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# Radical-mediated alkoxypolyhaloalkylation of styrenes with polyhaloalkanes and alcohols *via* C(sp$^3$)–H bond cleavage†

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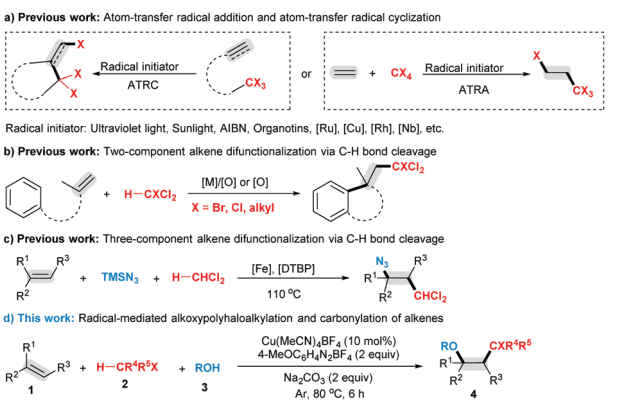
**We have developed a new radical-mediated alkoxypolyhaloalkylation of styrenes with polychloroalkanes and alcohols for the facile synthesis of complex polyhaloalkanes. 4-Methoxybenzenediazonium tetrafluoroborate is a good radical initiator for this transformation. This protocol is well applied to the late-stage functionalization of complex molecules, including vitamin E, estrone and cholesterol derivatives.**

Polychlorinated hydrocarbons, especially those bearing di- or trichloromethyl groups, are prevalent in natural products, pesticides and various bioactive molecules.[1] Since Kharasch reported the atom-transfer radical addition (ATRA) of terminal alkenes with bromotrichloromethane as the polychloromethyl unit,[2] the radical addition reaction of alkenes, including difunctionalization of alkenes, has become one of the most powerful strategies for the rapid assembly of diverse complex polyhaloalkyl-containing molecules from abundant and readily available starting materials.[3] Traditional radical-mediated alkene difunctionalization methods for the synthesis of polyhaloalkyl-containing compounds usually focus on the one-component atom-transfer radical cyclization (ATRC) and two-component atom-transfer radical addition (ATRA) processes through C–halogen bond homolysis using photocatalysts (*e.g.*, ultraviolet light, sunlight), thermocatalysts, and/or highly toxic organic initiators (*e.g.*, azobisisobutyronitriles, organotins) (Scheme 1a).[4] To expand their applications, recent efforts have been devoted to the polychloroalkyl radical-initiated two- (Scheme 1b)[5] and three-component (Scheme 1c)[6] alkene difunctionalization reactions with polyhaloalkanes and nucleophiles (*e.g.*, aryl C(sp$^2$)–H bonds, $O^-$, $N_3^-$) beyond the ATRA and ATRC, which enable the preferential cleavage of the C–H bonds in polyhaloalkanes, leading to the formation of polychloroalkyl radicals, owing to the enthalpic and stereoelectronic effects.[5,6] Therefore, the transformation of alkenes into complex polyhaloalkanes through polychloroalkyl radicals is an appealingly straightforward tool in organic synthesis. To our knowledge, however, such available examples of the polychloroalkyl radical-initiated alkene difunctionalization reaction, especially including the three component intermolecular mode, remain rare.[6]

Here, we report a new, general radical-mediated alkoxypolyhaloalkylation of alkenes with polychloroalkanes and alcohols to produce various 1-alkoxyl-2-polyhaloalkyl alkanes *via* C(sp$^3$)–H bond cleavage (Scheme 1d). Using 4-methoxybenzenediazonium tetrafluoroborate as a radical initiator enables the construction of polyhaloalkyl radical intermediates, realizing the late-stage synthesis of natural products.

We initiated our investigations by exploring the alkoxypolyhaloalkylation of 1-methoxy-4-vinylbenzene (**1a**) with $CHCl_3$ (**2a**) and ethanol (**3a**) in the presence of 4-MeOC$_6$H$_4$N$_2$BF$_4$ (Table 1). After evaluating various reaction parameters, the




Scheme 1 Difunctionalization of alkenes initiated by polychloroalkyl radicals.

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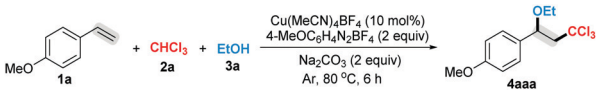
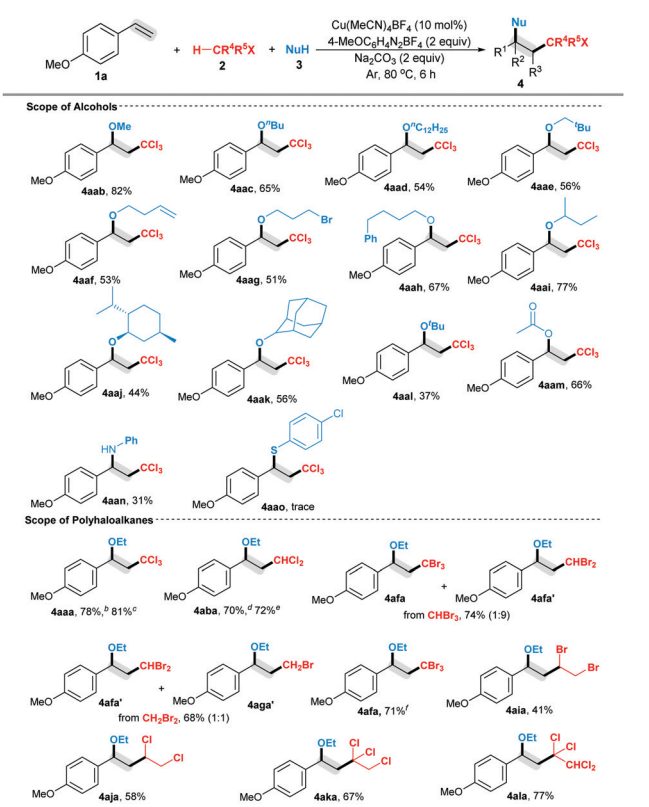
Table 1 Optimization of the reaction conditions[a]




| Entry | Variation from the standard conditions | Yield[b]/(%) |
|---|---|---|
| 1 | None | 91 |
| 2 | Without 4-$MeOC_6H_4N_2BF_4$ | 0 |
| 3 | Without $Cu(MeCN)_4BF_4$ | 63 |
| 4 | CuCl instead of $Cu(MeCN)_4BF_4$ | 83 |
| 5 | $CuCl_2$ instead of $Cu(MeCN)_4BF_4$ | 68 |
| 6 | $Cu(OTf)_2$ instead of $Cu(MeCN)_4BF_4$ | 67 |
| 7 | $FeCl_2$ instead of $Cu(MeCN)_4BF_4$ | 75 |
| 8 | $Ni(acac)_2$ instead of $Cu(MeCN)_4BF_4$ | 71 |
| 9 | Without $Na_2CO_3$ | 58 |
| 10 | $K_2HPO_4$ instead of $Na_2CO_3$ | 81 |
| 11 | 2,6-Lutidine instead of $Na_2CO_3$ | 63 |
| 12 | $NaHCO_3$ instead of $Na_2CO_3$ | 85 |
| 13 | KOH instead of $Na_2CO_3$ | 33 |
| 14 | $K_2CO_3$ instead of $Na_2CO_3$ | 73 |
| 15 | At room temperature for 24 h | 28 |
| 16 | At 70 °C | 69 |
| 17 | At 90 °C | 86 |
| 18[b] | None | 77 |
| 19[c] | None | 88 |
| 20[d] | None | 81 |

[a] Reaction conditions: **1a** (0.2 mmol), $CHCl_3$ **2a** (2 mL), **3a** (5 equiv.), $Cu(MeCN)_4BF_4$ (10 mol%), $Na_2CO_3$ (2 equiv.), 4-$MeOC_6H_4N_2BF_4$ (2 equiv.), 80 °C, argon, and 6 h. [b] 4-$MeOC_6H_4N_2BF_4$ (1.5 equiv.). [c] 4-$MeOC_6H_4N_2BF_4$ (2.5 equiv.). [d] **1a** (1 mmol).

Table 2 Variation of polyhaloalkanes (**2**) and nucleophiles (**3**)[a]




[a] Reaction conditions: **1a** (0.2 mmol), **2** (2 mL), **3** (3 equiv.), $Cu(MeCN)_4BF_4$ (10 mol%), $Na_2CO_3$ (2 equiv.), 4-$MeOC_6H_4N_2BF_4$ (2 equiv.), $CHCl_3$ (2 mL), 80 °C, argon, and 6 h. [b] $CBrCl_3$ (**2b**, 2 mL). [c] $CCl_4$ (**2c**, 2 mL). [d] $CHBrCl_2$ (**2d**, 2 mL). [e] $CH_2Cl_2$ (**2e**, 2 mL). [f] $CBr_4$ (**2h**, 2 mL).

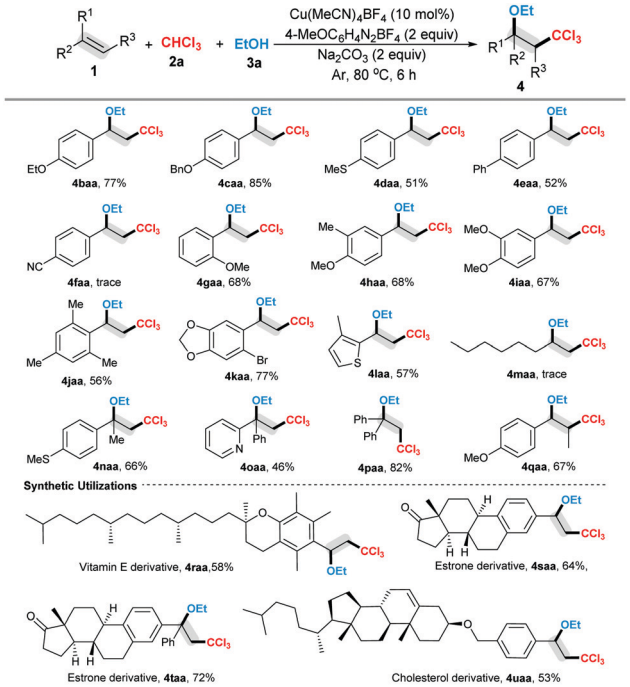
optimal reaction conditions were determined to be as follows: 10 mol% $Cu(MeCN)_4BF_4$, 2 equiv. 4-$MeOC_6H_4N_2BF_4$ and 2 equiv. $Na_2CO_3$ at 80 °C for 6 h (entry 1). The results showed that 4-$MeOC_6H_4N_2BF_4$ was crucial since its omission led to no reaction (entry 2). We were delighted to find that the reaction could be promoted by Lewis acids (entries 4–8), such as CuCl, $CuCl_2$, $Cu(OTf)_2$, $FeCl_2$ and $Ni(acac)_2$, by comparison with the results in the absence of $Cu(MeCN)_4BF_4$ (entry 3). It was noted that bases could promote the reaction (entry 9). Replacement of $Na_2CO_3$ with other bases, including $K_2HPO_4$, 2,6-lutidine, $NaHCO_3$, KOH and $K_2CO_3$, was adverse (entries 10–14). Reducing or increasing the reaction temperature resulted in decreasing yields (entries 15–17). The yield of the reaction did not improve when 1.5 or 2.5 equiv. of 4-$MeOC_6H_4N_2BF_4$ was used (entries 18 and 19). Notably, this reaction was applicable to the reaction with a 1 mmol scale of alkene **1a**, providing polychloroalkanes **4aaa** in 81% yield (entry 20).

With the optimized reaction conditions in hand, we set out to investigate the substrate scope of the alkene alkoxypolychloroalkylation (Table 2). Firstly, a variety of alcohols **3b–l** were investigated. Primary alcohols **3b–3h** bearing linear carbon chains, bromo, alkenyl, and bulky *tert*-butyl and phenyl smoothly gave the corresponding products **4aab–aah** in 51–82% yields. Secondary alcohols, including butan-2-ol (**3i**), DL-Menthol (**3j**) and adamantanol (**3k**), all reacted well, giving the corresponding polychloroalkanes **4aai–aak** in 77%, 44% and 56% yields, respectively. *tert*-Butanol (**3l**) was compatible with the alkoxypolychloroalkylation reaction, but the activity decreased. Other nucleophiles including acetic acid (**3m**) and aniline (**3n**) were tested, providing the corresponding products **4aam** and **aan** in 66% and 31% yields, respectively. Regrettably, 4-chlorobenzenethiol (**3o**) was inert to this transformation.

We next turned our attention to examine the scope of polyhaloalkanes and found that a series of mono-, di- and tri-substituted polyhaloalkanes **2b–l** were compatible with this reaction. Bromotrichloromethane (**2b**) and tetrachloride (**2c**) were suitable substrates, providing the same product **4aaa** in good yield *via* C–Cl and C–Br bond cleavage, respectively. The same product **4aba** was obtained by using bromodichloromethane (**2d**) and dichloromethane (**2e**) as polyhalomethanes. By employing other polyhalomethanes, such as bromoform (**2f**) and dibromomethane (**2g**), mixed products were afforded. When using perbromomethane (**2h**) as a substrate, the tribromoalkane **4afa** was smoothly obtained in 71% yield. Performing the alkoxypolyhaloalkylation of alkenes with 1,2-dibromoethane (**2i**), 1,2-dichloroethane (**2j**), 1,1,2-trichloroethane (**2k**) and 1,1,2,2-tetrachloroethane (**2l**) selectively cut C–H bonds, providing products **4aia–ala** in 41–77% yields, implying that the stability of polychloroalkyl radicals increases as the number of chlorine atoms increases.

Subsequently, a series of alkenes were examined (Table 3). Styrenes **1b–e** and **1g** with electron-rich groups, such as OEt, OBn, SMe, Ph and OMe, efficiently realized the alkoxypolychloroalkylation

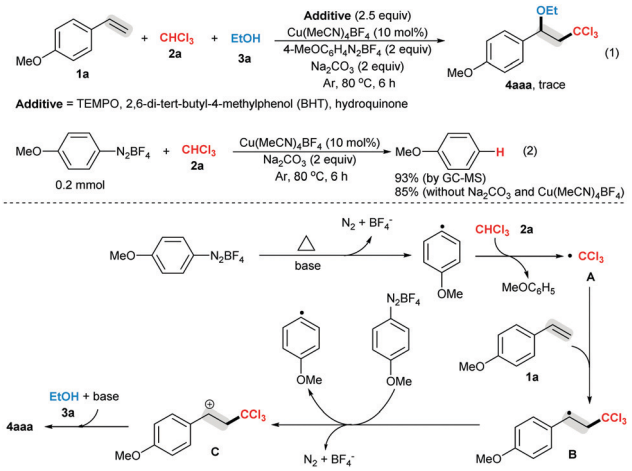
Table 3 Variation of alkenes (**1**)[a]




[a] Reaction conditions: **1a** (0.2 mmol), **2** (2 mL), **3** (3 equiv.), $Cu(MeCN)_4BF_4$ (10 mol%), $Na_2CO_3$ (2 equiv.), $4\text{-}MeOC_6H_4N_2BF_4$ (2 equiv.), $CHCl_3$ (2 mL), 80 °C, argon, and 6 h.

reaction to obtain the target products **4baa–eaa** and **4gaa** in 51–85% yields. Styrene **1f** with a strong electron withdrawing group on the benzene ring, such as CN, was inert; this may be due to the fact that the cyano group is unfavorable for the stability of the carbocation intermediate. Styrenes **1h–k** bearing polysubstituted functional groups on the benzene ring were suitable substrates, giving the corresponding products **4haa–4kaa** in 56–77% yields. Thiophene-containing trichloroalkane **4laa** was also provided in 57% yield by this method. Unfortunately, alkyl alkene **1m** was inert to this transformation. 1-Methyl-1-aryl alkene **1n** was a suitable substrate for this transformation, giving the corresponding product **4naa** in 65% yield. Nonsymmetric 1,1-diaryl alkene **1o** was also efficient for this transformation, offering product **4oaa** in 46% yield. 1, 1-Diphenylethylene (**1p**) was converted to the corresponding product **4paa** in 82% yield. Internal styrene **1q** was reactive, giving the corresponding product **4qaa** in 67% yield. It is worth noting that the alkene alkoxypolyhaloalkylation was suitable for the late-stage modification of complex molecules, including vitamin E derivative **1r**, estrone derivatives **1s** and **t** and cholesterol derivative **1u**, providing the corresponding complex polychloroalkanes **4raa–uaa** in moderate to good yields.

In order to explore the reaction mechanism, we performed control experiments; the addition of radical inhibitors, including TEMPO, 2,6-di-*tert*-butyl-4-methylphenol (BHT) and hydroquinone, prohibited the transformation under the standard conditions (eqn (1), Scheme 2), suggesting that the reaction proceeds through a radical mechanism. Subsequently, we investigated the conditions for the generation of aryl radicals, and experiments




Scheme 2 Control experiments and possible mechanism.

showed that the copper catalyst and base did not play any key role (eqn (2), Scheme 2). Simultaneously, 4-vinylbenzonitrile (**1f**) and oct-1-ene (**1m**) were inert to the alkoxypolyhaloalkylation, indicating that a carbocation intermediate might be generated.

On the basis of our preliminary experiments and previous reports,[5–8] a plausible mechanism is depicted in Scheme 2. Firstly, $4\text{-}MeOC_6H_4N_2BF_4$ salt generates an aryl radical under heating,[7] which reacts with chloroform (**2a**) to generate the trichloromethyl radical **A** and anisole. Then, radical **A** attacks the C=C bond and affords radical **B**, which can be oxidized by $4\text{-}MeOC_6H_4N_2BF_4$ salt to produce the cationic intermediate **C** and aryl radical. The cationic intermediate **C** is captured by nucleophile cation scavenger **3a** to afford **4aaa**.[8]

In summary, we have disclosed a new radical-mediated alkoxypolyhaloalkylation of styrenes for the facile synthesis of complex polyhaloalkanes *via* $C(sp^3)$–H bond cleavage. Commercially available polyhaloalkanes, including DCM and 1,2-DCE, are suitable substrates for generating polyhaloalkyl radical intermediates in the presence of 4-methoxybenzenediazonium tetrafluoroborate. This reaction is also applicable to the late-stage functionalization of complex molecules including vitamin E, estrone and cholesterol derivatives. The further application of this strategy for modifying bioactive molecules is currently underway in our laboratory.

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## Conflicts of interest

There are no conflicts to declare.

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