Article

Synthesis of phenyliodine(III) bis(3-bromopropionate) for an organocatalyzed Markovnikov-type bromo-aminoxidation of vinylarenes

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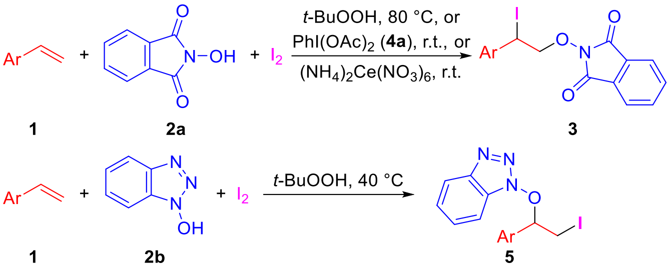
ABSTRACT

An organocatalytic Markovnikov-type bromo-aminoxidation of styrene derivatives was achieved by using in-situ-generated bromine and *N*-oxyl radicals, and the desired products were obtained in moderate to good yields. The bromine radical was generated from phenyliodine(III) bis(3-bromopropionate) through consecutive decarboxylation and deethylenation reactions *via* an organocatalytic SET process. Phenyliodine(III) bis(3-bromopropionate) also served as the oxidant for the generation of the *N*-oxyl radicals, while the *N*-oxyl radical was also crucial for the generation of the key β-enaminyl radical intermediate that was required for the SET process.

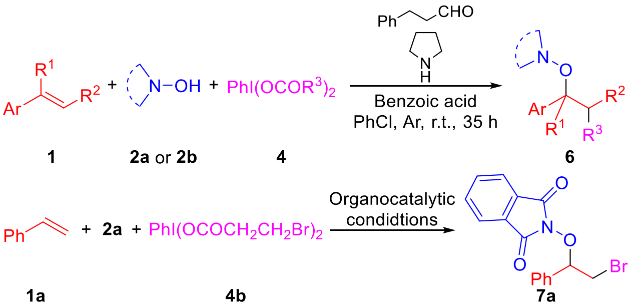
1. Introduction

Oxybromination of alkenes is highly synthetically useful because the reaction products can serve as versatile starting materials in organic synthesis. Moreover, the bromine atom in the products is also a very good leaving group for further elaborations. For example, bromohydrins [1-14] and bromohydrin ethers [10-20] can be directly synthesized from alkenes using many different reagents [1-20] and procedures [21-23], and these products have broad applications in organic synthesis [21,24]. Besides bromohydrins and bromohydrin ethers, methods for the synthesis of bromohydrin esters [9,15,18,25-28], β-bromonitrates [29,30], β-bromosulfonates [29], and β-bromohydroperoxides [31] from alkenes are also known. In contrast, reported methods for the β-halo-aminoxidation of alkenes are rare [32-35]. In 2018, Xu [33] and Terent'ev [34] independently reported the β-iodo-oxyimidation of alkenes using iodine and *N*-hydroxyphthalimide (NHPI, **2a**), with *t*-BuOOH [33] or PhI(OAc)$_2$ (**4a**) [34] as the oxidant, respectively (Scheme 1, top equation). Later on, Terent'ev and co-workers also demonstrated that a similar reaction could be achieved using CAN as the oxidant (Scheme 1, top equation) [35]. All these reactions yield regioselectively the *anti*-Markovnikov-type products **3** [33-35]. It is interesting to point out that when HOBt (**2b**) is used as the reagent, a similar reaction with *t*-BuOOH as the oxidant yields the Markovnikov-type product **5** instead (Scheme 1, bottom equation) [33]. Nonetheless, to our knowledge, similar direct β-bromo-aminoxidation of alkenes has not been reported. Only Dixon and Weiss have reported a single example of synthesizing an *anti*-Markovnikov type β-bromo-oxyimidation product from a bromohydrin [36].

We recently reported an organocatalytic alkylation-aminoxidation of styrene derivatives using NHPI (**2a**) or HOBt (**2b**) and phenyliodine(III) dicarboxylates (**4**) (Scheme 2, top equation) [37]. The reaction involves the generation of the *N*-oxyl radicals from **2a** or **2b** *via* the oxidation of **4a** and the generation of the alkyl radicals from **4** *via* the SET reduction by the β-enaminyl radical [38-41] and subsequent decarboxylation of the initial acyloxy radical [37]. The β-enaminyl radical, in turn, is generated from a HAT reaction between the enamine (formed from the aldehyde and the organocatalyst) and the *N*-oxyl radicals of **2** [37].



Scheme 1. Reported examples of *anti*-Markovnikov- and Markovnikov-type β-iodo-aminoxidations of alkenes.



Scheme 2. Organocatalyzed alkylation-aminoxidation and bromo-aminoxidation of styrene derivatives.

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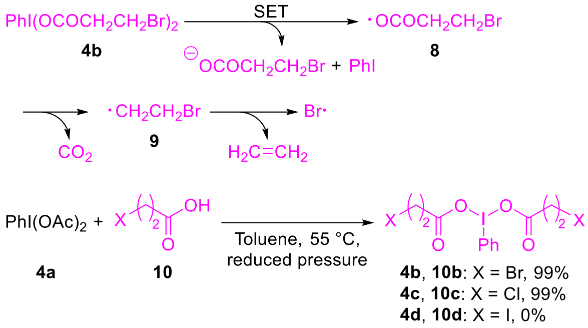
Interestingly, when we applied phenyliodine(III) bis(3-bromopropionate) (**4b**) as the oxidant and radical source in the above reaction, we did not obtain the expected alkylation-aminoxidation product **6**. Instead, we obtained the β-bromo-aminoxidation product **7a** when styrene (**1a**) and NHPI (**2a**) were used as the substrates (Scheme 2, bottom equation). According to the mechanism we proposed for the alkylation-aminoxidation reaction [37], the formation of **7a** is most likely due to an addition of the bromine radical to styrene (**1a**) followed by the phthalimide *N*-oxyl (PINO) radical. Formation of the bromine radical from **4b** can be rationalized by the consecutive decarboxylation and deethylenation [42] of 3-bromopropanoyloxy radical (**8**), which is formed from the SET reduction of **4b** (Scheme 3, top equation). Since this reaction provides easy access to the Markovnikov-type β-bromo-aminoxidation products from alkenes, it was studied in detail, and the results are summarized below.

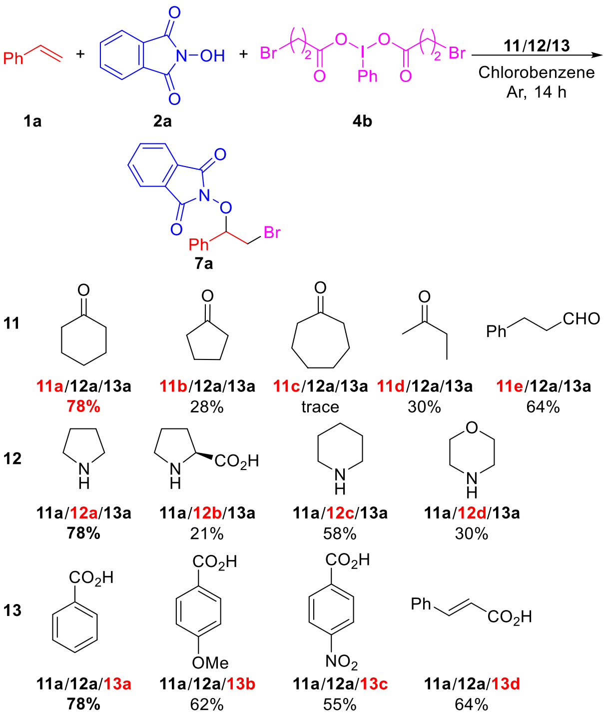


Scheme 3. Proposed formation of bromine radical from **4b** and the synthesis of **4b-d**.

2. Results and discussions

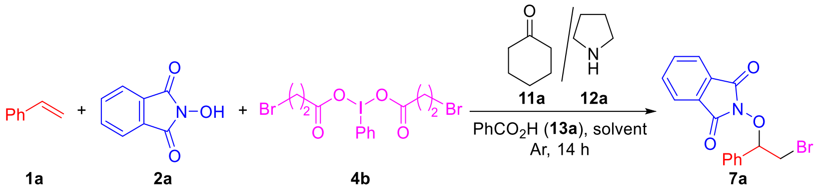
The required phenyliodine(III) dicarboxylates, such as **4b**, were synthesized from **4a** and the corresponding 3-halopropionic acids **10** *via* the ester exchange reaction (Scheme 3, bottom equation) [37,43]. In this way, **4b** and phenyliodine(III) bis(3-chloropropionate) (**4c**) were both obtained in almost quantitative yield. However, the synthesis of the corresponding iodo-derived **4d** in this manner failed completely (Scheme 3, bottom equation), most likely due to the oxidation of 3-iodopropionic acid by **4a**.

Once the starting materials were at hand, the reaction conditions were optimized by using styrene (**1a**), NHPI (**2a**), and **4b** as the substrates. Based on our previous results [37], cyclohexanone (**11a**) was used as the enamine precursor, pyrrolidine (**12a**) was used as the organocatalyst, and benzoic acid (**13a**) was used as the cocatalyst. The representative results are summarized in Table 1 and Scheme 4. When the reaction was carried out with 3.0 equiv. of **1a**, 2.0 equiv. of **4b**, 1.5 equiv. of **11a**, 0.20 equiv. of **12a** and **13a** (all relative to **2a**) in chlorobenzene at rt under argon, the desired product **7a** was obtained in 75% yield (Table 1, entry 1). Other common organic solvents turned out to produce lower yields of **7a** than chlorobenzene did under otherwise identical conditions (Table 1, entries 2-8). Conducting the reaction at 50 ºC led to a lower yield of 60% (Table 1, entry 9), while a similar reaction at 0 ºC gave only a trace amount of the desired compound (Table 1, entry 10). Similarly, conducting the reaction under air also led to a much lower yield of **7a** (15%, Table 1, entry 11). A slightly higher yield of **7a** (78%) could be achieved by using more cyclohexanone (**11a**), pyrrolidine (**12a**), and benzoic acid (**13a**) (3.0, 0.30, and 0.30 equiv., respectively) (Table 1, entry 12). Control reactions conducted under these conditions without either NHPI (**2a**), **4b**, **11a**, **12a**, or **13a** all failed to give any of the desired products (Table 1, entries 13-17), suggesting NHPI, **4b**, cyclohexanone (**11a**), pyrrolidine (**12a**), and benzoic acid (**13a**) are all crucial to the success of this reaction, which is not surprising since the reaction depends on the SET reduction of **4b** (Scheme 3) by the β-enaminyl radical derived from the enamine [37]. Thus, we next screened the enamine precursors (**11**), organocatalysts (**12**), and acid cocatalysts (**13**) under these conditions. As the results in Scheme 4 show, besides cyclohexanone (**11a**), 3-phenylpropanal (**11e**) also produced a good, but slightly lower, yield of **7a** (64%) as the enamine precursor. In contrast, all the other cyclic and acyclic ketones (**11b-d**) we screened all gave much lower yields of **7a** (Scheme 4). Thus, cyclohexanone (**11a**) was identified as the best enamine precursor for this reaction. Similarly, our screening of several readily available amine catalysts (**12a-d**) identified pyrrolidine (**12a**) as the best amine catalyst for this reaction (Scheme 4). Finally, several acid cocatalysts were screened (**13b-d**), and they all led to slightly inferior yields than benzoic acid (**13a**) did and, therefore, benzoic acid (**13a**) was identified as the best acid cocatalyst. In summary, this screening identified the combination of **11a**, **12a**, and **13a** is the best for obtaining product **7a**.



Scheme 4. Screening of the enamine precursors (**11**), amine catalysts (**12**), and acid cocatalysts (**13**). Reaction conditions: Styrene (**1a**, 3.0 mmol), NHPI (**2a**, 1.0 mmol), $PhI(O_2CCH_2CH_2Br)_2$ (**4b**, 2.0 mmol), the enamine precursor **11** (3.0 mmol), the amine catalyst **12** (0.30 mmol, 10 mol % of **11**), and the acid cocatalyst **13** (0.30 mmol, 10 mol % of **11**) in chlorobenzene (4.0 mL) under argon at room temperature for 14 h.

Table 1
Optimization of the reaction conditions [a]



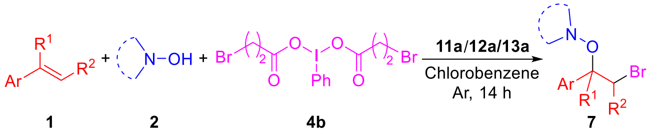
Entry	**1a** (equiv.)	**4b** (equiv.)	**11a** (equiv.)	**12a** (equiv.)	**13a** (equiv.)	Solvent	Yield of **7a** (%) [b]
1	3.0	2.0	1.5	0.20	0.20	Chlorobenzene	75
2	3.0	2.0	1.5	0.20	0.20	CH_3CN	26
3	3.0	2.0	1.5	0.20	0.20	THF	25
4	3.0	2.0	1.5	0.20	0.20	1,4-doxane	62
5	3.0	2.0	1.5	0.20	0.20	DMSO	20
6	3.0	2.0	1.5	0.20	0.20	EtOAc	37
7	3.0	2.0	1.5	0.20	0.20	Ether	43
8	3.0	2.0	1.5	0.20	0.20	$CHCl_3$	16
9 [c]	3.0	2.0	1.5	0.20	0.20	Chlorobenzene	60
10 [d]	3.0	2.0	1.5	0.20	0.20	Chlorobenzene	Trace
11 [e]	3.0	2.0	1.5	0.20	0.20	Chlorobenzene	15
12	3.0	2.0	3.0	0.30	0.30	Chlorobenzene	78
13 [f]	3.0	2.0	3.0	0.30	0.30	Chlorobenzene	---
14	3.0	---	3.0	0.30	0.30	Chlorobenzene	---
15	3.0	2.0	---	0.30	0.30	Chlorobenzene	---
16	3.0	2.0	3.0	---	0.30	Chlorobenzene	---
17	3.0	2.0	3.0	0.30	---	Chlorobenzene	---
18	3.0	2.0	1.5	0.30	0.30	Chlorobenzene	77
19	3.0	2.0	1.0	0.30	0.30	Chlorobenzene	62

[a] Unless otherwise indicated, all reactions were carried out using styrene (**1a**, 3.0 equiv.), NHPI (**2a**, 1.0 mmol), $PhI(O_2CCH_2CH_2Br)_2$ (**4b**), cyclohexanone (**11a**), pyrrolidine (**12a**, 20 mol % of **11a**), and benzoic acid (**13a**, 20 mol % of **11a**) in chlorobenzene (4.0 mL) under argon at room temperature for 14 h.
[b] Yield of the isolated product after column chromatography.
[c] Reaction was performed at 50 ˚C.
[d] Reaction was performed at 0 ˚C.
[e] Reaction was performed under air.
[f] NHPI (**2a**) was not added.

Finally, the loadings of **11a**, **12a**, and **13a** were optimized and it was found that an almost identical yield (77%) could be achieved with just 1.5 equiv. of cyclohexanone (**11a**), and 0.30 equiv. of pyrrolidine (**12a**) and benzoic acid (**13a**) each (Table 1, entry 18). Nevertheless, further dropping the loading of **11a** led to a lower yield of **7a** (Table 1, entry 19). Thus, the conditions in Table 1, entry 18 was identified as the best conditions for this reaction and they were adopted for the substrate scope study.

The results of the substrate scope study are summarized in Table 2. Besides styrene (**1a**, entry 1), substituted styrenes are also good substrates for this reaction (entries 2-7, 9), and good yields were obtained for styrenes with both an electron-donating (entries 2 and 3) and an electron-withdrawing group (entries 4-7, 9) at the *para*-position of the phenyl ring. There are no significant electronic effects on the yield of this reaction, although slightly lower yields are generally obtained with substrates with an electron-withdrawing group (entries 4-7, 9). Surprisingly, for an unknown reason, *para*-cyano-substituted styrene failed to give the desired product (entry 8). From the results obtained for 4-methoxy- (entry 3), 2-methoxy- (entry 10), and 3-methoxystyrenes (entry 11), as well as 4-fluoro- (entry 4) and 2-fluorostyrenes (entry 12), it is evident that the position of the substituent on the styrene phenyl ring has almost no effect on this reaction. Moderate to good yields were also obtained for the desired products of 2,3,4,5,6-pentafluorostyrene (58%, entry 13), 2-vinylnaphthalene (80%, entry 14), and α-phenylstyrene (64%, entry 15). Nevertheless, no desired product could be obtained from 2-vinylpyridine (entry 16). In addition to NHPI, HOBt could also be applied as the *N*-oxy radical precursor, and the corresponding β-bromo-aminoxidation products were obtained from several styrene derivatives (entries 17-21). The yields are mostly comparable to those of the NHPI products. It should be pointed out that we did not observe a regioselectivity switch in our reaction when HOBt was employed as the *N*-oxy radical precursor, as reported by Xu and co-workers [33].

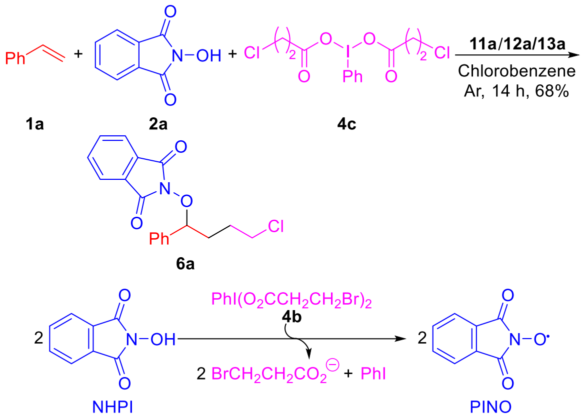
Table 2
Substrate scope study [a].



Entry	2	Ar	R^1	R^2	7/Yield (%)[b]
1	NHPI	Ph	H	H	**7a**/77
2	NHPI	4-MeC_6H_4	H	H	**7b**/74
3	NHPI	4-$MeOC_6H_4$	H	H	**7c**/71
4	NHPI	4-FC_6H_4	H	H	**7d**/64
5	NHPI	4-ClC_6H_4	H	H	**7e**/65
6	NHPI	4-BrC_6H_4	H	H	**7f**/68
7	NHPI	4-IC_6H_4	H	H	**7g**/72
8	NHPI	4-CNC_6H_4	H	H	**7h**/--
9	NHPI	4-$NO_2C_6H_4$	H	H	**7i**/61
10	NHPI	2-$MeOC_6H_4$	H	H	**7j**/70
11	NHPI	3-$MeOC_6H_4$	H	H	**7k**/72
12	NHPI	2-FC_6H_4	H	H	**7l**/63
13	NHPI	C_6F_5	H	H	**7m**/58
14	NHPI	2-Naphthyl	H	H	**7n**/80
15	NHPI	Ph	Ph	H	**7o**/64
16	NHPI	2-Pyridinyl	H	H	**7p**/--
17	HOBt	Ph	H	H	**7q**/75
18	HOBt	4-MeC_6H_4	H	H	**7r**/66
19	HOBt	4-BrC_6H_4	H	H	**7s**/71
20	HOBt	2-Naphthyl	H	H	**7t**/68
21	HOBt	Ph	Ph	H	**7u**/57

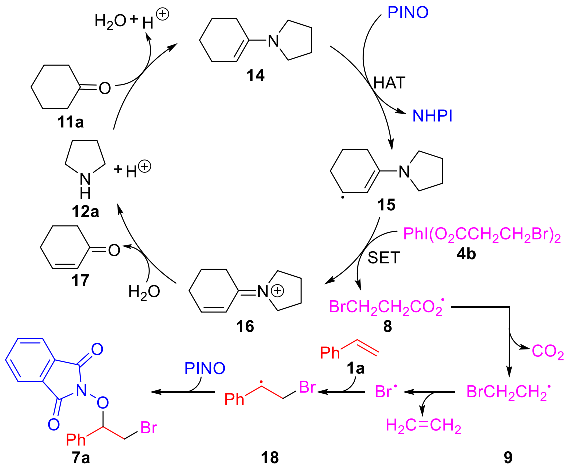
[a] Unless otherwise indicated, all reactions were carried out using **2** (1.0 mmol), alkene **1** (3.0 equiv.), $PhI(O_2CCH_2CH_2Br)_2$ (**4b**, 2.0 mmol), cyclohexanone (**11a**, 1.5 mmol.), pyrrolidine (**12a**, 0.30 mmol, 20 mol % of **11a**), and benzoic acid (**13a**, 0.30 mmol, 20 mol % of **11a**) in chlorobenzene (4.0 mL) under argon at the room temperature for 14 h.
[b] Yield of the isolated product after column chromatography.

In contrast, when phenyliodine(III) bis(3-chloropropionate) (**4c**) was applied under the optimized conditions with styrene (**1a**) and NHPI (**2a**), we did not obtain the corresponding β-chloro-aminoxidation product. Instead, we obtained the normal alkylation-aminoxidation [37] product **6a** in 68% yield (Scheme 5, top equation). Apparently, in the case of phenyliodine(III) bis(3-chloropropionate) (**4c**), the corresponding 3-chloropropanoyloxy radical does not undergo deethylenation to yield the chlorine radical.



Scheme 5. The reaction of **4c** and the generation of the PINO radical.

Based on the mechanism we proposed for the organocatalytic alkylation-aminoxidation reaction [37], a similar mechanism is proposed for the current organocatalytic Markovnikov-type β-bromo-aminoxidation reaction. As shown in Scheme 6, cyclohexanone (**11a**) reacts with the organocatalyst pyrrolidine (**12a**) and the acid cocatalyst to form the enamine intermediate **14**. Simutaneously, NHPI (**2a**) is oxidized by **4b** to the PINO radical (Scheme 5, bottom equation). The HAT reaction between **14** and PINO leads to the key β-enaminyl radical intermediate **15**. A SET between **15** and **4b** yields the 3-bromopropanoyloxy radical (**8**), which produces the bromine radical after decarboxylation and deethylenation [42]. Meanwhile, intermediate **15** is oxidized to the iminium intermediate **16**, which upon hydrolysis gives 2-cyclohexenone (**17**) and completes the catalytic cycle. The bromine radical adds to styrene (**1a**) regioselectively to form the more stable benzylic radical **18**, which catches the PINO radical to yield the final Markovnikov-type β-bromo-aminoxidation product **7a**.



Scheme 6. Proposed mechanism for the organocatalytic Markovnikov-type β-bromo-aminoxidation reaction.

3. Conclusion

We have demonstrated that phenyliodine(III) bis(3-bromopropionate) could be used as the bromine source for an unprecedented organocatalytic Markovnikov-type β-bromo-aminoxidation of styrene derivatives, and the desired products were obtained in moderate to good yields. An organocatalytic system consisting of cyclohexanone, pyrrolidine, and benzoic acid was used to release bromine radicals from phenyliodine(III) bis(3-bromopropionate) *via* a SET reaction. Phenyliodine(III) bis(3-bromopropionate) also served as the oxidant for generating the required *N*-oxyl radicals, which were needed for the formation of the key β-enaminyl radical intermediate and the final products.

4. Experimental

4.1. General Information

Unless otherwise specified, all reactions were monitored by TLC on aluminum-backed silica gel plates (200 μm) and visualized by UV. Column chromatography was performed on silica gel (40-63 μm). ^{1}H NMR spectra were recorded on a 500 MHz or a 300 MHz spectrometer (126 MHz or 75 MHz for ^{13}C NMR). All deuterated solvents were purchased from Cambridge Isotope Laboratories. Infrared spectra were recorded on a Bruker Vector 22 instrument. Melting points were recorded on a MEL-TEMP melting point apparatus in open capillaries and uncorrected. HRMS were conducted by the RCMI Core Facilities, Department of Chemistry, UTSA. Unless specified below, all chemicals are commercial products and were used as received.

*4.2. General Procedure for the Synthesis of **4b** and **4c** [37,43]*

To a 50-mL flame-dried round bottom flask were added $PhI(OAc)_2$ (**4a**, 0.322 mg, 1.0 mmol), 3-bromopropanoic acid (1**0b**) [or 3-chloropropanoic acid (**10c**)] (2.1 mmol 2.1 equiv.), and 20 mL toluene. The flask was then attached to a rotary evaporator, and toluene and the *in-situ* generated acetic acid were evaporated at 55 °C for 10 min. Then another 20 mL of toluene was added to the flask and the solvent was again evaporated at the same temperature for the same amount of time. This procedure was repeated another three times to get the desired phenyliodine (III) dicarboxylate with a 99% yield. The newly synthesized phenyl-λ^3-iodanediyl bis(3-halopropanoate)s are pure enough to be used in the halogenation reactions without any further purification.

4.3. General Experimental Procedure for the Markovnikov-Type Bromo-Aminoxidation Reaction

An oven-dried round-bottom flask was evacuated and backfilled with argon (repeated for three times). To this flask were added chlorobenzene (5 mL), cyclohexanone (**11a**, 147 mg, 1.5 mmol), pyrrolidine (**12a**, 21 mg, 0.30 mmol, 20 mmol % of **11a**), and benzoic acid (**13a**, 37 mg, 0.30 mmol, 20 mmol % of **11a**). The resulted solution was stirred for 15 min at room temperature under argon. Then styrene (**1a**, 312 mg, 3.0 mmol) and $PhI(O_2CCH_2CH_2Br)_2$ (**4b**, 1,016 mg, 2.0 mmol) were added followed by NHPI (**2a**, 163 mg, 1.0 mmol). The reaction mixture was further stirred under the same conditions for 14 h (monitored by TLC). Upon the completion of the reaction, the organic volatiles were evaporated under reduced pressure. The resulted crude reaction mixture was purified by flash column chromatography using 2 to 5% EtOAc in hexane to give product **7a** as white solid (265 mg, 77% yield).

Declaration of competing interests

John C.G. Zhao is an editorial member for *Green Synthesis and Catalysis* and was not involved in the editorial review or the decision to publish this article. All authors declare that there are no competing interests.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at

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