# Efficient Conversion of Epoxides into β-Hydroperoxy Alcohols Catalyzed by Antimony Trichloride/$SiO_2$

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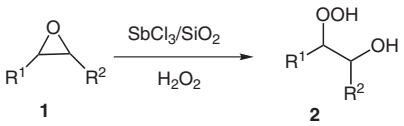
**Abstract:** Efficient ring-opening of various epoxides with hydrogen peroxide, catalyzed by antimony trichloride/$SiO_2$, afforded the corresponding β-hydroperoxy alcohols in good to excellent yields under mild reaction conditions. The reactions were efficiently promoted by ultrasound irradiation.

**Key words:** epoxides, β-hydroperoxy alcohols, antimony trichloride, ultrasound

Epoxides are versatile and important intermediates in organic synthesis that can undergo regio- and stereoselective ring-opening reactions to give β-substituted alcohols with a variety of nucleophilic species.[1] The opening of epoxides with hydrogen peroxide is the easiest and most straightforward synthetic procedure for the preparation of β-hydroperoxy alcohols. Such alcohols are useful in the field of pharmaceuticals and natural products,[2–3] especially for the synthesis of 1,2,4-trioxanes,[4] which are being actively investigated as antimalarial agents.[5] They can also serve as effective tridentate oxygen donors for epoxidation of ene diols.[6] This ring-opening reaction can be catalyzed by $HClO_4$,[7] $CF_3CO_2H$,[8] molybdenyl acetylacetonate,[9] and methyltriotylammonium tetrakis(oxodiperoxotungsto)phosphate.[10] However, these procedures are associated with disadvantages such as the use of strong acid,[7–8] or unsatisfactory yields,[9] or require special efforts to prepare the catalyst.[10] Thus, an improved protocol for the conversion of epoxides into the corresponding β-hydroperoxy alcohols is still actively pursued.

In recent years, antimony trichloride has been used as a catalyst in organic synthesis because this compound is not only commercially available and inexpensive, but is also easier to handle than other metal halides such as $InCl_3$, $GdCl_3$ and $TiCl_4$.[11] Antimony trichloride has been utilized as a catalyst for many important organic transformations, including selective cleavage of trityl ethers,[12] Michael addition,[13] the ring opening of epoxides with anilines,[14] synthesis of acylals,[15] benzo[b]1,4-diazepines,[16] and bis(indolyl)methanes.[17] Use of silica-supported reagents as recoverable and reusable catalysts in organic synthesis has received considerable attention. In a continuation of our work to develop new synthetic methodologies,[18] we report herein a simple and practical method for conversion of epoxides into β-hydroperoxy alcohols catalyzed by antimony trichloride/$SiO_2$ (Scheme1).




1 2

**Scheme 1**

Initially, the catalytic activities of various catalysts such as $H_3BO_3$, *p*-toluenesulfonic acid, $InBr_3$, $In(OTf)_3$, $ZrCl_4$, $(NH_4)_2Ce(NO_3)_6$, $I_2$, $SbCl_3$, $HClO_4/SiO_2$, $HBF_4/SiO_2$, $NaHSO_4/SiO_2$ and $SbCl_3/SiO_2$ were investigated to pro-

**Table 1** Synthesis of 2-Hydroperoxy-2-phenylethanol (**2e**) under Different Conditions[a]

| Entry | Catalyst | Catalyst loading (mol%) | Time (h) | Yield (%)[b] |
|---|---|---|---|---|
| 1 | none | – | 24 | trace |
| 2 | $H_3BO_3$ | 5 | 8 | 51 |
| 3 | *p*-TsOH | 5 | 12 | 68 |
| 4 | $InBr_3$ | 5 | 36 | 43 |
| 5 | $In(OTf)_3$ | 5 | 24 | 38 |
| 6 | $ZrCl_4$ | 5 | 48 | 46 |
| 7 | $(NH_4)_2Ce(NO_3)_6$ | 5 | 15 | 52 |
| 8 | $I_2$ | 5 | 15 | 42 |
| 9 | $SbCl_3$ | 5 | 5 | 41 |
| 10 | $HClO_4/SiO_2$ | 5 | 8 | 53 |
| 11 | $HBF_4/SiO_2$ | 5 | 72 | 62 |
| 12 | $NaHSO_4/SiO_2$ | 5 | 5 | 68 |
| 13 | $SbCl_3/SiO_2$ | 1 | 10 | 80 |
| 14 | $SbCl_3/SiO_2$ | 5 | 8 | 82 |
| 15 | $SbCl_3/SiO_2$ | 10 | 5 | 85 |
| 16 | $SbCl_3/SiO_2$ | 20 | 5 | 79 |
| 17 | $SbCl_3/SiO_2$ | 10 | 1.5 | 85[c] |

[a] The reaction was carried out according to the general experimental procedure without sonication.
[b] Isolated yield.
[c] With sonication.

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mote the model reaction of 2-phenyloxirane (**1e**) and hydrogen peroxide; the results are presented in Table 1. The catalyst plays a crucial role in the success of the reaction both in terms of the rate and yields. In the absence of catalyst, only traces of product were obtained, even after 24 hours (Table 1, entry 1). Among the catalysts tested, $SbCl_3/SiO_2$ was found to be the most effective catalyst in terms of both reaction time and yield. Next, we optimized the quantity of the catalyst ($SbCl_3/SiO_2$) used in this reaction and it was observed that the use of just 10 mol% was sufficient to produce an excellent yield of the product (Table 1, entry 15). Higher amounts of the catalyst (20mol%) did not improve the result (Table 1, entry 16). Lower catalyst loading could be used with only a marginal drop in reaction rate. When the same reaction was performed under sonication in an ultrasonic bath, the reaction time was strikingly shortened from 5 hours to 1.5 hours (Table 1, entry 17). It is thus apparent that the reaction could be efficiently promoted by ultrasound irradiation.

In light of these results, subsequent studies were carried out in order to evaluate the scope of the catalyst's application under the optimized conditions. Various epoxides were treated with an excess amount of $H_2O_2$ in the presence of 10 mol% of $SbCl_3/SiO_2$ with or without ultrasonic conditions. The results, presented in Table 2, indicated that the $SbCl_3/SiO_2$-catalyzed ring-opening reaction of epoxides proceeded smoothly and produced the corresponding β-hydroperoxy alcohols in good to excellent yields. The unsymmetrical alkyl oxiranes **1i** and **1j** afforded β-hydroperoxy alcohols in a regioselective manner with preferential attack at the terminal position. For the styrene oxide and substituted styrene oxides (**1a**–**1h**), the reaction occurred on the more substituted carbon, since the benzyl position of the epoxides are more positive and thus more prone to attack by $H_2O_2$. With cyclohexene oxide, the ring opening took place completely via a *trans*-stereospecific pathway and gave only the *trans* isomer (Table 2, entry k).

In summary, we have reported a novel, mild and highly efficient procedure for the synthesis of β-hydroperoxy alcohols. The use of an inexpensive and easily available catalyst, the high yields obtained, and the relatively short reaction times, together with the potential usefulness of the process for industrial applications are all attractive features of this method.

**Table 2** Synthesis of β-Hydroperoxy Alcohols in the Presence of $SbCl_3/SiO_2$

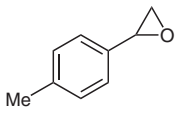
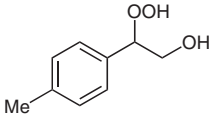
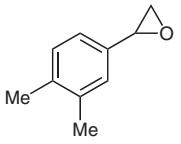
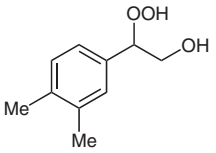
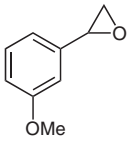
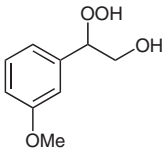
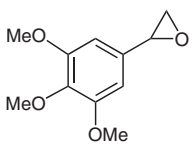
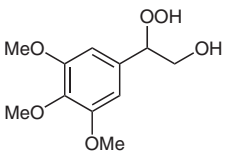
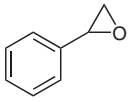
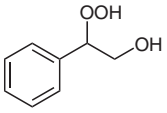
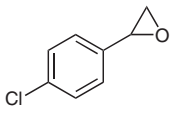
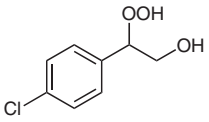
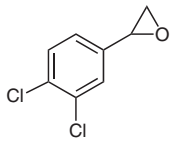
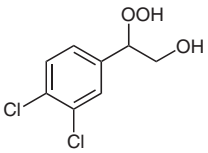
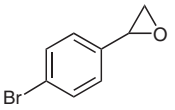
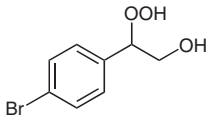
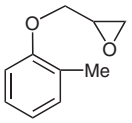
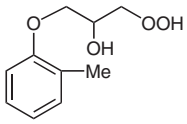
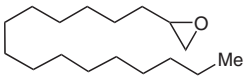
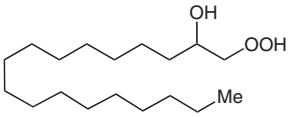
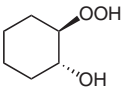
| Entry | Substrate **1** | Product **2** | Without sonication | | With sonication | |
|---|---|---|---|---|---|---|
| | | | Time (h) | Yield (%)[a] | Time (h) | Yield (%)[a] |
| **a** |  |  | 9 | 85 | 1.5 | 86 (95:5)[b] |
| **b** |  |  | 12 | 72 | 2.0 | 75 |
| **c** |  |  | 6 | 85 | 1.0 | 85 (96:4)[b] |
| **d** |  |  | 2 | 85 | 0.5 | 77 |
| **e** |  |  | 5 | 85 | 1.5 | 85 |

**Table 2** Synthesis of β-Hydroperoxy Alcohols in the Presence of $SbCl_3/SiO_2$ (continued)

| Entry | Substrate **1** | Product **2** | Without sonication | | With sonication | |
|---|---|---|---|---|---|---|
| | | | Time (h) | Yield (%)[a] | Time (h) | Yield (%)[a] |
| **f** |  |  | 5 | 86 | 1.0 | 85 |
| **g** |  |  | 5 | 78 | 1.0 | 78 (93:7)[b] |
| **h** |  |  | 10 | 81 | 2.0 | 85 |
| **i** |  |  | 8 | 82 | 1.5 | 82 (94:6)[b] |
| **j** |  |  | 12 | 80 | 2.0 | 81 |
| **k** |  |  | 12 | 82 | 2.0 | 83 |

[a] Isolated yield.
[b] Regioselectivity determined by $^1H$ NMR.

IR spectra were obtained using a Shimadzu FTIR-8900 spectrometer. $^1H$ NMR spectra were taken with Varian 400 or Bruker DRX-500 spectrometers as $CDCl_3$ solutions with TMS as internal standard. Elemental analyses were performed on a Vario EL III CHNOS elemental analyzer. Sonication was performed in a KQ-250E ultrasonic clearer with a frequency of 40 kHz and an output power of 250 W.

### Preparation of Antimony Trichloride Adsorbed on Silica Gel ($SbCl_3/SiO_2$)

The preparation of $SbCl_3/SiO_2$ was carried out following a reported procedure.[19] $SbCl_3$ (2.28 g, 10 mmol) was added to a suspension of $SiO_2$ (300–400 mesh, 27.8 g) in EtOH (50.0 mL). The mixture was stirred at r.t. for 1 h then the solvent was removed with a rotary evaporator and the residue was heated at 100 °C under vacuum for 5 h to furnish $SbCl_3/SiO_2$ as a free-flowing powder.

### Synthesis of β-Hydroperoxy Alcohols; General Procedure

**Caution:** Since organic peroxides are potentially hazardous compounds, they must be handled with due care; avoid exposure to strong heat or light, mechanical shock, oxidizable organic materials, or transition-metal ions. A safety shield should be used for all reactions involving hydrogen peroxide. No particular difficulties were experienced in handling any of the hydroperoxy alcohols prepared in this work using the reaction scales and procedures described below together with the safeguards mentioned above.

Hydrogen peroxide (8.5 mL, 30% in $H_2O$) was saturated with NaCl and the solution was extracted with $Et_2O$ (4 × 30 mL). Behind a safety shield, the ethereal solution was dried ($MgSO_4$) and evaporated in a rotavapor using a cold water bath until the solution volume reached 20 mL (~70 mmol $H_2O_2$). This solution was introduced to a flask containing epoxide (7.5 mmol) at r.t., $SbCl_3/SiO_2$ (2.25 g, 10 mol%) was added and the mixture was stirred or irradiated in the ultrasonic bath (the progress of the reaction was monitored by TLC). Upon completion of the reaction, the catalyst was removed by filtration and the filtrate was diluted with $Et_2O$ (100 mL), washed with $H_2O$ (2 × 50 mL) and brine (50 mL), dried ($MgSO_4$) and evaporated to give the crude β-hydroperoxy alcohol. Further purification was achieved by silica gel chromatography (EtOAc–cyclohexane) to afford the pure product.

### 2-Hydroperoxy-2-*p*-tolylethanol (2a)

Colorless needles; mp 81–82 °C.

IR (KBr): 3369, 2976, 2869, 1519, 1423, 1305, 1242, 1089, 1053, 1037, 908, 817, 767, 723 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ = 2.36 (s, 3 H), 3.80 (dd, *J* = 12.8, 3.6 Hz, 1 H), 3.93 (dd, *J* = 12.8, 8.4 Hz, 1 H), 5.12 (dd, *J* = 8.4, 3.6 Hz, 1 H), 7.21 (d, *J* = 6.0 Hz, 2 H), 7.26 (d, *J* = 6.0 Hz, 2 H), 8.13 (br s, 1 H).

Anal. Calcd for $C_9H_{12}O_3$: C, 64.27; H, 7.19. Found: C, 64.02; H, 7.42.

**2-(3,4-Dimethylphenyl)-2-hydroperoxyethanol (2b)**
Colorless platelets; mp 82–84 °C.

IR (KBr): 3369, 3138, 2808, 1616, 1504, 1403, 1338, 1245, 1093, 1056, 1039, 954, 769 $cm^{-1}$.

$^1H$ NMR (300 MHz, $CDCl_3$): δ = 2.26 (s, 3 H), 2.27 (s, 3 H), 3.79 (dd, *J* = 12.4, 3.6 Hz, 1 H), 3.91 (dd, *J* = 8.4, 12.4 Hz, 1 H), 5.08 (dd, *J* = 3.6, 8.4 Hz, 1 H), 7.08–7.18 (m, 3 H), 8.66 (br s, 1 H).

Anal. Calcd for $C_{10}H_{14}O_3$: C, 65.91; H, 7.74. Found: C, 66.02; H, 7.58.

**2-Hydroperoxy-2-(3-methoxyphenyl)ethanol (2c)**
Colorless needles; mp 66–68 °C.

IR (KBr): 3400, 2937, 2837, 1602, 1587, 1490, 1456, 1436, 1261, 1155, 1039, 873, 785, 698 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ = 3.78 (dd, *J* = 12.8, 3.6 Hz, 1 H), 3.82 (s, 3 H), 3.90 (dd, *J* = 12.8, 8.0 Hz, 1 H), 5.13 (dd, *J* = 8.0, 3.6 Hz, 1 H), 6.89–6.85 (m, 3 H), 7.32 (t, *J* = 5.7 Hz, 1 H).

Anal. Calcd for $C_9H_{12}O_4$: C, 58.69; H, 6.57. Found: C, 58.90; H, 6.38.

**2-Hydroperoxy-2-(3,4,5-trimethoxyphenyl)ethanol (2d)**
Colorless cubics; mp 116–118 °C.

IR (KBr): 3411, 3253, 2931, 2842, 1595, 1510, 1421, 1342, 1325, 1240, 1122, 1066, 981, 837, 785, 707 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ = 3.80 (dd, *J* = 12.8, 3.6 Hz, 1 H), 3.85 (s, 3 H), 3.88 (s, 6 H), 3.92 (dd, *J* = 12.8, 8.4 Hz, 1 H), 5.09 (dd, *J* = 8.4, 3.6 Hz, 1 H), 6.59 (s, 2 H), 8.22 (br s, 1 H).

Anal. Calcd for $C_{11}H_{16}O6$: C, 54.09; H, 6.60. Found: C, 54.26; H, 6.48.

**2-Hydroperoxy-2-phenylethanol (2e)**
Colorless platelets; mp 63–64 °C (Lit.[8] 60–61 °C).

IR (KBr): 3379, 3107, 2972, 1492, 1452, 1417, 1247, 1099, 1074, 1028, 916, 826, 756, 702 $cm^{-1}$.

$^1H$ NMR (500 MHz, $CDCl_3$): δ = 3.83 (dd, *J* = 12.5, 3.5 Hz, 1 H), 3.92 (dd, *J* = 12.5, 8.0 Hz, 1 H), 5.16 (dd, *J* = 8.0, 3.5 Hz, 1 H), 7.24–7.43 (m, 5 H), 9.03 (br s, 1 H).

Anal. Calcd for $C_8H_{10}O_3$: C, 62.33; H, 6.54. Found: C, 62.55; H, 6.38.

**2-(4-Chlorophenyl)-2-hydroperoxyethanol (2f)**
Colorless platelets; mp 102–104 °C.

IR (KBr): 3425, 3070, 1681, 1593, 1490, 1282, 1232, 1091, 1014, 979, 825, 761 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ = 3.80 (dd, *J* = 12.4, 3.6 Hz, 1 H), 3.88 (dd, *J* = 12.4, 7.6 Hz, 1 H), 5.11 (dd, *J* = 7.6, 3.6 Hz, 1 H), 7.30 (d, *J* = 8.4 Hz, 2 H), 7.37 (d, *J* = 8.4 Hz, 2 H), 8.46 (br s, 1 H).

Anal. Calcd for $C_8H_9ClO_3$: C, 50.94; H, 4.81. Found: C, 51.12; H, 4.68.

**2-(3,4-Dichlorophenyl)-2-hydroperoxyethanol (2g)**
Colorless platelets; mp 106–108 °C.

IR (KBr): 3384, 3109, 2987, 2785, 1589, 1562, 1425, 1382, 1342, 1240, 1062, 1045, 864, 829, 773 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ = 3.73 (dd, *J* = 12.4, 7.6 Hz, 1 H), 3.88 (dd, *J* = 12.4, 2.8 Hz, 1 H), 5.55 (dd, *J* = 7.6, 2.8 Hz, 1 H), 7.30 (dd, *J* = 8.4, 1.5 Hz, 1 H), 7.41 (d, *J* = 1.5 Hz, 1 H), 7.44 (d, *J* = 8.4 Hz, 1 H), 8.34 (br s, 1 H).

Anal. Calcd for $C_8H_8Cl_2O_3$: C, 43.08; H, 3.62. Found: C, 43.25; H, 3.50.

**1-Hydroperoxy-3-*o*-tolyloxypropan-2-ol (2i)**
Colorless platelets; mp 67–68 °C.

IR (KBr): 3489, 3203, 2979, 1602, 1496, 1458, 1402, 1244, 1126, 1049, 881, 752 $cm^{-1}$.

$^1H$ NMR (400 MHz, $CDCl_3$): δ = 2.23 (s, 3 H), 4.04–4.12 (m, 2 H), 4.20 (dd, *J* = 12.8, 6.4 Hz, 1 H), 4.30 (dd, *J* = 12.8, 3.6 Hz, 1 H), 4.39–4.45 (m, 1 H), 6.83 (d, *J* = 7.6 Hz, 1 H), 6.90 (t, *J* = 7.6 Hz, 1 H), 7.14–7.18 (m, 2 H), 8.78 (br s, 1 H).

Anal. Calcd for $C_{10}H_{14}O_4$: C, 60.59; H, 7.12. Found: C, 60.80; H, 6.98.

**1-Hydroperoxyoctadecan-2-ol (2j)**
Colorless platelets; mp 74–75 °C.

IR (KBr): 3232, 2979, 1467, 1377, 1143, 1072, 993, 873, 721 $cm^{-1}$.

$^1H$ NMR (500 MHz, $CDCl_3$): δ = 0.88 (t, *J* = 7.0 Hz, 3 H), 1.26–1.45 (m, 30 H), 3.46 (dd, *J* = 11.0, 8.0 Hz, 1 H), 3.72–3.75 (m, 1 H), 3.73 (dd, *J* = 8.0, 3.0 Hz, 1 H).

Anal. Calcd for $C_{18}H_{38}O_3$: C, 71.47; H, 12.66. Found: C, 71.68; H, 12.46.

**2-Hydroperoxycyclohexanol (2k)**
Colorless viscous liquid.

IR (film): 3379, 2937, 2860, 1452, 1234, 1124, 1070, 1026, 925, 835 $cm^{-1}$.

$^1H$ NMR (500 MHz, $CDCl_3$): δ = 1.15–1.34 (m, 4 H), 1.65–1.72 (m, 2 H), 1.98–2.09 (m, 2 H), 3.64 (td, *J* = 10.5, 5.0 Hz, 1 H), 3.73 (td, *J* = 10.5, 5.0 Hz, 1 H), 7.42 (br s, 1 H).

Anal. Calcd for $C_6H_{12}O_3$: C, 54.53; H, 9.15. Found: C, 54.72; H, 8.98.

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