Synthesis of Enantiopure *tert*-Butanesulfinamide from *tert*-Butanesulfinyloxazolidinone

Yong Qin,^{*,†} Canhui Wang,[‡] Zhiyan Huang,[†] Xue Xiao,[†] Yaozhong Jiang^{*,‡}

[†]Department of Chemistry of Medicinal Natural Products, West China School of Pharmacy,

Sichuan University, Chengdu 610041, P. R. China

[‡]Chengdu Institute of Organic Chemistry, Graduate School of Chinese Academy of Sciences,

Chengdu 610041, P. R. China

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General methods.

All commercially available reagents were used without further purification. All solvents were dried and distilled before use: THF was distilled from sodium/benzophenone ketyl; dichloromethane were distilled from calcium hydride; toluene was distilled from sodium. Chromatography was conducted by using 200-300-mesh silica gel. IR spectra were recorded on a FT IR spectrometer. NMR spectra were acquired on a 300 MHz NMR spectrometer. HRMS spectra were obtained by the FAB method. HPLC analyses were conducted by using a Diacel Chiralpak AS column.

General procedure for the preparation of N-alkoxycabonyl-1, 2-diphenylaminoethanols

1.

To a stirred solution of (1R, 2S)-1,2-diphenylaminoethanol (2.13g, 10 mmol) and Na_2CO_3 (2.11g, 20 mmol) in 120 ml of THF and H_2O (3:1) at 0°C was added a solution of TsCl (Boc₂O, CbzCl, or MeOCOCl, 11 mmol) in 5 mL of THF. After being stirred for 3 h, the mixture was poured into 100 mL of H_2O and extracted with dichloromethane to afford crude **1**, which was pure enough for next step in most cases. Further purification could be achieved by recrystallization from ethanol.

1a: yield 93%, mp 229-230 °C; $[\alpha]_{\text{D}}^{25} = -97.0$ (c 0.1, THF); IR: 3465, 3324, 1455, 1405, 1313, 1288, 1252, 1154, 1088, 1056 cm^{-1} ; ^1H NMR (CDCl_3) δ 2.33 (s, 4 H), 4.55 (dd, $J = 4.4, 7.8$ Hz,

1 H), 5.00 (m, 1 H), 5.23 (d, $J = 7.8$ Hz, 1 H), 6.8 (d, $J = 7.4$ Hz, 2 H), 6.93 (d, $J = 7.7$ Hz, 2 H), 7.00-7.26 (m, 8 H), 7.50 (d, $J = 8.2$ Hz, 2 H) ppm.

1b: yield 97%, mp 192-193 °C; $[\alpha]_D^{25} = -57.6$ (c 1, CHCl₃); IR: 3378, 2927, 1683, 1523, 1456, 1369, 1293, 1252, 1172 cm⁻¹; ¹H NMR (CDCl₃) δ 1.26 (s, 9 H), 2.60 (s, br, 1 H), 4.97 (s, br, 1 H), 5.00 (s, br, 1 H), 5.30 (s, br, 1 H), 7.00 (m, 4 H), 7.20 (m, 6 H) ppm.

1c: yield 94%, mp 172-173 °C; $[\alpha]_D^{25} = -67.4$ (c 1, CHCl₃); IR: 3348, 3063, 1690, 1540, 1496, 1453, 1441, 1352, 1292, 1258, 1153, 1060, 1034 cm⁻¹; ¹H NMR (CDCl₃) δ 2.40 (s, br, 1 H), 5.05 (m, 4 H), 5.6 (s, br, 1 H), 6.90-7.12 (m, 4 H), 7.23-7.26 (m, 6 H), 7.30-7.65 (m, 5 H).

1d: yield 99%, mp 175-176 °C; $[\alpha]_D^{25} = -62.1$ (c 1, CHCl₃); IR: 3352, 3032, 2944, 1691, 1541, 1498, 1454, 1352, 1309, 1289, 1260, 1216, 1193, 1080, 1060 cm⁻¹; ¹H NMR (CDCl₃) δ 2.38 (s, br, 1 H), 3.65 (s, 3 H), 5.02 (m, 1 H), 5.08 (m, 1 H), 5.48 (m, 1 H), 7.02 (m, 4 H), 7.25 (m, 6 H) ppm.

Typical procedure for the preparation of 4,5-diphenyl-1,2,3-oxathiazolidine-2-oxides **2**.

Under N₂, to a stirred solution of **1** (1 mmol) and 4-(N, N-dimethylamino)pyridine (3 mmol) in 30 mL of dried CH₂Cl₂ at -45°C was added SOCl₂ (1 mmol). After being stirred for 1 h, the mixture was quenched with H₂O (5 mL) at -45°C and was allowed to warm to room temperature. The aqueous phase was extracted with CH₂Cl₂, the combined organic phases were dried with MgSO₄ and evaporated to afford a crude mixture of **2**-endo and **2**-exo. The

ratio of endo-**2** and exo-**2** (table 2) was determined by ^1H NMR of crude **2** with integration of proton at position-5. Pure endo-**2d** and exo-**2d** were obtained by chromatography (EtOAc/petroleum 1:5). For the inseparable endo and exo mixture of **2b**, endo and exo mixture of **2c**, purification was achieved by recrystallization (after chromatography, EtOAc/petroleum 1:7) to afford pure endo-**2b** (EtOAc/petroleum 1:12) and pure endo-**2c** (EtOAc/petroleum 1:12) respectively.

For endo-**2b**: mp 188-189 °C; $[\alpha]_D^{25} = -277.7$ (c 1, CHCl_3); IR: 2979, 1727, 1700, 1456, 1369, 1324, 1186, 1140 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.49 (s, 9 H), 5.34 (d, $J = 6.7$ Hz, 1 H), 5.95 (d, $J = 6.6$ Hz, 1 H), 7.01 (d, $J = 6.0$ Hz, 2 H), 7.16 (m, 8 H) ppm; ^{13}C NMR (CDCl_3) δ 28.1, 63.7, 84.2, 89.4, 126.8, 126.9, 127.6, 127.9, 127.9, 128.2, 128.5, 133.2, 134.7 ppm; Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{N}_1\text{O}_4\text{S}_1$: C, 63.49; H, 5.89; N, 3.90; S, 8.92. Found: C, 63.22; H, 5.89; N, 3.94; S, 9.22.

For exo-**2b**: ^1H NMR (CDCl_3) δ 1.30 (s, 9 H), 5.34 (d, $J = 6.7$ Hz, 1 H), 6.57 (s, br, 1 H), 6.80 (d, $J = 8.1$ Hz, 2 H), 6.94 (d, $J = 8.0$ Hz, 2 H), 7.10 (m, 6 H) ppm.

For endo-**2c**: mp 127-128 °C; $[\alpha]_D^{25} = -242.6$ (c 1, CHCl_3); IR: 3030, 1702, 1498, 1456, 1394, 1322, 1186, 1153, 1080, 1033, 956, 939 cm^{-1} ; ^1H NMR (CDCl_3) δ 5.22 and 5.30 (AB, $J = 12$ Hz, 2 H), 5.39 (d, $J = 6.6$ Hz, 1 H), 5.98 (d, $J = 6.7$ Hz, 1 H), 6.98 (d, $J = 6.6$ Hz, 1 H), 7.01 (d, $J = 7.8$ Hz, 2 H), 7.14 (m, 9 H), 7.20 (m, 4 H) ppm; ^{13}C NMR (CDCl_3) δ 63.8, 68.7, 89.6, 126.7, 127.0, 127.8, 128.0, 128.3, 128.5, 128.6, 128.6, 132.9, 134.4, 135.0 ppm; Anal. Calcd for $\text{C}_{22}\text{H}_{19}\text{N}_1\text{O}_4\text{S}_1$: C, 67.16; H, 4.87; N, 3.56; S, 8.15. Found: C, 67.11; H, 4.88; N, 3.60; S, 7.94.

For exo-**2c**: ^1H NMR (CDCl_3) δ 5.22 and 5.30 (AB, $J = 12$ Hz, 2 H), 5.39 (d, $J = 6.6$ Hz, 1 H), 6.60 (s, br, 1 H), 6.80 (d, $J = 6.6$ Hz, 2 H), 7.02 (d, $J = 6.6$ Hz, 2 H), 7.10 (m, 7 H), 7.26 (m, 4 H) ppm.

For endo-**2d**: mp 143-144 °C; $[\alpha]_D^{25} = -299.3$ (c 1, CHCl₃); IR: 2958, 1712, 1496, 1437, 1328, 1277, 1187, 1145, 1084 cm⁻¹; ¹H NMR (CDCl₃) δ 3.83 (s, 3 H), 5.34 (d, *J* = 6.7 Hz, 1 H), 5.95 (d, *J* = 6.7 Hz, 1 H), 6.99 (d, *J* = 6.0 Hz, 2 H), 7.12 (m, 8 H) ppm; ¹³C NMR (CDCl₃) δ 54.1, 63.8, 89.5, 126.7, 127.8, 128.0, 128.0, 128.3, 128.7, 132.9, 134.2 ppm; Anal. Calcd for C₁₆H₁₅N₁O₄S₁: C, 60.55; H, 4.76; N, 4.41; S, 10.10. Found: C, 60.65; H, 4.80; N, 4.44; S, 10.32.

For exo-**2d**: mp 165-166 °C; $[\alpha]_D^{25} = -104.6$ (c 0.78, CHCl₃); IR: 2953, 1746, 1495, 1453, 1440, 1326, 1196, 1135, 1063, 1028, 993, 953, 925 cm⁻¹; ¹H NMR (CDCl₃) δ 3.81 (s, br, 3 H); 5.35 (s, br, 1 H), 6.56 (d, *J* = 6.6 Hz, 1 H), 6.77 (s, *J* = 6.3 Hz, 2 H), 6.90 (d, *J* = 7.0 Hz, 2 H), 7.10 (m, 6 H) ppm; ¹³C NMR (CDCl₃) δ 53.9, 64.8, 89.5, 126.7, 128.0, 128.1, 128.2, 128.3, 128.7, 128.8, 131.9, 133.9 ppm; Anal. Calcd for C₁₆H₁₅N₁O₄S₁: C, 60.55; H, 4.76; N, 4.41; S, 10.10. Found: C, 60.71; H, 4.82; N, 4.48; S, 10.34.

Typical procedure for the syntheses of sulfinate **3**, sulfonamide **4** and oxazolidinone **5**.

Under N₂, to a stirred solution of 4, 5-Diphenyl-1,2,3-oxathiazolidine-2-oxides **2** (endo isomer, 1 mmol) in 10 mL of dry THF at -45°C was added *tert*-butylmagnesium chloride (1.5 mmol, 0.75 mL of 2M solution in THF). After addition, the reaction was stirred at -45°C for 2-4h monitored by TLC analysis. Once the starting material was consumed completely, the reaction was quenched with saturated NH₄Cl aqueous solution (1 mL) and allowed to warm to room

temperature. The mixture was diluted with 30 mL of ethyl acetate and washed with brine, and dried with MgSO_4 . Concentration and chromatography gave sulfinate **3**, sulfonamide **4** and oxazolidinone **5** as depicted in table 3.

For sulfinate **3b**: mp 195-196 °C; $[\alpha]_D^{25} = -114.9$ (c 0.5, CHCl_3); IR: 3749, 3423, 1694, 1513, 1457, 1390, 1365, 1294, 1245, 1169, 1125 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.20 (s, 9 H), 1.43 (s, 9 H), 4.96 (d, $J = 7.8$ Hz, 1 H), 5.54 (s, br, 1 H), 6.10 (m, 1 H), 6.92 (m, 4 H), 7.25 (m, 6 H) ppm; ^{13}C NMR (CDCl_3) δ 21.8, 28.4, 58.1, 59.8, 79.7, 84.6, 126.9, 127.4, 127.5, 128.0, 128.2, 128.7, 138.0, 139.0, 155.1 ppm. HRMS-FAB calcd for $\text{C}_{23}\text{H}_{31}\text{N}_1\text{O}_4\text{S}_1$ (M+Na) 440.1873, found 440.1879.

For sulfinamide **4b**: mp 186-187 °C; $[\alpha]_D^{25} = +77$ (c 0.5, CHCl_3); IR: 3297, 2977, 1734, 1457, 1392, 1367, 1280, 1255, 1162, 1064, 977 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.1 (s, 9 H), 1.35 (s, 9 H), 3.70 (s, 1 H), 4.72 (dd, $J = 2.7, 6.6$ Hz, 1 H), 5.82 (d, $J = 6.6$ Hz, 1 H), 7.28 (m, 10 H) ppm; ^{13}C NMR (CDCl_3) δ 22.5, 27.6, 55.7, 62.1, 80.7, 82.6, 127.5, 128.0, 128.2, 128.5, 128.8, 129.4, 136.0, 136.4, 152.8 ppm; HRMS-FAB calcd for $\text{C}_{23}\text{H}_{31}\text{N}_1\text{O}_4\text{S}_1$ (M+Na) 440.1873, found 440.1871.

For sulfinate **3c**: mp 153-154 °C; $[\alpha]_D^{25} = -112.8$ (c 1, CHCl_3); IR: 3401, 2926, 1701, 1519, 1288, 1243, 1219, 1124, 978 cm^{-1} ; ^1H NMR (CDCl_3) δ 1.20 (s, 9 H), 5.06 (AB, $J = 12$ Hz, 2 H), 5.10 (d, $J = 3.6$ Hz, 1 H), 5.6 (s, 1 H), 6.50 (d, $J = 7.5$ Hz, 1 H), 6.95 (m, 4 H), 7.10-7.35 (m, 11 H) ppm; ^{13}C NMR 21.6, 58.1, 60.1, 66.8, 84.7, 126.7, 127.5, 127.6, 127.9, 128.0, 128.3, 128.4, 128.7, 135.8, 136.6, 136.7, 155.7 ppm; Anal. Calcd for $\text{C}_{26}\text{H}_{29}\text{N}_1\text{O}_4\text{S}_1$: C, 69.15; H, 6.47; N, 3.10; S, 7.10. Found: C, 68.86; H, 6.53; N, 3.14; S, 6.85.

For sulfinamide **4c**: mp 168-169 °C; $[\alpha]_D^{25} = +65.4$ (c 0.5, CHCl₃); IR: 3297, 2958, 2926, 1739, 1456, 1386, 1365, 1269, 1064 cm⁻¹; ¹H NMR (CDCl₃) δ 1.08 (s, 9 H), 3.61 (s, 1 H), 4.74 (dd, $J = 2.1, 6.6$ Hz, 1 H), 5.06 (AB, $J = 12$ Hz, 2 H), 5.85 (d, $J = 6.6$ Hz, 1 H), 7.22 (m, 15 H) ppm; ¹³C NMR (CDCl₃) δ 22.4, 55.7, 62.0, 69.8, 82.1, 127.4, 128.1, 128.3, 128.5, 128.6, 129.0, 129.2, 135.0, 135.5, 136.2 ppm; HRMS-FAB calcd for C₂₆H₂₉N₁O₄S₁ (M+Na) 474.1725, found 474.1715.

For sulfinate **3d**: mp 182-183 °C; $[\alpha]_D^{25} = -135.1$ (c 0.6, CHCl₃); IR: 3358, 2955, 1729, 1702, 1530, 1456, 1290, 1251, 1101, 981 cm⁻¹; ¹H NMR (CDCl₃) δ 1.21 (s, 9 H), 3.67 (s, 3 H), 4.99 (dd, $J = 1.6, 6.6$ Hz, 1 H), 5.56 (d, $J = 1.8$ Hz, 1 H), 6.50 (d, $J = 7.0$ Hz, 1 H), 6.9 (m, 4 H), 7.20 (m, 6 H) ppm; ¹³C NMR (CDCl₃) δ 21.8, 52.3, 58.2, 60.0, 85.2, 126.6, 127.5, 127.6, 128.0, 128.3, 128.7, 135.8, 136.8 ppm; Anal. Calcd for C₂₀H₂₅N₁O₄S₁: C, 63.97; H, 6.71; N, 3.73; S, 8.54. Found: C, 63.84; H, 6.64; N, 3.80; S, 8.66.

For sulfinamide **4d**: mp 161-162 °C; $[\alpha]_D^{25} = +79.6$ (c 1, CHCl₃); IR: 3298, 2925, 2854, 1744, 1444, 1272, 1063, 979 cm⁻¹; ¹H NMR (CDCl₃) δ 1.11 (s, 9 H), 3.59 (s, 1 H), 3.68 (s, 3 H), 4.77 (dd, $J = 2.7, 6.6$ Hz, 1 H), 5.83 (d, $J = 6.3$ Hz, 1 H), 7.20-7.34 (m, 10 H) ppm; ¹³C NMR (CDCl₃) δ 22.4, 55.0, 55.7, 61.9, 77.0, 82.1, 127.4, 128.2, 128.3, 128.6, 129.0, 129.1, 135.5, 136.3, 154.9 ppm; HRMS-FAB calcd for C₂₀H₂₅N₁O₄S₁ (M+Na) 398.1395, found 398.1402.

For oxazolidinone **5**: mp 181-182 °C; $[\alpha]_D^{25} = 51.5$ (c 1, CHCl₃); IR: 1746, 1454, 1338, 1289, 1216, 1184, 1130, 1101, 1013 cm⁻¹; ¹H NMR (CDCl₃) δ 1.38 (s, 9 H), 5.22 (d, $J = 6.6$ Hz, 1 H), 5.95 (d, $J = 6.6$ Hz, 1 H), 6.92 (m, 4 H), 7.11 (m, 6 H) ppm; ¹³C NMR (CDCl₃) δ 22.3, 61.1, 64.1, 82.3, 126.2, 126.5, 128.0, 128.1, 128.4, 132.4, 134.8, 156.2 ppm; Anal. Calcd for

C₁₉H₂₁N₁O₃S₁: C, 66.45; H, 6.16; N, 4.08; S, 13.98. Found: C, 64.35; H, 6.36; N, 4.28; S, 14.11.

HRMS-FAB calcd for C₁₉H₂₁N₁O₃S₁ (M+Na) 366.1134, found 366.1140.

Ammonolysis of oxazolidinone **5**.

To a solution of liquid ammonia (20 mL) was added a few crystals of Fe(NO₃)₃·9H₂O, followed by addition of lithium pieces (0.184g, 8 mmol) in a controlled manner while keeping the internal temperature at around -45°C. When all lithium was added and the solution became gray, the mixture was stirred for additional 1 h. The mixture was cooled to -78°C and was added a solution of **5** (0.687g, 2 mmol) in 5 mL of THF. After being stirred for 0.5 h, the mixture was added solid NH₄Cl in small portion. The mixture was warmed to room temperature and the ammonia gas escaped from the reaction was trapped by water. The residue was added 2 mL of H₂O and 20 mL of ethyl acetate, and stirred vigorously. The organic phase was separated and the aqueous phase was washed with EtOAc (2 × 10 mL). The combined organic phases were washed with brine and dried with MgSO₄ and evaporated to dryness. The residue was subjected to chromatography (EtOAc) to afford **7** (0.464g, 91%) and (R)-**6** (0.216g, 89%) with 98.6% ee. A single recrystallization from hexane afforded (R)-**6** with > 99% ee. The absolute stereochemistry and enantiomeric excess of (R)-**6** was established by Chiral HPLC analysis (Diacel Chiralpak AS column, hexane/ethanol 95:5; 1 mL/min, 222 nm, (R)-**6**, *r*_t=9.0 min; (S)-**6**, *r*_t=13.2 min). mp 101-102 °C; ¹H NMR (CDCl₃) δ 1.18 (s, 9 H), 3.82 (s, br, 2 H) ppm.

For compound **7**, mp 221-222 °C; $[\alpha]_{\text{D}}^{25} = -79.2$ (c 1, CHCl₃); IR: 3277, 2924, 1748, 1712, 1455, 1426, 1352, 1235, 1094, 1072 cm⁻¹; ¹H NMR (CDCl₃) δ 5.20 (d, *J* = 8.1 Hz, 1 H), 5.39 (s, br, 1 H), 5.97 (d, *J* = 8.2 Hz, 1 H), 6.97 (m, 4 H), 7.10 (m, 6 H) ppm; ¹³C NMR (CDCl₃) δ 61.4, 82.3, 126.1, 126.9, 127.9, 128.1, 128.3, 134.3, 136.0, 159.5 ppm; Anal. Calcd for C₁₅H₁₃N₁O₃: C, 75.30; H, 5.48; N, 5.85. Found: C, 75.17; H, 5.50; N, 5.91.

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Project Name: Defaults
Reported by User: System

Breeze

SAMPLE INFORMATION

Sample Name:	wch-1	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	6/29/04 11:05:53 AM
Vial:	1	Acq. Method:	100A222
Injection #:	2	Date Processed:	6/29/04 11:24:56 AM
Injection Volume:	20.00 ul	Channel Name:	2487Channel 1
Run Time:	180.00 Minutes	Sample Set Name:

	RT (min)	Area ($\mu V \cdot sec$)	% Area	Height (μV)	% Height
1	9.098	8137503	98.66	444754	99.28
2	13.287	110689	1.34	3241	0.72

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Project Name: Defaults
Reported by User: System

Breeze

SAMPLE INFORMATION

Sample Name: wch-2
Sample Type: Unknown
Vial: 1
Injection #: 3
Injection Volume: 20.00 μ l
Run Time: 180.00 Minutes

Acquired By: System
Date Acquired: 6/29/04 11:26:17 AM
Acq. Method: lwg
Date Processed: 6/29/04 11:43:54 AM
Channel Name: 2487Channel 1
Sample Set Name:

	RT (min)	Area (μ V*sec)	% Area	Height (μ V)	% Height
1	1.127	144916	3.47	5621	2.59
2	2.182	502251	12.02	9036	4.16
3	2.882	290116	6.22	11004	5.07
4	3.121	226099	5.41	19361	8.91
5	3.383	586877	14.04	12309	5.67
6	4.271	155908	3.73	7453	3.43
7	4.607	423305	10.13	6427	2.96
8	9.300	1890347	44.99	146025	67.22