50 °C, ~4 h, 7.46 F mol^{-1}. [b] Isolated yields.

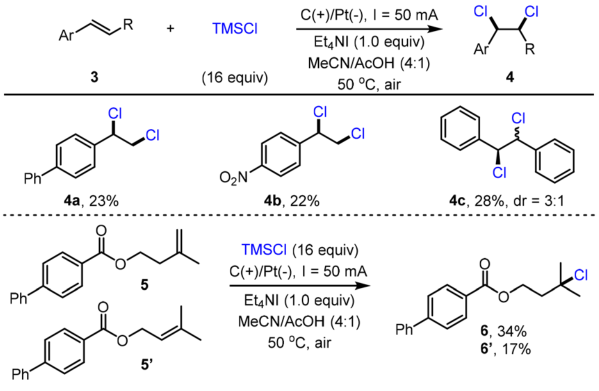


Scheme 2 Electrochemical vicinal dichlorination of unactivated alkenes. Reaction conditions: the same as Table 1.

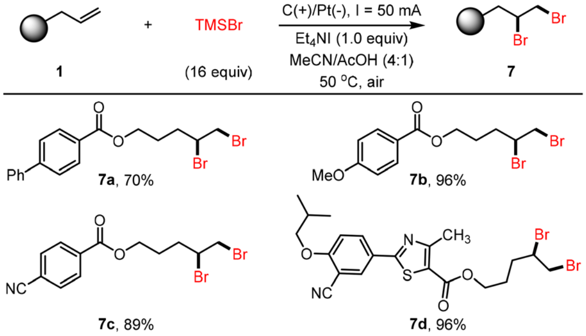
dichlorinated products **2v** and **2w** in relatively low yield probably due to steric hindrance. Other classes of unactivated alkenes furnished the corresponding products with moderate efficiency (**2y–2z**). Heterocyclic substrates, including glycine derivatives (**2s**), quinoline (**2t**), and thiophene (**2u**), were compatible with the protocol. Notably, derivatives of pharmaceutically relevant molecules such as ibuprofen could also be converted to the corresponding vicinal dichlorides (**2aa–2ae**) in moderate yields. Notably, under standard reaction conditions, substrate **1ae** affords the target product **2ae** in relatively low yield. This is mainly attributed to its facile oxidation under electrochemical conditions, leading to substantial byproduct formation.

Furthermore, we evaluated the electrochemical chlorination of styrenes and aliphatic di- and trisubstituted alkenes **3** (Scheme 3). For these activated alkenes, the yields of vicinal dichlorides were generally lower than those observed for unactivated alkenes. *para*-Phenyl- and *para*-nitro-substituted styrenes furnished dichlorides **4a** and **4b** in 23% and 22% yield, respectively, whereas stilbene afforded **4c** in 28% yield with a 3 : 1 dr. Aliphatic disubstituted alkene **5** and trisubstituted alkene **5′** gave mainly monochlorinated products under the standard electrolysis conditions. Importantly, replacement of TMSCl with TMSBr under otherwise identical conditions enabled an efficient vicinal dibromination, affording **7a–7d** in 70–96% yields (Scheme 4).

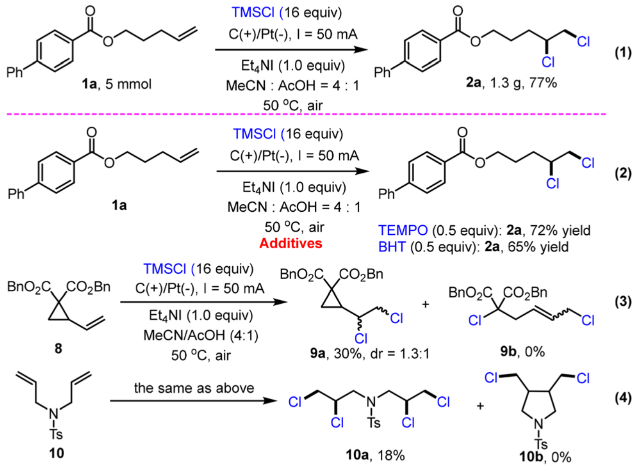
To demonstrate the practicality of the method, a scale-up experiment was conducted using **1a** (5.0 mmol) under the optimized dichlorination conditions, which furnished 1.3 g of **2a** in 77% yield (Scheme 5(1)). To gain insight into the reaction mechanism, a series of control experiments was performed. The addition of radical scavengers such as TEMPO or BHT to the standard electrolysis conditions did not significantly affect the yield of **2a**, suggesting that the process likely proceeds *via* an ionic rather than a radical pathway (Scheme 5(2)). In radical clock experiments, *N*-tosyl diallylamine and a vinylcyclopropane derivative gave only the corresponding vicinal dichlorides **9a** and **10a**, without formation of the ring-opened product **9b** or the cyclized product **10b**, further arguing against the involvement of $Cl^{\bullet}$ species (Scheme 5(3) and (4)). Cyclic voltammetry (CV) measurements revealed an anodic oxidation peak at 0.85 V (*vs.* SCE) in the presence of Et_4NI alone. The CV trace of a mixture of the alkene and Et_4NI closely resembled that of Et_4NI alone, whereas no obvious oxidation peak was observed for the alkene when nBu_4NBF_4 was used as the electrolyte (see the SI for details). These results suggest that free I^- in solution is oxidized at the anode, while the alkene itself is not directly oxidized.



Scheme 3 Electrochemical vicinal dichlorination of activated alkenes. Reaction conditions: the same as Table 1.

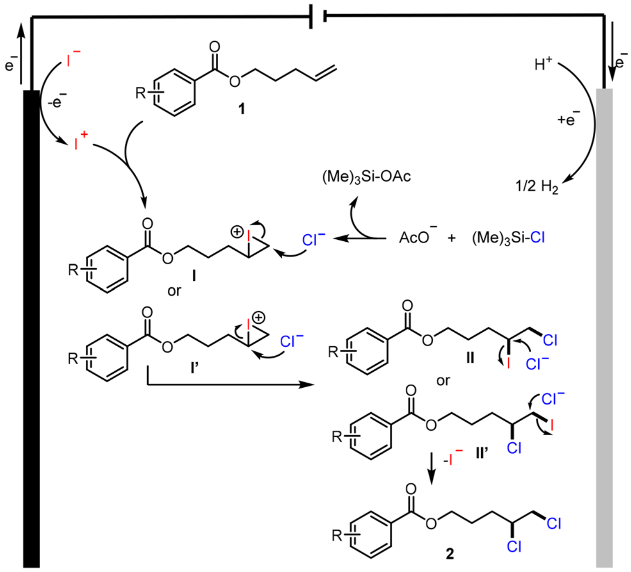


Scheme 4 Electrochemical vicinal dibromination of unactivated alkenes. Reaction conditions: a platinum plate as cathode (1.0 cm × 1.0 cm × 0.1 mm) and a graphite plate as anode (1.0 cm × 1.0 cm × 2.0 mm), constant current = 50 mA, **1a** (0.5 mmol), TMSBr (16 equiv.), Et_4NI (0.5 mmol), MeCN/AcOH (4/1, 5.0 mL), undivided cell, open to air, 50 °C. Isolated yields.



Scheme 5 Gram-scale reaction and mechanistic experiments.

On the basis of literature precedent and the above mechanistic studies, a plausible mechanism is proposed (Scheme 6). At the anode, I^- is oxidized to an electrophilic iodine species (I^+), which reacts with the alkene **1** to form an iodonium ion intermediate **I** or **I′**. Concurrently, TMSCl reacts with AcOH to generate Cl^-. Nucleophilic attack of Cl^- on the iodonium ion **I** yields a chloroiodinated intermediate **II** or **II′**. A second attack of Cl^- on this intermediate displaces I^-, delivering the vicinal dichloride product **2**. At the cathode, proton reduction occurs to evolve H_2. Reaction condition screening results (Table 1,



Scheme 6 Plausible mechanistic pathway.

entries 7–9) suggest this reaction may also proceed *via* a competitive mechanism. Specifically, chloride anions are oxidized to cations at the anode, react with alkenes to form chloronium intermediates, and subsequent nucleophilic addition gives the target product **2**.

Conclusions

In summary, we have developed an environmentally benign electrochemical method for the vicinal dichlorination and dibromination of unactivated alkenes, employing TMSCl and TMSBr as halogen sources, respectively, under undivided electrolytic conditions. The use of Et_4NI-mediated electrophilic addition facilitates efficient halogenation, providing a straightforward route to dichloroalkanes and dibromoalkanes. Mechanistic investigations reveal that the reaction proceeds *via* an ionic pathway, with *in situ* generation of iodide ions contributing to the halogenation process. Importantly, gram-scale reactions demonstrate the scalability and practical applicability of this approach.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information is available. See DOI: **https://doi.org/10.1039/d6qo00047a**.

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