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## ORIGINAL ARTICLE

# Oxidative esterification of aldehydes with alcohols using imidazolium perrhenate catalysts

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## KEYWORDS

Imidazolium perrhenate;  
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**Abstract** Four imidazolium perrhenates were synthesized, characterized and used as efficient catalysts for the oxidative esterification of benzaldehydes with alcohols. The perrhenates showed high catalytic activity toward oxidative esterifications, whereas good yields could be obtained for a series of aldehyde and alcohol substrates. The results indicate that the steric effects of functional groups on the aromatic ring play important roles in the formation of esters. Moreover, the alkyl structures of the alcohols also dramatically affect the product yields.

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## 1. Introduction

Carboxylic esters are very important key raw materials in organic synthesis. In industry, esters are mainly produced by the esterification of carboxylic acid (or their derivatives) with alcohols using Brønsted or Lewis acids as catalysts [1–3]. However, these traditional catalytic methods need a multi-step process and are air polluted, corrosive and produce a large amount of unwanted by-products [4,5]. Esters can be also obtained through the cross-coupling of aryl halides with alcohols, in which the carbonylation of halides is involved. Nevertheless, this reaction has to be carried out under high

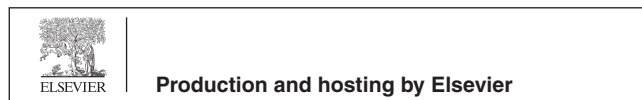
temperature and CO pressure [6]. Recently, the direct oxidative esterification of aldehydes with alcohols using transition metal catalysts under mild conditions has received considerable attention. Some simple transition metal salts such as Cu (ClO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O [7], In Br<sub>3</sub> [7], ZnBr<sub>2</sub> [8], ZnCl<sub>2</sub> [8] and Fe (ClO<sub>4</sub>)<sub>3</sub>·xH<sub>2</sub>O [9] can be applied as effective catalysts for this transformation using tert-butyl hydrogen peroxide (TBHP) or aqueous hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) as oxidants, with a good to excellent yield obtained for a variety of aldehyde substrates. Many organometallic catalysts such as methyltrioxorhenium (MTO) [10], IrCl(cod)<sub>2</sub> [11], (t-Bu<sub>2</sub>POH)<sub>2</sub>PdCl<sub>2</sub> [12], RuH<sub>2</sub>(PPh<sub>3</sub>)<sub>4</sub> [13], Ru<sub>3</sub>(CO)<sub>12</sub> [14] and HRh(CO)(PPh<sub>3</sub>)<sub>3</sub> [15] also display excellent catalytic activity toward oxidative esterification of aldehydes. However, these organometallic catalysts are normally difficult to prepare and also thermally unstable in a homogeneous catalytic system. MTO combining with a co-catalyst such as bromide or chloride ions, with hydrogen peroxide as oxidant was found to be active for oxidative esterification of aldehydes to esters [10]. However, MTO displays low stability in the examined catalytic systems and decomposes

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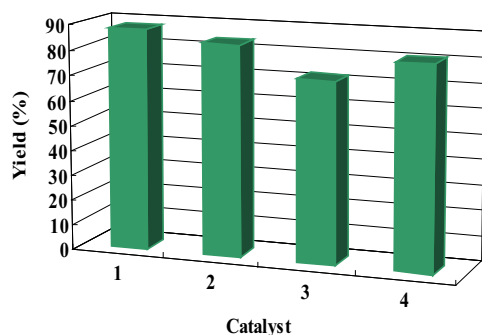


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**Figure 1** Oxidative esterification of benzaldehyde with catalysts 1–4. Reaction condition: benzaldehyde (2 mmol, 212 mg), MeOH (2 mL), catalyst (8 mol%), UHP (4 equiv.) at 80 °C for 24 h.

readily into inactive perrhenates. For long time, it was believed that perrhenates were inactive for catalytic oxidations. Surprisingly, it has been found recently that some perrhenate based inorganic-organic hybrid compounds could promote olefin epoxidation [16], sulfide oxidation [17] and even the hydrolysis of cellulose [18]. Comparing to organometallic catalysts, these organic perrhenates are more stable – an important advantage for the catalyst. In continuation of the previous studies, we were interested in further examining the catalytic behavior of these organic perrhenates in oxidative esterifications. In this regard, for (see Fig. 1) the first time, the oxidative esterification of aldehyde with alcohol using organic perrhenates as catalyst in the presence of urea hydrogen peroxide (UHP) as oxidants is reported herein.

## 2. Results and discussion

### 2.1. Synthesis and characterization

Four kinds of organic perrhenate catalysts have been synthesized. The structures are shown in Scheme 1. The compounds were characterized by Fourier Transform Infrared Spectroscopy (FT-IR), Nuclear Magnetic Resonance spectra ( $^1\text{H}$  NMR) and elemental analysis (EA) to confirm their structures. Despite compounds 1–4 show “ionic liquid – like” structures, the melting points are all above 100 °C and they present as white solids at room temperature. The thermogravimetric analysis (TGA) data indicated that all the compounds show negligible volatility and high thermal stability with a decomposition onset temperature near 300 °C. All three compounds showed similar IR bands at around  $910\text{ cm}^{-1}$ , indicating a

typical  $\text{Re}=\text{O}$  stretching vibrations of  $\text{ReO}_4^-$  anion [16–18]. The cation structure of each organic perrhenate salt could be identified by  $^1\text{H}$  NMR spectra, and the purity of compounds was determined by EA. Detailed characteristic data can be found in the experimental section.

### 2.2. Oxidative esterification of aldehydes with alcohols

Compounds 1–4 were used as catalysts for oxidative esterification of aldehydes with alcohols. The reaction was optimized by using benzaldehyde and methanol as substrates, and compound 1 as catalyst. Blank reaction showed 52% of methyl benzoate yield when no catalyst was applied (Table 1, entry 1). Meanwhile, only 5% of methyl benzoate could be detected when no oxidant was involved in the reaction, indicating that

**Table 1** Optimization of the reaction.<sup>a</sup>

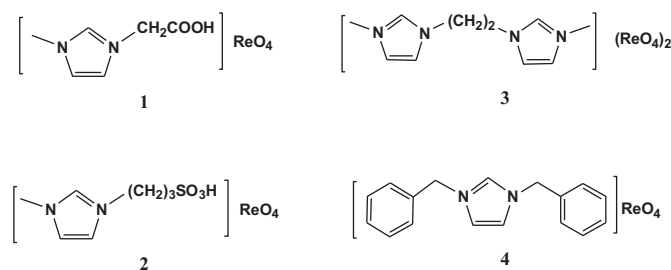
Entry	Catalyst (mol%)	Oxidant (equiv.)	Temp. (°C)	Conv. <sup>b</sup> (%)	Yield <sup>b</sup> (%)
1	0	UHP (3)	80	75	52
2	8	–	80	14	5
3	1	UHP (3)	80	80	63
4	3	UHP (3)	80	82	72
5	5	UHP (3)	80	86	75
6	8	UHP (3)	80	90	82
7	10	UHP (3)	80	88	81
8	8	$\text{H}_2\text{O}_2$ (3)	80	86	75
9	8	TBHP (3)	80	15	2
10	8	UHP (3)	40	29	21
11	8	UHP (3)	60	77	62
12	8	UHP (1)	80	76	63
13	8	UHP (2)	80	85	77
14	8	UHP (4)	80	98	88
15 <sup>c</sup>	8	UHP (4)	80	84	72
16 <sup>d</sup>	8	UHP (4)	80	91	84

<sup>a</sup> The reactions were carried out using benzaldehyde (2 mmol, 212 mg), MeOH (2 mL), 1 as catalyst for 24 h.

<sup>b</sup> The conversion and yield were determined by GC.

<sup>c</sup> The reaction was carried out without MeOH using 40 equivalent of UHP, and benzoic acid was obtained as the product.

<sup>d</sup> Benzoic acid as the substrate.



**Scheme 1** Structures of perrhenate catalysts 1–4.

benzaldehyde could not be oxidized in air (Table 1, entry 2). However, the yield increased gradually with the increase of compound **1** loading (Table 1, entries 3–7) and the highest methyl benzoate yield (82%) was obtained with 8 mol% of compound **1** applied. Different oxidants including hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>, 30%), TBHP and UHP were compared. However, with H<sub>2</sub>O<sub>2</sub>, the yield was lower than UHP and, with TBHP, no significant yield of methyl benzoate product was obtained (Table 1, entries 6, 8 and 9). The reactions were further studied with the variation of temperature and amount of UHP. The results indicated that the favorable reaction condition was 8 mol% of catalyst **1**, 4 equivalent of UHP at 80 °C. However, a small amount of benzoic acid could be detected as the byproduct in all examined reactions.

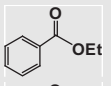
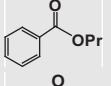
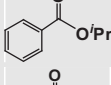
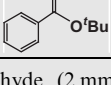
Patel et al. reported the oxidative esterification of aldehydes with V<sub>2</sub>O<sub>5</sub>-H<sub>2</sub>O<sub>2</sub> [19]. In their catalytic system, the oxidation of aldehydes in an alcoholic medium was assumed to proceed through a formation of hemiacetal, then the interaction of hemiacetal with the peracid (formed through reaction of H<sub>2</sub>O<sub>2</sub> with V<sub>2</sub>O<sub>5</sub>), and finally the elimination of intermediate to ester product. To study the reaction mechanism, the oxidation of benzaldehyde was carried out in the absence of methanol over excessive amount of UHP (Table 1, entry 15). Benzoic acid was found as the main product according to GC analysis. Moreover, benzoic acid was found to be able to be transferred into methyl benzoate in a good yield under the optimal conditions (Table 1, entry 16). It is important to note that these experimental results were in good accordance with those obtained for imidazolium polyoxometalate ionic liquid catalyzed oxidative esterification of aldehyde reported by Hou et al. [20]. Thus, the data indicated that the oxidative esterification reaction might proceed initially via the oxidation of benzaldehyde to benzoic acid, followed by the esterification of benzoic acid with methanol.

Based on the above studies, the oxidative esterification of benzaldehyde with methanol was performed using four different catalysts. The results indicated comparable catalytic performance for the examined four perrhenate catalysts. Nevertheless, compounds **1** and **2** bearing the acidic COOH or SO<sub>3</sub>H functional groups displayed a bit higher catalytic activity, indicating that the reaction could be further promoted by improving the catalyst acidity.

The reactions were extended to other alcohols using benzaldehyde as the standard aldehyde. The common alcohols such as ethanol, <sup>n</sup>PrOH, <sup>i</sup>PrOH could also be transformed into the corresponding esters in good yields (Table 2, entries 1–3). However, the esterification with <sup>t</sup>BuOH resulted in only 19% of ester yield (Table 2, entry 4), indicating that the steric tert-butyl group significantly affected the activity of the reaction.

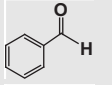
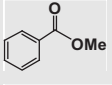
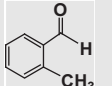
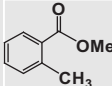
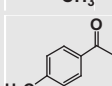
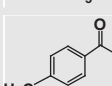
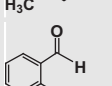
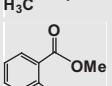
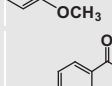
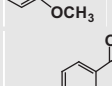
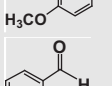
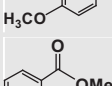
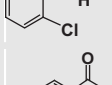
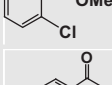
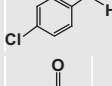
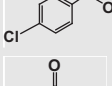
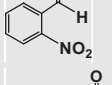
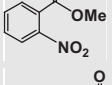
The oxidative esterification was then evaluated by using different aldehydes with methanol. Results are summarized in Table 3. Benzaldehyde derivatives bearing different functional groups, including methyl (Me), methoxyl (OMe), nitro (NO<sub>2</sub>) and chloro (Cl) groups on either *ortho* or *para* position of the aromatic ring were investigated under standard conditions. The reactions were significantly affected by the steric effect of functional groups on the aromatic ring of benzaldehydes. All reactions resulted in good ester yields (≥85%) for the substrates bearing the functional groups at the *para* position of the aromatic ring. However, poor yields were obtained with functional groups located at the *ortho* positions. The electronic

**Table 2** Oxidative esterification of benzaldehyde with different alcohols using **1** as catalyst.<sup>a</sup>

Entry	Alcohols	Esters	Yield (%)
1	EtOH		81
2	<i>n</i> -PrOH		76
3	<i>i</i> -PrOH		64
4	<i>t</i> -BuOH		19

<sup>a</sup> Reaction condition: benzaldehyde (2 mmol, 212 mg), ROH (2 mL), **1** (8 mol%), UHP (4 equiv.) at 80 °C for 24 h.

**Table 3** Oxidative esterification of benzaldehyde derivatives with methanol using **1** as catalyst.<sup>a</sup>

Entry	Aldehydes	Esters	Yield (%)
1			88
2			66
3			85
4			21 (74 <sup>b</sup> )
5			65
6			56 (30 <sup>b</sup> )
7			87
8			31 (50 <sup>b</sup> )
9			89

<sup>a</sup> Reaction condition: benzaldehyde (2 mmol, 212 mg), MeOH (2 mL), **1** (8 mol%), UHP (4 equiv.) at 80 °C for 24 h.

<sup>b</sup> Unreacted substrate.

effect on the reaction seems not dominating. As in the case of different functional groups at *para* positions, the reactions resulted in similar ester yields. For the benzaldehydes with functional groups at *ortho* positions, the obtained yields were in the order of Me > Cl > NO<sub>2</sub> > OMe.

### 3. Experimental section

#### 3.1. General remarks

All chemicals were purchased from the Sinopharm Chemical Reagent Co. Ltd of China or Acros and used without further purification.  $^1\text{H}$  NMR spectra were recorded on a Bruker Avance DPX-500 spectrometer. IR spectra were recorded on a PerkinElmer Frontier FT-IR spectrometer. Catalytic runs were monitored by gas chromatography (GC) methods on an Agilent 6890 instrument equipped with a FID and a capillary column (30 m  $\times$  0.25 mm  $\times$  0.25  $\mu\text{m}$ ). Microanalyses of the obtained products were performed with a Flash EA 1112 series elemental analyzer. TGA and differential scanning (DSC) analysis were conducted utilizing a Netzsch-STA 409 PC system, and typically about 10 mg of each sample were heated from 25  $^\circ\text{C}$  to 1000  $^\circ\text{C}$  at 10 K  $\text{min}^{-1}$ . The 1,2-di(methyl-1H-imidazol-1-yl)ethane bromide and 1,3-dibenzylimidazolium chloride salts were prepared according to the literatures [21,22].

#### 3.2. Preparation of imidazolium perrhenates

**1-( $\alpha$ -methylcarboxylic)-3-methylimidazolium perrhenate (1):** Ethyl chloroacetate (0.490 g, 4 mmol) and equal equiv. of 1-methylimidazole (0.328 g, 4 mmol) were mixed in 20 mL ethanol under Argon, and then stirred at 70  $^\circ\text{C}$  for 24 h. The obtained white precipitate was filtered, washed with ether for three times, and dried under vacuum at 70  $^\circ\text{C}$ . The resulting acetate functionalized intermediate was then dissolved in acetonitrile, treated with equal equiv. of concentrated hydrochloric acid (37%, 0.33 mL) at 70  $^\circ\text{C}$  for 12 h. 1-( $\alpha$ -methylcarboxylic)-3-methylimidazolium chloride was obtained after removal of solvent. In the next step, 1-( $\alpha$ -methylcarboxylic)-3-methylimidazolium chloride was dissolved in acetonitrile, added with equal equiv. of  $\text{AgReO}_4$  and stirred at room temperature for 24 h. The resultant mixture was filtrated to remove  $\text{AgCl}$ . The pure compound **1** (white solid) was obtained by removal of solvent, washed with ethanol, and dried under vacuum at 70  $^\circ\text{C}$ .  $\text{C}_6\text{H}_9\text{N}_2\text{O}_6\text{Re}$  (391.35), elemental analysis calcd.: C, 18.41, H, 2.32, N, 7.16 found: C, 18.40, H, 2.34, N, 7.12; IR ( $\text{cm}^{-1}$ ): 3413, 3107, 2916, 1742 (COOH), 1619, 1404, 1167, 913 (Re=O);  $^1\text{H}$  NMR (DMSO, 500 MHz, r.t., ppm): 3.91 (3H, s,  $\text{CH}_3$ ), 5.13 (2H, s,  $\text{CH}_2$ ), 7.71 (2H, s, CH), 9.08 (1H, s, CH), 13.71 (1H, s, OH).

**1-(1-propylsulfonic)-3-methylimidazolium perrhenate (2):** 1,3-propanesulfone (0.489 g, 4 mmol) and equal equiv. of 1-methylimidazole (0.328 g, 4 mmol) were mixed in 20 mL ethanol under Argon, and then stirred at 70  $^\circ\text{C}$  for 24 h. The obtained white precipitate was then filtered, washed with ether for three times, and dried under vacuum at 70  $^\circ\text{C}$  for 2 h. The resulted sulfonated intermediate was then dissolved in toluene, treated with equal equiv. of concentrated hydrochloric acid (37%, 0.33 mL) at 70  $^\circ\text{C}$  for 12 h. 1-(1-propylsulfonic)-3-methylimidazolium chloride was obtained after removal of solvent. In the next step, 1-(1-propylsulfonic)-3-methylimidazolium chloride was dissolved in acetonitrile, added with equal equiv. of  $\text{AgReO}_4$  and stirred at room temperature for 24 h. The resulted mixture was filtrated to remove  $\text{AgCl}$ , and the pure compound **2** (white solid) was obtained by removal of solvent, washed with ethanol, and dried under vacuum at 70  $^\circ\text{C}$ .

$\text{C}_7\text{H}_{13}\text{N}_2\text{SO}_7\text{Re}$  (455.46), elemental analysis calcd.: C, 18.46, H, 2.88, N, 6.15; found: C, 18.51, H, 2.93, N, 6.10; IR ( $\text{cm}^{-1}$ ): 3447, 3155, 3113, 1582, 1207 (S=O), 1057, 907 (Re=O);  $^1\text{H}$  NMR ( $\text{D}_2\text{O}$ , 500 MHz, r.t., ppm): 2.19–2.25 (2H, *m*,  $\text{CH}_2$ ), 2.81–2.84 (2H, *t*,  $\text{CH}_2$ ), 3.80 (3H, *s*,  $\text{CH}_3$ ), 4.25–4.28 (2H, *t*,  $\text{CH}_2$ ), 7.35 (1H, *d*, CH), 7.43 (1H, *d*, CH), 8.66 (1H, *s*, CH).

**1,2-di(methyl-1H-imidazol-1-yl)ethane perrhenate (3):** 1,2-di(methyl-1H-imidazol-1-yl)ethane bromide (0.695 g, 1 mmol) and 2.4 equiv. of  $\text{AgReO}_4$  (0.86 g, 2.4 mmol) were mixed in 20 mL acetonitrile under Argon and stirred at 60  $^\circ\text{C}$  for 24 h. The slurry was then filtrated with a Gooch funnel to remove  $\text{AgBr}$  and the unreacted  $\text{AgReO}_4$ . The pure compound **3** (white solid) was obtained by recrystallization in acetonitrile, washing three times with *n*-hexane and drying under oil pump vacuum.  $\text{C}_{10}\text{H}_{16}\text{N}_4\text{O}_8\text{Re}_2$  (692.67), elemental analysis calcd.: C, 17.34, H, 2.33, N, 8.09; found: C, 17.30, H, 2.35, N, 8.11; IR ( $\text{cm}^{-1}$ ): 3139, 3084, 1648, 1566, 1449, 1168, 916 (Re=O), 826, 618;  $^1\text{H}$  NMR (DMSO, 500 Hz, r.t., ppm): 3.85 (3H, *s*, N- $\text{CH}_3$ ), 4.66 (2H, *t*,  $\text{CH}_2$ ), 7.58 (1H, *d*,  $\text{mz-H}^5$ ), 7.71 (1H, *d*,  $\text{mz-H}^4$ ), 8.99 (1H, *s*,  $\text{mz-H}^2$ ).

**1,3-dibenzylimidazolium perrhenate (4):** The 1,3-dibenzylimidazolium chloride salt (0.5 g, 1 mmol) and 1.2 equiv. of  $\text{AgReO}_4$  (0.43 g, 1.2 mmol) were mixed in 20 mL acetonitrile under Argon and stirred at 60  $^\circ\text{C}$  for 24 h. The slurry was then filtrated with a Gooch funnel to remove  $\text{AgCl}$  and the unreacted  $\text{AgReO}_4$ . The pure compound **4** (white solid) was obtained by re-crystallization in acetonitrile, washing three times with *n*-hexane and drying under oil pump vacuum.  $\text{C}_{17}\text{H}_{17}\text{N}_2\text{O}_4\text{Re}$  (499.53), elemental analysis calcd.: C, 40.87, H, 3.43, N, 5.61; found: C, 40.58, H, 3.45, N, 5.65; IR ( $\text{cm}^{-1}$ ): 3139, 3092, 1621, 1557, 1494, 1449, 1159, 907 (Re=O), 699, 636;  $^1\text{H}$  NMR (DMSO, 500 Hz, r.t., ppm): 5.42 (4H, *s*,  $\text{CH}_2$ ), 7.40–7.46 (10H, *m*, Ar-H), 7.83 (2H, *d*,  $\text{mz-H}^{4,5}$ ), 9.38 (1H, *s*,  $\text{mz-H}^2$ ).

#### 3.3. General procedure for oxidative esterification

In a typical reaction, the perrhenate catalyst (8 mol%) was dissolved into 2 mL of  $\text{CH}_3\text{OH}$  in the reaction flask in air. Benzaldehyde (0.212 g, 2 mmol) was added to the flask successively, followed by the addition of UHP (0.376 g, 4 mmol) at 80  $^\circ\text{C}$  to start the reaction. The course of the reaction was monitored by quantitative GC analysis on an Agilent 7890 instrument equipped with a HP-5 capillary column (30 m  $\times$  0.25 mm  $\times$  0.25  $\mu\text{m}$ ), and a flame ionization detector. Samples were taken at regular time intervals, diluted with acetonitrile, and treated with a catalytic amount of  $\text{MgSO}_4$  and  $\text{MnO}_2$  to remove water and to destroy the unreacted peroxide. The resulting mixture was filtered and injected onto a GC column. The conversion of benzaldehyde and the formation of methyl benzoate were calculated from calibration curves ( $r^2 > 0.999$ ) recorded prior to the reaction.

### 4. Conclusions

The oxidative esterification of benzaldehydes with alcohols was studied using imidazolium perrhenates as efficient catalysts in the presence of UHP as oxidant. The synthesized imidazolium perrhenates were fully characterized by FT-IR,  $^1\text{H}$  NMR and EA to confirm their structures. When using these

organic perrhenates as catalysts, good to excellent ester yields could be obtained for various benzaldehyde and alcohol derived substrates. Steric effects of functional groups on the aromatic ring of benzaldehydes, and the alkyl structures of alcohols are assumed to play important roles in the formation of esters.

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