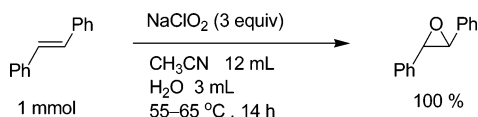# JOC*Note*

A Simple Method for Epoxidation of Olefins Using Sodium Chlorite as an Oxidant without a Catalyst

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Sodium chlorite has been demonstrated to be capable of epoxidizing a variety of olefins at 55−65 °C (oil bath). Chlorine dioxide is believed to be the pivotal epoxidizing agent in the reaction on the basis of the mechanistic studies.

Epoxidation of olefins is a key transformation in organic synthesis,[1] because epoxides are widely utilized in the laboratory as versatile building blocks and to manufacture different chemicals.[2] Nowadays, the development of nonasymmetric epoxidation methods to provide racemic epoxides is still important for both laboratory and industrial use. A number of nonasymmetric metal-catalyzed epoxidation reactions have been discovered in the past decade;[3] especially noteworthy was the development of redox active tungsten-containing-polyoxometalate-catalyzed reactions with hydrogen peroxide as the oxidant.[4] Meanwhile, several interesting small-organic-molecule-catalyzed[5] epoxidation reactions have been developed, with ketone- and amine-catalyzed reactions being typical examples. On the other hand, a more traditional and still currently used way to synthesize racemic epoxides is the treatment of olefins with a stoichiometric amount of peracids without the involvement of a catalyst, with *m*-chloroperbenzoic acid being the most often used oxidant.[6] This catalyst-free epoxidation reaction possesses advantages with respect to its being simple, environmentally benign, and easily handled.

Sodium chlorite ($NaClO_2$), a very cheap oxidizing agent, has been extensively used in water treatment and as a bleaching agent in the paper, pulp, and textiles industries.[7] However, in the field of synthetic organic chemistry, applications of sodium chlorite are not that broad. The most impressive use of sodium chlorite is its efficient oxidation of aldehydes to the corresponding carboxylic acids in acidic aqueous media.[8] More recently, there is increasing attention focused on its utilization in organic syntheses, including the oxidation of sulfides to the corresponding sulfoxides,[9] the chlorination of activated arenes[10] and ketones[11] with manganese complexes

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as catalysts in the presence of alumina, the oxidation of saturated hydrocarbons using manganese porphyrin as a catalyst,[12] the oxidation of primary alcohols to the corresponding carboxylic acids catalyzed by TEMPO and sodium hypochlorite,[13] the dihydroxylation of olefins with osmium as a catalyst,[14] and the transformation of 3,4-disubstituted furans into γ-hydroxybutenolides in acidic aqueous alcohol.[15] To the best of our knowledge, the use of sodium chlorite to epoxidize olefins to the corresponding epoxides in synthetic useful yields has never been reported before. Herein, we report a simple and easy protocol for the epoxidation of olefins that employs sodium chlorite as the oxidant in the homogeneous solvent mixture of acetonitrile and water at 55–65 °C (oil bath).

Initially, the epoxidation of *trans*-stilbene was examined using sodium chlorite (2 equiv) in the homogeneous solvent mixture of acetonitrile and water[16] (4/1, v/v) at room temperature. It was encouraging to observe the formation of *trans*-stilbene oxide in 81% yield, though the reaction was very slow (72 h). When the reaction temperature was elevated from room temperature to 55–65 °C,[17] the reaction was completed within 24 h, and afforded the epoxide in 84% yield. Further increasing the amount of sodium chlorite to 3 equiv resulted in a complete reaction within 14 h, and furnished the quantitative yield of *trans*-stilbene oxide.

A solvent-screening study was conducted using 1-phenylcyclohexene as the test substrate. Water was checked alone, and no reaction was observed.[18] Among screened solvent systems, acetonitrile–water was the solvent system of choice, in which an 81% yield of 1-phenylcyclohexene oxide was obtained after 5 h. DMF, DME, DMSO, and 1,4-dioxane afforded worse results compared to those for acetonitrile, giving the 1-phenylcyclohexene oxide in 54%, 50%, 38%, and 40% yield, respectively. Other solvents, including *t*-BuOH, THF, and acetone, were poor choices that yielded no or trace amounts of the desired epoxide. A dichloromethane–water system gave no reaction at all.

Consequently, the optimal reaction system was composed of 3 equiv of sodium chlorite and a solvent mixture of acetonitrile and water (4/1, v/v), with the oil bath temperature ranging from 55 to 65 °C (Scheme 1).

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(17) When the epoxidation of *trans*-stilbene was carried out using sodium chlorite (3 equiv) in 12 mL of CH₃CN and 3 mL of H₂O at 35–45 °C (oil bath) for 14 h, only a 15% yield of *trans*-stilbene oxide was obtained.
(18) We also investigated the epoxidation of *trans*-stilbene (1 mmol) using sodium chlorite (3 equiv) and Aliquat 336 (20 mol %) in 10 mL of aqueous Na₂·EDTA solution (2.5×10^{-4} M) at 55–65 °C for 12 h. An excellent yield (91%) of *trans*-stilbene oxide was obtained. Chalcone also gave 71% yield of the desired epoxide. However, the big problem with this system is its very limited substrate scope.
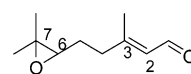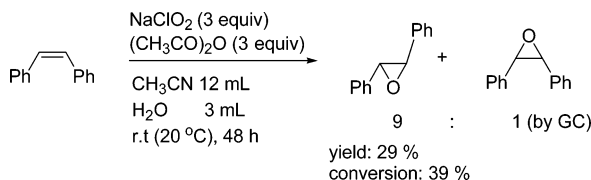
SCHEME 1. Optimal Epoxidation System of *trans*-Stilbene
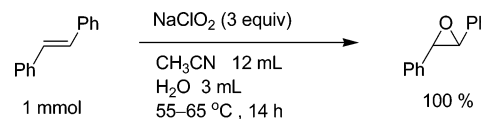
TABLE 1. Epoxidation of Olefins by Sodium Chlorite[a]

entry	olefin	time (h)	epoxide yield (%)[b]	conversion (%)
1	*trans*-stilbene (1)	14	100[c]	100
2	*cis*-stilbene (2)	23	78[d] (trans/cis = 7.5/1)	80
3[e]	*trans*-β-methylstyrene (3)	14	70[c]	95
4[e]	*cis*-β-methylstyrene (4)	17	52[f] (trans/cis = 10/1)	66
5	citral (5)	5	53	100
6	triphenylethylene (6)	15	92	100
7[g]	*trans*-α-methylstilbene (7)	21	70	99
8	citronellyl acetate (8)	15	65	100
9[h]	1-phenylcyclohexene (9)	5	81	100
10[i]	indene (10)	5	61	93
11	cyclooctene (11)	30	38	95
12[j]	chalcone (12)	48	55	58
13[k]	α-methylstyrene (13)	17	50	94
14[l]	styrene (14)	24	77	100
15[m]	cinnamyl alcohol (15)	6	51	100

[a] Unless otherwise indicated, the reaction was conducted with 1 mmol of olefin and 3 mmol of sodium chlorite in 12 mL of distilled acetonitrile and 3 mL of Na₂·EDTA aqueous solution (5×10^{-4} M) at 55–65 °C. *[b]* Isolated yield. *[c]* *trans*-Epoxide only. *[d]* Determined by GC. *[e]* Benzaldehyde was detected as a side product in low yield (5–10%). *[f]* Determined by ¹H NMR. *[g]* cis-α-Methylstilbene oxide was obtained in 15% yield. *[h]* One equivalent of sodium chlorite was used. *[i]* Two equivalents of sodium chlorite was used. *[j]* Four equivalents of sodium chlorite was used. *[k]* Benzophenone was produced in 15% yield. *[l]* Five millimoles of styrene was used. Benzaldehyde was observed as a side product in 8% yield. *[m]* Cinnamic aldehyde was obtained in 16% yield.

A variety of olefins (**1–15**), including 1,2-disubstituted olefins, trisubstituted olefins, cyclic olefins, one electron-deficient olefin, one 1,1-disubstituted olefin, one monosubstituted aromatic olefin, and an allylic alcohol, were tested using this new epoxidation method. It was found that epoxidation of *trans*-olefins (*trans*-stilbene and *trans*-β-methylstyrene) gave the corresponding *trans*-epoxides (Table 1, entries 1 and 3) exclusively, whereas *cis*-olefins (*cis*-stilbene and *cis*-β-methylstyrene) yielded *trans*-epoxides as the major product (entries 2 and 4). The nonstereospecificity of this reaction implied that the reaction proceeded stepwise. Moreover, it was disclosed that electron-rich olefins gave a faster epoxidation reaction, suggesting that the reaction was electrophilic. *cis*-Stilbene reacted slower than *trans*-stilbene because the electron density of the double bond of *cis*-stilbene is lower than that of *trans*-stilbene.[19] In the molecule of citral (entry 5), one trisubstituted 6,7-double bond was exclusively epoxidized because of its relatively greater electron density than that of another trisubstituted 2,3-double bond, to give the product **16**[20] in 53% yield.

Trisubstituted olefins were generally good substrates (Table 1, entries 5–9). In the case of *trans*-α-methylstil-

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(20) The structure of **16**:

SCHEME 2. Epoxidation of *cis*-Stilbene by Authentic Chlorine Dioxide Generated in Situ

FIGURE 1. (A) UV spectrum of $NaClO_2$ (3 mmol) in CH_3CN/H_2O (12 mL/3 mL) after being heated for 10 h at $55-65$ °C. (B) UV spectrum of ClO_2 from the reaction mixture of $NaClO_2$ (3 mmol) and $(CH_3CO)_2O$ (3 mmol)[25] in CH_3CN/H_2O (12 mL/3 mL) at room temperature.

bene, the desired epoxide was produced in 70% yield (entry 7) along with *cis*-α-methylstilbene oxide **17**,[21] obtained in 15% yield. Another trisubstituted aliphatic olefin **8** was epoxidized in 65% yield (entry 8). Cyclic 1,2-disubstituted olefin, indene, yielded the epoxide in 61% yield, using 2 equiv of sodium chlorite (entry 10). As for the electron-deficient olefin, chalcone was epoxidized in 55% yield, using 4 equiv of sodium chlorite after 2 days (entry 12). Epoxidation of styrene proceeded smoothly to afford styrene oxide in 77% yield (entry 14). However, 1-dodecene was completely inert under standard conditions. As an allylic olefin, the epoxidation of cinnamyl alcohol readily provided the desired epoxide in 51% yield (entry 15).

A large-scale epoxidation reaction of styrene (50 mmol) was performed to give styrene oxide in 75% yield under standard conditions after 2 days.

The mechanism of this new epoxidation reaction is worth considering. *cis*-Stilbene (1 mmol) remained unchanged after being heated in CH_3CN/H_2O (12 mL/3 mL) at $55-65$ °C for 24 h, and this proved thermal stability of *cis*-stilbene ensured that *trans*-stilbene oxide, a major product from the epoxidation of *cis*-stilbene under standard conditions (Table 1, entry 2), was not derived from the reaction pathway as follows: *cis*-stilbene → *trans*-stilbene → *trans*-stilbene oxide. The nonstereospecificity of our epoxidation reaction implied that the reaction might go on via free radical species. This was the case because the addition of a radical-trapping compound, 2,6-di-*tert*-butyl-4-methylphenol (1 equiv), in the epoxidation of *trans*-stilbene resulted in a significant decrease of the epoxide yield, to only 33%, compared to the quantitative yield attained under standard conditions (Table 1, entry 1).[22]

It was reported that chlorine dioxide (ClO_2) and hypochlorite were generally formed during the oxidation of an aldehyde to the corresponding acid by sodium chlorite.[8a,c] During the investigation of bleaching properties of chlorine dioxide (ClO_2), two papers mentioned that chlorine dioxide could epoxidize styrene[23] and *trans*-stilbene[24] to their corresponding epoxides in 26% and 36% yield, respectively. This observation suggested that chlo-

rine dioxide, a reasonably disproportionate product of sodium chlorite when heated and an inherent radical species itself, was quite possibly the key epoxidizing agent in our reaction. To make a convincing conclusion, we performed the epoxidation of *cis*-stilbene with authentic chlorine dioxide that was generated by mixing sodium chlorite and acetic anhydride (Scheme 2).[25] It was observed that *trans*-stilbene oxide was formed as the major epoxidation product, and that the ratio of *trans*- to *cis*-epoxide was 9/1. On the other hand, in the course of epoxidation of *cis*-stilbene under our standard conditions, except that sodium chlorite was replaced with sodium hypochlorite[26] as the oxidant after 48 h, it was found that *cis*-stilbene gave *trans*-stilbene oxide as the major product, but the ratio of *trans*- to *cis*-epoxide was 35/1, which was quite different from the ratio (7.5/1, Table 1, entry 2) observed in the chlorite epoxidation system. Upon comparison of the *trans*- to *cis*-epoxide ratio numbers in these three reactions, it was apparent that chlorine dioxide was more likely to be the pivotal epoxidizing agent in our epoxidation reaction.

To detect the presence of chlorine dioxide in our reaction, an ultraviolet−visible spectrometry experiment was carried out. The mixture of sodium chlorite (3 mmol) in acetonitrile (12 mL) and water (3 mL) was heated for 10 h at $55-65$ °C, and the resulting cooled yellow solution was measured by a UV spectrometer. The characteristic absorption peak of chlorine dioxide ($\lambda_{max} = 359$ nm)[27] appeared (Figure 1), indicating that chlorine dioxide was present in our reaction and therefore responsible for epoxidation reaction.

In conclusion, a simple epoxidation of olefins using sodium chlorite as an oxidant without the aid of a catalyst has been developed. The new reactivity disclosed here expands the synthetic application of sodium chlorite to include one that is readily available and very cheap.

(21) The structure of **17**:

(22) The epoxidation of *trans*-stilbene was carried out under conditions identical to those of Scheme 1 except for the addition of 15 mol % TEMPO. The reaction was affected dramatically, as *trans*-stilbene oxide was obtained in only 16% yield at 47% conversion of the starting material after 16 h, further indicating that a radical species was involved in our epoxidation reaction.

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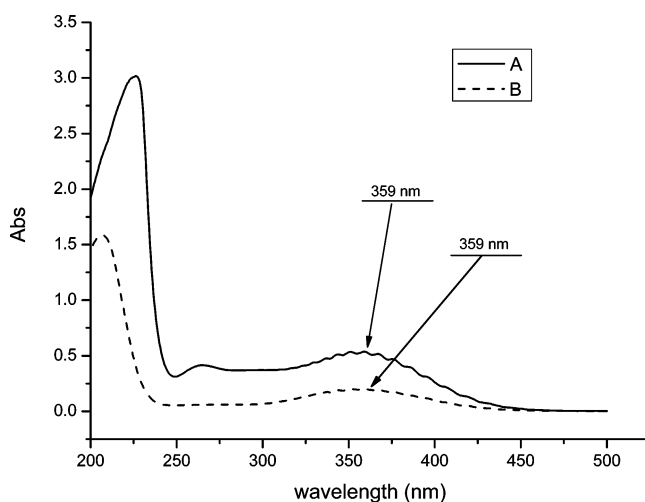(24) Lindgren, B. O.; Nilsson, T. *Acta Chem. Scand. B* **1974**, *28*, 847−852.

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(26) Sodium chlorate ($NaClO_3$) gave no reaction at all in the epoxidation of *tans*-stilbene under the standard conditions.

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Mechanistic studies show that chlorine dioxide is the key epoxidizing agent in the reaction.

Experimental Section

General Epoxidation Procedure. To a stirred mixture of olefin (1 mmol) in the homogeneous solvent mixture of distilled CH_3CN (12 mL) and aqueous $Na_2 \cdot EDTA$ solution (3 mL, 5 × 10^{-4} M) was added sodium chlorite (340 mg, 3 mmol) at room temperature. The reaction flask was then put into an oil bath with a temperature between 55 and 65 °C. The reaction was allowed to run at 55−65 °C (oil bath), and was monitored by TLC. The mixture turned from colorless to yellow in the course of the reaction. After completion, the reaction was quenched by a saturated aqueous solution of $Na_2S_2O_3$, and the mixture was then extracted with CH_2Cl_2 three times. The combined organic layer was washed with water and brine one time each, dried over anhydrous Na_2SO_4, filtered, and concentrated. The products were confirmed by comparison of ^1H NMR and ^{13}C NMR data with those reported in the literature.

Epoxidation of *trans*-Stilbene. To a stirred mixture of *trans*-stilbene (180 mg, 1 mmol) in the homogeneous solvent mixture of distilled CH_3CN (12 mL) and aqueous $Na_2 \cdot EDTA$ solution (3 mL, 5 × 10^{-4} M) was added sodium chlorite (340 mg, 3 mmol) at room temperature. The reaction flask was then put into an oil bath with a temperature between 55 and 65 °C (oil bath). The reaction was monitored by TLC. The mixture turned from colorless to yellow in the course of the reaction. After completion within 14 h, the reaction was quenched by a saturated aqueous solution of $Na_2S_2O_3$, and the mixture was then extracted with CH_2Cl_2 three times. The combined organic layer was washed with water and brine one time each, dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by flash column chromatography (2% EtOAc in petroleum ether) on silica gel to give *trans*-stilbene oxide (**1P**, ref: SI1; 196 mg, 100%) as a white solid: mp 66−67 °C; ^1H NMR (300 MHz, CDCl$_3$) δ 7.36 (m, 10H), 3.83 (s, 2H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 137.07, 128.48, 128.23, 125.44, 62.70;[28] IR (KBr) 1452, 1278, 860, 837, 745, 690 cm^{-1}; EI-MS (*m/z*) (M$^+$) 197 (26), 196 (12), 180 (16), 167 (100), 165 (70), 105 (40), 77 (36).

Epoxidation of Triphenylethylene. The epoxidation of triphenylethylene (256 mg, 1 mmol) was carried out following the typical procedure, with a reaction time of 15 h. The crude product was purified by preparative TLC plate (2% EtOAc in petroleum ether), which gave 2,2,3-triphenyloxirane (**6P**, 250 mg, 92% yield, ref: SI1) as a white solid: mp 75−76 °C; ^1H NMR (300 MHz, CDCl$_3$) δ 7.37−7.25 (m, 5H), 7.21 (m, 5H), 7.16−7.14 (m, 3H), 7.05−7.03 (m, 2H), 4.33 (s, 1H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 140.95, 135.76, 135.39, 129.15, 128.33, 127.81, 127.75, 127.68, 127.61, 127.52, 126.72, 126.30, 68.62, 68.00;[28] IR (KBr) 3027, 1605, 1460, 1394, 1280, 756, 690 cm^{-1}; EI-MS (*m/z*) (M$^+$) 272 (20), 271 (26), 243 (16), 166 (40), 165 (100), 105 (32).

Epoxidation of Chalcone. The epoxidation of chalcone (208 mg, 1 mmol) was carried out following the typical procedure, with a reaction time of 48 h. The crude product was purified by flash column chromatography (5% EtOAc in petroleum ether) on silica gel to give chalcone oxide (**12P**, ref: SI5, 123 mg, 55%) as a white solid: mp 81−82 °C; ^1H NMR (300 MHz, CDCl$_3$) δ 8.02−7.99 (dd, J = 8.4 Hz, 2H), 7.61−7.38 (m, 8H), 4.30 (d, J = 2.1 Hz, 1H), 4.07 (d, J = 1.8 Hz, 1H); ^{13}C NMR (75 MHz, CDCl$_3$) δ 193.09, 135.51, 134.03, 129.10, 128.92, 128.87, 128.81, 128.38, 125.84, 61.03, 59.41;[29] IR (KBr) 1685, 1449, 1405, 1235, 756, 694 cm^{-1}; EI-MS (*m/z*) (M$^+$) 224 (83), 223 (70), 196 (16), 167 (28), 118 (20), 105 (100), 77 (81).

Supporting Information Available: General information, specific examples of epoxidation, NMR spectra of epoxides, and analytic data of compound **17**. This material is available free of charge via the Internet at http://pubs.acs.org.

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