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Tandem base-free synthesis of β-hydroxy sulphides under ultrasound irradiation

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Abstract. Rongalite® promotes cleavage of diaryl disulphides generating the corresponding thiolate species *in situ* which then undergo facile ring-opening of epoxides in a regioselective manner under ultrasound irradiation, affording β-hydroxy sulphides in good to excellent yields. The important features of this methodology are base-free, odourless, high yield, reasonably rapid reaction rate, simple workup, high regioselectivity, cost-effective and no requirement of transition metal catalysts. It is noteworthy that ring-opening reaction of 1,2-diphenyldiselane with 2-(phenoxymethyl)oxirane are also conducted smoothly to afford β-hydroxy selenide in excellent yield under the standard conditions.

Keywords. Ultrasound irradiation; Rongalite®; β-hydroxy sulphides; epoxides; ring-opening reaction.

1. Introduction

β-Hydroxy sulphides possess unique physical properties, which have become increasingly important in medicinal chemistry and organic synthesis for the preparation of building blocks and target molecules.[1] On the other hand, β-hydroxy sulphides are excellent ligands for transition-metal-based asymmetric catalysis.[2] As a consequence, development of new methods for the synthesis of β-hydroxy selenides has received much attention. Classical methods for the synthesis of β-hydroxy sulphides involve the ring opening of an epoxide by an excess of thiols, which inevitably gives unpleasant odour, either catalysed by Lewis acid,[3] PBu_3[4] or under microwave irradiation conditions.[5] Recently, we have studied the ring-opening reaction of epoxides in ionic liquids without any catalyst[6] or with gallium(III) triflate as a catalyst.[7]

Recent findings concerning the ring-open reaction of epoxides with *in situ*-generated thiolate species provide an alternative to the century-old reaction of epoxides with odour thiols. Disulphide bond cleavage could lead to interesting products by the reaction of the resulting nucleophilic sulphur[8] species to a variety of organic substrates. Thus, the method has been developed in recent years for the synthesis of β-hydroxy sulfides by the reaction of epoxides with disulphides using different promoting agents. These promoting agents include tetrathiomolybdate,[9] sulfite/base in DMF,[10] $NaBH_4$/amberlite IRA 400,[11] ytterbium(III) chalcogenolate complexes[12] InI–$InCl_3$,[13] Zn–$Bi(OTf)_3$ or Zn–$Bi(TFA)_3$.[14] However, these methods usually suffer from one or more limitations such as the use of unpleasant odour substrates,[3–7] expensive, toxic or metallic catalysts,[10,12–14] long reaction times,[10–13] unsatisfactory yields[12,13] as well as elevated temperature.[11,13,14] Therefore, developing versatile approaches to synthesize β-hydroxy sulphides selectively still remains a highly desired goal in organic synthesis.

Ultrasound has increasingly been used in organic synthesis in the last three decades. A large number of organic reactions can be carried out in higher yields, shorter reaction time and milder conditions under ultrasound irradiation.[15]

Rongalite® (sodium hydroxymethanesulfinate or sodium formaldehyde sulfoxylate) is commercially available material used in the textile industry as a decolourizing agent, and it has also been used in organic synthesis.[16] Very recently, we have reported that Rongalite® promoted cleavage of diaryl disulphides generating the thiolate species *in situ* which then undergo facile ring-opening of epoxides,[17] thia-Michael addition,[18] and acylation.[19] As a continuation of our research in this area, we report here a tandem base-free synthesis

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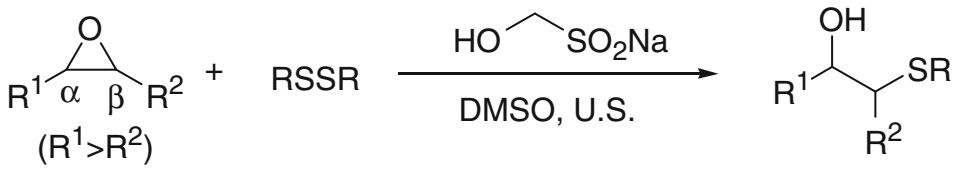


Scheme 1. Synthesis of β-hydroxy sulphides.

of β-hydroxy sulphides from the reaction of epoxides with diaryl disulphides under ultrasound irradiation in the presence of Rongalite® (scheme 1).

2. Experimental

All reagents were purchased and used without further purification. Melting points were recorded on Digital Melting Point Apparatus WRS-1B and uncorrected. IR spectra were recorded on an AVATAR 370 FI-Infrared Spectrophotometer. NMR spectroscopy was performed on a Bruker-300 spectrometer or Bruker-500 spectrometer using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as an internal standard at room temperature. Mass spectrometric analysis was performed on GC–MS analysis (SHIMADZU GCMS-QP2010). Elemental analysis was determined on a Carlo-Erba 1108 instrument. Ultrasonication was performed in a KQ-300VDE ultrasound cleaner with a frequency of 45, 80 and 100 kHz and an output power 300 W. The reaction flask was located in the water bath of the ultrasonic cleaner, where the surface of reactants is slightly lower than the level of the water. The reaction temperature was controlled at 22–25°C by addition or removal of water from ultrasonic bath.

2.1 *General procedure for the synthesis of β-hydroxy sulphides*

A mixture of epoxides **1** (0.5 mmol), disulphides **2** (0.2 mmol) (0.2 mmol), Rongalite® (3 equiv, 0.6 mmol) and DMSO (2 mL) was irradiated under ultrasound in an open vessel at room temperature (22–25°C) for the appropriate time. After completion of the reaction as indicated by TLC, ethyl acetate (10 mL) was then added to the mixture. The mixture was washed with brine. The organic layer was separated and dried with sodium sulphate, filtered and concentrated. Further purification was achieved by silica gel chromatography using ethyl acetate/cyclohexane as eluent to afford pure product.

2.1a *1-(2-Aminophenylthio)-3-phenoxypropan-2-ol (**3e**)*: White solid, m.p. 65–68 °C; IR (KBr): 3405, 3333, 3055, 2928, 1660, 1593, 1514, 1454 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ ppm 7.42–7.39 (m, 1H), 7.27–7.22 (m, 2H), 7.11 (t, $J = 0.8$ Hz, 1H), 6.94–6.84 (m, 3 H), 6.71–6.69 (m, 2H), 3.99–3.93(s, 4H), 3.07–3.01 (m, 1H), 2.94–2.87 (m, 1H), ^{13}C NMR (125 MHz, $CDCl_3$)δ ppm 158.4, 148.2, 136.2, 130.1, 129.4, 121.1, 119.0, 117.1, 115.3, 114.5, 70.3, 68.8, 38.7; MS (EI, 70 eV) m/z (%): 275 (M^+, 40), 125 (100). Anal. Calcd. for $C_{15}H_{17}NO_2S$: C, 65.43; H, 6.22; Found: C, 65.49; H, 6.29.

2.1b *1-Phenoxy-3-(thiophen-2-ylthio)propan-2-ol (**3f**)*: Oil, ^1H NMR (500 MHz, $CDCl_3$)δ ppm 7.37–7.17 (m, 4H), 6.98–6.88 (m, 4H), 4.11–4.00 (m, 3H), 3 .09 (dd, $J = 13.5$ and 5.0 Hz, 1H), 3 .01 (dd, $J = 13.5$ and 7.5 Hz, 1H), 2.76 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$)δ ppm 158.4, 134.2,133.3, 129.8, 129.5, 127.7, 121.3, 114.6, 70.1, 68.6, 42.3; MS (ESI) m/z (%): 267 ($[M+1]^+$, 100). Anal. Calcd. for $C_{13}H_{14}O_2S_2$: C, 58.62; H, 5.30; Found: C,58.77, H, 5.42.

2.1c *1-Phenoxy-3-(pyridin-2-ylthio)propan-2-ol (**3g**)*: Oil, ^1H NMR (500 MHz, $CDCl_3$)δ ppm 8.34–8.33 (m, 1H), 7.50–7.47 (m, 1H), 7.30–7.24 (m, 4H), 7.03–7.00 (m, 1H), 6.95–6.92 (m, 3H); 4.35–4.31 (m, 1H), 4.09 (dd, $J = 9.0$ and 5.0 Hz, 1H), 4.02 (dd, $J = 9.0$ and 7.5 Hz, 1H), 3.55–3.49 (m, 1H), 3.39 (dd, $J = 14.5$ and 6.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$)δ ppm 158.9, 158.5, 148.6, 136.5, 129.3, 125.2, 122.8, 120.8, 120.0, 114.5, 70.0, 35.4; MS (ESI) m/z (%): 262 ($[M+1]^+$, 100). Anal. Calcd. for $C_{14}H_{15}NO_2S$: C, 64.34; H, 5.79; Found: C, 64.22, H, 5.88.

2.1d *1-(p-Tolylthio)octan-2-ol (**3i**)*: Oil, IR (KBr): 3287, 3023, 2983, 2884, 2835, 1725, 1560, 1472, 1431 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ ppm 7.31–7.26 (m, 2H), 7.10 (d, $J = 7.8$ Hz, 2H), 3.62 (s, 1 H), 3.10 (dd, $J = 13.8$ Hz and 3.1 Hz, 1H), 2.82–2.75 (m, 1H), 2.55 (s, 1H), 2.32 (s, 3H), 1.49–1.26 (m, 10H), 0.87 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$)δ ppm 136.8, 131.4, 130.9, 129.8, 69.2, 43.0, 36.0, 31.7, 29.2, 25.6, 22.5, 21.0, 14.0; MS (EI, 70 eV) m/z (%): 252 (M^+, 34), 138 (100). Anal. Calcd. for $C_{15}H_{24}OS$: C, 71.37; H, 9.58; Found: C, 71.45, H, 9.62.

2.1e *2-(4-Fluorophenylthio)-1-phenylethanol* (**3o**): Oil; IR (KBr): 3433, 3032, 2922, 1594, 1532, 1490, 1441 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ ppm 7.44–7.31 (m, 7H), 7.05–6.99 (m, 2H), 4.69 (dd, J = 9.2 and 3.7 Hz, 1H), 3.25 (dd, J = 13.7 and 3.7 Hz, 1H), 3.11–3.03 (m, 1H), 2.89 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$)δ ppm 163.8, 160.5, 142.0, 133.2, 133.1, 129.9, 129.9, 129.8, 128.5, 128.0, 125.8, 116.3, 116.1, 71.7, 45.1; MS (EI, 70 eV) m/z (%): 248 (M^+, 12), 142 (100). Anal. Calcd. for $C_{14}H_{13}FOS$: C, 67.72; H, 5.28; Found: C, 67.66; H, 5.33.

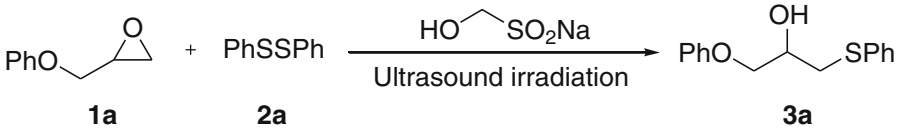
2.1f *1-Chloro-3-(p-tolylthio)propan-2-ol* (**3r**): Oil; ^1H NMR (300 MHz, $CDCl_3$)δ ppm 7.28 (d, J = 8.2 Hz, 2H), 7.08 (d, J = 8.0 Hz, 2 H), 3.88–3.83 (m, 1 H), 3.66–3.60 (m, 2 H), 3.12–2.95 (m, 2 H), 2.72 (d, J = 4.9 Hz, 1 H), 2.29 (s, 3 H); ^{13}C NMR (75 MHz, CDCl3) δ: 137.2, 130.9, 130.7, 130.0, 69.4, 47.9, 39.0, 21.0; IR (KBr, cm^{-1}): 3226, 3024, 2980, 2887, 1600, 1529, 1466, 1396; MS (EI, 70 eV) m/z (%): 218 ([M+2]$^+$, 19), 216 (M^+, 55), 137(100). $[\alpha]_D^{20}$ +26.8 (c 0.99 in $CHCl_3$), R; 98% ee [t_R(S) 39.50 min and t_R(R) 41.63 min].

3. Results and discussion

Initially, the efficacy of various solvents was investigated in the model reaction using 2-(phenoxymethyl)oxirane (**1a**) with 1,2-diphenyldisulphide (**2a**) under ultrasound irradiation at room temperature and the results are summarized in table 1. Initially, the effect of solvents was tested. Among the solvents screened (toluene, 2-methyl-tetrahydrofuran (2-MeTHF), CH_2Cl_2, C_2H_5OH, THF, CH_3CN, DMF, and 1,4-dioxaneoluene), the corresponding β-addition product 1-phenoxy-3-(phenylthio) propan-2-ol (**3a**) was obtained in low yield. When the reaction was performed in DMSO, **1a** undergoes the cleavage with **2a** to produce only β-addition product **3a** in 99% yield under ultrasound irradiation (table 1, entries 1–9).

We also have observed the effect of frequency of ultrasound irradiation on the model reaction. The reaction rate was compared at 45, 80 and 100 kHz having the same output power of 300 W. In the presence of ultrasound the yield was 99% only after 20 min for 80 kHz (table 1, entry 9). The yield decreased drastically without ultrasound irradiation (table 1, entries 12–13). Experiments performed with variable frequency (45 and 100 kHz) showed the same trend. It was indicated from table 1 that that there was remarkable ultrasonic effect on this reaction and there was an optimum frequency of 80 kHz for effective ring-opening reaction. The reason may be the phenomenon of cavitation produced by ultrasound. Because the super-high pressure and temperature generated by the collapse of the acoustic cavitations can never be gained in classical heating, the thermal effects of ultrasonic waves observably accelerate the reaction rate and enhance the yield.

Table 1. The synthesis of **3a** under different conditions[a].

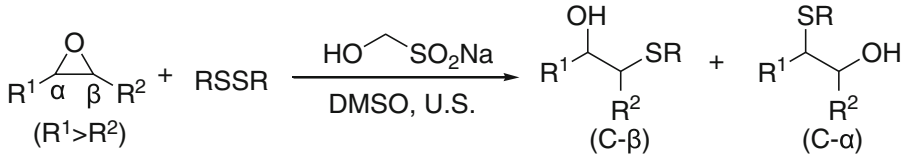


1a **2a** **3a**

Entry	Solvent	Ultrasound (kHz)	Time (min)	Yield (%)[b]
1	toluene	80	60	trace
2	2-Me-THF	80	60	14
3	CH_2Cl_2	80	60	10
4	C_2H_5OH	80	60	trace
5	THF	80	60	11
6	CH_3CN	80	60	trace
7	DMF	80	40	43
8	1,4-dioxane	80	60	15
9	DMSO	80	20	99
10	DMSO	45	20	98
11	DMSO	100	20	99
12	DMSO	without	20	trace
13	DMSO	without	300	16

[a]Reaction conditions: **1a** (0.5 mmol), **2a** (0.2 mmol), Rongalite® (0.6 mmol), under ultrasound irradiation at room temperature.
[b]Isolated yields.

Table 2. Ultrasound-assisted synthesis of β-hydroxy sulphides[a].



1 **2** **3** **4**

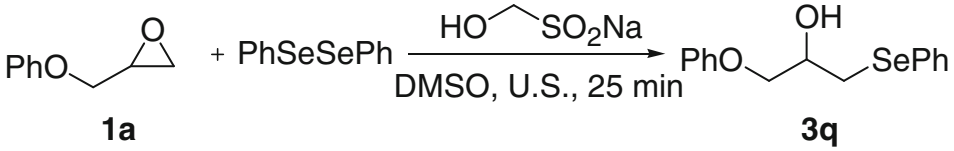
Entry	R^1	R^2	R	Product	Time (min)	Yield (%)[b]
1	$PhOCH_2$	H	Ph	**3a**	20	99
2	$PhOCH_2$	H	p-(Cl)C_6H_4	**3b**	20	96
3	$PhOCH_2$	H	p-(CH_3)C_6H_4	**3c**	15	98
4	$PhOCH_2$	H	p-(F)C_6H_4	**3d**	20	95
5	$PhOCH_2$	H	o-(NH_2)C_6H_4	**3e**	25	71
6	$PhOCH_2$	H	2-thienyl	**3f**	20	81
7	$PhOCH_2$	H	2-pyridyl	**3g**	20	94
8	n-$CH_3(CH_2)_5$	H	Ph	**3h**	20	89
9	n-$CH_3(CH_2)_5$	H	p-(CH_3)C_6H_4	**3i**	20	88
10	n-$CH_3(CH_2)_5$	H	p-(Cl)C_6H_4	**3j**	20	86
11	-$(CH_2)_4$-		Ph	**3k**	20	92
12	-$(CH_2)_4$-		p-(CH_3)C_6H_4	**3l**	20	94
13	Ph	H	Ph	**3m**	20	97
14	Ph	H	p-(CH_3)C_6H_4	**3n**	25	82
15	Ph	H	p-(F)C_6H_4	**3o**	20	86
16	Ph	H	o-(NH_2)C_6H_4	**3p**	25	70

[a]All reactions were run with epoxide **1** (0.5 mmol), disulfide **2** (0.2 mmol), and Rongalite® (0.6 mmol), in 2 mL of DMSO for the appropriate time under ultrasound irradiation at room temperature.
[b]Isolated yield.

The critical size and life time of the cavitation bubbles depend on the liquid and the frequency of ultrasound. On account of longer ultrasonic periods, the implosion time and the size of the cavitation bubbles and the mechanical mixing effects in the liquid increase with decreasing frequencies. Lower frequencies are preferred for the ring-opening reaction due to the more intense mechanical effects. Furthermore, the characteristics of the liquid (such as viscosity, temperature and vapour pressure) can affect cavitation as well, but the mechanisms are complicated.

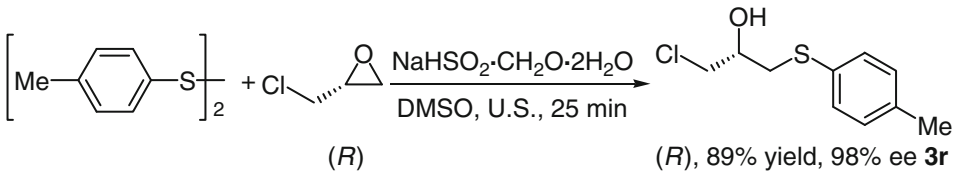
With the optimal reaction conditions in hand, the scope of epoxides was explored and the results are summarized in table 2. As shown in table 2, in the case of alkyl-substituted unsymmetrical epoxides, the reaction proceeds with a remarkable region-selectivity to give only one β-hydroxy sulphides isomer (**3a**–**j**) of the two possible regio-isomers (**3** and **4**) as a result of the exclusive attack of the sulphide anions on the less hindered carbon of the epoxide.

A series of various epoxides and disulphides bearing either electron-donating or electron-withdrawing groups were investigated. The substitution groups on the aromatic ring had no obvious effect on the yield. When less nucleophilic disulphides (R = p-FC_6H_4) (table 2, entries 4 and 15) was used, the reactions could also work efficiently compared to electron-rich disulphides (R = p-MeC_6H_4) (table 2, entries 3, 9, 12 and 14) under the same conditions. On the other hand, the chemoselective reaction in the presence of unprotected reactive functional groups such as -NH_2 also proved to be successful. The corresponding products of **3e** and **3p** were obtained in moderate yields (table 2, entries 5 and 16). Furthermore, we examined the reactivity

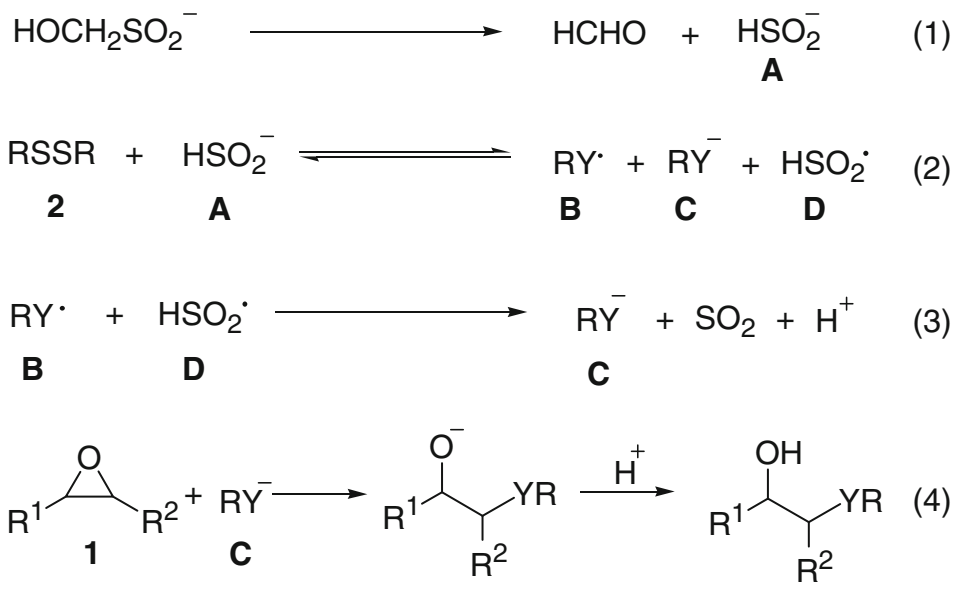


1a **3q**

Scheme 2. Synthesis of 1-phenoxy-3-(phenylselanyl)propan-2-ol (**3q**).



Scheme 3. Synthesis of (*R*)-2-(chloromethyl)oxirane (**3r**).



Scheme 4. A proposed mechanism.

of epoxides with heterocyclic disulphides such as 1,2-di(thiophen-2-yl)disulphide and 1,2-di(pyridin-2-yl)disulphide. Similarly, the corresponding products **3f** and **3g** were obtained with 89% and 94% yields, respectively. On the other hand, the ring-opening of symmetrical epoxide, such as cyclohexene oxide with 1,2-diphenyldisulphide and 1,2-dip-tolyldisulphides to afford *trans*-2-(phenylthio)cyclohexanol and *trans*-2-(*p*-tolylthio)cyclohexanol with high stereoselectivity in 92% and 94% yields, respectively (table 2, entries 11–12). The ^{1}H NMR spectra indicated that only the *trans*-isomer was formed.

As listed in scheme 2, ring-opening reaction of 2-(phenoxymethyl)oxirane with 1,2-diphenyl diselenide smoothly to afford 1-phenoxy-3-(phenylselanyl)propan-2-ol (**3q**) in 98% yields under ultrasound irradiation.

Finally, to extend the scope of this reaction, the thiolysis of chiral epoxide, such as (*R*)-2-(chloromethyl) oxirane (99% ee) was also examined under the optimized conditions. It was found that optically pure epoxide was converted into the corresponding *β*-hydroxy sulphide **3q** in good yield without any racemization or inversion (scheme 3).

A tentative mechanism for the formation of *β*-hydroxy selenides and *β*-hydroxy sulphides was proposed in scheme 4. According to the previous proposed mechanism,[20] Rongalite® can be readily decomposed into HCHO and HSO_2^- anion (**A**). Intermediate (**A**) then reacts with RYYR (**2**, Y = Se or S) to generate two radical intermediates (**B** and **D**) and an anion (**C**). The radical (**B**) can also be converted into the anion (**C**) by reacting with intermediate (**D**). Finally, the nucleophilic attack of the anion (**C**) to the less hindered position of the epoxide (**1**) affords the target product.

4. Conclusions

In conclusion, an efficient and simple method for the odourless tandem synthesis of *β*-hydroxy sulphides with high regioselectivity under ultrasound irradiation has been developed. Compared with the previously reported methods, the present method is bestowed with several advantages, such as odourless, high yield, reasonably rapid reaction rate, simple workup, high regioselectivity, cost-effective and no need for transition metal catalysts. This procedure would be a valuable addition to the current methodologies.

Acknowledgements

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