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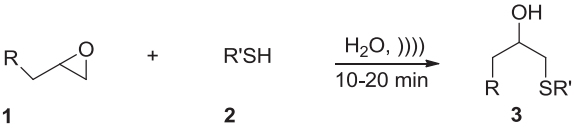
An environmentally friendly and efficient procedure is developed for ring opening of various epoxides with thiols under non-thermal conditions. Reactions take place by ultrasonic irradiation of the two reactants suspended in an additive-free aqueous medium. Consequently, high-yield formation of various $\beta$-hydroxy sulfides is quickly observed.




**Keywords:** thiolysis; green chemistry; aqueous conditions; sonochemistry; $\beta$-hydroxy sulfides

## 1. Introduction

Ring opening of epoxides with nucleophilic thiols is a commonly used process in organic chemistry with many applications in total synthesis of pharmaceuticals and natural molecules such as leukotrienes,[1] benzothiazepines,[2] and covalent immobilization of functionalized monolayers.[3] This reaction arguably constitutes one of the best routes to the synthesis of $\beta$-hydroxy sulfides. For this, many synthetic procedures have been offered so far based on the thiolysis of epoxides by using various methods that employ aqueous media,[4,5] heterogeneous conditions,[6,7] organocatalysts,[8] and Lewis acids.[9,10] However, several of these methods still involve the use of solvents, additives, or reagents that are not safe from the environmental points of view.

Due to increasing global environmental safety regulations, development of benign chemical processes using conditions with fewer hazards and disposal requirements is of prime importance to synthetic organic chemists nowadays. In this regard, numerous reactions have been conducted cleanly and efficiently in recent decades under aqueous conditions,[11] since water is an abundant, inexpensive, and environmentally safe medium. In another front, many organic transformations

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are performed using ultrasonic irradiation,[12] an activation source that usually lowers the consumption of energy. The advantages associated with these two approaches have led to dramatic upraise in the number of green processes carried out under aqueous and/or ultrasonic conditions, and in the majority of the cases, have caused remarkable rate and selectivity enhancements.[13,14] In the framework of our program to develop environmentally friendly processes [15,16] and in continuation of our studies on ring opening of epoxides,[17,18] we would like to herein report an efficient protocol for thiolysis of epoxides under aqueous/ultrasound conditions using no extra additive or catalyst. Search in the literature shows a precedent for the synthesis of $\beta$-hydroxy sulfides under ultrasonic irradiation.[19] However, this work was performed by using disulfides as the thiol source, sodium hydroxymethanesulfinate as the reductive agent, and DMSO as the solvent. The present work is different in a way that involves direct use of thiols for the ring opening of the epoxides without using any additives or catalysts. In addition, reactions go to completion in shorter time periods and under more benign conditions.

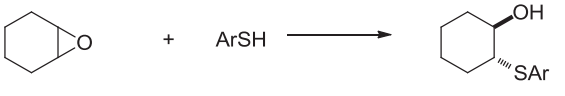
## 2. Results and discussion

We first optimized the conditions by subjecting cyclohexene oxide **1a** to react with thiophenol **2a** in aqueous media, as illustrated in Table 1. Sonication of a 1:1 mixture of the two reactants led to the formation of 93% **3aa** after 15 min irradiation (Entry 1). Use of both water and ultrasound waves were shown to be crucial for the thiolysis to proceed efficiently. This was illustrated by two test reactions in the absence of either the medium or the irradiation. Therefore, in these two tests much lower yields of **3aa** was obtained after several hours indicating the combined promoting effects of water and the energy source (Entries 2–3).

The amount of water was also optimized and it was found that the best ratio of epoxide/water is 3.0 mmol/2.0 mL. $^1$H NMR and GC analyzes of the crude mixture showed sole formation of the *trans*-$\beta$-hydroxy sulfide **3aa** as the only product of the reaction under the optimum conditions. This indicated that the nucleophilic attack of the thiol occurred stereoselectively from the opposite side of the epoxide ring.

Next, the optimized conditions were applied to the reactions of cyclohexene oxide with other thiols. As a result, addition of **2b**–**e** to **1a** proceeded equally well and gave good to high yields of the respective products within 10–20 min (Entries 4–7). To further demonstrate the generality of the method, we then evaluated the reactions of other epoxides with thiols **2a–e** (and furan-2-ylmethanethiol, **2f**) under the optimized conditions (Table 2). Use of epoxides **1b**–**1f** gave the

Table 1. Aqueous thiolysis of **1a** under ultrasonic irradiation.

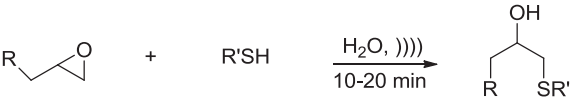



**1a** **2** **3**

| Entry | Thiol | Product | Conditions | Yield (%)[a] | Time (min) |
|---|---|---|---|---|---|
| 1 | **2a**, PhSH | **3aa** | $H_2O$, )))) | 93 | 15 |
| 2 | **2a** | **3aa** | $H_2O$ | <10 | 120 |
| 3 | **2a** | **3aa** | )))) | <10 | 45 |
| 4 | **2b**, 4-$ClC_6H_4SH$ | **3ab** | $H_2O$, )))) | 76 | 20 |
| 5 | **2c**, 4-$MeC_6H_4SH$ | **3ac** | $H_2O$, )))) | 93 | 15 |
| 6 | **2d**, 2-naphthySH | **3ad** | $H_2O$, )))) | 82 | 10 |
| 7 | **2e**, $C_6H_5CH_2SH$ | **3ae** | $H_2O$, )))) | 87 | 15 |

[a]Isolated yield.

Table 2. Thiolysis of various epoxides.



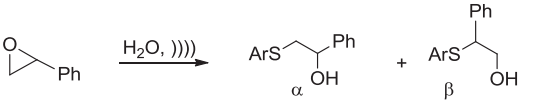

**1b**; R = PhO **2** **3**
**1c**; R = *i*-PrO
**1d**; R = *n*-BuO
**1e**; R = $CH_2$=$CHCH_2O$
**1f**; R = $CH_3$

| Entry | Reactants | R / R′ | Product | Yield (%)[a] | Time (min) |
|---|---|---|---|---|---|
| 1 | **1b** + **2a** | PhO / $C_6H_5$ | **3ba** | 99 | 15 |
| 2 | **1b** + **2b** | PhO / 4-$ClC_6H_4$ | **3bb** | 82 | 20 |
| 3 | **1b** + **2c** | PhO / 4-$MeC_6H_4$ | **3bc** | 94 | 15 |
| 4 | **1b** + **2d** | PhO / 2-naphthyl | **3bd** | 82 | 10 |
| 5 | **1b** + **2e** | PhO / $C_6H_5CH_2$ | **3be** | 90 | 15 |
| 6 | **1b** + **2f** | PhO / 2-furyl-$CH_2$ | **3bf** | 74 | 10 |
| 7 | **1c** + **2a** | *i*-PrO / $C_6H_5$ | **3ca** | 97 | 15 |
| 8 | **1c** + **2b** | *i*-PrO / 4-$ClC_6H_4$ | **3cb** | 80 | 20 |
| 9 | **1c** + **2c** | *i*-PrO / 4-$MeC_6H_4$ | **3cc** | 94 | 15 |
| 10 | **1c** + **2e** | *i*-PrO / $C_6H_5CH_2$ | **3ce** | 94 | 15 |
| 11 | **1c** + **2f** | *i*-PrO / 2-furyl-$CH_2$ | **3cf** | 79 | 10 |
| 12 | **1d** + **2a** | *n*-BuO / $C_6H_5$ | **3da** | 86 | 15 |
| 13 | **1d** + **2b** | *n*-BuO / 4-$ClC_6H_4$ | **3db** | 82 | 20 |
| 14 | **1d** + **2c** | *n*-BuO / 4-$MeC_6H_4$ | **3dc** | 88 | 15 |
| 15 | **1e** + **2a** | $CH_2$=$CHCH_2O$ / $C_6H_5$ | **3ea** | 96 | 15 |
| 16 | **1e** + **2b** | $CH_2$=$CHCH_2O$ / 4-$ClC_6H_4$ | **3eb** | 75 | 20 |
| 17 | **1e** + **2c** | $CH_2$=$CHCH_2O$ / 4-$MeC_6H_4$ | **3ec** | 97 | 15 |
| 18 | **1e** + **2e** | $CH_2$=$CHCH_2O$ / $C_6H_5CH_2$ | **3ee** | 97 | 15 |
| 19 | **1e** + **2f** | $CH_2$=$CHCH_2O$ / 2-furyl-$CH_2$ | **3ef** | 78 | 10 |
| 20 | **1f** + **2a** | $CH_3$ / $C_6H_5$ | **3fa** | 82 | 15 |
| 21 | **1f** + **2b** | $CH_3$ / 4-$ClC_6H_4$ | **3fb** | 80 | 15 |
| 22 | **1f** + **2c** | $CH_3$ / 4-$MeC_6H_4$ | **3fc** | 79 | 15 |

[a]Isolated yield.

respective products within 10–20 min irradiation. In each case, the structure of the major products was determined based on their physical and spectroscopic data and was verified by comparison with those reported in the literature. This comparison supported the regioselectivity of the process in favor of the attack of the thiol on the less hindered carbon of the epoxide.

When styrene oxide **1g** was used under the optimized conditions, thiolysis occurred resulting in the formation of both possible regioisomers. Due to lower steric hindrance, the $\alpha$ products were obtained as the major hydroxy sulfides of the reactions within 10–15 min, as shown in Scheme 1.




**1g**

**3ga** (Ar = $C_6H_5$); 88% (α:β = 4:1)
**3gb** (Ar = 4-$ClC_6H_4$); 85% (α:β = 4:1)
**3gd** (Ar = 2-naphthyl); 87% (α:β = 5:1)

Scheme 1. Thiolysis of styrene oxide under the optimized conditions.

In conclusion, we reported a new method for fast and efficient ring opening of epoxides with different thiols in aqueous media and under ultrasonic conditions. Environmental safety of the process, use of no additive or catalyst, and high selectivity of the reactions are the main advantages of this work. We can make a better conclusion by comparing the outcome of this method with some other procedures reported in the literature, as illustrated in Table 3 for the reaction of **1a** with thiophenol **2a**. It is obvious from the table that the reaction reaches to completion in a relatively shorter time by using the present conditions and without employing an additive or catalyst.

From mechanistic point of view, we propose a favorable hydrogen-bonded association which activates the starting epoxide for a more facile ring opening. This association combined with simultaneous sonication can lower the required activation energy for the process to overcome the transition state barrier under non-thermal conditions. This activation via hydrogen bonding is depicted in Figure 1 and is proposed in similar cases.[24]

## 3. Experimental

All reported yields are based on isolated products. Melting points were determined with a Buchi melting point apparatus and are uncorrected. TLC separations were carried out on silica gel plates with UV indicator; visualization was by UV fluorescence or by staining with iodine vapor. IR spectra were recorded on a FT-IR Bruker Vector 22 infrared spectrophotometer using KBr disks.

Table 3. Comparison of the present work with some other related procedures for the synthesis of **3aa**.

| Conditions | Time | Yield (%) | Reference |
|---|---|---|---|
| ))))), $H_2O$ | 15 min | 93 | This work |
| NaOH, 30°C, $H_2O$ | 3 h | 95 | [4] |
| $InCl_3$, $CH_2Cl_2$ | 4.5 h | 90 | [20] |
| $Zn(ClO_4)_2{\cdot}6H_2O$ | 30 min | 100 | [21] |
| $\beta$-Cyclodextrin, 60°C, $H_2O$ | 15 min | 80 | [22] |
| NaOH, MW (150°C), $H_2O$ | 5 min | 97 | [5] |
| $MgCl_2$, $CH_2Cl_2$ | 20 h | 77 | [10] |
| PhSZnBr, $H_2O$ | 15 min | 73 | [23] |

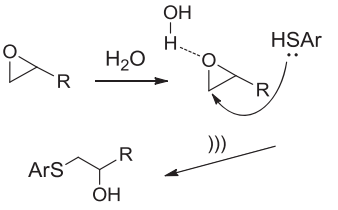



Figure 1. Suggested mechanism.



NMR spectra were recorded on FT-NMR Bruker Ultra Shield™ (500 MHz) as $CDCl_3$ solutions with TMS as internal reference. GC–MS were obtained on a Fisons 8000 Trio instrument at an ionization potential of 70 eV. All chemicals and reagents were purchased from commercial sources. Sonication was performed using a Sartorius Ultrasonic-homogenizer LABSONIC®P 230V/50 Hz. In all reactions the tip of the sonotrode was located in the same position just under the liquid surface in order to obtain optimal sonication and reproducible results.

### 3.1. *General procedure*

A mixture of epoxide (3.0 mmol) and thiol (3.0 mmol) in water (2 mL) in a test tube was sonicated for appropriate length of time (as indicated in Tables 1 and 2) until TLC and GC experiments showed complete disappearance of the starting epoxide. The mixture was extracted by minimum amounts of EtOAc (5 mL) and the organic phase was washed with saturated $NaHCO_3$ and dried over $Na_2SO_4$. Spectral data of the products were obtained and compared with those known in the literature.

### 3.2. *Typical spectral data*

#### 3.2.1. *trans-2-(p-Tolylthio)cyclohexanol (**3ac**)*

$^1$H NMR (500 MHz, $CDCl_3$) $\delta$ 1.24–1.36 (m, 4H), 1.68–1.75 (m, 2H), 2.08–2.16 (m, 2H), 2.37 (s, 3H), 2.71–2.74 (m, 1H), 2.98 (s, 1H), 3.33 (ddd, $J$ = 4.5, 10.0, 10.5 Hz, 1H), 7.15 (d, $J$ = 8 Hz, 2H), 7.40 (d, $J$ = 8 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ 21.5, 24.7, 26.6, 32.9, 34.2, 57.1, 72.2, 128.8, 130.1, 135.0, 136.6.

#### 3.2.2. *1-Phenoxy-3-(p-tolylthio)propan-2-ol (**3bc**)*

$^1$H NMR (500 MHz, $CDCl_3$) $\delta$ 2.37 (s, 3H), 2.85 (br s, 1H), 3.16 (dd, $J$ = 7.0, 14.0 Hz, 1H), 3.25 (dd, $J$ = 5.5, 14.0 Hz, 1H), 4.05–4.14 (m, 3H), 6.91–6.93 (m, 2H), 7.02 (t, $J$ = 7.5 Hz, 1H), 7.14 (d, $J$ = 8.0 Hz, 2H), 7.31 (d, $J$ = 8.0 Hz, 2H), 7.32–7.38 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ 21.5, 38.8, 69.0, 70.5, 115.0, 121.6, 129.9, 130.4, 131.7, 131.7, 137.4, 158.8.

#### 3.2.3. *1-((4-Chlorophenyl)thio)-3-isopropoxypropan-2-ol (**3cb**)*

$^1$H NMR (500 MHz, $CDCl_3$) $\delta$ 1.18 (d, $J$ = 6.0 Hz, 6H), 2.74 (br s, 1H), 3.09 (dd, $J$ = 7.0, 14.0 Hz, 1H), 3.12 (dd, $J$ = 6.0, 14.0 Hz, 1H), 3.48 (dd, $J$ = 6.0, 9.5 Hz, 1H), 3.50 (dd, $J$ = 9.5, 14.0 Hz, 1H), 3.56–3.63 (m, 1H), 3.85–3.87 (m, 1H), 7.28 (d, $J$ = 8.0 Hz, 2H), 7.35 (d, $J$ = 8.0 Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ 22.5, 37.9, 69.6, 70.8, 72.7, 129.5, 131.2, 132.7, 134.8.

### 3.2.4. *1-Isopropoxy-3-(p-tolylthio)propan-2-ol (**3cc**)*

$^1$H NMR (500 MHz, $CDCl_3$) $\delta$ 1.17 (d, $J = 6.0$ Hz, 6H), 2.34 (s, 3H), 2.75 (br s, 1H), 3.02 (dd, $J = 7.0$, 14.0 Hz, 1H), 3.08 (dd, $J = 6.0$, 14.0 Hz, 1H), 3.45 (dd, $J = 6.0$, 10.0 Hz, 1H), 3.54 (dd, $J = 4.0$, 10.0 Hz, 1H), 3.60 (dd, $J = 6.0$, 12.0 Hz, 1H), 3.84–3.88 (m, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 7.32 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ 21.4, 22.4, 38.6, 69.6, 70.8, 72.7, 130.2, 130.8, 132.3, 136.9.

### 3.2.5. *1-Butoxy-3-(p-tolylthio)propan-2-ol (**3dc**)*

$^1$H NMR (500 MHz, $CDCl_3$) $\delta$ 0.95 (t, $J = 7.5$, 3H), 1.37–1.39 (m, 2H), 1.56–1.59 (m, 2H), 2.35 (s, 3H), 2.65 (br s, 1H), 3.03 (dd, $J = 7.0$, 14.0 Hz, 1H), 3.07 (dd, $J = 6.0$, 14.0 Hz, 1H), 3.45–3.48 (m, 3H), 3.53 (dd, $J = 4.0$, 10.0 Hz, 1H), 3.85–3.89 (m, 1H), 7.13 (d, $J = 8.0$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ 14.3, 19.7, 21.4, 32.1, 38.7, 69.4, 71.8, 73.4, 130.2, 130.9, 132.2; 137.0.

### 3.2.6. *1-(Phenylthio)butan-2-ol (**3fa**)*

$^1$H NMR (500 MHz, $CDCl_3$) $\delta$ 1.00 (t, $J = 6.0$ Hz, 3H), 1.60–1.63 (m, 2H), 2.50 (br s, 1H), 2.89 (dd, $J = 8.5$, 13.0 Hz, 1H), 3.20 (dd, $J = 4.0$, 13.0 Hz, 1H), 3.64–3.66 (m, 1H), 7.24–7.26 (m, 1H), 7.30–7.35 (m, 2H), 7.42–7.44 (m, 2H); $^{13}$C NMR (125 MHz, $CDCl_3$) $\delta$ 10.4, 29.4, 42.2, 71.1, 127.0, 129.5, 130.4, 135.8.

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