Facile Epoxidation of α,β-Unsaturated Ketones with *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxolane as an Efficient Oxidant

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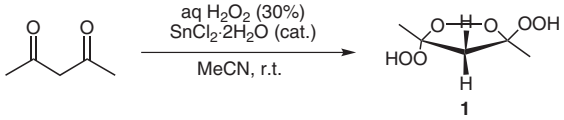
Abstract: Application of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane as an efficient oxygen source has been explored in the epoxidation of *trans*-chalcones. The reactions proceed under mild conditions at room temperature in alkaline solution to afford the corresponding epoxides in excellent yields.

Key words: α,β-unsaturated ketone, epoxide, dihydroperoxide, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane, epoxidation

Hydrogen peroxide (H_2O_2) is a well-known, versatile, and environmentally benign (only generates water as byproduct) oxygen source in various oxidative transformations.[1–3] However, the oxidation power of H_2O_2 is low for many purposes and requires to be activated by systems based on various acids such as AcOH,[4] TBHP/PTSA,[5] or transition-metal catalysts.[6,7] Many of these reagents and catalysts are subject to certain drawbacks such as toxicity of the transition metals present in these catalysts, long reaction times, and low yields.[8] One way to overcome these limitations is to convert H_2O_2 into dihydroperoxides.[9] In this respect, *gem*-dihydroperoxides have received considerable interest in recent years due to their relevance to peroxidic antimalarial agents,[10,11] which are structurally similar to hydroperoxides. In contrast to H_2O_2, *gem*-dihydroperoxides act more efficiently as high-oxygen-content oxidants for transferring oxygen to sulfides, α,β-unsaturated ketones, amines, and other substrates.[12–19] Anomeric hydroperoxides derived from 3,4,6-tri-*O*-benzyl-galactose and glucose were used for enantioselective epoxidation of naphthoquinone, chalcones, (*E*)-1,2-dibenzoyl ethylene, and (*E*)-iso-butyrylphenyl ethylene.[20] Most recently, we reported a novel use of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane in selective sulfoxidation of sulfides.[21] In the last few years, great attention has been paid to epoxidation of α,β-unsaturated ketones due to the important role of epoxides,[22,23] especially chiral epoxides, as useful building blocks in organic synthesis, for example, in the synthesis of pharmaceutical and natural products.[24–29] Also, these compounds have been used as intermediates in flavonoid chemistry,[30–32] in the synthesis of natural products and drug molecules.[33]

Several approaches to epoxides have been investigated using various systems such as, *m*-chloroperbenzoic acid,[34] hydrogen peroxide,[35–40] peroxide–ammonium fluoride,[41] sodium perborate,[42,43] tetra-*n*-butyl ammonium peroxydisulfate,[44] and H_2O_2/[bmim]BF_4.[45] In addition, several hydroperoxides have been reported in epoxidation reactions including *tert*-butylhydroperoxide,[46–50] PEG-supported cinchona ammonium salt catalyzed hydroperoxides,[51] and diketopiperazine-derived hydroperoxides.[52] As known from literature survey, very few reports have been recorded on application of organic dihydroperoxides in epoxidation of α,β-unsaturated ketones.[44]

In our ongoing research in the synthesis of *gem*-dihydroperoxides,[53,54] and their applications as oxidants for various organic transformations,[21] we became interested in using these dihydroperoxides as potential oxidants for epoxidation of *trans*-chalcones. Herein, we wish to report for the first time the application of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**) in the epoxidation of α,β-unsaturated ketones. *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxolane has been prepared in this laboratory following the reported procedure as a white crystalline compound in high yield (Scheme 1).[21]



Scheme 1

We preliminary studied the Weitz–Scheffer epoxidation reaction of *trans*-chalcone **2a** as the test compound using *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**) and 1.0 M aqueous KOH as the base at room temperature. To study the effect of the solvent on the reaction, various solvents including *n*-hexane, toluene, dichloromethane, ethanol *N*,*N*-dimethyl formamide (DMF), acetonitrile, 1,2-dimethoxyethane (DME), and 1,4-dioxane were examined and the results are summarized in Table 1. As could be seen in Table 1, the polarity of the solvent plays a crucial role in the reaction yield. So that, nonpolar *n*-hexane and toluene were found not suitable for this reaction, since no epoxide was obtained in these two solvents after a long reaction time (entries 1 and 2). Nevertheless, the reaction did happen in polar solvents, CH_2Cl_2 (entry 3), EtOH (entry 4), and DMF (entry 5) to give the desired epoxide **3a** in 35%, 60%, and 85% yields, respectively. Water-soluble polar solvents MeCN (entry 6), DME (entry 7), and 1,4-dioxane (entry 10) produced the epoxide

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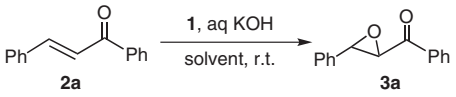
even in better 87%, 96%, and 90% yields, respectively. Among the solvents examined, 1,2-dimethoxyethane (DME) proved to be the solvent of choice for this reaction in producing the epoxide in almost quantitative yield (96%, entry 7) within two hours without posing any toxicity.

Table 1 Solvent Screening in the Epoxidation of *trans*-Chalcone **2a** with *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**)[a]

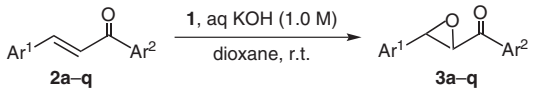


Entry	Solvent	Time (h)	Yield (%)[b]	Oxidant (mmol)
1	*n*-hexane	26	0	1
2	toluene	26	0	1
3	CH_2Cl_2	24	35	1
4	EtOH	12	60	1
5	DMF	4	85	1
6	MeCN	7	87	1
7	DME	2	96	1
8	DME	2	92	2
9	DME	3	90	0.5
10	1,4-dioxane	2	90	1
11	1,4-dioxane	2	85	0.5
12	DME[c]	2	36	1
13	DME[c]	2	42	2

[a] All reactions were carried out with *trans*-chalcone **2a** (1.0 mmol), aq KOH (1.0 M, 1.0 mL), and the solvent (5.0 mL) at r.t.
[b] Isolated yield.
[c] With 30% H_2O_2 as the oxidant.

It was observed that, using lower or higher amounts of the oxidant **1** did not show any improving effect on the yield (entries 8 and 9). Also, the effect of the base on the reaction was investigated using different bases such as aqueous NaOH, KOH, hexamethylenetetramine, and *n*-Bu_4NOH. Among these, KOH was found to be the most convenient in terms of yield (96%) and reaction time (2 h), whereas no detectable amount of epoxide was formed when hexamethylenetetramine and *n*-Bu_4NOH were used as the bases. The role of the base used in the reaction was substantiated by conducting the reaction in the absence of KOH that resulted in only trace amount of the expected epoxide. In order to compare the oxidative power of the oxidant **1** with that of H_2O_2, the reaction was carried out by using 30% H_2O_2 in DME under similar reaction conditions and lower yields of the epoxide were obtained (entries 12 and 13).

To develop the scope of reaction, we were encouraged to extend this reaction to a variety of other substituted *trans*-chalcones **2b–q** under the optimized conditions (r.t., 1.0 M aq KOH in DME) (Scheme 2), and the results are summarized in Table 2.[55] As shown in Table 2, the oxidant **1** has conveniently accomplished the conversion of all *trans*-chalcones **2a–q** into their corresponding epoxides **3a–q** in relatively short reaction times with excellent yields (82–96%, Table 2). Also, it should be mentioned that in all the reactions acetylacetone was recovered in high yield after the reaction as evidenced by the spectral analysis. The structures of the products **3a–q** were fully established by analysis of their spectral (^1H NMR, ^{13}C NMR, IR) and physical data and compared with those reported.[45,56]



Scheme 2

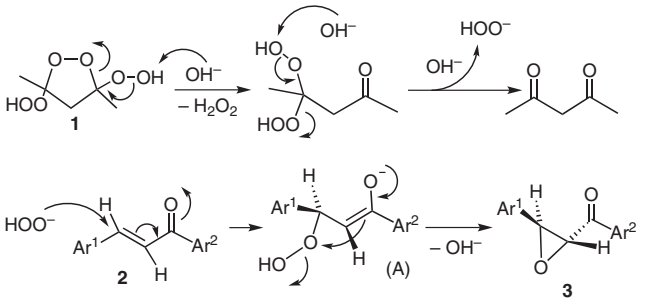
Table 2 Epoxidation of α,β-Unsaturated Ketones **2a–q** with *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**) under the Optimized Conditions[a]

Entry	Ar^1	Ar^2	Product	Time (h)	Yield (%)[b]
1	Ph	Ph	**3a**	2	96
2	Ph	2-ClC_6H_4	**3b**	3	92
3	Ph	4-$MeOC_6H_4$	**3c**	4	90
4	Ph	2-naphthyl	**3d**	3	93
5	4-ClC_6H_4	Ph	**3e**	2.5	95
6	4-ClC_6H_4	4-ClC_6H_4	**3f**	3	92
7	4-ClC_6H_4	4-MeC_6H_4	**3g**	2	94
8	2-ClC_6H_4	4-MeC_6H_4	**3h**	3	94
9	4-ClC_6H_4	2-naphthyl	**3i**	3	94
10	3-ClC_6H_4	2-naphthyl	**3j**	3	90
11	4-ClC_6H_4	2-thionyl	**3k**	2.5	91
12	4-ClC_6H_4	2-furyl	**3l**	2.2	90
13	4-MeC_6H_4	Ph	**3m**	3	90
14	4-$MeOC_6H_4$	Ph	**3n**	5	82
15	4-$O_2NC_6H_4$	Ph	**3o**	2	95
16	3-$O_2NC_6H_4$	Ph	**3p**	2.5	94
17	4-$O_2NC_6H_4$	2-naphthyl	**3q**	2.5	92

[a] All reactions were carried out with α,β-unsaturated ketone (1.0 mmol), *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**, 1.0 mmol), aq KOH (1.0 M, 1.0 mL), and 1,4-dioxane (5.0 mL) at r.t.
[b] Isolated yield.

On the basis of the experimental evidences, a probable mechanism to explain the stereoselective formation of *trans*-epoxides **3** is depicted in Scheme 3. As shown in

this scheme, the reaction likely takes place preliminary with in situ generation of hydroperoxide anion in two successive steps upon the effect of KOH on *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**). Subsequently, the nucleophilic addition of the hydroperoxide anion to the β-carbon of the *trans*-chalcone takes place to yield the intermediate anion **A**, which then stereochemically undergoes an S_N2-like cyclization by pushing out the hydroxide ion. This is consistent with the observed stereoselectivity of the reactions in only affording *trans*-epoxide **3**.



Scheme 3

In conclusion, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**) has been conveniently used as an effective and high-oxygen-content oxidant in epoxidation of variously substituted *trans*-chalcones to corresponding epoxides. The reactions proceed under mild conditions at room temperature to afford the epoxides in excellent yields. This protocol may be considered as environmentally benign since no additional catalyst is necessary for activation of the oxidant, and also the acetylacetone used for preparation of the oxidant may be recovered after the reaction.

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(55) **Caution**
Although we did not encounter any problem with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2 dioxolane (**1**), it is potentially explosive and should be handled with precautions; all reactions should be carried out behind a safety shield inside a fume hood and transition-metal salts or heating should be avoided.
Preparation of *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxolane (1)[21]
To a stirred solution of acetylacetone (100 mg, 1 mmol) in MeCN (5 mL) was added $SnCl_2{\cdot}2H_2O$ (45 mg, 0.2 mmol), and stirring of the reaction mixture was continued for 5 min at r.t. Then, aq 30% H_2O_2 (5 mmol) was added to the reaction mixture and was allowed to stir for 12 h at r.t. After completion of the reaction as monitored by TLC, H_2O (15 mL) was added, and the product was extracted into EtOAc (2 ´ 10 mL). The combined organic layer was dried over anhyd $MgSO_4$ and evaporated under reduced pressure to give almost pure white crystalline product **1**, in 85% yield (140 mg); mp 98–100 °C.
General Experimental Procedure
To a mixture of *trans*-chalcone **2** (1.0 mmol) and *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane (**1**, 166 mg, 1 mmol) in DME (5 mL) was added 1.0 M aq KOH solution (1 mL). The reaction mixture was stirred at r.t. for 2 h. After completion of the reaction (TLC), the remaining peroxide was neutralized with aq Na_2SO_3 solution. The resulting mixture was diluted with Et_2O (15 mL) and washed with H_2O (10 mL). The organic layer was dried over anhyd $MgSO_4$, and then the solvent was removed under reduced pressure. The remaining crude product was purified by column chromatography (eluent hexane–EtOAc, 90:10) to provide pure epoxide **3** (Table 2). Physical and spectroscopic (IR, ^{1}H NMR, and ^{13}C NMR) data for some selected compounds are given.
Compound **3g**: white solid, mp 72–74 °C. ^{1}H NMR (90 MHz, $CDCl_3$–TMS): δ = 7.10–8.00 (m, 8 H), 4.42 (d, 1 H), 4.03 (d, 1 H), 2.21 (s, 3 H). ^{13}C NMR (22.5 MHz, $CDCl_3$): δ = 195.1, 143.6, 136.0, 134.2, 129.0, 125.8, 70.3, 63.0, 25.5. IR (KBr): ν_{max} = 3069, 1677, 1605, 1415, 1236, 1162, 1012, 899, 766, 674 cm^{-1}. Anal. Calcd (%) for $C_{16}H_{13}ClO_2$: C, 70.59; H, 4.78. Found: C, 70.52.14; H, 4.74. MS (EI): m/z = 272 [M$^+$].
Compound **3m**: white solid, mp 78–80 °C. ^{1}H NMR (90 MHz, $CDCl_3$–TMS): δ = 7.20–8.00 (m, 9 H), 4.36 (d, 1 H), 4.04 (d, 1 H), 2.37 (s, 3 H). ^{13}C NMR (22.5 MHz, $CDCl_3$): δ = 193.5, 155.0, 150.8, 142.4, 136.0, 131.05, 129.7, 126.0, 114.4, 72.5, 62.1, 55.6. IR (KBr): ν_{max} = 3020, 2983, 2867, 1667, 1583, 1502, 1455, 1305, 1279, 1165, 1140, 1054, 827, 731, 693 cm^{-1}. Anal. Calcd (%) for $C_{16}H_{14}O_2$: C, 80.67; H, 5.88. Found: C, 80.63; H, 5.82. MS (EI): m/z = 238 [M$^+$].
Compound **3o**: white solid, mp 146–148 °C. ^{1}H NMR (90 MHz, $CDCl_3$–TMS): δ = 7.50–8.30 (m, 9 H), 4.30 (d, 1 H), 4.18 (d, 1 H). ^{13}C NMR (22.5 MHz, $CDCl_3$): δ = 192.2, 153.8, 143.5, 140, 138.0, 130.7, 128.5, 125.0, 119.6, 70.1, 62.5. IR (KBr): ν_{max} = 3063, 1659, 1594, 1493, 1395, 1333, 1128, 824, 742, 687 cm^{-1}. Anal. Calcd (%) for $C_{15}H_{11}NO_4$: C, 66.91; H, 4.09; N, 5.20. Found: C, 66.82; H, 4.02; N, 5.15. MS (EI): m/z = 269 [M$^+$].
Compound **3p**: white solid, mp 117–119 °C. ^{1}H NMR (90 MHz, $CDCl_3$–TMS): δ = 7.50–8.03 (m, 9 H), 4.32 (d, 1 H), 4.22 (d, 1 H). ^{13}C NMR (22.5 MHz, $CDCl_3$): δ = 198.2, 153.8, 145.7, 137.4, 134.3, 133.0, 129.5, 126.8, 121.7, 71.1, 62.0. IR (KBr): ν_{max} = 3061, 3025, 2923, 1797, 1595, 1495, 1395, 1337, 1139, 848, 747, 690 cm^{-1}. Anal. Calcd (%) for $C_{15}H_{11}NO_4$: C, 66.91; H, 4.09; N, 5.20. Found: C, 66.85; H, 4.04; N, 5.17. MS (EI): m/z = 269 [M$^+$].
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