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Highly efficient epoxidation of electron-deficient olefins with tetrabutylammonium peroxydisulfate

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Abstract—The epoxidation of α,β-unsaturated carbonyl compounds such as enones and enals was efficiently achieved with tetrabutylammonium peroxydisulfate in the presence of equimolar amounts of hydrogen peroxide and NaOH in acetonitrile or methanol at 25 °C in excellent yields. Base-sensitive substrate such as cinnamaldehyde could be successfully epoxidized in short reaction time under mild reaction conditions.

1. Introduction

Epoxidation of electron-deficient olefins has become one of the most significant organic reactions in recent years not only because the resulting epoxy compounds could be directly used in organic synthesis as essential building blocks, but also because they could be readily transformed into various synthetically useful intermediates, e.g., α- and β-hydroxy carbonyls, α,β-epoxy alcohols, allylic alcohols, and 1,3-diols, etc.[1]

The epoxidation of general and electron-rich olefins is generally achieved with electrophilic oxidants such as peroxy acids.[2] By contrast, epoxidation of electron-deficient olefins bearing an electron-withdrawing group at the double bond generally requires nucleophilic oxidation under basic conditions. Representative examples of such nucleophilic oxidants are H_2O_2/Bu_4NF,[3a] *t*-BuOOH/DBU,[1c] *m*-CPBA/KOH,[3b] NaOCl/cinchona alkaloid,[3c] or $NaBO_3$/THAHS.[3d] Recent reports of epoxidation of electron-deficient olefins with iodosyl benzene[4a] and dioxirane[4b] are noteworthy to be mentioned since the reactions are carried out under neutral conditions.

We have been interested in developing efficient epoxidizing systems/reagents for electron-deficient olefins such as α,β-unsaturated ketones. In our earlier work, we have failed to epoxidize α,β-unsaturated ketones with peroxysulfur intermediate generated in situ from superoxide anion and nitrobenzenesulfonyl chloride.[5] In continuation of this kind of work, we have successfully prepared tetrabutylammonium peroxydisulfate ($TBA_2S_2O_8$),[6] which turns out to be a versatile reagent for the useful transformations, e.g., cleavage of C=N bond,[7a] tetrahydrofuranylation/tetrahydropyranylation of alcohols,[6,7b,c] and α-iodination of functionalized ketones.[7d]

Recently, we have shown that various α,β-unsaturated ketones were smoothly epoxidized with $TBA_2S_2O_8/H_2O_2$ under basic conditions in excellent yields.[8] This initial result prompted us to thoroughly study this reaction using different solvents/various electron-deficient carbonyl compounds such as enones and enals. Herein, we wish to disclose the details of this research and data of the reaction products.

2. Results and discussion

Various α,β-unsaturated carbonyl compounds **3** (enones and enals) have been selected for epoxidation reaction according to the protocol described in Scheme 1.

In order to obtain the basic information regarding the optimum reaction conditions, α,β-unsaturated ketone **3a** was attempted first as a model compound under the various reaction conditions, and the results are summarized in Table 1.

When **3a** was reacted with equimolar amounts of **1** and NaOH without H_2O_2 in acetonitrile at 25 °C, very low reaction progress was observed (run 1). On the other hand, the same reaction conducted using equimolar amounts of H_2O_2 and NaOH in the absence of **1** provided the corresponding epoxide **4a** in ca. 30% yield (run 2). However, combined use of equimolar amounts of H_2O_2, NaOH, and **1** in acetonitrile at 25 °C resulted in excellent yield of

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Keywords: Epoxidation; Electron-deficient olefins; Tetrabutylammonium peroxydisulfate; Hydrogen peroxide.

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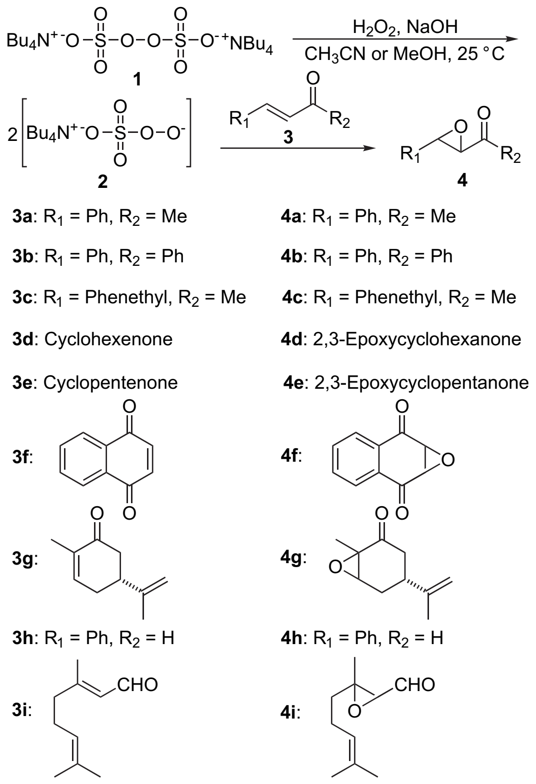


Scheme 1. Epoxidation of α,β-unsaturated carbonyl compounds with $TBA_2S_2O_8$, **1**.

epoxide **4a** (95%, run 3). In the case of K_2CO_3 being used as a base under the similar reaction conditions as run 3, the yield of **4a** was diminished to 50% (run 4). In the variation of solvents, several other organic solvents, e.g., benzene, CH_2Cl_2, $CHCl_3$, and methanol were also tested as the reaction solvent (run 5, 6, 7, and 8). Among other solvents examined, methanol demonstrated the best results in terms of reaction time and yield most probably due to the increased solubility of base in methanol (run 8). When **3a** was reacted with 0.5 equiv of each **1**, H_2O_2, and NaOH in methanol, epoxide **4a** was obtained in 90% yield (25 °C, 10 min, run 9). Thus, acetonitrile and methanol have been finally chosen as the reaction solvent, and reaction conditions (run 3 and 8) were adopted as the standard protocol for epoxidation reaction. The epoxidation reaction of various electron-deficient olefins was carried out first using acetonitrile as the reaction solvent under the standard reaction conditions (1 equiv of each **1**, H_2O_2, and NaOH, 25 °C), and the experimental results are summarized in Table 2 as reported earlier.[8]

Table 1. Attempted epoxidation of α,β-unsaturated ketone (**3a**) under various reaction conditions

$$\mathbf{3a} \xrightarrow[\text{Solvent}]{\mathbf{1}/H_2O_2/\text{Base}} \mathbf{4a}$$

Run	**1**/H_2O_2 (equiv)	Base (equiv)	Solvent	Time (min)	Yield[a] (%)
1	1/0	NaOH (1)	CH_3CN	60	Trace
2	0/1	NaOH (1)	CH_3CN	60	30
3	1/1	NaOH (1)	CH_3CN	30	95
4	1/1	K_2CO_3 (1)	CH_3CN	60	50
5	1/1	NaOH (1)	Benzene	60	10
6	1/1	NaOH (1)	CH_2Cl_2	60	65
7	1/1	NaOH (1)	$CHCl_3$	60	73
8	1/1	NaOH (1)	MeOH	5	98
9	0.5/0.5	NaOH (0.5)	MeOH	10	90

[a] Isolated yield.

Table 2. Epoxidation of α,β-unsaturated carbonyl compounds (**3**) in CH_3CN at 25 °C[a]

Run	Substrate	Time (min)	Product	Yield[b] (%)
1	**3a**	30	**4a**	95
2	**3c**	150	**4c**	88
3	**3d**	30	**4d**	95
4	**3e**	30	**4e**	85
5	**3f**	30	**4f**	95
6	**3g**	120	**4g**	92

[a] The ratio of **1**/**3**/H_2O_2/NaOH=1:1:1:1.
[b] Isolated yield.

The double bonds of acyclic and cyclic α,β-unsaturated ketones were smoothly epoxidized in good to excellent yields.

Methanol was then tested as the reaction solvent to expand the generality of this new epoxidation protocol, and the results are summarized in Table 3.

When methanol instead of acetonitrile was used as the reaction solvent, the reaction time reduced dramatically though the yields improved slightly in general probably due to the increased solubility of NaOH in methanol.

The double bonds of acyclic α,β-unsaturated ketones can be readily epoxidized (run 1 and 2), while the double bond in a long chain of acyclic α,β-unsaturated ketone required longer reaction time (run 3). In the case of cyclic α,β-unsaturated ketones (run 4, 5, 6, and 7), the epoxidation generally proceeded more rapidly than acyclic counterparts with comparable yields. The epoxidation of (−)-carvone **3g** is noteworthy to be mentioned since it clearly demonstrates that the double bond conjugated with carbonyl group can be epoxidized regioselectively in the presence of a general double bond even though it has an α-substituent (run 7). This result is quite interesting since β-substituted α,β-unsaturated ketones such as α-ionone and pulegone were failed to be epoxidized under the standard reaction conditions probably owing to the steric hindrance of β-substituent.[9]

Epoxidation of aldehydes is of special attention because earlier works on the epoxidation of cinnamaldehyde reported low yields or long reaction time.[1c,3a,d,10] Following our standard reaction conditions, however, cinnamaldehyde could be

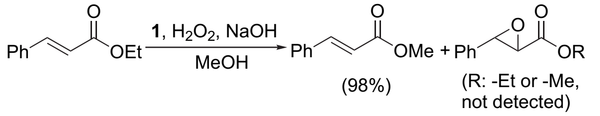
Table 3. Epoxidation of α,β-unsaturated carbonyl compounds (**3**) in CH_3OH at 25 °C[a]

Run	Substrate	Time (min)	Product	Yield[b] (%)
1	**3a**	10	**4a**	98
2	**3b**	20	**4b**	91
3	**3c**	30	**4c**	95
4	**3d**	5	**4d**	95
5	**3e**	10	**4e**	92
6	**3f**	10	**4f**	95
7	**3g**	60	**4g**	92
8	**3h**	20	**4h**	90
9	**3i**	60	**4i**	91

[a] The ratio of **1**/**3**/H_2O_2/NaOH=1:1:1:1.
[b] Isolated yield.

epoxidized in short reaction time in excellent yield (run 8, 90%, 20 min). Presumably, rapid reaction progress and mild reaction conditions enable the epoxidation reaction successful by allowing aldehyde moiety being intact during the short reaction time. The successful epoxidation of **3i** (run 9), an acyclic β-substituted α,β-unsaturated aldehyde, is also quite contrasted with the aforementioned negative results in the epoxidation of β-substituted α,β-unsaturated ketones such as α-ionone and pulegone.

We have also attempted epoxidation of α,β-unsaturated ester and amide in methanol under the similar reaction conditions, but epoxidation did not occur at all. In the case of α,β-unsaturated ester, transesterification took place almost quantitatively (Scheme 2).[11]



Scheme 2. Attempted reaction of α,β-unsaturated ester with **1**/H_2O_2/NaOH in CH_3OH.

Concerning the reaction mechanism, there seems to be two competitive epoxidating routes plausible: one is direct epoxidation by HOO^-, and the other is epoxidation by peroxysulfate anion intermediate **2** (Fig. 1).

The latter, however, appears to be the main route from the following experimental results (Table 1): (i) use of equivalent amount of each peroxydisulfate **1** and NaOH without H_2O_2 gave trace amount of epoxide **4a** (run 1); (ii) use of equivalent amount of each H_2O_2 and NaOH in the absence of **1** resulted in 30% yield of epoxide **4a** (run 2); (iii) use of equivalent amount of each **1**, H_2O_2, and NaOH afforded 95% yield of epoxide **4a** (run 3); (iv) use of 0.5 equiv of each **1**, H_2O_2, and NaOH provided 90% of epoxide **4a** (run 9). Result (iv) is highly informative about the reaction mechanism since it strongly indicates that 0.5 equiv of each **1** and HOO^- may form stoichiometric amount of peroxysulfate anion intermediate **2** as a reactive intermediate. The stronger nucleophile (HOO^-) rather than OH^- may be attributed to form peroxysulfate anion intermediate **2** more easily, which also shows a strong nucleophilic oxidizing ability toward substrates **3**. Therefore, epoxidation may be initiated by peroxysulfate anion intermediate **2** via 1,4-addition of **2** onto α,β-unsaturated carbonyls **3**, and then following electronic movement to afford the epoxides **4** together with tetrabutylammonium sulfate **5**. Peroxysulfate anion intermediate **2** has been neither isolated nor confirmed, but sulfate **5** was isolated in ca. 80% yield and confirmed.

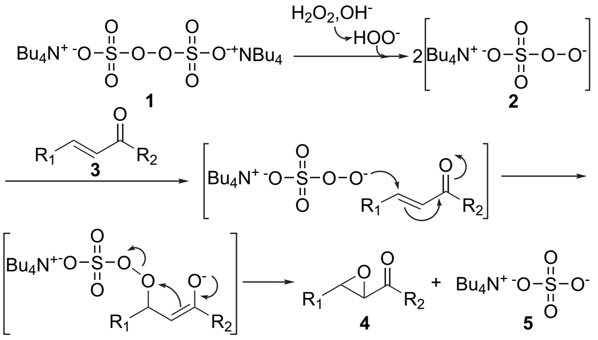


Figure 1. Plausible reaction mechanism for epoxidation.

In conclusion, both acyclic and cyclic α,β-unsaturated ketones (enones) were smoothly oxidized into the corresponding epoxides in excellent yields. α,β-Unsaturated aldehydes (enals) including base-sensitive cinnamaldehyde could also be epoxidized efficiently in short reaction time. Considering several advantages of our approach over the reported methods,[1c,3a,d,10,12,13] e.g., easy preparation of the requisite reagent ($TBA_2S_2O_8$), mild reaction conditions and simple reaction procedures, highly improved yields and dramatically reduced reaction time, use of acetonitrile and preferably MeOH as the reaction solvent, and furthermore inertness of acetal, ketal, and thioether functionalities toward **1** found in our earlier works,[6,7b,c] our epoxidating approach could be a method of choice in the epoxidation of α,β-unsaturated carbonyl compounds having various functional groups such as acetal, ketal, and thioether moieties without affecting these functional groups.

3. Experimental

3.1. General

All reactions were carried out in oven-dried glassware under dry N_2 or argon atmosphere. Melting points were taken on an Electro-thermal melting point apparatus and are uncorrected. ^1H NMR (200, 300 or 400 MHz) and ^{13}C NMR (75 or 100 MHz) spectra were obtained on Bruker FT spectrometers in $CDCl_3$, and chemical shifts (δ) were reported in parts per million downfield from tetramethylsilane as an internal standard. GC spectra were recorded from GC fitted with OV-17 packed column. High-resolution mass spectra (HRMS) were measured with VG Autospec Ultima instrument in EI (70 eV) mode at KAIST. Flash column chromatography was performed on silica gel (Merck, 230–400 mesh) using organic solvents as eluents. Starting α,β-unsaturated carbonyl compounds were purchased from commercial sources or prepared according to the known method. Methanol, acetonitrile, methylene chloride, and diethyl ether were purified by the usual methods.

3.1.1. Tetrabutylammonium peroxydisulfate (1). Tetrabutylammonium hydrogensulfate (21.2 g, 64.0 mmol) and potassium persulfate (8.70 g, 32.0 mmol) were dissolved in 140 mL of distilled water and the solution was stirred for 30 min at room temperature. The solution was extracted with CH_2Cl_2 (30 mL×3), and the combined organic layers were washed with distilled water (30 mL×3), dried over anhydrous $MgSO_4$, and filtered. Evaporation of the solvent in vacuo and subsequent drying under high vacuum gave **1** as a white solid in 95% yield. Mp 118–120 °C (dec); ^1H NMR (300 MHz) δ 3.2 (t, 8H), 1.6 (m, 8H), 1.2 (m, 8H), 0.8 (t, 12H); IR (NaCl) 2951, 2874, 1476, 1382, 1063, 1041, 676 cm^{-1}. Anal. Calcd for $C_{32}H_{72}N_2O_8S_2$: C, 56.7; H, 10.7; N, 4.14. Found: C, 56.9; H, 10.8; N, 4.15. The peroxydisulfate **1** turns out to be stable for a couple of months at room temperature, and can be stored in refrigerator permanently.

3.2. General procedure for the epoxidation in CH_3CN (procedure A)

To a stirred solution of α,β-unsaturated ketone (1.0 mmol) in anhydrous acetonitrile (1 mL) were added a mixture of **1** (667 mg, 1.0 mmol) and sodium hydroxide (40 mg, 1.0 mmol) in anhydrous acetonitrile (4 mL), and then hydrogen peroxide (100 μL, 30%, 1.0 mmol) slowly at 25 °C. The reaction mixture was stirred for 30–150 min at 25 °C with monitoring on TLC, and extracted with CH_2Cl_2 (30 mL×3). The combined organic layers were washed with water, dried over anhydrous $MgSO_4$, and concentrated in vacuo. The crude product was purified by flash column chromatography on SiO_2 to afford a pure product.

3.3. General procedure for the epoxidation in MeOH (procedure B)

To a stirred solution of α,β-unsaturated carbonyl compound (0.50 mmol) in 2 mL of methanol were added **1** (334 mg, 0.50 mmol), H_2O_2 (50 μL, 0.50 mmol, 30% in H_2O), and NaOH (20.0 mg, 0.5 mmol) successively at room temperature. After stirring for 5–60 min at 25 °C, the reaction mixture was poured into saturated aq NH_4Cl solution (30 mL), and extracted with diethyl ether (30 mL×3). The combined ethereal layers were washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure to provide a crude product, which was purified by flash column chromatography on SiO_2 to provide a pure product.

3.3.1. 3,4-Epoxy-4-phenyl-2-butanone (4a). Mp 53–55 °C (lit.[13a] 52–54 °C); ^{1}H NMR (300 MHz) δ 7.35–7.24 (m, 5H), 3.98 (d, J=1.8 Hz, 1H), 3.47 (d, J=1.8 Hz, 1H), 2.16 (s, H); ^{13}C NMR (75 MHz) δ 204.0, 134.9, 128.9, 128.6, 125.6, 63.4, 57.6, 24.7; IR (NaCl) 1712 cm^{-1}; HRMS (EI) *m/z* calcd for $C_{10}H_{10}O_2$ 162.0681, found 162.0682.

3.3.2. 2,3-Epoxy-1,3-diphenylpropanone (4b). Mp 90–91 °C (lit.[13b] 89–90 °C); ^{1}H NMR (300 MHz) δ 8.00–7.98 (m, 2H), 7.60–7.58 (m, 1H), 7.49–7.44 (m, 2H), 7.39–7.35 (m, 5H), 4.28 (d, J=1.9 Hz, 1H), 4.06 (d, J=1.9 Hz, 1H); ^{13}C NMR (75 MHz) δ 193.0, 135.4, 128.8, 128.7, 128.3, 125.7, 61.0, 59.3; IR (NaCl) 1689 cm^{-1}; HRMS (EI) *m/z* calcd for $C_{15}H_{12}O_2$ 224.0837, found 224.0838.

3.3.3. 3,4-Epoxy-6-phenyl-2-hexanone (4c). ^{1}H NMR (400 MHz) δ 7.30–7.16 (m, 5H), 3.15 (d, J=2.0 Hz, 1H), 3.09–3.05 (dt, 1H), 2.81–2.74 (m, 1H), 1.99 (s, 3H), 1.96–1.90 (m, 2H); ^{13}C NMR (100 MHz) δ 205.4, 140.3, 128.5, 128.4, 128.3, 128.2, 126.3, 59.8, 57.4, 33.3, 31.9, 24.7; IR (NaCl) 1711 cm^{-1}; HRMS (EI) *m/z* calcd for $C_{12}H_{14}O_2$ 190.0994, found 190.0998.

3.3.4. 2,3-Epoxycyclohexanone (4d). ^{1}H NMR (300 MHz) δ 3.56–3.54 (m, 1H), 3.19 (d, J=3.9 Hz, 1H), 2.55–2.46 (dt, 1H), 2.26–2.04 (m, 1H), 1.95–1.66 (m, 2H), 1.67–1.61 (m, 1H); ^{13}C NMR (75 MHz) δ 205.6, 55.6, 36.3, 22.8, 16.9; IR (NaCl) 1711 cm^{-1}; HRMS (EI) *m/z* calcd for $C_6H_8O_2$ 112.0524, found 112.0526.

3.3.5. 2,3-Epoxycyclopentanone (4e). ^{1}H NMR (300 MHz) δ 3.73–3.72 (m, 2H), 3.10–3.08 (m, 1H), 2.18–2.02 (m, 2H), 1.96–1.82 (m, 2H); ^{13}C NMR (75 MHz) δ 209.3, 57.2, 54.0, 29.7, 22.4.

3.3.6. 2,3-Epoxy-2,3-dihydro-1,4-naphthoquinone (4f). Mp 135–136 °C (lit.[13b] 134–136 °C); ^{1}H NMR (300 MHz) δ 7.98–7.95 (m, 2H), 7.76–7.73 (m, 2H), 4.00 (s, 2H); ^{13}C NMR (75 MHz) δ 190.5, 134.5, 131.6, 127.0, 55.1; HRMS (EI) *m/z* calcd for $C_{10}H_6O_3$ 174.0317, found 174.0317.

3.3.7. 2,3-Epoxy-5-isopropenyl-2-methylcyclohexanone (4g). ^{1}H NMR (300 MHz) δ 4.63–4.57 (d, 2H), 3.31 (t, 1H), 2.55–2.52 (m, 1H), 2.45–2.37 (m, 1H), 2.25–2.16 (m, 1H), 1.92–1.83 (dd, 1H), 1.81–1.72 (m, 1H), 1.57 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (75 MHz) δ 204.8, 146.0, 110.1, 60.9, 58.3, 41.4, 34.7, 28.3, 20.2, 14.9; IR (NaCl) 1705 cm^{-1}; HRMS (E1) *m/z* calcd for $C_{10}H_{14}O_2$ 166.0994, found 166.0986.

3.3.8. 2,3-Epoxy-3-phenylpropanal (4h). ^{1}H NMR (300 MHz) δ 9.16 (d, J=6.0 Hz, 1H), 7.36–7.24 (m, 5H), 4.14 (d, J=1.8 Hz, 1H), 3.43 (dd, J_1=6.0 Hz, J_2=1.8 Hz, 1H); ^{13}C NMR (75 MHz) δ 196.7, 134.0, 129.0, 128.6, 125.5, 62.7, 56.4; IR (NaCl) 1730 cm^{-1}; HRMS (EI) *m/z* calcd for $C_9H_8O_2$ 148.0524, found 148.0522.

3.3.9. 2,3-Epoxy-3,7-dimethyl-6-octenal (4i). ^{1}H NMR (300 MHz, mixture of cis and trans) δ 9.38–9.32 (m, 1H), 4.98–4.95 (m, 1H), 3.10–3.05 (m, 1H), 2.05–1.98 (m, 2H), 1.76–1.33 (m, 11H); ^{13}C NMR (75 MHz, mixture of cis and trans) δ 199.3, 198.6, 133.0, 132.5, 122.4, 122.2, 64.4, 64.3, 63.9, 63.3, 38.1, 33.2, 25.47, 25.42, 24.0, 23.2, 21.9, 17.47, 17.41, 17.0; HRMS (E1) *m/z* calcd for $C_{10}H_{16}O_2$ 168.1150, found 168.1157.

3.3.10. Isolation of tetrabutylammonium hydrogensulfate (5). In the above experiment (procedure A), the aqueous solution was saturated with sodium chloride and extracted with methylene chloride (30 mL×4). The CH_2Cl_2 layer was washed with aq HCl (30 mL, 1 N), then with distilled water and filtered. The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated in vacuo. Further drying under high vacuum gave pure tetrabutylammonium hydrogensulfate as a white solid in 80% yield, mp 170–171 °C (lit.[14] 169–171 °C).

Acknowledgements

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