Stille Coupling of Stereochemically-defined α -Sulfonamidoorganostannanes

Kevin W. Kells and J. Michael Chong*

Guelph-Waterloo Centre for Graduate Work in Chemistry and Biochemistry, (GWC)²

Department of Chemistry, University of Waterloo, Waterloo, Ontario, Canada

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General Experimental

All reactions were performed using flame-dried glassware under an argon atmosphere. Toluene and THF were freshly distilled from Na/benzophenone. Dichloromethane was distilled from calcium hydride. (*R*)- and (*S*)-*t*-butanesulfinamide were prepared from the appropriate *cis*-1-amino-2-indanol as reported by the Sepracor group.¹ ¹H and ¹³C NMR spectra were recorded in CDCl₃ at 300 and 75 MHz, respectively. Optical rotations were recorded in cells with 10 cm path length on a Perkin-Elmer 241 digital polarimeter. Chiral HPLC analysis was performed using a ChiralCel OD column using 1% *i*PrOH/Hex as eluent (1.0 mL/min). ¹³C NMR spectra show Sn-C couplings over one, two, and three bonds due to ¹¹⁷Sn and ¹¹⁹Sn. Elemental analyses were performed by MHW Laboratories, Phoenix, Arizona.

General procedure for synthesis of enantiomerically pure sulfinimines. *t*-Butanesulfinimines were prepared by condensation of (*R*)- or (*S*)-*t*-butanesulfinamide with aldehydes as described by Ellman.² Our (*R*)-*t*-butanesulfinamide showed [α]_D = +4.6 (c 0.8, CHCl₃); lit²: [α]_D = +4.9 (c 1.0, CHCl₃). Our (*S*)-*t*-butanesulfinamide showed [α]_D = -5.1 (c 0.6, CHCl₃). Spectral data are as follows:

(*R*_S)-*N*-(Benzylidene)-*t*-butanesulfinamide (1a)

$[\alpha]_D = -95.7$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.56 (1H, s), 7.80 (2H, d, $J = 7.8$ Hz), 7.42 (3H, m), 1.22 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 162.7, 132.3, 129.2, 129.1, 128.8, 57.6, 22.4.

(*R*_S)-*N*-(*p*-Methylbenzylidene)-*t*-butanesulfinamide (1b)

$[\alpha]_D = -200.0$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.52 (1H, s), 7.72 (2H, d, $J = 7.9$ Hz), 7.23 (2H, d, $J = 7.9$ Hz), 2.38 (3H, s), 1.22 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 162.4, 143.1, 131.6, 129.6, 129.3, 57.6, 22.5, 21.6; IR (neat) 1600, 1568 cm^{-1} ; MS (EI) m/z 223.12 (M); Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{NOS}$: C, 64.53; H, 7.67; N, 6.27. Found: C, 64.60; H, 7.79; N, 6.08.

(*R*_S)-*N*-(*p*-Methoxybenzylidene)-*t*-butanesulfinamide (1c)

$[\alpha]_D = -62.4$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.48 (1H, s), 7.78 (2H, d, $J = 8.7$ Hz), 6.95 (2H, d, $J = 8.7$ Hz), 3.84 (3H, s), 1.22 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 163.0, 161.7, 131.3, 127.3, 114.3, 57.5, 55.5, 22.5.

(*R*_S)-*N*-(*p*-Dimethylaminobenzylidene)-*t*-butanesulfinamide (1d)

$[\alpha]_D = +41.6$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.38 (1H, s), 7.66 (2H, d, $J = 8.5$ Hz), 6.63 (2H, d, $J = 8.5$ Hz), 2.98 (6H, s), 1.18 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 161.5, 152.9, 131.0, 122.2, 111.2, 57.1, 39.9, 22.4.

(*R*_S)-*N*-(*p*-Chlorobenzylidene)-*t*-butanesulfinamide (1e)

$[\alpha]_D = -93.1$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.46 (1H, s), 7.69 (2H, d, $J = 8.4$ Hz), 7.38 (2H, d, $J = 8.4$ Hz), 1.17 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 161.2, 138.4, 132.3, 130.3, 129.1, 57.7, 22.4.

(*R*_S)-*N*-(*p*-Bromobenzylidene)-*t*-butanesulfinamide (1f)

$[\alpha]_D = -49.2$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.48 (1H, s), 7.66 (2H, d, $J = 8.4$ Hz), 7.55 (2H, d, $J = 8.4$ Hz), 1.21 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 161.5, 132.8, 132.2, 130.6, 127.1, 57.8, 22.5.

(*S*)-*N*-(*p*-Trifluoromethylbenzylidene)-*t*-butanesulfinamide (1g)

$[\alpha]_D = +74.9$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 8.55 (1H, s), 7.88 (2H, d, $J = 8.1$ Hz), 7.63 (2H, d, $J = 8.1$ Hz), 1.18 (9H, s); ^{13}C NMR (75 MHz, CDCl_3) δ 161.3, 136.7, 133.0 (q, $J_{\text{C-F}} = 32.9$ Hz), 129.4, 125.7 (q, $J_{\text{C-F}} = 3.8$ Hz), 57.9, 22.4.

General procedure for the synthesis of α -sulfinamidostannanes.

Method A. A flame dried round bottom flask equipped with a stir bar and septum was charged with dry THF (10 mL) while being kept under argon. Upon cooling the solution to 0 °C, diisopropylamine (4.0 mmol, 1.3 eq) was added via syringe, followed by *n*-butyllithium (1.6 M, 4.0 mmol, 1.3 eq). The solution was stirred for 15 min. at 0 °C. Tributyltin hydride (4.0 mmol, 1.3 eq) was then added via syringe and the solution stirred for another 15 min. at 0 °C before cooling to –78 °C. The sulfinimine (3.64 mmol) was dissolved in dry THF (1 mL) and added dropwise to the stirring solution. Upon completion of the addition, the solution was stirred at –78 °C for 15 min to 1 h. The reaction was quenched cold with methanol (2 mL), then saturated aqueous ammonium chloride (5 mL), and allowed to warm to room temperature. The mixture was diluted

with diethyl ether (100 mL) and water (15 mL), and the ether layer washed with brine (20 mL), dried with sodium sulfate, and filtered through a pad of Celite[®]. The ether layer was evaporated under reduced pressure to afford a clear yellowish oil, which was purified via flash column chromatography (30 g of silica/g of crude material, 4:1 to 6:1 hexane:ethyl acetate) to provide sulfinamides.

Method B. A flame dried round bottom flask equipped with a stir bar and septum was charged with dry THF (10 mL) while being kept under argon. Upon cooling the solution to 0 °C, hexabutylditin (4.0 mmol, 1.3 eq) was added via syringe, followed by *n*-butyllithium (1.6 M, 4.0 mmol, 1.3 eq). The solution was stirred for 15 min. at 0 °C. Diethylzinc (1.0 M, 4.0 mmol, 1.3 eq) was then added via syringe and the solution stirred for another 45 min. at 0 °C before cooling to –78 °C. The sulfinimine (3.64 mmol) was dissolved in dry THF (1 mL) and added dropwise to the stirring solution. Upon completion of the addition, the solution was stirred at –78 °C for 1 h. The rest of the procedure was followed as in Method A.

The sulfinamides could not be analyzed by mass spectrometry, as the ionized species were too unstable with all available techniques. Spectral data are as follows:

(*R,S*)-*N*-(1-Tributylstannylbenzyl)-*t*-butanesulfinamide (2a)

$[\alpha]_D = -42.1$ ($c = 1.0$, CHCl_3); $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.12 (5H, m), 4.43 (1H, d, $J = 4.0$ Hz), 3.84 (1H, d, $J = 4.0$ Hz), 1.32 (6H, m), 1.23 (9H, s), 1.18 (6H, m), 0.82 (15H,

m); ^{13}C NMR (75 MHz, CDCl_3) δ 144.7, 128.2, 125.1, 125.0, 56.2, 51.6, 28.6, 27.2, 22.8, 13.5, 10.1; IR (neat) 3455, 1458, 1076 cm^{-1} .

(*S,S,R*)-*N*-(1-Tributylstannyl-*p*-methylbenzyl)-*t*-butanesulfinamide (2b)

$[\alpha]_{\text{D}} = -49.1$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.03 (4H, s), 4.41 (1H, d, $J = 4.0$ Hz), 3.81 (1H, d, $J = 4.0$ Hz), 2.27 (3H, s), 1.30 (12H, m), 1.25 (9H, s), 0.83 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 141.7, 134.7, 129.0, 125.1, 56.3, 51.4, 28.8, 27.3, 23.0, 20.9, 13.6, 10.2; IR (neat) 3437, 1073 cm^{-1} .

(*S,S,R*)-*N*-(1-Tributylstannyl-*p*-methoxybenzyl)-*t*-butanesulfinamide (2c)

$[\alpha]_{\text{D}} = -55.8$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.03 (2H, d, $J = 7.6$ Hz), 6.76 (2H, d, $J = 7.6$ Hz), 4.37 (1H, d, $J = 4.1$ Hz), 3.78 (1H, d, $J = 4.1$ Hz), 3.72 (3H, s), 1.30 (12H, m), 1.22 (9H, s), 0.80 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 157.3, 136.8, 126.3, 113.8, 56.2, 55.1, 51.0, 28.8, 27.3, 22.9, 13.5, 10.1; IR (neat) 3422, 1508, 1245 cm^{-1} ; Anal. Calcd for $\text{C}_{24}\text{H}_{45}\text{NO}_2\text{SSn}$: C, 54.35; H, 8.55; N, 2.64. Found: C, 54.74; H, 9.19; N, 2.38.

(*S*,*R*)-*N*-(1-Tributylstannyl-*p*-dimethylaminobenzyl)-*t*-butanesulfinamide (2d)

$[\alpha]_D = -16.3$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.05 (2H, d, $J = 7.7$ Hz), 6.68 (2H, d, $J = 7.7$ Hz), 4.40 (1H, s), 3.82 (1H, s), 2.91 (6H, s), 1.35 (12H, m), 1.27 (9H, s), 0.87 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 148.7, 132.7, 126.3, 113.0, 56.1, 51.1, 40.7, 28.8, 27.3, 22.9, 13.5, 10.1; IR (neat) 3420, 1663, 1372 cm^{-1} ; Anal. Calcd for $\text{C}_{25}\text{H}_{48}\text{N}_2\text{OSSn}$: C, 55.25; H, 8.90; N, 5.15. Found: C, 55.33; H, 8.52; N, 4.95.

(*S*,*R*)-*N*-(1-Tributylstannyl-*p*-chlorobenzyl)-*t*-butanesulfinamide (2e)

$[\alpha]_D = -32.2$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.16 (2H, d, $J = 7.3$ Hz), 7.03 (2H, d, $J = 7.3$ Hz), 4.38 (1H, d, $J = 4.5$ Hz), 3.79 (1H, d, $J = 4.5$ Hz), 1.30 (12H, m), 1.23 (9H, s), 0.82 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 143.5, 128.3, 126.2, 125.0, 56.2, 51.0, 28.7, 27.2, 22.8, 13.5, 10.2; IR (neat) 3429, 1464 cm^{-1} .

(*S,S*)-*N*-(1-Tributylstannyl-*p*-bromobenzyl)-*t*-butanesulfinamide (2f)

$[\alpha]_D = -32.0$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.32 (4H, m), 4.43 (1H, d, $J = 4.0$ Hz), 3.83 (1H, d, $J = 4.0$ Hz), 1.30 (12H, m), 1.23 (9H, s), 0.83 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 144.8, 132.2, 128.4, 126.8, 56.4, 51.7, 28.6, 27.2, 22.8, 13.6, 10.2; IR (neat) 3416, 1704 cm^{-1} .

(*R,S*)-*N*-(1-Tributylstannyl-*p*-trifluoromethylbenzyl)-*t*-butanesulfinamide (2g)

$[\alpha]_D = +23.0$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.47 (2H, d, $J = 8.1$ Hz), 7.22 (2H, d, $J = 8.1$ Hz), 4.48 (1H, d, $J = 4.7$ Hz), 3.85 (1H, d, $J = 4.7$ Hz), 1.30 (12H, m), 1.24 (9H, s), 0.83 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 149.4, 127.2 (q, $J_{\text{C-F}} = 32.4$ Hz), 126.1, 125.4 (q, $J_{\text{C-F}} = 4.3$ Hz), 124.9, 56.5, 51.6, 28.7, 27.3, 22.8, 13.5, 10.3; IR (neat) 3419, 1614, 1325 cm^{-1} ; Anal. Calcd for $\text{C}_{24}\text{H}_{42}\text{F}_3\text{NOSSn}$: C, 50.72; H, 7.45; N, 2.46. Found: C, 50.89; H, 7.36; N, 2.28.

General procedure for the synthesis of α -sulfonamidostannanes. The procedure described by Weinreb³ was modified slightly, as follows. Into a round bottom flask

equipped with a stir bar was added the sulfinamide (2.00 mmol). The flask was then charged with dry CH₂Cl₂ (20 mL) and then *m*CPBA (4.00 mmol, 2 eq) was added in one portion at room temperature. The mixture was stirred at room temperature for 1 hour and then quenched with a 1:1 mixture of saturated aqueous NaHCO₃ and saturated aqueous NaHSO₃ (50 mL). The layers were separated and the aqueous layer was washed twice with CH₂Cl₂ (50 mL each). The CH₂Cl₂ was washed with a 1:1 mixture of saturated aqueous NaHCO₃ and saturated aqueous NaHSO₃ (20 mL) and then saturated bicarbonate solution, dried with sodium sulfate, and filtered through a pad of Celite®. The solvent was evaporated under reduced pressure to afford a clear oil, which was purified via flash column chromatography (30 g of silica/g of crude material, 5:1 to 10:1 hexane:ethyl acetate) to provide sulfonamides. Spectral data are as follows:

(*S*)-*N*-(1-Tributylstannylbenzyl)-*t*-butanesulfonamide (3a)

The following compound was prepared in 87% yield according to the general procedure.

[α]_D = +41.1 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.13 (5H, m), 4.55 (1H, d, J = 9.3 Hz), 4.46 (1H, d, J = 9.3 Hz), 1.37 (6H, m), 1.25 (9H, s), 1.20 (6H, m), 0.84 (15H, m); ¹³C NMR (75 MHz, CDCl₃) δ 145.3, 128.6, 124.6, 124.0, 59.6, 47.9, 28.8, 27.3, 24.2, 13.5, 9.6; IR (neat) 3284, 1304, 1126 cm⁻¹; MS (CI) *m/z* 535.3 (M+NH₄); HRMS Calcd for C₁₉H₃₄NO₂S¹¹⁶Sn (M-C₄H₉): 456.1328. Found: 456.1335.

(S)-N-(1-Tributylstannyl-*p*-methylbenzyl)-*t*-butanesulfonamide (3b)

The following compound was prepared in 78% yield according to the general procedure.

$[\alpha]_D = +49.1$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.03 (2H, d, $J = 7.9$ Hz), 6.91 (2H, d, $J = 7.9$ Hz), 4.54 (1H, d, $J = 13.6$ Hz), 4.51 (1H, d, $J = 13.6$ Hz), 2.27 (3H, s), 1.30 (12H, m), 1.24 (9H, s), 0.83 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 142.1, 134.0, 129.2, 124.0, 59.6, 47.7, 28.7, 27.3, 24.2, 20.8, 13.5, 9.5; IR (neat) 3282, 1307, 1126 cm^{-1} ; MS (CI) m/z 549.3 ($\text{M}+\text{NH}_4$); HRMS Calcd for $\text{C}_{20}\text{H}_{36}\text{NO}_2\text{S}^{116}\text{Sn}$ ($\text{M}-\text{C}_4\text{H}_9$): 470.1485. Found: 470.1472.

(S)-N-(1-Tributylstannyl-*p*-methoxybenzyl)-*t*-butanesulfonamide (3c)

The following compound was prepared in 95% yield according to the general procedure.

$[\alpha]_D = +59.3$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 6.93 (2H, d, $J = 8.0$ Hz), 6.77 (2H, d, $J = 8.0$ Hz), 4.64 (1H, d, $J = 9.4$ Hz), 4.45 (1H, d, $J = 9.4$ Hz), 3.73 (3H, s), 1.32 (12H, m), 1.23 (9H, s), 0.82 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 156.9, 137.4, 125.2, 114.0, 59.5, 55.1, 47.2, 28.7, 27.3, 24.2, 13.5, 9.5; IR (neat) 3402, 1510, 1302, 1246, 1126 cm^{-1} ; MS (CI) m/z 565.3 ($\text{M}+\text{NH}_4$); Anal. Calcd for $\text{C}_{24}\text{H}_{45}\text{NO}_3\text{SSn}$: C, 52.76; H, 8.30; N, 2.56. Found: C, 52.74; H, 8.50; N, 2.65.

(S)-N-(1-Tributylstannyl-*p*-chlorobenzyl)-*t*-butanesulfonamide (3e)

The following compound was prepared in 75% yield according to the general procedure.

$[\alpha]_D = +65.4$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.21 (2H, d, $J = 8.4$ Hz), 6.97 (2H, d, $J = 8.4$ Hz), 4.52 (2H, m), 1.32 (12H, m), 1.26 (9H, s), 0.84 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 144.2, 128.6, 125.2, 124.0, 59.8, 47.4, 28.7, 27.3, 24.2, 13.5, 9.6; IR (neat) 3281, 1304, 1126 cm^{-1} ; MS (CI) m/z 569.2 ($\text{M}+\text{NH}_4$); Anal. Calcd for $\text{C}_{23}\text{H}_{42}\text{ClNO}_2\text{SSn}$: C, 50.15; H, 7.69; N, 2.54. Found: C, 49.90; H, 7.75; N, 2.72.

(R)-N-(1-Tributylstannyl-*p*-trifluoromethylbenzyl)-*t*-butanesulfonamide (3g)

The following compound was prepared in 77% yield according to the general procedure.

^1H NMR (300 MHz, CDCl_3) δ 7.50 (2H, d, $J = 8.1$ Hz), 7.15 (2H, d, $J = 8.1$ Hz), 4.60 (2H, m), 1.30 (12H, m), 1.29 (9H, s), 0.83 (15H, m); ^{13}C NMR (75 MHz, CDCl_3) δ 150.0, 126.8 (q, $J_{\text{C-F}} = 32.4$ Hz), 126.1, 125.7 (q, $J_{\text{C-F}} = 3.8$ Hz), 123.8, 59.9, 48.1, 28.7, 27.3, 24.2, 13.5, 9.8; IR (neat) 3420, 1327 cm^{-1} ; MS (CI) m/z 603.2 ($\text{M}+\text{NH}_4$); HRMS Calcd for $\text{C}_{20}\text{H}_{33}\text{F}_3\text{NO}_2\text{S}^{116}\text{Sn}$ ($\text{M}-\text{C}_4\text{H}_9$): 524.1201. Found: 524.1199.

General procedure for the coupling of α -sulfonamidostannanes with acid chlorides.

The stannane (0.20 mmol) was weighed into a flame-dried round bottom flask equipped with a stir bar and a condenser. The vessel was flushed with argon, and charged with toluene (10 mL). The palladium catalyst (10 mol %; 0.020 mmol in the case of $\text{Pd}(\text{PPh}_3)_4$, or 0.010 mmol of Pd_2dba_3 and 0.040 mmol of ligand) was added to the solution, followed by CuCN (5-10 mol %). The acid chloride (0.24 mmol, 1.2 equivalents) was added to the solution via syringe, and the reaction mixture was heated to 60-70 °C, stirring under argon, for 5 to 6 hours. The reaction was monitored by TLC and when all of the starting material was consumed, the reaction was allowed to cool, diluted with ethyl acetate (20 mL), and quenched with a 10% solution of ammonium hydroxide in saturated aqueous ammonium chloride (30 mL). The aqueous layer was washed twice with ethyl acetate (20 mL), and the combined organic layers were washed with brine (20 mL). The organic layer was dried with sodium sulfate, filtered through a pad of Celite®, and the solvent was removed under reduced pressure to afford a clear oil which was purified by flash column chromatography (30 g silica / g crude, 4:1 hexane:ethyl acetate) to afford α -amino ketones. Spectral data are as follows:

(*S*)-2-*t*-Butylsulfonamido-1,2-diphenylethanone (4a)

$[\alpha]_D = +121.2$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.92 (2H, d, $J = 7.2$ Hz), 7.35 (8H, m), 6.13 (1H, d, $J = 7.4$ Hz), 5.77 (1H, d, $J = 7.4$ Hz), 1.25 (9H, s); ^{13}C NMR

(75 MHz, CDCl₃) δ 195.6, 137.3, 133.8, 129.2, 129.0, 128.7, 128.5, 128.0, 127.6, 62.2, 59.8, 23.9; IR (neat) 3410, 1686, 1311, 1216 cm⁻¹; MS (CI) m/z 349.2 (M+NH₄); Anal. Calcd for C₁₈H₂₁NO₃S: C, 65.23; H, 6.39; N, 4.23. Found: C, 65.48; H, 6.48; N, 4.32.

(S)-2-*t*-Butylsulfonamido-2-(*p*-methylphenyl)-1-phenylethanone (4b)

[α]_D = +147.6 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.91 (2H, d, J = 7.4 Hz), 7.42 (3H, m), 7.20 (2H, d, J = 8.0 Hz), 7.07 (2H, d, J = 8.0 Hz), 6.09 (1H, d, J = 7.5 Hz), 5.72 (1H, d, J = 7.5 Hz), 2.23 (3H, s), 1.25 (9H, s); ¹³C NMR (75 MHz, CDCl₃) δ 195.7, 138.4, 134.2, 133.9, 133.7, 129.9, 128.9, 128.7, 127.9, 62.0, 59.8, 24.0, 21.0; IR (neat) 3303, 1686, 1311 cm⁻¹; MS (CI) m/z 363.2 (M+NH₄); Anal. Calcd for C₁₉H₂₃NO₃S: C, 66.06; H, 6.71; N, 4.05. Found: C, 66.28; H, 6.85; N, 4.07.

(S)-2-*t*-Butylsulfonamido-2-(*p*-methoxyphenyl)-1-phenylethanone (4c)

[α]_D = +118.6 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.89 (2H, d, J = 8.1 Hz), 7.47 (1H, t, J = 7.3 Hz), 7.35 (2H, t, J = 7.3 Hz), 7.22 (2H, d, J = 8.6 Hz), 6.78 (2H, d, J = 8.6 Hz), 6.07 (1H, d, J = 7.3 Hz), 5.70 (1H, d, J = 7.3 Hz), 3.69 (3H, s), 1.24 (9H, s); ¹³C

NMR (75 MHz, CDCl₃) δ 195.6, 159.6, 133.9, 133.7, 133.4, 129.3, 129.0, 128.6, 114.6, 61.7, 59.8, 55.1, 24.0; IR (neat) 3284, 1686 cm⁻¹; MS (CI) m/z 379.2 (M+NH₄); Anal. Calcd for C₁₉H₂₃NO₄S: C, 63.13; H, 6.41; N, 3.88. Found: C, 63.13; H, 6.45; N, 4.03.

(S)-2-*t*-Butylsulfonamido-2-(*p*-chlorophenyl)-1-phenylethanone (4e)

[α]_D = +144.7 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.89 (2H, d, J = 7.2 Hz), 7.40 (7H, m), 6.13 (1H, d, J = 7.1 Hz), 5.73 (1H, d, J = 7.1 Hz), 1.25 (9H, s); ¹³C NMR (75 MHz, CDCl₃) δ 195.3, 136.0, 134.1, 129.5, 129.4, 129.3, 129.1, 128.9, 128.1, 61.5, 60.0, 24.0; IR (neat) 3304, 1682 cm⁻¹; MS (CI) m/z 383.2 (M+NH₄); Anal. Calcd for C₁₈H₂₀ClNO₃S: C, 59.09; H, 5.51; N, 3.83. Found: C, 59.30; H, 5.70; N, 3.79.

(R)-2-*t*-Butylsulfonamido-2-(*p*-trifluoromethylphenyl)-1-phenylethanone (4g)

[α]_D = -8.4 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.90 (2H, d, J = 7.7 Hz), 7.50 (7H, m), 6.22 (1H, d, J = 7.2 Hz), 5.78 (1H, d, J = 7.2 Hz), 1.27 (9H, s); ¹³C NMR (75 MHz, CDCl₃) δ 195.0, 141.4, 134.4, 133.5, 130.8 (q, J_{C-F} = 32.8 Hz), 129.1, 129.0, 128.7, 128.4, 126.3 (q, J_{C-F} = 3.1 Hz), 61.6, 60.1, 24.0; IR (neat) 3413, 1687, 1326, 1216 cm⁻¹;

MS (CI) m/z 417.1 (M+NH₄); HRMS Calcd for C₁₂H₁₅F₃NO₂S (M-C₇H₅O): 294.0776.

Found: 294.0782.

(S)-2-*t*-Butylsulfonamido-1-(*p*-chlorophenyl)-2-phenylethanone

[α]_D = +237.6 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.84 (2H, d, J = 8.4 Hz), 7.30 (7H, m), 6.07 (1H, d, J = 7.4 Hz), 5.72 (1H, d, J = 7.4 Hz), 1.24 (9H, s); ¹³C NMR (75 MHz, CDCl₃) δ 194.5, 140.4, 137.0, 132.2, 130.4, 129.4, 129.1, 128.7, 128.0, 62.3, 59.9, 23.9; IR (neat) 3326, 1682, 1178 cm⁻¹; MS (CI) m/z 383.2 (M+NH₄); Anal. Calcd for C₁₈H₂₀ClNO₃S: C, 59.09; H, 5.51; N, 3.83. Found: C, 59.12; H, 5.61; N, 3.65.

(R)-2-*t*-Butylsulfonamido-1,4-diphenyl-3-buten-2-one

[α]_D = -75.3 (c = 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃) δ 7.70 (1H, d, J = 15.9 Hz), 7.35 (10H, m), 6.65 (1H, d, J = 15.9 Hz), 5.75 (1H, d, J = 6.0 Hz), 5.49 (1H, d, J = 6.0 Hz), 1.22 (9H, s); ¹³C NMR (75 MHz, CDCl₃) δ 193.6, 145.2, 136.9, 133.8, 131.1, 129.3, 128.9, 128.6, 128.3, 127.7, 121.6, 65.4, 59.8, 24.0; IR (neat) 3296, 1610, 1307, 1065 cm⁻¹; MS (CI) m/z 375.2 (M+NH₄); Anal. Calcd for C₂₀H₂₃NO₃S: C, 67.20; H, 6.49; N, 3.92. Found: C, 67.47; H, 6.68; N, 4.12.

(*R*)-1-*t*-Butylsulfonamido-1-phenyl-2-pentanone

$[\alpha]_D = -54.5$ ($c = 1.0$, CHCl_3); ^1H NMR (300 MHz, CDCl_3) δ 7.35 (5H, m), 5.60 (1H, d, $J = 5.9$ Hz), 5.21 (1H, d, $J = 5.9$ Hz), 2.31 (2H, m), 1.30 (2H, m), 1.17 (9H, s), 0.76 (3H, t, $J = 7.3$ Hz); ^{13}C NMR (75 MHz, CDCl_3) δ 172.8, 137.0, 131.1, 129.2, 128.1, 66.1, 59.7, 41.3, 24.0, 17.1, 13.4; IR (neat) 3414, 1610, 1304, 1216 cm^{-1} ; MS (CI) m/z 315.2 ($\text{M}+\text{NH}_4$); HRMS Calcd for $\text{C}_{11}\text{H}_{16}\text{NO}_2\text{S}$ ($\text{M}-\text{C}_4\text{H}_7\text{O}$): 226.0902. Found: 226.0905.

Synthesis of (*R*)-*N*-Boc-2-phenylglycine. (*R*)-2-Phenylglycine (33.08 mmol) was dissolved in 300 mL of a 1:1 mixture of 1,4-dioxane and saturated aqueous sodium bicarbonate. Boc_2O (66.15 mmol, 2 eq) was added to the solution, which was stirred overnight at room temperature. The bulk of the solvent was removed under reduced pressure and to the residue was added an excess of saturated aqueous ammonium chloride. The aqueous layer was extracted 5 times with ethyl acetate (75 mL each), and the combined organic layers were washed with brine, dried with sodium sulfate, and the solvent was removed in vacuo to provide a thick oil which was used in the next step without further purification. The compound showed spectral data consistent with that found in the literature.⁴

Synthesis of (*R*)-*N*-Boc-*N'*-methyl-*N'*-methoxy-2-phenylglycinamide. The procedure of ⁵ was modified as follows. A dichloromethane solution of (*R*)-*N*-Boc-2-phenylglycine (8.65 mmol), HBTU (8.65 mmol), HOBt (8.65 mmol) and diisopropylethylamine (8.65 mmol) was cooled to 0 °C. A mixture of *N,O*-dimethylhydroxylamine hydrochloride (9.52 mmol, 1.1 eq) and diisopropylethylamine (9.52 mmol, 1.1 eq) in dichloromethane was added slowly in one portion. The mixture was stirred at room temperature overnight. The solvent was removed in vacuo, and the residue was dissolved in ethyl acetate (100 mL) and saturated aqueous ammonium chloride (50 mL). The organic phase was washed with saturated aqueous ammonium chloride (25 mL), saturated aqueous sodium bicarbonate (25 mL), and brine (25 mL), dried with sodium sulfate, and the solvent was removed under reduced pressure. The crude product was purified via flash column chromatography (2:1 hexane:ethyl acetate) to afford a thick oil (94% yield) which showed spectral data consistent with the literature.⁶

Synthesis of (*R*)-*N*-Boc-2-amino-1,2-diphenylethanone. To a THF solution of (*R*)-*N*-Boc-*N'*-methyl-*N'*-methoxy-2-phenylglycinamide (2.28 mmol) cooled to 0 °C was added freshly prepared⁷ phenyl magnesium chloride (2M in THF, 11.40 mmol, 5 eq) via syringe. The solution was allowed to warm to room temperature and stirred overnight. The reaction was quenched with saturated aqueous ammonium chloride (50 mL). The aqueous layer was extracted twice with ethyl acetate (50 mL each) and the combined organic layers washed with brine. The solvent was dried with sodium sulfate and removed under reduced pressure to afford a thick oil. The crude compound was purified

via flash column chromatography (4:1 hexane:ethyl acetate) to afford a white solid (98% yield) that showed spectral data which was in agreement with the literature.⁸

Synthesis of (*R*)-2-*t*-Butylsulfinamido-1,2-diphenylethanone. To a dichloromethane solution of (*R*)-*N*-Boc-2-amino-1,2-diphenylethanone (0.68 mmol) cooled to 0 °C was added anisole (1.02 mmol, 1.5 eq) and trifluoroacetic acid (13.6 mmol, 20 eq). The solution was allowed to warm to room temperature and stirred for 2 hours, monitoring by TLC analysis. Once the reaction was complete, the solution was cooled to 0 °C. A solution of *t*-butylsulfinyl chloride⁹ in dichloromethane (0.7 M, 0.82 mmol, 1.2 eq) was added via syringe, followed by diisopropylethylamine (5.44 mmol, 8 eq). The solution was allowed to warm to room temperature and stirred overnight. The mixture was washed with saturated aqueous ammonium chloride. The aqueous layer was extracted twice with 20 mL of dichloromethane, and the combined organic layers were washed with water (20 mL), dried with sodium sulfate, and removed under reduced pressure. The crude product was purified by flash column chromatography (2:1 hexane:ethyl acetate) and used immediately in the next step.

Synthesis of (*R*)-2-*t*-Butylsulfonamido-1,2-diphenylethanone. The sulfinamide was oxidized with *m*CPBA according to the same procedure as with the α -stannyl sulfinamides.

References

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C-13 with Decoupling AVANCE-300B

Current Data Parameters
NAME k4301
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040905
Time 22:42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 10240
DS 2
SWH 17730.485 Hz
FIDRES 0.541032 Hz
AQ 0.0241075 sec
RG 916.2
DE 28.200 usec
TE 300.2 K
D1 2.00 usec
D11 2.0000000 sec
DELTA 0.0300000 sec
DELTA 1.8992998 sec
NUPRST 0.0000000 sec
NUPRST 0.0150000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
SF01 75.4756615 MHz

===== CHANNEL f2 =====
CDDPRG2 waltz16
NUC2 1H
P2 80.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SF02 300.1318008 MHz

F2 - Processing parameters
SI 32768
SF 75.467497 MHz
WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR Plot parameters
CX 20.00 cm
CY 12.50 cm
F1 200.000 ppm
F2 13093.35 Hz
F3 -5.000 ppm
F4 -377.34 Hz
NUC1 13C
NUC2 1H
77.3.54449 Hz/cm

¹³C observe with ¹H decoupling

Current Data Parameters
 NAME Kk597
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040831
 Time 23.06
 INSTRUM Avance300
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 5120
 DS 4
 SWH 20703.934 Hz
 FIDRES 0.315917 Hz
 AQ 1.5827444 sec
 RG 20642.5
 DM 24.150 usec
 DE 37.82 usec
 TE 300.0 K
 D1 5.0000000 sec
 d11 0.0300000 sec
 d12 0.0002000 sec

===== CHANNEL f1 =====
 NUC1 ¹³C
 P1 5.20 usec
 PL1 -2.00 dB
 SF01 75.4756682 MHz

===== CHANNEL f2 =====
 CDPORG2 waltz16
 NUC2 ¹H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 21.80 dB
 PL13 26.41 dB
 SF02 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677519 MHz
 MDW EN
 SSB 0
 LB 2.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 15.00 cm
 F1P 200.000 ppm
 F1 15093.55 Hz
 F2P -5.000 ppm
 F2 -377.34 Hz
 PPMCH 10.25000 ppm/cm
 HZCH 773.54449 Hz/cm

C-13 with Decoupling AVANCE-300B

Current Data Parameters
 NAME k1887a
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040827
 Time 23:39
 INSTRUM av300
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TO 32768
 SOLVENT CDCl3
 NS 1024
 DS 2
 SWH 17730.496 Hz
 FIDRES 0.541092 Hz
 AQ 0.9241076 sec
 RG 11595.2
 JW 28.200 usec
 JE 6.00 usec
 TE 298.0 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 %REST 0.00000000 sec
 %CHRG 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 9.80 usec
 PL1 -2.00 dB
 SF1 75.4758615 MHz

***** CHANNEL f2 *****
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PL2 -2.00 dB
 PL12 15.00 dB
 PL13 15.00 dB
 SF02 300.1318008 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677508 MHz
 WDW EM
 SSF 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CZ 12.50 cm
 FIP 200.000 ppm
 F1P 1509.150 Hz
 F2P 327.34 MHz
 F2 10.25000 ppm/cm
 PCNOM 773.5449 Hz/cm

C-13 with Decoupling AVANCE-300B

Current Data Parameters
 NAME: K881purs
 EXPNO: 2
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20040824
 Time: 14.37
 INSTRUM: av300
 PROBHD: 5 mm BBO BB-1H
 PULPROG: zgpg30
 TO: 32768
 SOLVENT: CDCl3
 NS: 622
 DS: 2
 SWH: 17730.485 Hz
 FIDRES: 0.341092 Hz
 AQ: 0.9241076 sec
 RG: 11385.2
 DW: 28.200 usec
 DE: 6.00 usec
 TE: 295.0 K
 D11: 2.00000000 sec
 DELTA: 0.03000000 sec
 ACQRES: 1.699598 sec
 MREST: 0.00000000 sec
 MCPR: 0.01500000 sec

===== CHANNEL f1 =====
 NUC1: 13C
 P1: 9.80 usec
 PL1: -2.00 dB
 SF01: 75.4756515 MHz

===== CHANNEL f2 =====
 CPDPRG2: waltz16
 NUC2: 1H
 P2: 80.00 usec
 PL2: -2.00 dB
 PL12: 15.00 dB
 PL13: 15.00 dB
 SF02: 300.1318008 MHz

F2 - Processing parameters
 SI: 32768
 SF: 75.4677519 MHz
 EQ: 0
 NSB: 0
 LB: 1.00 Hz
 GB: 0
 PC: 1.40

1D NMR plot parameters
 CX: 20.00 cm
 CY: 12.50 cm
 F1P: 200000 ppm
 F1: 15093.35 Hz
 F2P: -51000 ppm
 F2: -3777.34 Hz
 PRGM: 10 25000 ppm/cm
 HZCM: 775.54449 Hz/cm

C-13 with Decoupling AVANCE-300B

Current Data Parameters
NAME k8650ure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040724
Time 16.29
INSTRUM av-300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 257
DS 2
SWH 17730.496 Hz
FIDRES 0.541092 Hz
AQ 0.9241076 sec
RG 11505.2
CW 28.200 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
ACREST 0.00000000 sec
MCWK 0.01500000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
SF01 75.475615 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SF02 300.1318008 MHz

F2 - Processing parameters
SI 32768
SF 75.4677535 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
SC 1.40

ID NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 200.000 mm
F1 15093.55 Hz
F2 -5.000 mm
F2 -377.34 Hz
PPMCM 10.25000 ppm/cm
HZCM 773.54449 Hz/cm

¹³C observe with ¹H decoupling

Current Data Parameters
NAME kk889pure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040908
Time 1.29
INSTRUM Avance300
PROBHD 5 mm GNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1314
DS 4
SWH 20703.334 Hz
FIDRES 0.315917 Hz
AQ 1.5827444 sec
RG 20642.5
DM 24.150 usec
DE 37.82 usec
TE 300.0 K
D1 5.0000000 sec
d11 0.0300000 sec
d12 0.0002000 sec

===== CHANNEL f1 =====
NUC1 ¹³C
P1 5.20 usec
PL1 -2.00 dB
SF01 75.4756682 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 21.80 dB
PL13 26.41 dB
SF02 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 75.4677512 MHz
WDW EN
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 15093.95 Hz
F2P -5.000 ppm
F2 -377.34 Hz
PPMCM 10.25000 ppm/cm
HZCM 773.54449 Hz/cm

C-13 with Decoupling AVANCE-300B

Current Data Parameters
NAME k4854pure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040908
Time 12.44
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TO 32768
SOLVENT CDCl3
NS 470
DS 2
SWH 17730.496 Hz
FIDRES 0.541092 Hz
AQ 0.9241076 sec
RG 11595.2
DM 28.200 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRR 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
SF01 75.4758615 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SF02 300.1318008 MHz

F2 - Processing parameters
SI 32768
SF 75.4677503 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
FIP 200.000 ppm
F1 15093.55 Hz
F2 -5.000 ppm
F20 -372.34 Hz
PPMCM 10.25000 ppm/cm
HZCM 773.54449 Hz/cm

C-13 with Decoupling AVANCE-300B

Current Data Parameters
NAME kk868pure
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040909
Time 10.00
INSTRUM av300
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 660
DS 2
SWH 17730.496 Hz
FIDRES 0.541092 Hz
AQ 0.9241076 sec
RG 11595.2
DM 28.200 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89299998 sec
MPCST 0.00000000 sec
MCPRK 0.01500000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 9.80 usec
PL1 -2.00 dB
SF01 75.4756615 MHz

***** CHANNEL f2 *****
CPOPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SF02 300.1318008 MHz

F2 - Processing parameters
SI 32768
SF 75.4677589 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P -5.000 ppm
F2 -377.34 Hz
SFOCM 10.25000 ppm/cm
AQCM 773.54456 Hz/cm

¹³C observe with ¹H decoupling

¹³C observe with ¹H decoupling