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# Synthesis of $\alpha$ -ester $-\beta$ -keto peroxides via iron-catalyzed carbonylation–peroxidation of $\alpha$ , $\beta$ -unsaturated esters

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# ABSTRACT

A general and practical method for the synthesis of  $\alpha$ -ester $-\beta$ -keto peroxides has been achieved by ironcatalyzed three-component reactions of alkenes, aldehydes, and TBHP. A wide variety of functionalized organic peroxides were synthesized efficiently and selectively.

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#### 1. Introduction

Organic peroxides have received considerable attention in pharmacy, biochemistry and food chemistry because of their close relationship with drug design, cell damage, and food safety.<sup>1</sup> Organic peroxides also play as key reactive intermediates in synthetic chemistry.<sup>2</sup> Hydroperoxides and cycloperoxides are typically obtained by the radical processes in the presence of  $O_2$  or  $H_2O_2$ (Scheme 1, Method A).<sup>3</sup> Kharasch and co-workers were the pioneers to introduce peroxy into organic molecules by the use of hydroperoxides in the presence of various transition-metal catalysts.<sup>4</sup> Since these pioneering works, this strategy has been developed as an efficient and powerful method for peroxidation (Scheme 1, Method B).<sup>5</sup> In addition, the addition of peroxy radicals to alkenes (Scheme 1, Method C) and the base-catalyzed nucleophilic reactions of hydroperoxides with various electrophiles (Scheme 1, Method D) were also developed to generate the mixed peroxides.<sup>6</sup> However, the established methods have more-limited scope. Therefore, new peroxidation methods are highly desirable and valuable. Recently, we reported an iron-catalyzed three-component carbonylation-peroxidation reaction of alkene, aldehyde and hydroperoxide.<sup>7</sup> The methodology allows a carbonyl group<sup>8</sup> and a peroxy group to add across C=C bonds of styrene derivatives selectively.<sup>9</sup> The generated mixed peroxides could be

efficiently and selectively transformed into multisubstituted  $\alpha$ carbonyl epoxides in the presence of a base catalyst.<sup>7</sup> These preliminary results prompt us to further investigate iron-catalyzed three-component reactions of  $\alpha$ , $\beta$ -unsaturated esters, aldehydes, and *tert*-butyl hydroperoxide (TBHP). The study highlights that the densely functionalized peroxides,  $\alpha$ -ester- $\beta$ -keto peroxides, which are difficult to be obtained through the conventional methods, were efficiently and selectively built up from three-component assembling (Scheme 1, Method E).



Scheme 1. General methods for synthesis of organic peroxides.





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### 2. Results and discussion

#### 2.1. Optimization of the reaction conditions

The reaction of benzyl methacrylate (**1a**) with benzaldehyde (**2a**) and TBHP (**3**) was chosen to establish the suitable reaction conditions (Table 1). The yields of the desired carbon-ylation-peroxidation product **4aa** were substantially improved through increasing the amount of **2a** and **3** (entries 1–4). It is worth noting that **4aa** was obtained in the absence of catalyst, albeit in 31% yield (41% for 6 h) (entry 5). Furthermore, the catalyst screening with various iron sources (entries 6–13) as well as other metal salts (entries 14–17) indicated FeCl<sub>2</sub> as the best catalyst for the transformation.<sup>10</sup> To our satisfied, an excellent result was obtained using 4.0 equiv of TBHP (**3**) in CH<sub>3</sub>CN at 85 °C (91%, entry 18).

#### Table 1

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



| Entry | <b>2a</b> (equiv) | 3 (equiv) | Catalyst (2.5 mol %)                 | <b>4aa</b> yield (%) <sup>b</sup> |
|-------|-------------------|-----------|--------------------------------------|-----------------------------------|
| 1     | 1                 | 1         | FeCl <sub>2</sub>                    | 10                                |
| 2     | 1                 | 3         | FeCl <sub>2</sub>                    | 28                                |
| 3     | 3                 | 3         | FeCl <sub>2</sub>                    | 74                                |
| 4     | 5                 | 3         | FeCl <sub>2</sub>                    | 82                                |
| 5     | 5                 | 3         | _                                    | 31(41) <sup>c</sup>               |
| 6     | 5                 | 3         | FeCl <sub>3</sub>                    | 73                                |
| 7     | 5                 | 3         | Fe(OAc) <sub>2</sub>                 | 68                                |
| 8     | 5                 | 3         | $Fe_2(CO)_9$                         | 70                                |
| 9     | 5                 | 3         | FeBr <sub>2</sub>                    | 56                                |
| 10    | 5                 | 3         | FeCl <sub>3</sub> ·6H <sub>2</sub> O | 50                                |
| 11    | 5                 | 3         | FeSO <sub>4</sub> ·7H <sub>2</sub> O | 41                                |
| 12    | 5                 | 3         | Fe(acac) <sub>2</sub>                | 30                                |
| 13    | 5                 | 3         | Fe(acac) <sub>3</sub>                | 40                                |
| 14    | 5                 | 3         | CuCl                                 | 72                                |
| 15    | 5                 | 3         | CuBr <sub>2</sub>                    | 49                                |
| 16    | 5                 | 3         | CoCl <sub>2</sub>                    | 52                                |
| 17    | 5                 | 3         | $Mn(OAc)_2$                          | 74                                |
| 18    | 5                 | 4         | FeCl <sub>2</sub>                    | 91                                |

 $^{\rm a}$  Conditions: 1a (0.5 mmol), catalyst (2.5 mol %), MeCN (3 mL), 85 °C, 1 h, under nitrogen atmosphere.

<sup>b</sup> Reported yields were based on **1a** and determined by <sup>1</sup>H NMR using an internal standard. <sup>c</sup> 6 h.

# 2.2. The scope of aldehydes

With the optimized conditions in hand, the scope of aldehydes (2) was investigated by the use of **1a** as a model substrate (Table 2). Both aromatic and aliphatic aldehydes were successfully applied to the present transformation. Electron-donating substituted benzaldehydes (2b and 2c) afforded the desired peroxides in excellent yields (entries 1 and 2), while benzaldehydes with electronwithdrawing group (2d-f) retarded the efficiency of the transformation (entries 3–5). Heteroaromatic aldehydes (**2g**–**i**) also led to the corresponding peroxides (entries 6–8). 1-Naphthaldehyde (2j) showed a lower efficiency due to the steric effect (entry 9). Aliphatic aldehydes could also be used to give the desired peroxides under the standard conditions (entries 10-12). Importantly, the reactions of formamides (2n and 2o) with 1a led to the expected peroxides, albeit with the use of large excess amount of formamides in the absence of MeCN (entries 13-14). Interestingly, the decarbonylation product (5) instead of the desired carbonvlation-peroxidation product was obtained quantitatively when pivaldehyde **2p** was applied under the standard conditions (Eq. 1).

# Table 2

The scope of aldehydes<sup>a</sup>

|       | )Bn FeCl <sub>2</sub><br>+RCHO+ <i>t</i> -BuOOH MeC | (2.5 mol 9<br>CN (3 mL) | %)<br>R | DO-t-Bu                    |
|-------|---|-------------------------|---------|----------------------------|
| Entrv | 2 3 00  | ) C, TH                 | 4       | O<br>Yield(%) <sup>b</sup> |
| 1     | MeO   | 2b                      | 4ab     | 89(75)                     |
| 2     | Me  | 2c                      | 4ac     | 92(78)                     |
| 3     | Br  | 2d                      | 4ad     | 80(72)                     |
| 4     | AcHN  | 2e                      | 4ae     | 70(58)                     |
| 5     | NC  | 2f                      | 4af     | 55(46)                     |
| 6     | O<br>HN   | 2g                      | 4ag     | 73(63)                     |
| 7     | (s) o   | 2h                      | 4ah     | 66(56)                     |
| 8     |   | 2i                      | 4ai     | 55(45)                     |
| 9     | °   | 2j                      | 4aj     | 27(13)                     |
| 10    |   | 2k                      | 4ak     | 73(72)                     |
| 11    |   | 21                      | 4al     | 58(46)                     |
| 12    |   | 2m                      | 4am     | 84(80)                     |
| 13    | Me<br>N<br>Me                                       | 2n                      | 4an     | 60(45) <sup>c</sup>        |
| 14    | Et<br>N<br>Et                                       | 20                      | 4a0     | 57(51) <sup>c</sup>        |

<sup>a</sup> Conditions: **1a** (0.5 mmol), 85 °C, 1 h, under nitrogen atmosphere.

<sup>b</sup> Reported yields were based on **1a** and determined by <sup>1</sup>H NMR using an internal standard; isolated yields were given in parentheses.

2k (10 mmol); without MeCN.

$$1a + \bigcup_{\substack{P \\ P}} + 3 \xrightarrow{FeCl_2 (2.5 \text{ mol }\%)}_{MeCN, 85 °C, 1 h} \xrightarrow{t-BuOO}_{Me} + 100 \text{ mol s} (1)$$

This result indicated that decarbonylation competes with carbonylation kinetically depended on the nature of substituent of aldehydes.<sup>11</sup>

#### 2.3. The scope of terminal alkenes

Subsequently, the scope of  $\alpha$ -substituted acrylates was investigated under the standard conditions (Table 3). A verity of functional groups was well compatible with this transformation under optimized conditions and afforded the corresponding peroxides in good to excellent yields (entries 1–9). The scope of this carbonylation–peroxidation could be extended to the Baylis–Hillman products bearing *O*-protected groups, however, the transformation showed no diastereoselectivity (entries 10–12). 3-Methylenedihydrofuran-2,5-dione **1n** also reacted smoothly with **2a** (entry 13). It should be noted that a dimeric product **6** was obtained in 20% yield together with the desired peroxide **40a** when methyl acrylate was tested under the standard reaction conditions

#### Table 3

The reactions of terminal alkenes<sup>a</sup>





<sup>a</sup> Conditions: **1** (0.5 mmol), 85 °C, 1 h.

<sup>b</sup> Yields were based on 1 and determined by <sup>1</sup>H NMR using an internal standard; isolated yields were given in parentheses.

<sup>c</sup> Two diastereomers (3:2).

<sup>d</sup> Two diastereomers (2:1).

e Two diastereomers (1:1).

<sup>f</sup> 3 h.



(Eq. 2). This result indicated that  $\alpha$ -substituent is important to gain the high selectivity.

#### 2.4. The scope of internal alkenes

It is encouraging that the implementation of this synthetic strategy to tri-/tetra-substituted acrylates could also give the desired carbonylation—peroxidation products (Table 4), which validates the reaction as a general and practical method for synthesis of multifunctional organic peroxides. In most cases, the corresponding peroxides were efficiently obtained using internal alkenes bearing a  $\beta$ -alkyl (entries 1–3, 7–11),  $\beta$ -phenyl (entries 4–6, 13),  $\beta$ -ester (entry 12) in excellent yields. Noticeably, aromatic aldehydes showed lower reactivity than aliphatic aldehydes in some cases, for examples, entry 1 versus entries 2–3 and entry 4 versus entries 5–6. Methylenyl Meldrum's acid derivatives (**1x**–**z**) were also applicable for the present transformation (entries 14–17). Importantly,  $\beta$ , $\beta$ -disubstituted substrate **1z** reacted with **2a** to generate two vicinal quaternary carbon centers, albeit with a much lower efficiency (entry 17).

#### Table 4

The reactions of internal alkenes<sup>a</sup>



| Entry  | 1              |    | 2        | <b>4</b> , yield(%) <sup>b</sup>                                     |
|--------|----------------|----|----------|--|
| 1      | COOMe<br>Me Me | 1p | 2a       | <b>4pa</b> , 74(55) <sup>c</sup>                                     |
| 2<br>3 |                |    | 2k<br>2m | <b>4pk</b> , 96(64) <sup>c</sup><br><b>4pm</b> , 96(92) <sup>c</sup> |
| 4      | Ph COOMe       | 1q | 2a       | <b>4qa</b> , 36(30)  |
| 5<br>6 | 0005           |    | 2k<br>2l | <b>4qk</b> , 85(75)<br><b>4ql</b> , 87(74)                           |
| 7      | Me COOEt       | 1r | 2k       | <b>4rk</b> , 99(91)  |
| 8      |                |    | 21       | <b>4rl</b> , 94(87)  |
| 9      | Pr COOEt       | 1s | 21       | <b>4sl</b> , 99(87)  |
| 10     | Me<br>Me<br>Me | 1t | 21       | <b>4tl</b> , 74(64)  |
| 11     | Me COOEt       | 1u | 21       | <b>4ul</b> , 71(63) <sup>d</sup>                                     |
| 12     | MeOOC<br>MeOOC | 1v | 2a       | <b>4va</b> , 99(90) <sup>d</sup>                                     |
| 13     | Ph CN          | 1w | 2a       | <b>4wa</b> , 99(90) <sup>d</sup>                                     |
| 14     | Me             | 1x | 2a       | <b>4xa</b> , 90(90)  |
| 15     | MeO            |    | 21       | <b>4xl</b> , 68(63)  |
| 16     | Ph O           | 1у | 2a       | <b>4ya,</b> 87(81)   |
| 17     |                | 1z | 2a       | <b>4za</b> , 18(18)  |

<sup>a</sup> Conditions: **1** (0.5 mmol), 85 °C, 1 h, under nitrogen atmosphere.

<sup>c</sup> Two diastereomers (2:1).

# 2.5. A proposed reaction mechanism

Based on our results and literature reports,<sup>12</sup> a tentative reaction mechanism for iron-catalyzed three-component reactions of  $\alpha$ , $\beta$ unsaturated esters, aldehydes, and hydroperoxide is given in Scheme 2.<sup>7</sup> Alkyloxy and alkylperoxy radicals are generated from a serious of steps, including steps (a) and (b), by iron catalyst. Subsequently, hydrogen abstraction by oxyl radicals, such as *tert*butoxyl radical, gives acyl radical (step c). The nucleophilic radical addition of acyl radical to C=C bond leads to a regioselective  $\alpha$ ester radical (step d). Finally, the desired peroxide is formed by radical coupling (step e).<sup>5f,13</sup>



**Scheme 2.** A tentative reaction mechanism for iron-catalyzed carbonylation—peroxidation.

#### 3. Conclusion

A general and practical method for the synthesis of multifunctional peroxides has been developed by iron-catalyzed threecomponent reactions of  $\alpha$ , $\beta$ -unsaturated esters, aldehydes, and hydroperoxide. The densely functionalized peroxides, 2-peroxy-1,4-dicarbonyls, were efficiently and selectively synthesized with a broad scopes of the substrates. The results are the basis of the studies on these organic peroxides. The works on the reactivity of the obtained peroxides are undergoing in this lab.

#### 4. Experimental section

#### 4.1. General information

<sup>1</sup>H NMR spectra were recorded on Bruker 400 MHz spectrometer and the chemical shifts were reported in parts per million ( $\delta$ ) relative to internal standard TMS (0 ppm) for CDCl<sub>3</sub>. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; g, guartet. The coupling constants, J, are reported in Hertz (Hz). <sup>13</sup>C NMR spectra were obtained at Bruker 100 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl<sub>3</sub>). CDCl<sub>3</sub> was used as the NMR solvent. Mass spectra were obtained on a VG ZAB-HS mass spectrometer. APEX II (Bruker Inc.) was used for HRMS and ESI-MS. IR spectra were recorded by a Nicolet 5MX-S infrared spectrometer. Flash column chromatography was performed over silica gel 200-300. All reagents were weighed and handled in air at room temperature. Unless otherwise noted, all reactions were performed under a nitrogen atmosphere. All reagents were purchased from Alfa, Acros, Aldrich, and TCI and used without further purification.

#### 4.2. General procedure for the products 4

To a mixture of alkene 1 (0.5 mmol), aldehyde 2 (2.5 mmol), and FeCl<sub>2</sub> (1.6 mg, 0.0125 mmol), acetonitrile (3.0 mL) was

<sup>&</sup>lt;sup>b</sup> Reported yields were based on **1** and determined by <sup>1</sup>H NMR using an internal standard; isolated yields were given in parentheses.

added under nitrogen at room temperature. Then *tert*-butyl hydroperoxide **3** (TBHP, 2.0 mmol, 5–6 M in decane) was dropped into the mixture under nitrogen at room temperature. The resulting mixture was stirred under 85 °C for 1 h. The temperature of reaction was cooled to room temperature. The resulting reaction solution was directly filtered through a pad of silica by ethyl acetate. The solvent was evaporated in vacuo to give the crude products. NMR yields are determined by <sup>1</sup>H NMR using dibromomethane as an internal standard. Solvent was evaporated and the residue was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether as eluent to afford the pure product **4**.

4.2.1. Benzyl 2-(tert-butylperoxy)-2-methyl-4-oxo-4phenylbutanoate (**4aa**). Isolated by flash column chromatography (ethyl acetate/petroleum ether=1:10,  $R_{\rm f}$ =0.5). IR (neat):  $v_{\rm max}$  2958, 2934, 1750, 1716 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.95 (d, *J*=7.2 Hz, 2H), 7.54 (t, *J*=7.2 Hz, 1H), 7.43 (t, *J*=7.2 Hz, 2H), 7.38–7.26 (m, 5H), 5.20 (s, 2H), 3.73 (d, *J*=16.4 Hz, 1H), 3.42 (d, *J*=16.4 Hz, 1H), 1.67 (s, 3H), 1.13 (s, 9H); <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  196.7, 171.3, 137.1, 135.8, 133.1, 128.4, 128.3, 128.2, 128.0, 127.9, 82.6, 80.0, 66.6, 43.9, 26.3, 20.7; MS(EI) m/z (%): 282 (M<sup>+</sup>–88), 191, 174, 162, 143, 120, 105, 91 (100), 77, 73, 57, 43, 29; HRMS calcd for C<sub>22</sub>H<sub>26</sub>NaO<sub>5</sub> (M<sup>+</sup>+Na): 393.1678; found:393.1673.

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#### Supplementary data

Experimental details, characterization data, copies of <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra for all products. Supplementary data related to this article can be found at http://dx.doi.org/10.1016/ j.tet.2012.09.110.

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