Rongalite promoted highly regioselective synthesis of *β*-hydroxytellurides

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Ditellurides undergo facile cleavage on treatment with rongalite (sodium hydroxymethanesulfinate) to generate tellurium anions in situ, which undergo ring opening of epoxides in high regioselective manner. A simple, cost-effective method has been developed to prepare *β*-hydroxytellurides with good yields.

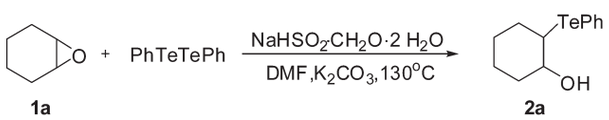
Keywords: *β*-hydroxytellurides, rongalite, expoxide, ditellurides

Tellurium compounds contain an extremely rich chemistry and are widely applied in many areas, such as metallurgy, industrial chemicals and electronic conductors.[1–4] Organotellurium compounds have become attractive synthetic targets because of their chemo-, regio-, and stereoselective reactions, in the presence of a wide variety of functional groups, thus avoiding the need for protecting group chemistry. They have useful biological activities.[5–7] β-Hydroxytellurides are important intermediates comparable to β-hydroxy selenides[8–12] and β-hydroxy sulfides,[13–17] but fewer methods have been developed to synthesise organotellurium compounds. The classical synthesis of β-hydroxytellurides involves the ring opening of an epoxide by a telluride in the presence of NaBH4/EtOH system[18] or from ditellurides with a $NaBH_4$/EtOH system,[19] a $AlH(Bu\text{-}i)_2$/THF system,[20] a MeOH-$NaClO_4$-(Pt) system[21] or a $LiAlH_4$/THF system.[22] Other methods to obtain β-hydroxytellurides involving some of these reagents have their own advantages. However, these procedures possess one or more disadvantages such as expensive metallic reagents,[20] water sensitivity,[20,22] or unusual instruments[21] which might limit the scale of an experiment. Thus, there is still a need to find new methodologies for the synthesis of β-hydroxytellurides.

Based on the fact that rongalite (sodium formaldehyde sulfoxylate, $NaHSO_2{\cdot}CH_2O{\cdot}2H_2O$ as an inexpensive reagent) promotes the formation of thiolate anions or selenolate anions[23], we now report that tellurium anions generated from ditellurides by the action of rongalite, will open epoxide rings and produce β-hydroxytellurides efficiently and regioselectively.

Initial studies were carried out using 7-oxabicyclo[4.1.0] heptane (1a) with 1,2-diphenylditelluride as a model substrate to explore the reaction conditions (Scheme 1). The results were shown in Table 1.

Compared with the ring opening of epoxides with disulfides or diselenides mediated by the rongalite which occurs at room temperature,[24,25] it was found that the reaction did not occur satisfactorily until the reaction temperature was raised to 130 °C (Table 1, entries 1–3). The amount of rongalite affected the yield dramatically (Table 1, entries 4–6). When 4 equiv. of rongalite was added to the reaction system and the reaction time was shortened to 10 min, 82% yield of the product was obtained. Among the solvents that were screened, (acetonitrile, water, THF, Toluene and DMF) (Table 1, entries 7–11), DMF afforded a useful result. It should be especially noted that



1a **2a**

Scheme 1

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Table 1 Effects of rongalite, solvent, and reaction temperature on the ring opening of epoxide with diphenylditelluride

Entry	Rongalite /equiv.	Solvent	Time/min	T/°C	Yield/%[a]
1	3	DMF	5	r. t.	0
2	3	DMF	5	70	0
3	3	DMF	5	100	0
4	3	DMF	5	130	50
5	4	DMF	10	130	82
6	5	DMF	10	130	87
7	6	DMF	5	130	88
8	5	H_2O	5	130	NR
9	5	THF	5	130	Trace
10	5	CH_3CN	5	130	Trace
11	5	Toluene	5	130	0
12	5	DMF	5	130	87[b]

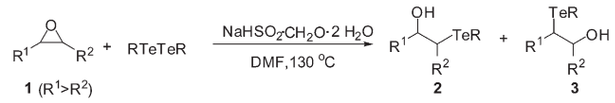
[a]Based on isolated product.
[b]Without K_2CO_3.

the yield was not affected when the reaction was carried out without the addition of K_2CO_3 (Table 1, entries 12). This was different from the ring opening reaction of epoxides with diphenyldisulfides or diphenyldiselenides which did not take place in absence of base.[24,25] We consider that the high temperature promoted the formation of tellurium anions directly without the help of a base.

To establish the generality of the methodology, a variety of epoxides were treated with the diphenylditellurides under the optimised condition to afford *β*-hydroxytellurides. The results are listed in Table 2.

As shown in Table 2, the reaction of epoxides **1a–g** with diphenylditellurides mediated by rongalite resulted in the formation of the corresponding β-hydroxytellurides **2a–m** in good yield. It should be noted that the ring-opening of epoxides produced only one product **2** in our procedure, as the result of the epoxides undergoing facile ring opening in the usual manner at the less hindered carbon centre. The reaction showed excellent regioselectivity. However, if the oxirane had two bulky groups on 2, 3 position separately (Table 2, entries 14, 15), the reaction did not proceed smoothly even if the reaction time was prolonged to 24 hours. This can be explained on the basis of two factors. One is that the bulky groups prevented the attack of nucleophile, the other is that the telluride anion is itself a bulky group, compared to the other chalcogen anion such as the sulfide, selenide anion.

In conclusion, an efficient and simple method for the synthesis of β-hydroxytellurides with high regioselectivity by ring opening of epoxides with diphenylditellurides promoted by inexpensive rongalite in the absence of metal and base has been developed. Compared to the previous reports, our method has advantages such as high regioselectivity, high yields, simple operations and no requirement for expensive metallic reagents.



1 (R^1>R^2) **2** **3**

Table 2 Ring-opening of epoxides with diphenylditellurides in the presence of rongalite

Entry	Epoxide		R	Product	Yield/%[a]
	R^1	R^2			
1	-$(CH_2)_4$-		C_6H_5	**2a**	90
2	-$(CH_2)_4$-		*p*-(F) C_6H_4	**2b**	86
3	$C_6H_5OCH_2$	H	C_6H_5	**2c**	85
4	$C_6H_5OCH_2$	H	*p*-(F) C_6H_4	**2d**	87
5	n-C_6H_{13}	H	C_6H_5	**2e**	80
6	n-C_6H_{13}	H	*p*-(F) C_6H_4	**2f**	80
7	$ClCH_2$	H	C_6H_5	**2g**	85
8	$ClCH_2$	H	*p*-(F) C_6H_4	**2h**	84
9	C_6H_5	H	C_6H_5	**2i**	72
10	C_6H_5	H	*p*-(F) C_6H_4	**2j**	50
11			C_6H_5	**2k**	88
12			*p*-(F) C_6H_4	**2l**	65
13			C_6H_5	**2m**	83
14	C_6H_5	C_2H_5OCO	C_6H_5	**2n**	–
15	C_6H_5CO	C_6H_5	C_6H_5	**2o**	–

[a] Based on an isolated product. The regioisomer 3 was not detected by ^{1}HNMR.

Experimental

Chemicals and solvents were either purchased or purified by standard techniques. IR spectra were recorded on a NICOLET IS10-Infrared Spectrophotometer. NMR spectroscopy was performed on both a Bruker-300 spectrometer operating at 300 MHz (^{1}H NMR), 500 MHz (^{1}H NMR) and 125 MHz (^{13}C NMR). TMS (tetramethylsilane) was used as an internal standard and $CDCl_3$ was used as the solvent. Mass spectrometric analysis was performed on GC–MS analysis (SHIMADZU GCMS-QP2010). HRMS (EI) analysis was performed by the analytical centre at the Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences.

Preparation of **2a–m***; general procedure*

A mixture of epoxides (0.5 mmol), diphenylditellurides (0.2 mmol), and rongalite (1.0 mmol) in DMF (2.0 mL) was stirred at 130 °C for 10 min under air. The reaction mixture was washed with ethyl acetate. After adding enough silica gel to the mixture, the combined washings was evaporated under vacuum directly. The residue was purified by flash column chromatography to afford the desired product **2**.

Trans-2-(phenyltellanyl)cyclohexanol (**2a**):[26] ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.83–7.80 (m, 2H), 7.37–7.32 (m, 1H), 7.24–7.20 (m, 2H), 3.40 (m, 1H), 3.08 (ddd, *J* = 3.9, 12.6, 16.2 Hz 1H), 2.73 (br, 1H, OH), 2.30 (d, *J* = 12.6 Hz, 1H), 2.16–2.12 (m, 1H), 1.78–1.76 (m, 1H), 1.56–1.48 (m, 2H), 1.32–1.19 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 141.0, 129.2, 128.3, 109.6, 73.7, 39.8, 35.2, 34.3, 27.9, 24.8.

Trans-2-(4-fluorophenyltellanyl)cyclohexanol (**2b**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.81–7.76 (m, 2H), 6.92 (t, *J* = 8.7 Hz, 2 H), 3.42–3.34 (m, 1H), 3.05 (ddd, *J* = 3.9, 14.1, 15.9 Hz, 1H), 2.64 (br s, 1H, OH), 2.26 (d, *J* = 12.0 Hz, 1H), 2.17–2.12 (m, 1H), 1.79–1.77 (m, 1H), 1.52–1.46 (m, 2H), 1.33–1.19 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 163.2 (d, $J_{C\text{-}F}$ = 247.4 Hz), 143.2 (d, $J_{C\text{-}F}$ = 7.6 Hz), 116.5 (d, $J_{C\text{-}F}$ = 20.7 Hz) 103.2 (d, *J* = 3.7 Hz), 73.60, 39.4, 35.0, 34.4, 27.7, 24.7; IR(liquid film): 3448 cm^{-1}, 2928 cm^{-1}, 2854 cm^{-1}, 1578 cm^{-1}, 1483 cm^{-1}, 1228 cm^{-1}, 1219 cm^{-1}, 1107 cm^{-1}; MS (EI, 70eV) *m/z* (%): 324 (9), 226 (9), 95 (26), 81 (100), 55 (22). HRMS: Calcd for $C_{12}H_{15}FOTe$: 324.0169. Found: 324.0168.

1-Phenoxy-3-(phenyltellanyl)propan-2-ol (**2c**): ^{1}H NMR (300 MHz,$CDCl_3$) δ: 7.74 (dd, *J* = 1.2, 8.1Hz, 2H), 7.28–7.14 (m, 5H), 6.95 (t, *J* = 7.3 Hz, 1H), 6.84 (dd, *J* = 0.9, 8.7 Hz, 2H), 4.16 (m, 1H),4.03 (dd, *J* = 3.9, 9.3 Hz, 1H), 3.97 (dd, *J* = 6.3, 9.3 Hz, 1H), 3.18 (d, *J* = 6.3 Hz, 2H), 2.76 (br, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 158.2, 138.4, 129.4, 129.3, 127.8, 121.2, 114.5, 111.4, 71.5, 70.1, 13.6. HRMS: Calcd for $C_{15}H_{16}O_2Te$: 358.0213. Found: 358.0212.

1-(4-Fluorophenyltellanyl)-3-phenoxypropan-2-ol (**2d**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.72 (t, *J* = 6.6 Hz, 2H), 7.26 (t, *J* =7.5 Hz, 2H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.93–6.82 (m, 4H), 4.15–4.13 (m, 1H), 4.04–3.94 (m, 2H), 3.14 (d, *J* = 6.1 Hz, 2H), 2.77 (br, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 163.0 (d, $J_{C\text{-}F}$ = 246.7 Hz), 158.2, 140.9 (d, $J_{C\text{-}F}$ = 7.6 Hz), 129.5, 121.2, 116.7 (d, $J_{C\text{-}F}$ = 20.8 Hz) 114.5, 105.0, 71.4, 70.0, 14.2; IR(liquid film): 3415 cm^{-1}, 2851 cm^{-1}, 2159 cm^{-1}, 1585 cm^{-1}, 1364 cm^{-1}, 1079 cm^{-1}, 822 cm^{-1}, 690 cm^{-1}; MS (EI, 70eV) *m/z* (%): 376 (38), 372 (22), 225 (27), 133 (54), 107 (100) 77 (84), 51 (18). HRMS: Calcd for $C_{15}H_{15}FO_2Te$: 376.0118. Found: 376.0119.

1-*(Phenyltellanyl)octan-2-ol* **2e**: ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.75–7.73 (m, 2H), 7.30–7.26 (m, 1H), 7.21–7.18 (m, 2H), 3.73–3.68 (m, 1H), 3.16 (dd, *J* = 4.0, *J* = 12.0 Hz, 1H), 2.98 (dd, *J* = 8.0, 12.0 Hz, 1H), 2.24 (br, 1H, OH), 1.54–1.53 (m, 2H), 1.28–1.25 (m, 7H), 0.88–0.85 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 138.4, 129.2, 127.8, 111.3, 71.1, 37.9, 31.7, 29.2, 25.9, 22.5, 20.3, 14.0; HRMS: Calcd for $C_{14}H_{22}OTe$: 336.0733. Found: 336.0734.

1-(4-Fluorophenyltellanyl)octan-2-ol (**2f**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.75–7.70 (m, 2H), 6.90 (t, *J* = 8.7 Hz, 2H), 3.69 (m, 1H), 3.12 (dd, *J* = 3.9, 9.3 Hz, 1H), 2.96 (dd, *J* = 7.2, 12.0 Hz, 1H), 2.28 (d, *J* = 4.0 Hz, OH), 1.82–1.52 (m, 2H),1.49–1.25 (m, 8H), 0.87 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 162.9 (d, $J_{C\text{-}F}$ = 246.2 Hz), 140.9 (d, $J_{C\text{-}F}$ = 7.5 Hz), 116.6 (d, $J_{C\text{-}F}$ = 20.0 Hz), 104.9 (d, $J_{C\text{-}F}$ = 7.5 Hz), 71.1, 37.9, 31.7, 29.1, 25.8, 22.5, 20.7, 14.0; IR (liquid film): 3428 cm^{-1}, 2927 cm^{-1}, 2855 cm^{-1}, 2026 cm^{-1}, 1702 cm^{-1}, 1580 cm^{-1}, 1484 cm^{-1}, 1085 cm^{-1}; MS (EI, 70eV) *m/z* (%): 354 (18), 240 (21), 109 (14), 95 (34), 69 (100), 55 (46). HRMS: Calcd for $C_{14}H_{21}FOTe$: 354.0639. Found:354.0637.

1-Chloro-3-(phenyltellanyl)propan-2-ol (**2g**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.75 (d, *J* = 7.5Hz,2H 2H), 7.32–7.18 (m, 3H), 4.00–3.96 (m, 1H), 3.69–3.60 (m, 2H), 3.09 (d, *J* = 6.4 Hz, 2H), 2.71 (br, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 138.5, 129.3, 128.0, 111.0, 71.3, 49.4, 14.0; IR(liquid film): 3735 cm^{-1}, 3650 cm^{-1}, 3009 cm^{-1}, 2860 cm^{-1}, 2158 cm^{-1}, 1716 cm^{-1}, 1363 cm^{-1}, 1223 cm^{-1}; MS (EI, 70eV) *m/z* (%): 302 ([M+2]+, 5), 300 (M+, 22), 224 (19), 205 (16), 130 (8), 91 (34), 77 (100), 51 (46). HRMS: Calcd for $C_9H_{11}ClOTe$, 299.9561. Found:299.9563.

1-Chloro-3-(4-fluorophenyltellanyl)propan-2-ol (**2h**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.77–7.72 (m, 2H), 6.92 (t, *J* = 8.8 Hz, 2H), 3.99–3.97 (m, 1H), 3.67–3.64 (m, 2H), 3.07 (d, *J*=6.3 Hz, 2H), 2.68 (d, *J* = 4.0 Hz, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 163.1 (d, $J_{C\text{-}F}$ = 247.1 Hz), 141.2 (d, $J_{C\text{-}F}$ = 7.6 Hz), 116.8 (d, $J_{C\text{-}F}$ = 20.8 Hz) 104.6 (d, *J* = 3.6Hz), 71.2, 49.4, 14.5; IR(liquid film): 3447 cm^{-1}, 2931 cm^{-1}, 1578 cm^{-1}, 1484 cm^{-1}, 1388 cm^{-1}, 1228 cm^{-1}, 1160 cm^{-1}, 1062 cm^{-1}; MS (EI, 70eV) *m/z* (%): 320 ([M+2]+, 12), 318 (M+, 47), 242 (38), 225 (39), 95 (100), 75 (44), 57 (31). HRMS: Calcd for $C_9H_{10}ClFOTe$: 317.9466. Found: 317.9467.

1-Phenyl-2-(phenyltellanyl)ethanol (**2i**)**:**[27] ^{1}H NMR (300 MHz, $CDCl_3$) : δ (ppm) 7.70 (d, *J* = 6.9 Hz 2H), 7.32–7.15 (m, 8H), 4.86 (m, 1H), 3.28–3.25 (m, 2H), 2.76 (br, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 143.5, 138.4, 129.2, 128.5, 127.8, 127.7, 125.6, 111.5, 73.6, 20.7.

1-(4-Fluorophenyl)-2-(phenyltellanyl)ethanol (**2j**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.68–7.63 (m, 2H), 7.32–7.25 (m, 5H), 6.88 (t, *J* = 8.7 Hz 2H), 4.88–4.83 (m, 1H), 3.28–3.23 (m, 2H), 2.65 (br, 1H, OH); ^{13}C NMR (125 MHz, $CDCl_3$) δ: 162.9 (d, $J_{C\text{-}F}$ = 246.2 Hz), 143.4, 140.9 (d, $J_{C\text{-}F}$ = 7.5 Hz), 128.5, 127.9, 125.6, 116.6 (d, $J_{C\text{-}F}$ = 20.0 Hz) 105.0, 73.7, 21.1; IR(liquid film): 3443 cm^{-1}, 2928 cm^{-1}, 2856 cm^{-1}, 2159 cm^{-1}, 1578 cm^{-1}, 1483 cm^{-1}, 1453 cm^{-1}, 821 cm^{-1}; MS (EI, 70eV) *m/z* (%): 346 (23), 240 (30), 225 (21), 104 (71), 103 (100), 77 (96), 51 (42). HRMS: Calcd for $C_{14}H_{13}FOTe$: 346.0013. Found: 346.0011.

*1-(Phenyltellanyl)dec-9-en-2-ol (***2k**): Yellow oil;^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.76 (d, *J* = 7.1 Hz 2H), 7.31–7.17 (m, 3H), 5.87–5.73 (m, 1H), 5.02–4.91 (m, 2H), 3.70 (m, 1H), 3.16 (dd, *J* = 3.9, 12.3 Hz,1H), 2.97 (dd, *J* = 8.1, 12.3 Hz, 1H), 2.20 (br, 1H, OH), 2.02 (q, *J* = 6.9 Hz, 2H), 1.55–1.50 (m, 2H),1.35–1.26 (m, 8H). 13C NMR (125 MHz CDCl3): δ (ppm) 139.0, 138.4, 129.2, 127.7, 114.2, 111.3, 71.1, 37.9, 33.7, 29.3, 28.9, 28.8, 25.9, 20.3; IR(liquid film): 3428 cm^{-1}, 2925 cm^{-1}, 2854 cm^{-1}, 2159 cm^{-1}, 1702 cm^{-1}, 1575 cm^{-1}, 1434 cm^{-1}, 1113 cm^{-1}; MS (EI, 70eV) *m/z* (%): 362 (15), 207 (26), 205 (18), 81 (47), 77 (65), 55 (100). HRMS: Calcd for $C_{16}H_{24}OTe$: 362.0889. Found: 362.0888.

1-(4-Fluorophenyltellanyl)dec-9-en-2-ol (**2l**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.75–7.71 (m, 2H), 6.90 (t, *J* = 8.7 Hz, 2H), 5.87–5.73 (m, 1H), 5.01–4.82 (m, 2H), 3.69 (m, 1H), 3.12 (dd, *J* = 3.9, 12.0 Hz, 1H), 2.95 (dd, *J* = 7.8, 12.0 Hz, 1H),2.16 (br, 1H, OH), 2.02 (q, *J* = 6.9Hz, 2H), 1.54–1.27 (m, 10H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 163.2 (d, $J_{C\text{-}F}$ = 245.0 Hz), 141.1 (d, $J_{C\text{-}F}$ = 7.6 Hz), 139.0, 116.7(d, $J_{C\text{-}F}$ = 20.8 Hz), 114.2, 104.8 (d, *J* = 3.6 Hz), 71.1, 37.9, 33.7, 29.7, 29.3, 29.0, 28.8, 25.9, 20.8; IR(liquid film): 3566 cm^{-1}, 2925 cm^{-1}, 2854 cm^{-1},1715 cm^{-1}, 1575 cm^{-1}, 1484 cm^{-1}, 1363 cm^{-1}, 1223 cm^{-1}; MS (EI, 70eV) *m/z* (%): 380 (26), 240 (9), 225 (28), 95 (90), 55 (100). HRMS: Calcd for $C_{16}H_{23}FOTe$: 380.0795. Found: 380.0796.

1-(Allyloxy)-3-(phenyltellanyl)propan-2-ol (**2m**): Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.75–7.72 (m, 2H), 7.27–7.16 (m, 3H), 5.87–5.81 (m, 1H), 5.28–5.16 (m, 2H), 3.96–3.94 (m, 3H), 3.53 (dd, *J* = 3.6, 9.3 Hz, 1H), 3.44 (dd, *J* = 6.3, 9.3 Hz, 1H), 3.07 (d, *J* = 6.3 Hz, 2H), 2.83 (d, *J* = 2.4 Hz, 1H, OH). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 138.2, 134.2, 129.1, 127.6, 117.3, 111.7, 73.9, 72.1, 70.3, 13.7; IR (liquid film): 3419 cm^{-1}, 2920 cm^{-1}, 2855 cm^{-1}, 1709 cm^{-1}, 1575 cm^{-1}, 1433 cm^{-1}, 1364 cm^{-1}, 1018 cm^{-1}; MS (EI, 70eV) *m/z* (%): 322 (24), 207 (39), 117 (29), 91 (36), 77 (100), 51 (32). HRMS: Calcd for $C_{12}H_{16}O_2Te$: 322.0213. Found: 322.0214.

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