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# Electrochemically driven bromochlorination of alkenes

 Chang-Hui Liu,<sup>ab</sup> Su-Yang Xu,<sup>ab</sup> Xue-Ting Li,<sup>ab</sup> Heng Liu,<sup>a</sup> Hao Zheng<sup>\*a</sup> and Qing-An Chen<sup>ib</sup> <sup>\*ab</sup>

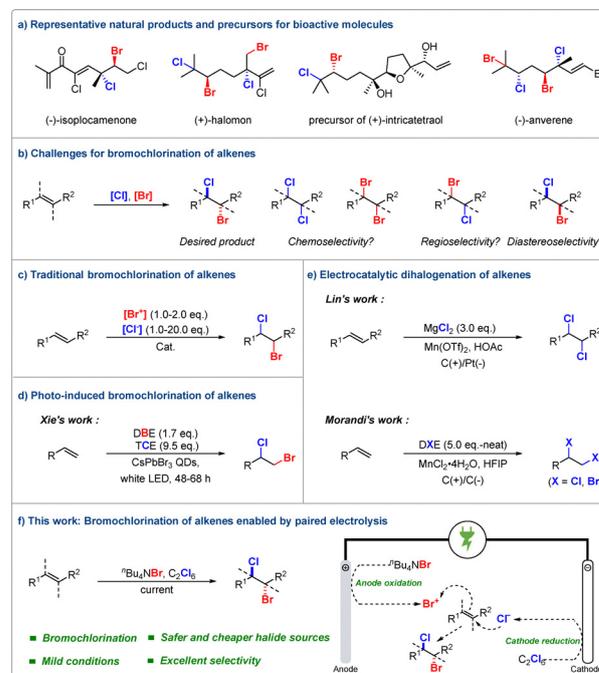
**Organic halides are vital in the synthesis of pharmaceuticals, agrochemicals, and materials. While conventional alkene dihalogenation uses active halogen reagents, we developed an electrochemical strategy for selective bromochlorination. This approach enables precise halogenation under mild conditions.**

Organic halides are common structural motifs in many bioactive natural products,<sup>1</sup> pharmaceuticals,<sup>2</sup> commodity chemicals<sup>3</sup> and polymers.<sup>3b,4</sup> Additionally, organic halides are utilized as important reaction intermediates in organic synthesis due to their ability to convert into various functional groups for the creation of complex organic compounds.<sup>5</sup> Bromochlorides constitute a significant subset within the realm of organic halides, showcasing distinctive physiological activities (Fig. 1a).<sup>6</sup> Moreover, bromochlorides containing two distinct halogen atoms can undergo sequential reactions to introduce two different molecular fragments, which significantly enhances the diversity and flexibility in the synthesis of complex molecules.<sup>7</sup>

The bromochlorination of alkenes represents an efficient and step-economical strategy for the incorporation of chlorine and bromine.<sup>8</sup> The key to this strategy involves the provision of two halogen intermediates with different electronic properties. In addition, the nucleophilic competition of halogen anions (Br<sup>-</sup>, Cl<sup>-</sup>), site selectivity, and stereoselectivity all increase the difficulty in obtaining a single bromochloride product (Fig. 1b).<sup>9</sup> The traditional approach for bromochlorination of alkenes entails the direct addition of disparate halogen sources to the alkenes in the presence of well-designed catalysts (Fig. 1c).<sup>10–12</sup> Nevertheless, a limitation of this methodology is the use of highly active reagents. Xie and colleagues have reported a photocatalytic shuttle strategy to realize bromochlorination of alkenes with perovskite quantum dots (QDs) as catalysts (Fig. 1d).<sup>13</sup> However, an excess of

1,1,2,2-tetrachloroethane (TCE) was required to obtain bromochloride products under long time LED irradiation.

Electrochemistry can provide an effective and sustainable alternative to traditional chemical methods for organic redox transformations.<sup>14</sup> Lin's and Morandi's group have independently utilized Mn-catalyzed electrolysis to achieve the dihalogenation of alkenes by employing MgCl<sub>2</sub> and dihalogen ethane as halide sources (Fig. 1e).<sup>15,16</sup> Nevertheless, there is still a significant knowledge gap in utilizing electrochemistry for the bromochlorination of alkenes. Inspired by these precedents and our continued interest in alkene functionalization,<sup>17</sup> we employed an undivided cell to generate two distinct types of halogen intermediates through paired electrolysis (Fig. 1f).<sup>18</sup>



**Fig. 1** Representative vicinal bromochlorides and related synthesis from alkenes.

<sup>a</sup> Dalian Institute of Chemical Physics, Chinese Academy of Sciences, 457 Zhongshan Road, Dalian 116023, China. E-mail: hzheng@dicp.ac.cn, qachen@dicp.ac.cn

<sup>b</sup> University of Chinese Academy of Sciences, Beijing 100049, China



These intermediates were then reacted with alkenes, enabling the efficient synthesis of bromochlorides with high chemo-, regio-, and diastereoselectivity. Besides, the reaction was characterized by its simplicity, occurring under catalyst or additive-free conditions. Initially, styrene **1a** was chosen as the model substrate for optimization (Table 1). With hexachloroethane ( $C_2Cl_6$ ) as the chlorine source, bromochloride **2a** was obtained as the main product by using tetrabutylammonium bromide ( ${}^nBu_4NBr$ ) as the electrolyte and bromine source in DCM (entry 1). Conducting the electrolysis without  $C_2Cl_6$  resulted in a lower yield (entry 2). Replacing  $C_2Cl_6$  with TCE as the chlorine source was found to be slightly less effective in terms of activity and selectivity (entry 3). It is worth noting that when  $C_2Cl_6$  was added, DCM typically only served as a solvent instead of a chlorine source, which was further confirmed in subsequent mechanistic experiments (Fig. 3D and E). Using DCE as both the chlorine source and solvent resulted in poor selectivity of the reaction (entry 4). The use of alternative bromine sources, such as *N*-bromosuccinimide (NBS) or lithium bromide (LiBr), lowered the reaction efficiency with tetrabutylammonium hexafluorophosphate ( ${}^nBu_4NPF_6$ ) as the electrolyte (entries 5 and 6). The alteration of the ratio between  ${}^nBu_4NBr$  and  $C_2Cl_6$  led to a significant decrease in yield and favored the formation of the undesired dichloride **4a** (entry 7). Solvents such as methanol (MeOH), acetonitrile (MeCN), and *N,N*-dimethylformamide (DMF) were screened as well, but lower yields were obtained (entries 8–10). The electrochemical reaction could also be performed when using a glassy carbon (GC) electrode as the anode, although with a slight decrease in yield (entry 11). Severe corrosion of the lead cathode material occurred, which hindered the generation of chloride ions and thereby led to a decrease in both reaction activity and selectivity (entry 12). No

desired reaction occurred in the absence of current (entry 13). Finally, under ambient air conditions, the electrochemical reaction could also be conducted (entry 14).

With the optimized conditions in hand, the scope of terminal alkenes was investigated (Fig. 2A). Both electron-donating (**2b–2d**) and electron-withdrawing groups (**2e–2i**) at the *para*-position of the aryl ring were well tolerated, delivering the desired products in moderate to good yields with high selectivities. 1-Chloro-3-vinylbenzene also exhibited good activity and gave the bromochloride product in an 87% yield (**2j**), although with a slight decrease in the chemoselectivity. However, the steric hindrance resulted in a noticeable decrease in both yield and chemoselectivity (**2k**). Besides, this protocol could be successfully extended to bromochlorination of heterocycle derived substrates (**2l–2m**). To our delight, the electrolytic process showed good compatibility with vinyl ester type alkenes (**2n–2o**).

Additionally, the current electrochemical protocol was also suitable for the bromochlorination of internal alkenes (Fig. 2B). It was observed that both five-membered (**2aa** and **2ab**) and six-membered (**2ac**) cyclic internal alkenes gave good results. Free hydroxyl groups were all compatible with this process, leading

Table 1 Optimization of the reaction conditions<sup>a</sup>

Entry	Variation	Yield <sup>b</sup> [%]			
		2a	3a	4a	5a
1	None	91 (88) <sup>d</sup>	0	0	5
2	Without $C_2Cl_6$	43	0	0	8
3	TCE instead of $C_2Cl_6$	75	0	3	7
4	DCE instead of DCM, without $C_2Cl_6$	42	11	11	0
5 <sup>c</sup>	NBS instead of ${}^nBu_4NBr$	19	0	0	12
6 <sup>c</sup>	LiBr instead of ${}^nBu_4NBr$	24	0	4	0
7	${}^nBu_4NBr$ (2.0 eq.), $C_2Cl_6$ (1.5 eq.)	29	0	24	7
8	MeOH instead of DCM	2	0	0	0
9	MeCN instead of DCM	59	10	6	0
10	DMF instead of DCM	30	0	0	6
11	GC(+)/Pt(-) instead of C(+)/Pt(-)	70	0	0	0
12	C(+)/Pb(-) instead of C(+)/Pt(-)	35	0	4	19
13	Without current	0	0	0	0
14	Under air	62	0	5	3

<sup>a</sup> Conditions: unless otherwise noted, all reactions were performed with **1a** (0.20 mmol),  ${}^nBu_4NBr$  (1.5 eq.) and  $C_2Cl_6$  (1.5 eq.) in DCM (4.0 mL) at room temperature for 16 h. <sup>b</sup> Yields were determined by GC-FID. <sup>c</sup> Adding  ${}^nBu_4NPF_6$  (1.5 eq.) as an electrolyte. <sup>d</sup> Isolated yield.

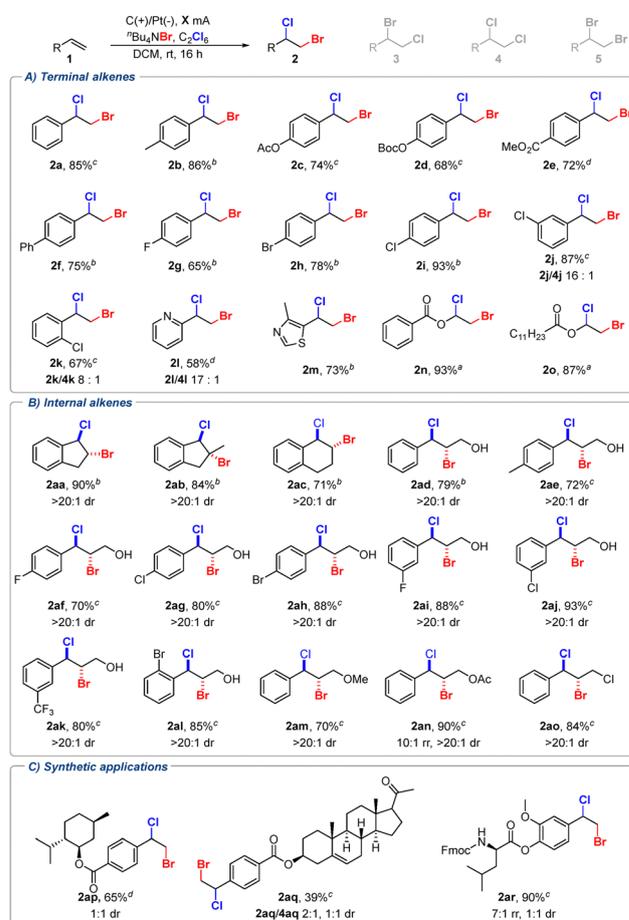


Fig. 2 Substrate scope for bromochlorination of alkenes. <sup>a</sup> Reaction with 3.0 mA, and 16 h (1.9 mA cm<sup>-2</sup>, 8.9 F mol<sup>-1</sup>). <sup>b</sup> Reaction with 4.0 mA, 16 h (2.5 mA cm<sup>-2</sup>, 11.9 F mol<sup>-1</sup>). <sup>c</sup> Reaction with 5.0 mA, 16 h (3.1 mA cm<sup>-2</sup>, 14.9 F mol<sup>-1</sup>). <sup>d</sup> Reaction with 6.0 mA, and 16 h (3.8 mA cm<sup>-2</sup>, 17.9 F mol<sup>-1</sup>).



to the bromochloride products (**2ad–2al**) in high yields and selectivities. The reaction also demonstrated good tolerance for cinnamyl ether (**2am**) and cinnamyl chloride (**2ao**). Cinnamyl ester also exhibited good activity and gave the bromochloride product in an 82% yield (**2an**), although with a decrease in regioselectivity. Finally, the electrolytic method successfully extended to alkenes bearing fragments of natural products (**2ap–2ar**), and these substrates could smoothly undergo bromochlorination with moderate to high yields (Fig. 2C).

To probe the mechanism of this electrochemically enabled bromochlorination, preliminary mechanistic investigations have been carried out (Fig. S2, SI). Control experiments were conducted by individually adding MeOH or bulky <sup>t</sup>BuOH to the reaction system (Fig. S2A, SI). The addition of MeOH gave bromochloride **2a** in a 30% yield accompanied by bromoalkoxylation product **6a** in a 21% yield (entry 2). Besides, the addition of <sup>t</sup>BuOH increased the yield of product **2a** to 56%, and a trace amount of bromoalkoxylation product **6b** was detected (entry 3). The above experiments suggest that a bromonium intermediate is involved in this reaction.<sup>19</sup> Furthermore, the scope of the substrate was successfully conducted with high diastereoselectivities, which further supports our hypothesis.<sup>20</sup>

After adding BHT or TEMPO as a radical scavenger, a significant decrease in the yield of product **2a** was observed. HRMS analysis of the reaction mixture revealed no adducts of TEMPO or BHT. Moreover, when conducting radical clock experiments with (1-cyclopropylvinyl)-benzene **1p**, ring-opening product **7p** was afforded in a 50% yield (Fig. S2B, SI). These results suggest that a bromine radical or cation may be involved in this reaction.<sup>21</sup>

In order to get insight into this electrochemical process, cyclic voltammetry (CV) studies were performed under different conditions (Fig. S2C, SI). A sharp oxidation peak of styrene with  $E_{1/2} = 2.06$  V (vs. SCE) was observed (curve II). Besides, two distinct oxidation peaks of the Br anion with  $E_{1/2} = 0.98$  V and  $E_{1/2} = 1.4$  V were exhibited, respectively (curve III). A comparison between curves II and III indicates that the Br anion is more susceptible to oxidation at the anode compared to styrene, and the generation of the Br cation may undergo two-step anodization. In order to track the source of chlorine, CV studies of DCM and C<sub>2</sub>Cl<sub>6</sub> were conducted (Fig. S2D, SI). C<sub>2</sub>Cl<sub>6</sub> showed a reduction peak with  $E_{1/2} = -1.46$  V (curve II) while DCM exhibited no apparent reduction peak from -3.0 to 0 V (curve III). Moreover, a similar result was obtained when adding C<sub>2</sub>Cl<sub>6</sub> and DCM (curve IV vs. III). These results reveal that chlorine mainly originates from the cathodic reduction of C<sub>2</sub>Cl<sub>6</sub>.

Kinetic studies were carried out to further gain mechanistic insight (Fig. S2E, SI). The consumption of styrene and C<sub>2</sub>Cl<sub>6</sub>, as well as the generation of tetrachloroethylene (C<sub>2</sub>Cl<sub>4</sub>), exhibits a similar trend with a close-to-stoichiometric relationship. It further confirms that the Br anion undergoes anodic oxidation to provide the Br cation, while C<sub>2</sub>Cl<sub>6</sub> is reduced to produce the Cl anion at the cathode. The desired bromochloride **2a** was not detected at the initial 1 h but then gradually increased. Throughout the entire process, the formation of side product **3a** was scarcely observed. Dibromide **5a** quickly formed, and the rate of formation reached a peak around 3 h and then gradually decreased. Although the Cl

anion had stronger nucleophilicity, the concentration of the Br anion is much greater than that of the Cl anion, resulting in similar rates of formation for bromochloride **2a** and dibromide **5a** at the early stage of reaction. As the reaction proceeded, the concentration of the Cl anion gradually increased, favoring the formation of bromochloride **2a**, and dibromide **5a** also gradually converted to bromochloride **2a**. Dichloride **4a** was not detected in the initial 3 h, and then its formation gradually increased until the rate of formation reached the peak at around 8 h. It is likely that dichloride **4a** results from the conversion of bromochloride **2a** or dibromide **5a**.

In order to demonstrate conversion between the products and establish their conversion relation, control experiments were conducted by replacing styrene **1a** with bromochloride **2a** or dibromide **5a** as a starting material under standard conditions (Fig. S2F, SI). A trace amount of bromochloride **2a** was transformed into dichloride **4a** (left, entry 1). However, when using tetrabutylammonium chloride (<sup>n</sup>Bu<sub>4</sub>NCl) as an electrolyte, the yield of dichloride **4a** increased to 18% (left, entry 2). The yield of dichloride **4a** was 8% without electricity (left, entry 3). No formation of dibromide **5a** was observed under the above conditions. When dibromide **5a** was used as a substrate, bromochloride **2a** was obtained in a 6% yield while dichloride **4a** was not detected (right, entry 1). However, when employing <sup>n</sup>Bu<sub>4</sub>NCl as an electrolyte, the yield of bromochloride **2a** increased to 53% accompanied by dichloride **4a** in a 5% yield (right, entry 2). In the absence of electricity, the yield of bromochloride **2a** was 14% along with trace amounts of dichloride **4a** (right, entry 3). The above results indicate that, in the presence of the Cl anion, bromochloride **2a** and dibromide **5a** can be further transformed into dichloride **4a** and bromochloride **2a**, respectively. Notably, electricity could accelerate the corresponding transformation. And the direct conversion of dibromide **5a** to dichloride **4a** is slow.

Based on the aforementioned experiments, a possible mechanism of this electrochemically driven bromochlorination of alkenes is proposed (Fig. 3). At the anode, the Br anion undergoes two-step oxidation to form the Br cation. Simultaneously, C<sub>2</sub>Cl<sub>6</sub> is reduced to generate a Cl anion and C<sub>2</sub>Cl<sub>4</sub> at the cathode. The Br cation then reacts with alkene **1** to form a bromonium intermediate **M**, which subsequently reacts with the Cl anion to obtain the desired product **2** (Path I). Besides, the Br anion can also react with the bromonium intermediate **M** to form dibromide product **5**. In the presence of electricity, dibromide product **5** can gradually convert into desired product **2** (path II). Meanwhile, a small amount of product **2** can transform into dichloride product **4**.

To further demonstrate the synthetic utility of this protocol, scale-up reaction and derivatizations have been performed (Fig. S7, SI). Gram-scale preparation of cinnamyl alcohol **1ad** was carried out to afford bromochloride **2ad** (1.5 g) in a 75% yield with excellent diastereoselectivity (>20:1). Next, the esterification of bromochloride **2ad** could occur and the configuration of halogenated ester **8** was further confirmed through single crystal X-ray diffraction.<sup>22</sup> Furthermore, reactions of bromochloride **2ad** with various nucleophilic reagents were conducted to synthesize 1,3-diol **9** and ester **10**.<sup>23</sup> Notably, these reactions could maintain high diastereoselectivities



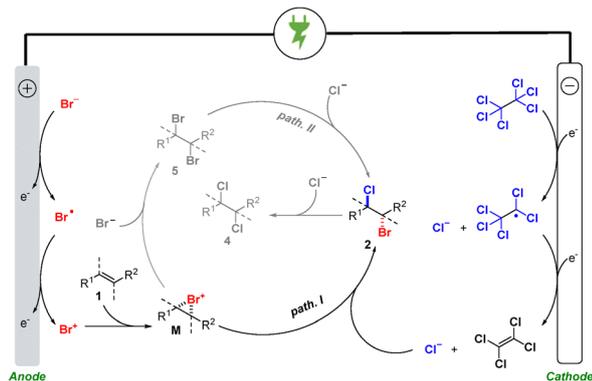


Fig. 3 Proposed mechanism.

(> 20 : 1). Finally, the elimination reaction of bromochloride **2ad** was performed, yielding the chlorinated allylic alcohol **11** (> 20 : 1 *E/Z*) in a 66% yield.

In conclusion, we developed an efficient electrolytic method for the catalyst-free bromochlorination of alkenes with high selectivities.  $C_2Cl_6$  as a halogen source demonstrates compatibility with multifarious functional groups such as free hydroxyl groups. Mechanistic studies revealed that the reaction involves the generation of a Br cation and Cl anion *via* paired electrolysis. Most importantly, the formation of a bromonium intermediate results in high selectivities. Besides, diverse synthetic transformations has been successfully achieved with high selectivities. We anticipate that this mild, efficient, and highly selective bromochlorination strategy will propel the development of other dihalogenations (such as bromofluorination), thereby providing a new paradigm for precise electrochemical synthesis of organic halides.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

The data underlying this study are available in the article and its supplementary information (SI). Supplementary information is available. See DOI: <https://doi.org/10.1039/d5cc04608d>.

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