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# IBX as a Catalyst for Dehydration of Hydroperoxides: Green Entry to $\alpha$ , $\beta$ -Unsaturated Ketones via Oxygenative Allylic Transposition

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A catalytic transformation of allylic hydroperoxides into  $\alpha,\beta$ -unsaturated carbonyl compounds using IBX as a dehydration catalyst is described. The combination of a singlet oxygen ene reaction and the IBX-catalyzed dehydration provides  $\alpha,\beta$ -unsaturated carbonyl compounds from alkenes via oxygenative allylic transposition with H\_2O as the only byproduct.

Green and sustainable transformations are of increasing importance in recent years,<sup>1</sup> which particularly spurred the pursuit of catalytic methods that realize direct and efficient C–H functionalization.<sup>2</sup> Because of the ready accessibility of the substrate as well as the versatile uses of the product, the allyic C–H oxygenation of an alkene to give an allylic alcohol or  $\alpha$ , $\beta$ -unsaturated carbonyl compound has been a rewarding subject in synthetic organic chemistry.<sup>3</sup>

Two logical pathways have arisen from the C–H oxygenation of an alkene with an unsymmetrical allylic motif (Scheme 1): the direct oxygenation at the allylic position of alkene 1 to give 2 (Type I),<sup>3,4</sup> and oxygenation involving the migration of alkene 1 to give 3 (Type II).<sup>5</sup>

A number of useful methods to conduct Type I allylic oxygenation have been developed, constituting a reliable



Scheme 1 Pathways of oxygenation of unsymmetrical alkenes

strategy in organic synthesis. On the other hand, there has been a paucity of methods for Type II allylic oxygenations to furnish either allylic alcohols or  $\alpha$ , $\beta$ -unsaturated carbonyl compounds. For the latter transformation, only the protocol by Mihelich and coworkers, consisting of a sequential singlet oxygen ene reaction (Schenck ene reaction)<sup>5b,6</sup> with an alkene and the following acetylation-dehydroacetoxylation of the allylic hydroperoxide, has been shown for Type II allylic oxygenation to obtain the corresponding  $\alpha$ , $\beta$ -unsaturated carbonyl compounds (Scheme 2).<sup>7,8</sup> Although Mihelich's protocol has been applied to the synthesis of complex molecules,<sup>9</sup> several drawbacks remain to be addressed: the dehydration of hydroperoxide 5 into  $\alpha,\beta$ -unsaturated carbonyl compound 4 does not proceed under mild conditions, thus requiring the use of a stoichiometric activating reagent such as Ac<sub>2</sub>O to promote this reaction by generating extremely reactive acetyl alkylperoxide 6, which potentially suffers from thermal runaway. The use of a stoichiometric activating reagent results in the generation of stoichiometric organic wastes.



Considering the homology to the Kornblum–DeLaMare reaction of dialkyl peroxide, which proceeds under relatively mild conditions including catalytic processes,<sup>10</sup> we envisaged the development of a catalytic transformation of allylic hydroperoxides to give  $\alpha,\beta$ -unsaturated carbonyl compounds (Scheme 3). Herein, we report the first organocatalytic transformation of allylic hydroperoxides to give  $\alpha,\beta$ -unsaturated carbonyl compounds using the hypervalent iodine reagent IBX. We also demonstrate that the IBX catalysis enabled

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a one-operation process of the Schenck ene reaction and concomitant fragmentation of the primarily generated allylic hydroperoxides to give  $\alpha,\beta$ -unsaturated carbonyl compounds via a Type II reaction.



Scheme 3 Fragmentation of alkyl peroxides

First, we explored basic or acidic catalysts that are used in a typical Kornblum-DeLaMare reaction to convert an allylic hydroperoxide into an  $\alpha,\beta$ -unsaturated carbonyl compound. However, these catalysts did not promote the reaction well (see ESI), prompting us to explore an alternative strategy to achieve this transformation (Scheme 4). We conceived that an electrophilic covalent catalysis would achieve the transformation of allylic hydroperoxides to give  $\alpha$ , $\beta$ unsaturated carbonyl compounds.<sup>11</sup> A hydroperoxide and an electrophile would react to provide a covalent intermediate 7. Then 7 would collapse into an enone (4) and a hydrate (9), via a proton transfer and O-O bond cleavage. Finally, the catalyst would regenerate by dehydration.



Scheme 4 Working hypothesis

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We screened electrophiles for the catalytic transformation of 2-cyclohexene-1-hydroperoxide (5a) into 2-cyclohexene-1-one (4a) (Table 1). Although treatment of hydroperoxide 5a with several classes of electrophiles at room temperature for 12 h resulted in almost no reaction (entries 1, 2), the use of PhI(OAc)<sub>2</sub> provided the desired enone in low yield (entry 3).<sup>12,13</sup> Then, we compared the reactivity of hypervalent iodine catalysts (entries 3-8). In the case of IBA and IBX, small amount of DMSO was added to dissolve the catalysts (entries 6 and 8). We found that IBX was the optimal electrophilic catalyst for this reaction (entry 8). We consider that higher electrophilicity of IBX compared to iodine(III) reagents realized the efficient catalytic reaction. The condition using DMP in CH<sub>2</sub>Cl<sub>2</sub> generated insoluble solid, which was estimated as IBX, and thus the reaction stopped at low conversion (entry 7). The use of DMSO as the sole solvent under more concentrated conditions gave higher yield of 4a (entry 9). Next, we examined several acidic additives to promote the reaction (entries 10-12).<sup>14</sup> Eventually, *p*-TsOH was identified as the best acid catalyst to complete this reaction within 4 h and enone 4a was obtained in good yield (entry 12). Decreasing the catalytic amount prolonged the reaction (entry 13). IBX-OTs<sup>15</sup> showed a similar reactivity to a combination of IBX and p-TsOH, but slight decomposition was observed (entry 14). The reaction did not proceed in the absence of IBX (entry 15).

	5a	catalyst (1 addii solvent,	0 mol %) ive rt, 12 h 4a	
entry	catalyst	solvent	additive	yield <sup>b</sup>
	-			(%)
1	(F <sub>3</sub> C) <sub>2</sub> C=O	$CH_2CI_2$		Trace
2	AZADO <sup>+</sup> BF <sub>4</sub> <sup>-</sup>	$CH_2CI_2$		7
3	PhI(OAc) <sub>2</sub>	$CH_2CI_2$		12
4	PhIO	$CH_2CI_2$		11
5	PhI(OH)OTs	$CH_2CI_2$		dec.
6	IBA	$CH_2CI_2^c$		37
7	DMP	$CH_2CI_2$		12
8	IBX	$CH_2CI_2^c$		59
9	IBX	DMSO		68
10	IBX	DMSO	TFA <sup>d</sup>	83
11	IBX	DMSO	(PhO)₂P(O)OH <sup>d</sup>	77
12	IBX	DMSO	p-TsOH∙H₂O <sup>e</sup>	83 (4 h)
$13^{f}$	IBX	DMSO	<i>p-</i> TsOH·H₂O	70
14	IBX-OTs	DMSO		68 (6 h)

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

<sup>*a*</sup>All reactions were carried out with 0.2 mmol of **5a** in 1 mL of CH<sub>2</sub>Cl<sub>2</sub> or 0.4 mL of DMSO. <sup>*b*</sup>Determined by <sup>1</sup>H-NMR analysis using mesitylene as an internal standard. <sup>*c*</sup> 5% DMSO was added. <sup>*d*</sup> 20 mol% <sup>*e*</sup> 10 mol% <sup>*f*</sup> 5 mol% IBX and *p*-TsOH-H<sub>2</sub>O were used. AZADO<sup>+</sup>BF<sub>4</sub><sup>-</sup> = 2-azaadamantane *N*-oxoammonuim tetrafluoroborate, IBA = 2-iodosobenzoic acid, DMP = Dess–Martin periodinane, IBX = 2-iodoxybenzoic acid, TFA = trifluoroacetic acid, *p*-TsOH = *p*-toluenesulfonic acid, IBX-OTs = 2-iodoxybenzoic acid tosylate.

 $p-TsOH \cdot H_2O^e$ 

0

DMSO

With the optimal conditions determined, we evaluated the scope of the substrate (Scheme 5). These conditions tolerated various functional groups including esters (**4b**–**e**), a nitrile (**4c**), a phthalimide (**4d**), an amide (**4f**), and a silyl ether (**4n**). The bulky substrate prolonged the reaction, but the resulting enone was obtained in good yield (**4g**). The hydroperoxides obtained from cyclic alkenes gave corresponding cyclic enones in moderate to good yield (**4i**–**n**). Cyclopentenone **4j**, which is the key intermediate in the synthesis of natural products or the ligand of organometallic complexes, was obtained in reasonable



Scheme 5 Substrate scope <sup>a</sup> Isolated as a 2,4-dinitrophenylhydrazone.

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yield.<sup>16</sup> The primary hydroperoxide was also applicable to this reaction to give the  $\alpha$ , $\beta$ -unsaturated aldehyde **4m**. Unfortunately, the acetal-protected substrate decomposed to give enone 4o in low yield.

Encouraged by the promising results, we investigated a oneoperation process of the singlet oxygen ene reaction and concomitant IBX-catalyzed transformation of an allylic hydroperoxide into an  $\alpha$ , $\beta$ -unsaturated carbonyl compound.<sup>17</sup> This method would provide a solution to the problems of handling the potentially explosive allylic hydroperoxide. It is noted that singlet oxygen has to be generated in the presence of photosensitizers, which are often deactivated by oxidants such as IBX. We screened the photosensitizers under O2 atmosphere with irradiation by a household fluorescent lamp to avoid the use of special equipment. As a result, it was found that  $C_{60}$  is the suitable photosensitizer to give enone **4k** in good yield (Scheme 6).<sup>18,19</sup> This method gave various cyclic enones including cyclopentenones (4p, 4i), cyclohexenones (4q, 4r), cycloheptenones (4s-u), and cyclooctenone 4v. Cyclohexenes 1q and 1r, and cyclooctene 1v reacted with singlet oxygen slowly; corresponding enones were obtained in low yield after 24 h with the recovered alkenes. Heterocyclic alkene was also applicable to give enone 4w. Acyclic enone 4h was obtained in moderate yield under these conditions because of the low selectivity of a singlet oxygen ene reaction of a trisubstituted alkene.

#### C<sub>60</sub> (1 mol %), O<sub>2</sub> (balloon) hv (22 W fluorescent lamp) IBX (10 mol %), p-TsOH·H2O (10 mol %) CHCl3-DMSO (4:1, 0.4 M), rt BZO BzC 4q: 30<sup>m</sup> (24 h) **4k**: 77% (10 h) **4p**: 46%<sup>a</sup> (12 h) **4i**: 57% (24 h) (10 mmol scale: 76%) (93% brsm) BzC BzC BzO BzO BzO-4r: 19% (24 h) quant<sup>a</sup> (12 h) 4u: 65% (24 h) 4s: 4t: 68% (24 h) (79% brsm) (85% brsm) BzC TsN BzO 4w: 70% (24 h) 4v: 37% (24 h) 4h: 43% (9 h) (77% brsm) (90% brsm)

Scheme 6 Scope of the one-operation reaction "Isolated as a 2,4dinitrophenvlhvdrazone.

Next, we confirmed the safety of this one-operation process. Monitoring of the reaction mixture by <sup>1</sup>H-NMR clearly indicated that a low concentration of allylic hydroperoxide 5k existed (Fig. 1). Thus, this method would provide a solution to the handling of the potentially explosive intermediate. Note that we consider hydroperoxide 5k was the reliable intermediate of this oneoperation process on the basis of the experiments shown below: (i) almost no reaction was observed when DABCO, which is known to quench singlet oxygen,<sup>20</sup> was added; (ii) hydroperoxide **5k** was obtained in the absence of IBX; (iii) trapping experiment of singlet oxygen using 9,10dimethylanthracene suggested that singlet oxygen was generated under these one-operation reaction conditions; (iv) no reaction was observed in dark (see section 4 in the ESI).



Fig. 1 Monitoring of reaction mixture <sup>a</sup> Determined by <sup>1</sup>H-NMR analysis using mesitylene as an internal standard.

To gain insight into the mechanism of the IBX-catalyzed dehydration, we carried out some experiments and calculations below (see the ESI for details): (i) <sup>18</sup>O-labelling experiments indicated that the oxygen atom of the enone products should come from molecular oxygen (Scheme 7a). (ii) to evaluate the feasibility of the mechanism in Scheme 4, the activation energy of the proton transfer and O-O bond cleavage step was estimated by DFT calculations. Although a six-membered transition state (cf. 7) gave a high activation energy ( $\Delta G^{\dagger} = 31.7$ kcal/mol), a ten-membered (or linear) transition state incorporating a DMSO molecule gave a reasonable activation energy ( $\Delta G^{\dagger}$  = 23.7 kcal/mol). Based on the above results, we propose a possible reaction mechanism (Scheme 7-b). An allylic hydroperoxide, generated by a singlet oxygen ene reaction of an alkene (1), would reacts with IBX to form an adduct (7'), which would collapse with the deprotonation by a DMSO (or another basic) molecule to afford an enone (4).



Scheme 7. Mechanistic Investigation

In conclusion, we have developed a method of catalytic transformation of allylic hydroperoxides into  $\alpha$ , $\beta$ -unsaturated carbonyl compounds, which are easily prepared by an ene reaction of singlet oxygen and alkenes, using IBX and p-TsOH as

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the catalysts. This reaction is the first example of not only the catalytic transformation of hydroperoxides into enones, but also the use of a hypervalent iodine catalyst other than in oxidation reaction. The combination of singlet oxygen ene reaction and this method enables oxygenative allylic transposition of alkenes into enones with water as the only stoichiometric waste. This reaction was applicable to the one-operation conversion of alkenes to enones. Further studiess to increase the efficiency of the one-operation reaction and to gain the insight into the mechanism are under way.

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#### **Conflicts of interest**

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There are no conflicts to declare.

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