Article

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Solvent- and Metal-free Oxidative Esterification of Aromatic Aldehydes Using Urea-2,2-dihydroperoxypropane as a New Solid Oxidant

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Urea-2,2-dihydroperoxypropane as a noble and solid *gem*-dihydroperoxide derivative was used to transform various aromatic aldehydes to their corresponding benzoate derivatives in the presence of HBr under mild conditions at room temperature in high yields and short reaction times.

Keywords: Oxidative esterification; Aromatic aldehydes; *gem*-Dihydroperoxide; Urea-2,2-dihydroperoxypropane; HBr.

INTRODUCTION

Direct esterification of aldehydes to esters is considered a useful reaction in organic synthesis[1,2] because of the wide application of esters in industry. The ester functionality is present in various structures of natural and synthetic compounds such as fragrances, medicines, polymer constructions,[3,4] dyes, agrochemicals, and natural products,[5] and as cross-coupling partners.[6,7] They are also used in the flavoring industry and have an important role as fixatives and carrier solvents. Therefore, chemists have been devoting much effort to find routes by which esters can be produced under mild conditions.[8–10]

Several methods have been reported in the literature to carry out this oxidative transformation. The traditional path to ester synthesis is the reaction of acids, acyl chlorides or anhydrides, and nitriles[11,12] with alcohols. These methods require stoichiometric amounts of heavy-metal oxidants such as $KMnO_4$,[13] CrO_3,[14] hydrogen peroxide,[15] ozone,[15] oxone,[15] *N*-iodosuccinimide,[16] sodium hypochlorite,[16] silver carbonate on celite,[16] 2,3-dichloro-5,6-dicyanobenzoquinone in the presence of amberlyst,[17] sodium metaperiodate,[17] 1,2-dimethylindazolium,[17] and mixtures of methane sulfonic acid and aluminum oxide,[18] or transition-metal catalysts such as vanadium,[1] rhenium,[19] silver,[20] palladium,[21] ruthenium,[22] rhodium,[23] copper,[24] titanium,[25] iridium,[26] iron,[27] nickel,[27] and zinc.[28] Among these methodologies, direct esterification of aldehydes has been the center of attention as an attractive route that affords the corresponding esters readily.[29] In a classical method that leads to ester production, the employed reagents are based on carboxylic acid activation and subsequent treatment with the desired alcohols.[3] This carboxylic activation can be carried out *in situ* using strong acids such as $SOCl_2$, CDI,[30] DEAD/PPh_3,[31] and DCC.[32] Many of these reported approaches suffer from several drawbacks including the use of large excess of reagents, long reaction times, or high temperature, reagent toxicity, over-oxidation, undesirable products, byproducts, co-catalysts, or hydrogen acceptors, poor to moderate yields, photochemical conditions, and dry solvents.

In the last 10–15 years, geminal dihydroperoxides have attracted much attention basically because of their antimalarial properties. These organic compounds have been employed in the synthesis of a variety of peroxides such as tetraoxanes,[33] spirobisperoxyketals,[34] silatetraoxanes,[35] 1,2,4,5-tetraoxacycloalkanes,[36] and bisperoxyketals.[37] They are also applicable in polymerization reactions as radical initiators,[37] in nucleophilic epoxidation and oxidation,[38,39] and in the synthesis of dicarboxylic acid di-esters as precursors.[40] Therefore, much effort has been expended to introduce new methodologies by which *gem*-dihydroperoxides could be prepared efficiently.

In continuation of our work on developing an efficient route for *gem*-dihydroperoxide synthesis and applications,[41] we synthesized urea-2,2-dihydroperoxypropane (UDHPP, Scheme 1) and used it as a

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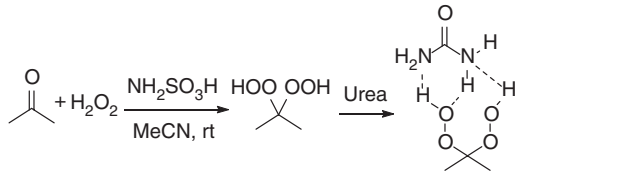


urea-2,2-dihydroperoxypropane (UDHPP)

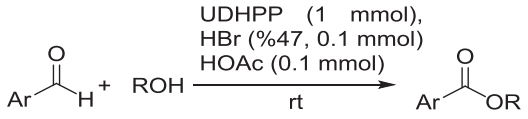
Scheme 1. Synthesis of urea-2,2-dihydroperoxypropane.

new solid oxidizing agent for the oxidative esterification of aromatic aldehydes (Scheme 2). Here we report on our studies on the facile and cost-effective ester synthesis under mild conditions at room temperature.

RESULTS AND DISCUSSION

First, as shown in Table 1, we investigated the effects of several parameters contributing to methylbenzoate synthesis. Based on the results by the model reaction of benzaldehyde with methanol, in the presence of UDHPP, it was found that the bromide anion was the best halide and hydrogen bromide was the best bromide source since methylbenzoate was formed only in trace amounts without any halide anions (Table 1, entry 13). It was notable that the reaction was highly accelerated in the presence of catalytic amount of acetic acid. It was seen that in the absence of peroxide, the reaction nearly stopped (Table 1, entry 14). Finally, some peroxides such as hydrogen peroxide and *tert*-butyl hydroperoxide (TBHP) were tested, and it was observed that UDHPP was the best oxidant. (Table 1, entries 1, 15, and 16) Therefore, the model reaction we chose based on the optimized conditions involved aldehyde (1 mmol), methanol (2 mL), UDHPP (1 mmol), acetic acid (0.1 mmol), and HBr (0.1 mmol) (Table 1, entry 1).

As shown in Table 2, a wide range of aromatic aldehydes were subjected to the optimized esterification conditions. Derivatives with both electron-withdrawing and electron-donating substituents were converted to their corresponding esters in high yields. The aldehydes bearing electron-donating substituents took longer reaction times and gave lower yields (Table 2, entries 9–13). In contrast, electron-withdrawing groups required shorter reaction times to afford the desired products (Table 2, entries 2–5, 8, and 15).



R= Me, Et, Penthyl, Benzyl

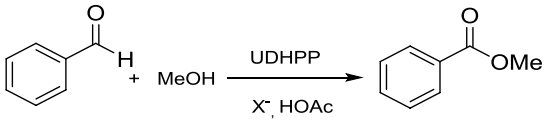
Scheme 2. Oxidative esterification of aromatic aldehydes to the corresponding esters by UDHPP/HBr.

Also, ortho substitution took longer reaction times and gave lower yields than para substitutions due to strain effects (Table 2, entries 3, 6–9, and 11). Similarly, alcohols with high steric congestion took longer reaction times than alcohols with less steric congestion (Table 2, entry 1). Because of the conjugation of the carbonyl group with double bond in cinnamaldehyde, it was converted to its corresponding methyl ester but took a long reaction time (Table 2, entry 16). In addition, heterocyclic aldehydes including 2-furylcarbaldehyde and 2-thiophen carbaldehyde were converted to their corresponding methyl esters successfully without over-oxidation in heterocyclic rings (Table 2, entries 17 and 18). Because of the electron-withdrawing nature of furyl ring, 2-furyl-carbaldehyde reacted faster than 2-thiphen carbaldehyde. 2-Naphthaldehyde was oxidized to the methyl ester with more difficulty than the model reaction due to the high steric effect of the naphthyl ring (Table 2, entry 19).

To study the chemoselectivity of the reaction, benzyl alcohol was selected (Table 2, entry 20). It was observed that benzyl alcohol was recovered unreacted, so no ester was detected. Also, the reaction was carried out with 1-pentanole as an aliphatic alcohol under the optimized conditions, and pentyl pentanoate was obtained as the predicted ester (Table 2, entry 21). In addition, acetaldehyde as a typical aliphatic aldehyde was oxidized to pentyl acetate successfully (Table 2, entry 22). All products were simply dissolved in chloroform and then extracted to yield the isolated products.

Studying the mechanism of the esterification reactions, all the observations mentioned above were proved. Initially, UDHPP converts HBr to BrOH, which is a Lewis acid. As HOAc accelerated the reaction, it seems that the generated BrOH is converted to BrOAc, which is more active.[41a] From the observed results in Table 2 and substitutions effects on the reaction rates and yields, it seems that, primarily, alcohol is added to aldehydes to generate hemiacetal reversibly (Scheme 3, compound **A**). Then, this hemiacetal is

Table 1. Optimization conditions



Entry	Methanol (mL)	Oxidant (mmol)	X^- (0.1 mmol)	HOAc (mmol)	Yield (%)	Time (h)
1	2	UDHPP (1)	HBr	0.1	87	6
2	2	UDHPP (1)	KBr	0.1	25	12
3	2	UDHPP (1)	NH_4Br	0.1	64	9
4	2	UDHPP (1)	NH_4Cl	0.1	41	11
5	2	UDHPP (1)	$MgBr_2$	0.1	30	12
6	2	UDHPP (0.5)	HBr	0.1	81	8
7	2	UDHPP (1.5)	HBr	0.1	83	6
8	2	UDHPP (1)	HBr	—	80	11
9	1	UDHPP (1)	HBr	0.1	50	14
10	3	UDHPP (1)	HBr	0.1	83	5
11	2	UDHPP (1)	HCl	0.1	30	11
12	2	UDHPP (1)	HI	0.1	85	10
13	2	UDHPP (1)	—	0.1	Trace	12
14	2	—	HBr	0.1	Trace	12
15	2	H2O2 (30%)	HBr	0.1	45	12
16	2	TBHP (70 %)	HBr	0.1	70	12

activated by BrOAc, which is an efficient Lewis acid, and converts hemiacetal **A** to the intermediate **B** irreversibly, which is unstable and is oxidized to the corresponding ester rapidly. Consequently, due to the intermediate **B**'s instability, it is understandable why electron-withdrawing substitutes carried out the reaction in short reaction times and electron-donating substitutions caused slow accomplishment of the reactions. In fact, generally, electron-withdrawing substitutes cause more instability in the intermediate **B**, and therefore it is oxidized to its corresponding ester faster.

Finally, the efficiency of this method of synthesis of methyl benzoate as the model reaction was compared with some reported methodologies, as shown in Table 3. Based on the obtained results in Table 3, clearly this method shows higher performance than the compared methodologies. In fact, this method improved the reaction times, yields, and reaction conditions.

Conclusion

In conclusion, we devised a new, clean, and mild approach for the esterification of aldehydes by UDHPP as a novel and low-cost oxidant in presence of catalytic amounts of HBr. UDHPP is a solid, powerful, and stable oxidant which can be synthesized in large scales and stored for several months at room temperature. The present protocol represents compatibility with a wide range of functional groups including electron-releasing and electron-withdrawing substituents. This protocol is efficient, environmentally benign, and straightforward which results in high yields and short reaction times.

EXPERIMENTAL

Solvents, reagents, and chemical materials were obtained from Aldrich and Merck and purified prior to use. Nuclear magnetic resonance spectra were recorded on a Bruker 300 MHz instrument using tetramethylsilane (TMS) as an internal standard. Infrared spectra were recorded on a Perkin Elmer GX FT IR spectrometer (KBr pellets).

Caution: Although we did not encounter any problems or explosion while working with *gem*-dihydroperoxides, peroxides are potentially explosive and should be handled with precaution. All reactions should be

Table 2. oxidative esterification of different aldehydes[a]

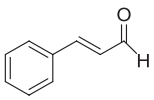
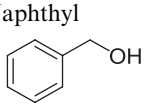
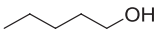
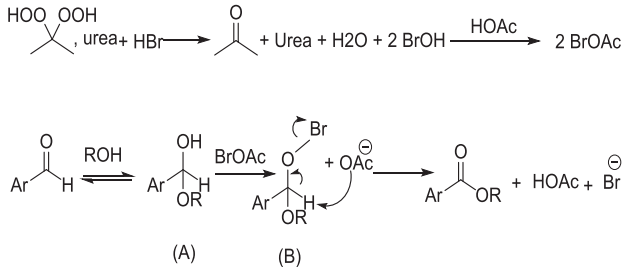
Entry	Ar	R	Time (h)	Yield (%)[b]	M.p. (°C)
1	C_6H_5	Me	6	87	–12.4
		Et	6	88	–34
		Pentyl	7	82	1
		Isopropyl	8	60	1
		Benzyl	7	80	21
2	3-NO_2-C_6H_4	Me	5	87	78
		Et	4.5	87	47
3	2-NO_2-C_6H_4	Me	4	70	78
		Et	4	71	47
		Pentyl	7	60	—
4	4-NO_2-C_6H_4	Me	3.5	85	96
		Et	3	86	57
		Pentyl	4	85	107
5	4-Cl-C_6H_4	Me	6	85	43.5
		Et	6	86	1
		Pentyl	7	80	61
6	2-Cl-C_6H_4	Me	7	85	1
		Et	7	82	1
		Phenyl	10	70	1
7	2,6-diCl-C_6H_3	Me	10	75	28
8	2,4-diCl-C_6H_3	Me	10	72	28
		Et	10	70	1
9	2-MeO-C_6H_4	Me	14	65	1
		Et	14	60	1
		Pentyl	16	50	60
10	4-MeO-C_6H_4	Me	13	78	49
		Et	13	80	7.5
		Pentyl	14	72	62
11	2-OH-C_6H_4	Me	13	68	−8
		Et	13	68	1
12	4-OH-C_6H_4	Me	12	80	131
		Et	12	81	117
		Phenyl	13	75	38
13	4-Me-C_6H_4	Me	7	85	33.2
		Et	7	87	<−10
		Pentyl	8	75	1
14	4-Br-C_6H_4	Me	5	82	79
15	4-F-C_6H_4	Et	4	85	26
16		Me	6	86	35
17	2-Furyl	Me	6	60	oil
18	2-Thienyl	Me	7	75	oil
19	2-Naphthyl	Me	10	70	77
20		Benzyl	12	—	—

Table 2. Continued

Entry	Ar	R	Time (h)	Yield (%)[b]	M.p. (°C)
21		Pentyl	12	75	Oil
22		Pentyl	10	80	Oil

[a] Conditions: aldehyde (1 mmol), alcohol (2 mL), UDHPP (1 mmol), acetic acid (0.1 mmol) and HBr (0.1 mmol), rt.
[b] Isolated yields.



Scheme 3. Suggested mechanism for oxidative esterification of aldehydes

carried out behind a safety shield inside a fume hood and heating should be avoided.

General procedure for the synthesis of urea-2,2-dihydrperoxypropane

Acetone (1 mmol, 0.074 mL) was added to acetonitrile (5 mL), followed by NH_2SO_3H (0.1 mmol, 0.01 g), which serves as the catalyst of this reaction. To this stirred solution, H_2O_2 30% (1 mL) was added and the mixture was stirred for 2 h at room temperature. After the completion of the reaction, as monitored by thin-layer chromatography (TLC), the mixture was diluted with water (5 mL), extracted using ethyl acetate (3 × 5 mL), and dried over $MgSO_4$; then urea was added (1 mmol). After evaporation of the solvent under reduced pressure, the pure crystalline product was obtained. The product was characterized on the basis of its melting point, elemental analysis, IR, ^{1}H-NMR, and ^{13}C-NMR spectroscopy, and the amount of peroxide in products was determined by iodometric titration.

Physical and spectral data of UDHPP

White crystal, m.p: 114–116°C. IR ν_{max} /cm^{-1} (KBr pellet): 3456, 3337, 3265, 2928, 2852, 1680, 1624, 1464, 1384, 1155, 1003, 787, 715, 715, 574, 503; ^{1}H-NMR (300 MHz, DMSO-d_6) δH: 1.26 (S, 6H, (CH3)), 5.49 (s, 4H, (NH_2)), 10.23 (s, br, 2 H, (OOH)). ^{13}C-NMR: (75 MHz, DMSO-d_6) δC: 21.2, 108.2, 160.6; Anal. Calcd (%) for $C_4H_{12}N_2O_5$: C, 28.57; H, 7.19; N, 16.66; Found: C: 28.10; H: 7.52; N, 17.10.

Table 3. Comparison of efficiency with some other reported methodologies

Entry	Oxidant	Cat.	Conditions	Time (h)	Yields (%)	Ref.
1	UDHPP	HBr/HOAc	rt	6	87	This method
2	Oxone	Graphite oxide	60°C bath ultrasonic	15 min	90	[42a]
3	H_2O_2 (30%)	$ZnBr_2$	rt	16	89	[28]
4	TBHP	$B(C_6F_5)_3$	Reflux	18	86	[42b]
5	H_2O_2 (30%)	V_2O_5	Reflux	3	100	[8]
6	H_2O_2 (30%)	$CaCl_2$	65°C	48	55	[42c]
7	H_2O_2 (30%)	$MgCl_2$	65°C	40	68	[42c]
8	DIB($PhI(OAc)_2$)	I_2	rt	13	83	[42d]

General procedure for the esterification of aromatic aldehydes

A mixture of aldehyde (1 mmol), alcohol (2 mL), acetic acid (glacial, 0.1 mmol), and UDHPP (1 mmol) was stirred at room temperature. After the peroxide was dissolved, HBr (47% aq., 0.1 mmol) was added. After the completion of the reaction, as monitored by TLC, the mixture was diluted with saturated NaCl solution (5 mL) and extracted with $CHCl_3$ (3 × 5 mL). Then the organic layer was separated, dried over anhydrous Mg_2SO_4, and evaporated under reduced pressure. The residue was purified by silica-packed column chromatography (hexane–EtOAc) to afford pure epoxides (Table 2). Products were characterized on the basis of their melting points, elemental analysis, and IR, ^{1}H-NMR, and ^{13}C-NMR spectroscopy.[1,8,28,42]

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Supporting information

Additional supporting information is available in the Appendix S1.

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