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Additive-Free Thiolysis of Epoxides in Water: A Green and Efficient Regioselective Pathway to β-Hydroxy Sulfides

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Additive-Free Thiolysis of Epoxides in Water: A Green and Efficient Regioselective Pathway to β-Hydroxy Sulfides

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Abstract: Room-temperature ring opening of various epoxides with aromatic thiols was carried out in water in the absence of any Lewis acid or additive. High yields of β-hydroxy sulfides with excellent regioselectivity were obtained under very environmentally friendly conditions.

Keywords: additive-free, aqueous conditions, epoxides, green chemistry, thiolysis

With increasing global environmental concerns, the design of green processes using alternative media with fewer hazards has gained special attention from synthetic organic chemists in recent years.[1] In this context, many reactions are found to proceed cleanly and efficiently in water,[2] a naturally abundant, environmentally safe, and inexpensive medium. Although the majority of organic compounds have limited solubility in water, there has been a dramatic increase in the number of reactions carried out under aqueous conditions due to rate and selectivity enhancements exhibited by

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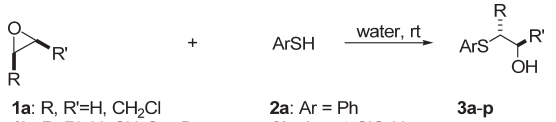
water.[3] Thus, development of convenient procedures under mild aqueous conditions is nowadays of prime importance in synthetic organic chemistry.

Ring opening of epoxides with thiols is an important class of organic transformations and has found many uses in pharmaceutical[4] and natural product chemistry,[5] particularly for the synthesis of leukotrienes.[6] Moreover, the resulting hydroxyl sulfide products are very convenient precursors to access allylic alcohols[7] and α-substituted ketones.[8] The classical approaches for the synthesis of β-hydroxy derivatives of sulfides involve thermal or Lewis acid–mediated nucleophilic ring opening of epoxides with thiols.[9] In many cases, the process is carried out in a halogenated solvent and normally requires a long treatment under refluxing temperatures or environmentally unfriendly conditions.

Recently, several reports were disclosed about thiolysis of epoxides under aqueous conditions. However, in these procedures, use of water has been combined with simultaneous application of extra additives[10] or microwave irradiation.[11] In continuation of our studies on the development of environmentally friendly processes,[12] we herein report an efficient and selective protocol for room-temperature thiolysis of epoxides in pure water, free from any additive or external stimulant (Scheme 1). This is the most inexpensive and environmentally friendly procedure offered so far for ring opening of epoxides with thiols.

We evaluated this chemistry by stirring a 1:1 mixture of 2-(chloromethyl) oxirane **1a** and thiophenol **2a** in pure water at room temperature. Thin-layer chromatograpy (TLC) experiments showed complete disappearance of the starting materials after 8 h. The ^{1}H NMR spectrum of the crude mixture showed the formation of β-hydroxy sulfide **3a** as the sole product of the reaction, indicating that the nucleophilic attack of the thiol occurred exclusively at the less-hindered side of the epoxide. The product was easily extracted with diethyl ether in a relatively pure form (Table 1, entry 1). Use of aqueous conditions proved to be crucial for thiolysis to proceed efficiently. A test reaction in the absence of water gave very low amounts of **3a** after several days, indicating the promoting effect of water.

Epoxide **1a** also reacted with 4-chlorothiophenol (entry 2) and 2-thionaphthol (entry 3) under similar conditions in slightly longer reaction times,



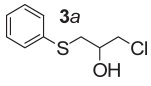
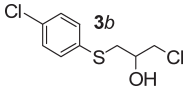
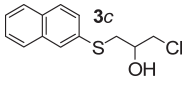
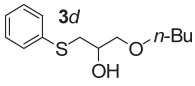
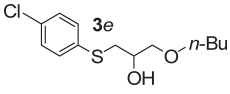
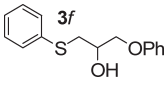
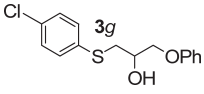
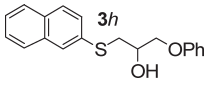
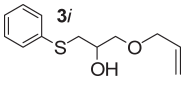
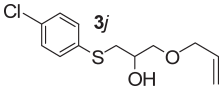
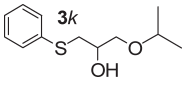
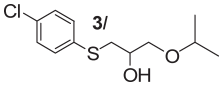
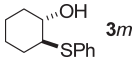
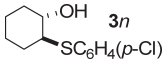
1a: R, R'=H, CH_2Cl
1b: R, R'=H, CH_2O-*n*-Bu
1c: R, R'=H, CH_2OPh
1d: R, R'=H, $CH_2OCH_2CH{=}CH_2$
1e: R, R'=H, CH_2OCHMe_2
1f: R, R'= -$(CH_2)_4$-

2a: Ar = Ph
2b: Ar = 4-ClC_6H_4
2c: Ar = 2-naphthyl

3a-p

Scheme 1.

Table 1. Aqueous thiolysis of 1,2-epoxides

Entry	Epoxide	Thiol	Product	Time (h)	Yield (%)[a]
1	**1a**	**2a**	 **3a**	8	98
2	**1a**	**2b**	 **3b**	10	95
3	**1a**	**2c**	 **3c**	14	91
4	**1b**	**2a**	 **3d**	16	97
5	**1b**	**2b**	 **3e**	18	95
6	**1c**	**2a**	 **3f**	17	93
7	**1c**	**2b**	 **3g**	24	94
8	**1c**	**2c**	 **3h**	28	97
9	**1d**	**2a**	 **3i**	30	96
10	**1d**	**2b**	 **3j**	24	97
11	**1e**	**2a**	 **3k**	30	95
12	**1e**	**2b**	 **3l**	36	94
13	**1f**	**2a**	 **3m**	28	94
14	**1f**	**2b**	 **3n**	36	91

[a]Isolated yields.

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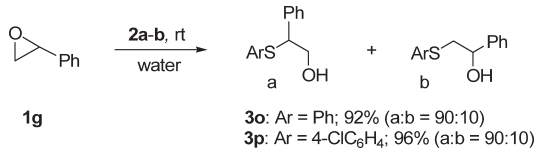

Scheme 2.

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producing 95% and 91% of **3b** and **3c**, respectively. Applicability of this procedure to other substrates was shown by subjecting epoxides **1b–e** to react with thiols. Consequently, **3d–3l** were obtained in excellent yields and within reasonable time periods (entries 4–12). In all instances, exclusive formation of single isomeric products shown in Table 1 was observed, illustrating the high regioselectivity of the ring-opening reactions. Under the cited conditions, reactions of 1,2-epoxycyclohexane with thiols **2a** and **b** gave more than 90% of **3m** and **n** with *trans* stereochemistry (entries 13 and 14).

When styrene oxide was treated under the same conditions, thiolysis occurred with reversed and slightly lower regioselectivity. Therefore, reactions of **1g** with thiols **2a** and **b** led to formation of **3o** and **p** as regioisomeric mixtures, with the major isomers having regiochemistry opposite that observed for cleavage of epoxides **1a–e** (Scheme 2). The reversed selectivity observed for the reactions of **1g** was quantified by analysis of the ^{1}H NMR spectrum of the mixtures and was attributed to favorable electronic effects associated with nucleophilic attack of the thiols on the benzylic carbon that can eventually stabilize the developing positive charge on this carbon.

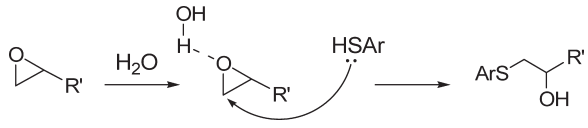
Because of importance of the selectivity issue in synthetic organic chemistry, competitive reactions were carried out to evaluate the relative reactivities of different epoxides toward thiophenol (Table 2). Under aqueous conditions, **1a** and **1c** did not show any significant difference when reacting with thiophenol (entry 1), while any of these two epoxides could selectively react with the same thiol when they compete with **1g** (entries 2 and 3).

The fact that these additive-free reactions proceed only in the presence of water could be rationalized by proposing a favorable hydrogen-bonded

Table 2. Competitive thiolysis of epoxides with thiophenol in water

Entry	Epoxides	Products	Ratio[a]
1	**1a**/**1c**	**3a**/**3f**	60:40
2	**1a**/**1g**	**3a**/**3o**	85:15
3	**1c**/**1g**	**3f**/**3o**	80:20

[a]Ratios determined by GC-MS.



Scheme 3.

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association of the reacting epoxides with water molecules. Such activation through hydrogen bonding,[13] as depicted in the Scheme 3, can stabilize the transition state similar to stabilization in a Lewis acid–mediated epoxide ring opening. This hypothesis can also be supported by a closer look at the reactions of styrene oxide (Scheme 2), where **1g** reacts with a regioselectivity similar to that observed in Lewis acid–catalyzed thiolysis[9] as opposed to thermal or base-promoted ring openings,[10e,11] which proceed with no significant regioselectivity.

In summary, the room-temperature ring opening of various epoxides with aromatic thiols in water was carried out within a few hours in the absence of any Lewis acid or additive. Reactions proceeded efficiently without the application of harsh conditions and tedious workup procedures. High yields of the products, excellent regioselectivity of the ring openings, and more important, the environmental safety of the process are all advantages of this methodology, which make it an attractive addition to the existing literature.

EXPERIMENTAL

All reported yields are isolated yields. IR spectra were recorded on a FT-IR Bruker Vector 22 infrared spectrophotometer. NMR spectra were recorded on a FT-NMR Bruker Ultra Shield^{TM} (500 MHz) as $CDCl_3$ solutions with TMS as internal reference. Gas chromatography-mass spectra (GC-MS) spectra were obtained on Fisons 8000 Trio instrument at an ionization potential of 70 eV.

Typical Procedure

A mixture of 3.0 mmol of epoxide and 3.0 mmol of thiophenol in distilled water (2 mL) was stirred at room temperature for an appropriate length of time until TLC showed completion of the reaction. The mixture was extracted with 10 mL of diethyl ether. The ethereal phase was washed with a saturated solution of $NaHCO_3$ and filtered through a short Na_2SO_4 column. The solvent was removed under reduced pressure, and the product was purified using bulb-to-bulb distillation or column chromatography over silica gel, if necessary. The NMR, IR, and GC-MS spectra of the products were obtained, and they matched perfectly those existing in the

literature.[9a,9k,12a] Elemental analysis for **3e** was performed using a Thermo Finnigan Flash EA 1112 instrument.

Selected Spectral Data

1-Butoxy-3-phenylthiopropan-2-ol (3d).[9j] ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.95 (t, $J = 7.5$, 3H), 1.35–1.42 (m, 2H), 1.55–1.60 (m, 2H), 2.82 (br s, 1H), 3.03 (dd, $J = 7$, 14 Hz, 1H), 3.11 (dd, $J = 6$, 14 Hz, 1H), 3.43–3.54 (m, 4H), 3.87–3.94 (m, 1H), 7.21–7.42 (m, 5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.3, 19.7, 32.1, 37.8, 69.4, 71.7, 73.3, 126.6, 129.4, 129.9, 136.2; MS: m/z, 240 [M^+], 222, 135, 113, 57; IR (KBr): $\tilde{\nu}$ 3442, 1480, 1119 cm^{-1}.

1-Butoxy-3-(4-chlorophenylthio)propan-2-ol (3e). ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.95 (t, $J = 7.5$, 3H), 1.35–1.43 (m, 2H), 1.55–1.61 (m, 2H), 2.82 (br s, 1H), 3.04 (dd, $J = 7$, 14 Hz, 1H), 3.11 (dd, $J = 6$, 14 Hz, 1H), 3.44–3.56 (m, 4H), 3.87–3.94 (m, 1H), 7.28 (d, $J = 8.5$ Hz, 2H), 7.34 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.3, 19.7, 32.1, 38.1, 69.4, 71.8, 73.3, 129.5, 131.3, 132.8, 134.8; MS: m/z, 274 [M^+], 257, 183, 113; IR (KBr): $\tilde{\nu}$ 3433, 1462, 1109 cm^{-1}. Calcd. for $C_{13}H_{19}ClO_2S$: C, 56.82; H, 6.97. Found: C, 56.88; H, 6.82.

1-(Naphthalen-2-ylthio)-3-phenoxypropan-2-ol (3h).[12b] ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.79 (br s, 1H), d 3.31 (dd, $J = 7$, 14 Hz, 1H), 3.41 (dd, $J = 5.5$, 14 Hz, 1H), 4.10–4.14 (m, 2H), 4.15–4.21 (m, 1H), 6.93 (d, $J = 7.5$ Hz, 2H), 7.00 (t, $J = 7.5$ Hz, 1H), 7.28–7.32 (m, 2H), 7.47–7.53 (m, 3H), 7.75–7.84 (m, 3H), 7.89 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 38.2, 69.0, 70.3, 114.9, 115.0, 121.8, 129.7, 129.8, 130.0, 130.1, 131.5, 133.1, 134.2, 158.7; MS: m/z, 310 [M^+], 217, 173, 160, 115; IR (KBr): $\tilde{\nu}$ 3437, 1595, 1087, 745 cm^{-1}.

1-(4-Chlorophenylthio)-3-isopropoxypropan-2-ol (3l).[12b] ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.18 (d, $J = 6.5$ Hz, 6H), 2.70 (br s, 1H), 3.05 (dd, $J = 7$, 14 Hz, 1H), 3.11 (dd, $J = 6$, 14 Hz, 1H), 3.48 (dd, $J = 6$, 9.5 Hz, 1H), 3.55 (dd, $J = 9.5$, 14 Hz, 1H), 3.60–3.65 (m, 1H), 3.85–3.90 (m, 1H), 7.28 (d, $J = 8.5$ Hz, 2H), 7.35 (d, $J = 8.5$ Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): 22.4, 37.9, 69.5, 70.6, 72.8, 129.5, 131.2, 132.7, 134.8; MS: m/z, 260 [M^+], 157, 143, 99, 43. IR (neat): $\tilde{\nu}$ 3443, 1575, 1093, 747 cm^{-1}.

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