Supporting Information for

Extending Pummerer Reaction Chemistry. Application to the Oxidative Cyclization of Tryptophan Derivatives.

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¹H NMR, ¹³C NMR, IR, MS, and selected combustion analyses for compounds **10-13, 15, 17, 18, 20, 21, 23, and 24.**

General Experimental	S2
¹ H NMR 10	S10
¹³ C NMR 10	S11
¹ H NMR 11	S12
¹³ C NMR 11	S13
¹ H NMR 12	S14
¹³ C NMR 12	S15
¹ H NMR 13	S16
¹³ C NMR 13	S17
¹ H NMR 15	S18
¹³ C NMR 15	S19
¹ H NMR 17	S20
¹³ C NMR 17	S21
¹ H NMR 18	S22
¹³ C NMR 18	S23
¹ H NMR 20	S24
¹³ C NMR 20	S25
¹ H NMR 21	S26
¹³ C NMR 21	S27
¹ H NMR 23	S28
¹³ C NMR 23	S29
¹ H NMR 24	S30
¹³ C NMR 24	S31

General Experimental. All reactions involving air and moisture sensitive reagents or solvents were performed in flame-dried glassware under an argon or nitrogen atmosphere. Tetrahydrofuran was distilled from sodium benzophenone ketyl under argon. Dichloromethane and acetonitrile were distilled from calcium hydride under argon. Dimethoxyethane was distilled from sodium and fluorenone under argon. All organic reagents were used as purchased. Flash chromatography was performed using 32 – 63 μm silica gel with the indicated solvent systems. The chromatography solvents used (hexanes, EtOAc, Et₂O) were distilled from CaH₂. All melting points are uncorrected. APCIMS, ESIMS and HRMS were obtained from the Mass Spectroscopy Laboratory at The Pennsylvania State University. Combustion analyses were performed by Midwest Microlab, IN. Copies of ¹H and ¹³C NMR spectra are provided as proof of purity.

2-Benzyloxycarbonylamino-3-(tert-butyl-dimethyl-silanyloxy)-3-[1-(toluene-4-sulfonyl)-1H-indol-3-yl]-propionic acid (10). Enoate **9** (2.0 g, 5.6 mmol) was suspended in *t*-BuOH/H₂O (26 mL, 1/1). A modified AD mix- α (potassium ferricyanide (4.40 g), potassium carbonate (1.86 g), (DHQ)₂PHAL (174 mg), and K₂OsO₂(OH)₄ (18 mg)) and methanesulfonamide (1.28 g, 13.4 mmol) were added in 4 equal portions at 1 hour intervals, and stirring was continued for 15 h. Sodium sulfite (8.4 g) was added, and the reaction solution was stirred for 30 min. The reaction solution was extracted with EtOAc (3 x 25 mL), the combined organic fractions were washed with 1 M KOH (25 mL), water, and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 40% EtOAc in hexanes as the eluent, providing 1.72 g (79%, > 99% ee¹) of the diol as a white solid. mp 48 – 51 °C; $[\alpha]_D^{25} + 30.7$ (c 1 CH₂Cl₂); IR (CDCl₃) 3534, 1738, 1598 cm⁻¹; ¹H NMR (360 MHz, CDCl₃) δ 7.97 (d, *J* = 8.3 Hz, 1H), 7.76 (s, 1H), 7.73 (d, *J* = 5.3 Hz, 2H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 1H), 7.21 (d, *J* = 7.3 Hz, 1H), 7.17 (d, *J* = 7.9 Hz, 2H), 5.25 (d, *J* = 5 Hz, 1H), 4.52 (dd, *J* = 5.1, 2.4 Hz, 1H), 3.78 (s, 3H), 3.50 (d, *J* = 4.9 Hz, 1H), 2.99 (d, *J* = 7.8 Hz, 1H), 2.29 (s, 3H); ¹³C NMR (90 MHz, CDCl₃) δ 173.2, 145.2, 135.3, 135.3, 130.1, 129.2, 127.0, 125.1, 124.5, 123.5, 121.7, 120.1, 113.9, 73.6, 68.6, 43.1, 21.7; APCI *m/z* (relative intensity) 372.2 (M + H – H₂O, 100%); Anal. Calcd for C₁₉H₁₉NO₆S: C, 58.60; H, 4.92; N, 3.60; S, 8.23; Found: C, 58.89; H, 5.30; N, 3.43; S, 7.93.

The diol (3.8 g, 9.7 mmol) was dissolved in CH₂Cl₂ (125 mL) and cooled to 0 °C. 4-Nitrobenzenesulfonyl chloride (2.2 g, 10 mmol) and Et₃N (2.85 mL, 20.5 mmol) were added and the reaction solution was stirred at 0 °C for 15 h. The reaction solution was added to 1 M HCl (50 mL), extracted with CH₂Cl₂ (3 x 50 mL), washed with water and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo to yield 4.2 g (75%) of a crude yellow-orange solid.

The crude nosylate (4.2 g, 7.4 mmol) was dissolved in DMF (40 mL), and sodium azide (534 mg, 8.2 mmol) was added. The mixture was heated at 45 °C for 15 h. The reaction solution was concentrated in vacuo, and the residue was added to 1 M H₃PO₄ (50

¹ The enantiomeric excess was determined by HPLC using an OJ-H column. The retention times using 93:7 hexanes as the eluent: isopropylalcohol were 133.1 min for the (2S, 3S) enantiomer and 172.9 min for the (2R, 3R enantiomer).

mL), extracted with CH₂Cl₂ (3 x 50 mL), washed with water and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo to yield 2.0 g (64%) of the crude azide.

2,6-Lutidine (1.5 mL, 13 mmol) was added to the crude azide (2.0 g, 4.8 mmol) in CH₂Cl₂ (100 mL) and the mixture was stirred for 5 min. Tertbutyldimethylsilyl trifluoromethanesulfonate (1.5 mL, 6.5 mmol) was added and stirring was continued for 15 h. The reaction solution was added to water (25 mL), extracted with CH₂Cl₂ (3 x 25 mL), dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 10% ether in hexanes as the eluent, providing 1.65 g (68%) of the TBS ether as a colorless oil. IR (CDCl₃) 2117, 1748 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.04 (d, *J* = 7.4 Hz, 1H), 7.79 (m, 3H), 7.64 (s, 1H), 7.38 (m, 4H), 5.24 (d, *J* = 7.7 Hz, 1H), 4.25 (d, *J* = 7.7 Hz, 1H), 3.75 (s, 3H), 2.33 (s, 3H), 0.84 (s, 9H), 0.06 (s, 3H), -0.28 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 168.9, 135.6, 135.1, 130.1, 130.0, 128.6, 126.8, 125.2, 125.1, 123.5, 121.7, 120.9, 114.0, 69.9, 66.8, 52.6, 25.6, 21.6, 18.2, -4.9, -5.5; ESI *m/z* (relative intensity) 551.1 (M + Na); HRMS calcd for C₂₅H₃₂N₄O₅SSi (M + NH₄) 546.2206, found 546.2164.

Trimethyl phosphine (3.5 mL, 1M in THF, 3.5 mmol) was slowly added to a solution of the TBS ether (1.55 g, 2.9 mmol) in THF (30 mL), and stirring was continued for 45 min. The reaction solution was cooled below -20 °C, benzyl chloroformate (440 μL, 3.1 mmol) was added, and the reaction solution was warmed to room temperature and stirred for 15 h. The reaction mixture was added to water (10 mL), extracted with CH₂Cl₂ (3 x 10 mL), washed with brine, dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 20% EtOAc in hexanes as the eluent, providing 650 mg (35%) of the CBz protected amine as a colorless oil. IR (CDCl₃) 3436, 1726 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.99 (d, *J* = 8.2 Hz, 1H), 7.84 (d, *J* = 7.7 Hz, 1H), 7.71 (m, 2H), 7.44 (s, 1H), 7.37 (m, 7H), 7.18 (d, *J* = 8.2 Hz, 2H), 5.68 (d, *J* = 7.7 Hz, 1H), 5.31 (d, *J* = 3 Hz, 1H), 5.15 (d, *J* = 12 Hz, 1H), 5.11 (d, *J* = 12 Hz, 1H), 4.73 (dd, *J* = 7.7, 3 Hz, 1H), 3.53 (s, 3H), 2.30 (s, 3H), 0.88 (s, 9H), 0.03 (s, 3H), -0.17 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 169.5, 155.7, 145.1, 136.3, 135.6, 135.4, 130.1, 130.0, 128.7, 128.4, 128.2, 126.9, 125.1, 124.4, 124.1, 123.0, 120.7, 113.9, 70.5, 67.2, 59.9, 52.1, 25.8, 21.7, 18.3, -4.8, -5.2; ESI *m/z* (relative intensity) 659.2 (M + Na, 100%), 654.2 (M + NH₄, 35%); HRMS calcd for C₃₃H₄₀N₂O₇SSi (M + NH₄) 654.2669, found 654.2627.

The CBz-protected amine (600 mg, 0.94 mmol) and LiOH•H₂O (120 mg, 3.86 mmol) were stirred for 15 h in THF/H₂O (15 mL, 4/1). The reaction mixture was acidified with 1 M HCl (10 mL), extracted with CH₂Cl₂ (3 x 10 mL), washed with water and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo, providing 500 mg (86%) of the product **10** as a white solid. mp 65 – 68 °C; IR (CDCl₃) 3690, 3433, 1716 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.98 (d, *J* = 8.4 Hz, 1H), 7.85 (d, *J* = 7.7 Hz, 1H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.50 (s, 1H), 7.38 (m, 7H), 7.15 (d, *J* = 8.2 Hz, 2H), 5.63 (d, *J* = 7.7 Hz, 1H), 5.35 (d, *J* = 4.5 Hz, 1H), 5.17 (d, *J* = 12 Hz, 1H), 5.13 (d, *J* = 12 Hz, 1H), 4.75 (dd, *J* = 7.3, 4.5 Hz, 1H), 2.27 (s, 3H), 0.90 (s, 9H), 0.07 (s, 3H), 0.04 (s, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 171.2, 155.8, 145.2, 136.2, 135.6, 135.2, 130.0, 128.8, 128.6, 128.5, 128.3, 126.9, 125.2, 124.6, 123.8, 122.6, 120.5, 114.0, 70.3, 67.4, 59.7, 25.9, 21.7, 18.4, -4.7, -5.2; APCI *m/z* (relative intensity) 645.2 (M + Na, 100%); HRMS calcd for C₃₂H₃₈N₂O₇SSi (M + NH₄) 640.2513, found 640.2496.

[4a-Bromo-4-(*tert*-butyl-dimethyl-silanyloxy)-2-oxo-9-(toluene-4-sulfonyl)-2,3,4,4a,9,9a-hexahydro-pyrano[2,3-*b*]indol-3-yl]-carbamic acid benzyl ester (11). N-bromosuccinimide (250 mg, 1.5 mmol) was added to a solution of **10** (200 mg, 0.32 mmol) and NaHCO₃ (120 mg, 1.4 mmol) in *t*-BuOH (5.5 mL)/H₂O (1.3 mL) and stirred for 15 h. The reaction solution was concentrated in vacuo and purified by flash chromatography using 33% ether in hexanes as the eluent, providing 150 mg (67%) of lactone **11**, as a white solid. mp 92 – 95 °C; IR (CDCl₃) 3426, 1764, 1718 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.53 (d, 8.3 Hz, 2H), 7.42 (m, 9H), 6.62 (s, 1H), 5.61 (d, *J* = 4.4 Hz, 1H), 5.23 (m, 3H), 4.32 (dd, *J* = 4.4, 1.4 Hz, 1H), 2.40 (s, 3H), 0.95 (s, 9H), 0.27 (s, 3H), 0.09 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 165.4, 155.6, 145.3, 139.9, 135.8, 135.2, 131.9, 130.3, 130.0, 128.8, 128.6, 128.3, 128.1, 125.8, 124.9, 115.2, 98.1, 74.1, 67.5, 63.5, 54.3, 26.1, 21.9, 18.6, -3.5, -4.1; APCIMS *m/z* (relative intensity) 701.1 (*M* + *H*, 85%), 703.1 (⁸¹Br, 100%) HRMS calcd for C₃₂H₃₇BrN₂O₇SSi (*M* + *H*) 701.1352, found 701.1373; Anal. Calcd for C₃₂H₃₇BrN₂O₇SSi: C, 54.77; H, 5.31; N, 3.99; S, 4.57; Found: C, 54.76; H, 5.30; N, 3.91; S, 4.57.

2-Benzylloxycarbonylamino-3-[1-(toluene-4-sulfonyl)-1*H*-indol-3-yl]-3-triisopropylsilanyloxy-propionic acid (12). Freshly prepared *t*-butyl hypochlorite (2.0 mL, 18 mmol) was added to a solution of benzyl carbamate (2.69 g, 17.8 mmol) in 0.4 M sodium hydroxide (35 mL) and *n*-propanol (25 mL) in a flask covered with aluminum foil. This mixture was stirred for 5 min. A solution of (DHQD)₂AQN (150 mg, 0.17 mmol) in *n*-propanol (25 mL), **9** (1.71 g, 4.82 mmol), and a solution of K₂OsO₂(OH)₄ (80 mg, 0.21 mmol) in 0.4 M NaOH (5 mL) were added, and this solution was stirred for 22 h. Sodium bisulfite (2.5 g) was added to the reaction mixture which was then extracted with EtOAc (3 x 30 mL), washed with water and brine, dried over MgSO₄, filtered, and concentrated in vacuo. The products were purified by flash chromatography using 20 % EtOAc in hexanes as the eluent to remove excess benzyl carbamate, then 50% EtOAc in hexanes as the eluent to yield 1.72 g (69% > 99% ee²) of the protected amino alcohol as a white solid. mp 66 – 68 °C; [α]_D²⁵ – 34.6 (c 1 CH₂Cl₂); IR (CDCl₃) 3436, 1712 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.97 (d, *J* = 9.7 Hz, 1H), 7.73 (d, *J* = 8.1 Hz, 2H), 7.68 (s, 1H), 7.57 (d, *J* = 7.1 Hz, 1H), 7.34 (m, 7H), 7.13 (d, *J* = 8.1 Hz, 2H), 5.86 (d, *J* = 8.9 Hz, 1H), 5.54 (d, *J* = 2.2 Hz, 1H), 5.02 (m, 2H), 4.77 (dd, *J* = 8.9, 2.2 Hz, 1H), 3.73 (s, 3H), 3.37 (s, 1H), 2.27 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ, 171.5, 156.9, 145.4, 136.4, 135.5, 135.4, 130.3, 129.0, 128.9, 128.6, 128.2, 127.2, 125.4, 124.4, 123.9, 122.0, 120.2, 114.1, 68.5, 67.5, 58.9, 53.2, 21.9; ESI *m/z* (relative intensity) 540.2 (*M* + NH₄⁺, 100%), 505.2 (*M* + *H* – H₂O, 20%); HRMS calcd for C₂₇H₂₆N₂O₇S (*M* + NH₄⁺) 540.1804, found 540.1786.

A solution of the protected amino alcohol (1.11 g, 2.1 mmol) and 2,6-lutidine (730 μL, 6.3 mmol) in CH₂Cl₂ (20 mL) were stirred for 5 min. Triisopropylsilyl trifluoromethanesulfonate (750 μL, 2.8 mmol) was added and stirring was continued for 15 h. The reaction mixture was added to water (10 mL), extracted with CH₂Cl₂ (3 x 10 mL), dried over MgSO₄, filtered, and concentrated in vacuo. The products were purified

² The enantiomeric excess was determined by HPLC using an OJ-H column. The retention times using 93:7 hexanes as the eluent: isopropylalcohol were 228.7 min for the (2*R*, 3*S*) enantiomer and 303.3 min for the (2*S*, 3*R*) enantiomer.

by flash chromatography using 20% EtOAc in hexanes as the eluent to yield 1.25 g (88%) of the TIPS ether as a colorless oil. IR (CDCl₃) 3442, 1728 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.99 (d, *J* = 8.3 Hz, 1H), 7.75 (d, *J* = 7.9 Hz, 2H), 7.60 (m, 2H), 7.39 (m, 6H), 7.24 (t, *J* = 7.1 Hz, 1H), 7.16 (d, *J* = 8.1 Hz, 2H), 5.67 (d, *J* = 3 Hz, 1H), 5.56 (d, *J* = 9.6 Hz, 1H), 5.08 (d, *J* = 12.3 Hz, 1H), 5.01 (d, *J* = 12.3 Hz, 1H), 4.56 (dd, *J* = 9.6, 3 Hz, 1H), 3.80 (s, 3H), 2.31 (s, 3H), 0.95 (m, 21H); ¹³C NMR (75 MHz, CDCl₃) δ 171.1, 156.5, 145.4, 136.6, 135.7, 135.5, 130.2, 129.0, 128.8, 128.6, 128.5, 127.1, 125.5, 124.7, 123.9, 123.3, 120.0, 114.3, 69.4, 67.5, 60.0, 52.9, 22.0, 18.3, 12.8; ESI *m/z* (relative intensity) 701.2 (M + Na, 100%), 696.3 (M + NH₄, 30%); HRMS calcd for C₃₆H₄₆N₂O₇SSi (M + NH₄) 696.3139, found 696.3190.

The TIPS ether (32 mg, 0.05 mmol) and LiOH•H₂O (7 mg, 0.2 mmol) were stirred for 15 h in THF/H₂O (0.5 mL, 4/1). The reaction solution was acidified with 1 M HCl (2 mL), extracted with CH₂Cl₂ (3 x 5 mL), dried over MgSO₄, filtered, and concentrated in vacuo, to yield 25 mg (80 %) of **12** as a white solid: mp 72 – 75 °C (sublime); IR (CDCl₃) 3419, 3035, 1770, 1719 cm⁻¹; ¹H NMR (360 MHz, CDCl₃) δ 7.97 (d, *J* = 8.2 Hz, 1H), 7.70 (d, *J* = 8.2 Hz, 2H), 7.60 (s, 1H), 7.41 (m, 7H), 7.18 (m, 3H), 5.77 (d, *J* = 3.4 Hz, 1H), 5.42 (d, *J* = 7.6 Hz, 1H), 5.14 (d, *J* = 12 Hz, 1H), 5.06 (d, *J* = 12 Hz, 1H), 4.70 (dd, *J* = 7.3, 3.5 Hz, 1H), 2.31 (s, 3H), 1.05 (m, 21H); ¹³C NMR (125 MHz, CDCl₃) δ 171.1, 156.2, 145.3, 136.1, 135.1, 135.0, 130.1, 129.1, 128.9, 128.5, 128.4, 127.0, 125.3, 124.6, 123.8, 121.5, 119.5, 114.1, 68.0, 67.4, 59.4, 21.8, 18.0, 12.3; ESI *m/z* (relative intensity) 663.3 (M – H, 100%), 507.1 (M – TIPS, 10%); HRMS calcd for C₃₅H₄₄N₂O₇SSi (M – H) 663.2560, found 663.2513; Anal. Calcd for C₃₅H₄₄N₂O₇SSi: C, 63.23; H, 6.67; N, 4.21; S, 4.82; Found: C, 63.31; H, 6.97; N, 4.01; S, 4.79.

Oxindole 13. N-bromosuccinimide (16 mg, 0.09 mmol) was added to a solution of **12** (30 mg, 0.045 mmol) and sodium bicarbonate (8 mg, 0.09 mmol) in *t*-BuOH (340 μL)/H₂O (80 μL) and stirred for 15 h. The reaction solution was concentrated in vacuo and the residue was purified by flash chromatography using 33% ether in hexanes as the eluent, providing 10 mg (33%) of the product, as a white solid. mp 82 – 85 °C (sublime), IR (CDCl₃) 3452, 1810, 1772, 1725 cm⁻¹; ¹H NMR (360 MHz, CDCl₃) δ 7.97 (m, 3H), 7.53 (m, 2H), 7.42 (m, 1H), 7.36 (m, 7H), 5.50 (d, *J* = 7.4 Hz, 1H), 5.35 (d, *J* = 9 Hz, 1H), 5.22 (d, *J* = 12 Hz, 1H), 5.09 (d, *J* = 12 Hz, 1H), 4.61 (m, 1H), 2.42 (s, 3H), 0.84 (m, 21H); ¹³C NMR (100 MHz, CDCl₃) δ 174.5, 171.7, 156.0, 147.0, 146.6, 141.3, 136.0, 135.0, 132.7, 130.3, 129.0, 128.9, 128.3, 126.1, 125.9, 124.0, 114.3, 83.1, 78.4, 68.0, 57.9, 22.1, 17.9, 12.6; ESI *m/z* (relative intensity) 679.2 (M + H, 35%), 696.3 (M + NH₄, 100%), 701.2 (M + Na, 70%); HRMS calcd for C₃₅H₄₂N₂O₈SSi (M + NH₄) 696.2775, found 696.2778; Anal. Calcd for C₃₅H₄₂N₂O₈SSi: C, 61.92; H, 6.24; N, 4.13; S, 4.72; Found: C, 61.31; H, 6.13; N, 4.01; S, 4.68.

Sulfoxide 15. A solution of phenylsulfonyl chloride (1.25 mL, 8.7 mmol) in CH₂Cl₂ (30 mL) was added to a solution of *N*-tert-butoxycarbonyl-L-tryptophan (2.5 g, 8.2 mmol) and triethylamine (1.25 mL, 9.0 mmol) in CH₂Cl₂ (30 mL) and stirred for 90 min. The reaction solution was concentrated and the residue was purified by flash chromatography using 5% MeOH in 1/1 EtOAc/hexanes as the eluent to yield 3.04 g (90%) of the 2-substituted indole as a white solid. mp 76 – 78 °C; IR (CDCl₃) 3459, 1756, 1711 cm⁻¹;

^1H NMR (300 MHz, CDCl_3) δ 10.67 (s, 1H), 7.76 (d, J = 8.1 Hz, 1H), 7.38 (d, J = 8.1 Hz, 1H), 7.28 (m, 2H), 7.18 (m, 5H), 5.84 (d, J = 7.7 Hz, 1H), 4.54 (m, 1H), 3.50 (dd, J = 14, 6 Hz, 1H), 3.34 (dd, J = 14, 6 Hz, 1H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, d^6 -acetone) δ 173.5, 155.9, 138.4, 137.9, 129.9, 128.8, 127.6, 126.6, 124.0, 123.8, 120.2, 120.1, 119.0, 114.9, 79.0, 55.2, 28.3, 15.5; APCI m/z (relative intensity) 413.3 ($\text{M} + \text{H}$, 100%); HRMS calcd for $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_4\text{S}$ ($\text{M} + \text{H}$) 413.1535, found 413.1520.

The 2-substituted indole (2.83 g, 6.9 mmol) was cooled to 0 °C in EtOAc (65 mL) and triethylamine (965 μL , 6.9 mmol) followed by TBSCl (1.08 g, 7.1 mmol) were added and the reaction mixture was stirred at 0 °C for 2 h. The reaction solution was filtered to remove triethylamine hydrochloride and concentrated to yield 3.43 g of the TBS protected ester as a highly unstable beige solid.

The TBS ester (3.43 g, 6.5 mmol) was dissolved in CH_2Cl_2 (120 mL) and cooled to 0 °C. *m*-Chloroperbenzoic acid (1.08 g, 6.3 mmol) was added and the reaction solution was stirred at 0 °C for 3 h. The reaction solution was added to saturated NaHCO_3 (10 mL), extracted with CH_2Cl_2 (3 x 20 mL), dried over Na_2SO_4 , filtered, and concentrated in vacuo to yield 3.4 g (100%, 2 steps) of **15** as a highly unstable light brown solid (mixture of diastereomers). IR (CDCl_3) 3442, 1716 cm^{-1} ; ^1H NMR (360 MHz, CDCl_3) δ 7.71 (m, 4H), 7.59 (m, 2H), 7.39 (m, 6H), 7.30 (m, 2H), 7.19 (m, 2H), 7.06 (m, 4H), 5.35 (d, J = 8 Hz, 1H), 5.29 (d, J = 8 Hz, 1H), 4.60 (m, 2H), 3.61 (m, 1H), 3.43 (m, 3H), 1.33 (s, 9H), 1.31 (s, 9H), 0.87 (s, 9H), 0.86 (s, 9H), 0.23 (s, 3H), 0.20 (s, 3H), 0.19 (s, 3H), 0.17 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.6, 172.4, 155.7, 155.5, 143.5, 143.4, 138.3, 138.2, 133.9, 133.7, 131.4, 129.8, 129.6, 127.4, 127.0, 126.2, 125.8, 125.6, 125.4, 120.8, 120.7, 120.6, 120.5, 117.2, 116.7, 112.3, 80.2, 80.2, 55.8, 28.8, 28.6, 26.2, 26.1, 25.9, 25.8, 18.5, 18.0, -3.1, -4.4; APCI m/z (relative intensity) 543.2 ($\text{M} + \text{H}$, 100%), 443.1 ($\text{M} - \text{Boc}$, 45%); HRMS calcd for $\text{C}_{28}\text{H}_{38}\text{N}_2\text{O}_5\text{SSi}$ ($\text{M} + \text{H}$) 543.2349, found 543.2341.

Spirocyclic butyrolactone 17. A solution of trifluoroacetic anhydride (175 μL , 1.24 mmol) in CH_2Cl_2 (20 mL) at -78 °C was added to a solution of **15** (500 mg, 0.93 mmol) and Hunig's base (400 μL , 2.30 mmol) in CH_2Cl_2 (20 mL) at -78 °C, and the solution was stirred at -78 °C for 2 h. The reaction solution was warmed to room temperature and added to saturated NaHCO_3 (5 mL), extracted with CH_2Cl_2 (3 x 10 mL), dried over Na_2SO_4 , filtered, and concentrated in vacuo. The crude products were purified by flash chromatography using 1/1 ether/hexanes as the eluent to yield 80 mg (21%) of **17** as a white solid (1:1 mixture of diastereomers). From this mixture, a small amount (30 mg) of the less polar diastereomer was isolated by flash chromatography as a white solid. IR (CDCl_3) 3434, 1798, 1714, 1525 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.69 (m, 2H), 7.54 (m, 4H), 7.36 (m, 2H), 7.23 (m, 1H), 5.32 (d, J = 7.1 Hz, 1H), 4.93 (m, 1H), 3.06 (dd, J = 13.7, 9.5 Hz, 1H), 2.83 (m, 1H), 1.52 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 179.9, 173.8, 155.1, 153.6, 134.5, 131.2, 129.9, 129.6, 126.1, 126.0, 123.4, 119.8, 112.9, 90.4, 81.1, 50.6, 36.9, 28.3.

For the mixture: ESI m/z (relative intensity) 411.1 ($\text{M} + \text{H}$, 100%); HRMS calcd for $\text{C}_{22}\text{H}_{22}\text{N}_2\text{O}_4\text{S}$ ($\text{M} + \text{H}$) 411.1379, found 411.1351; Anal. Calcd for $\text{C}_{22}\text{H}_{22}\text{N}_2\text{O}_4\text{S}$: C, 64.37; H, 5.40; N, 6.82; S, 7.81; Found: C, 64.49; H, 5.75; N, 6.50; S, 7.70.

Oxindole 18. Cerium (IV) ammonium nitrate (868 mg, 1.58 mmol) was added to a solution of **17** (550 mg, 1.34 mmol) in CH₃CN/H₂O (60 mL, 5/1) and stirred for 15 h. The aqueous layer was extracted with EtOAc (3 x 10 mL), and the combined organic layers were dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 25% hexanes in Et₂O as the eluent to yield 370 mg (87%) of **18**, as a white solid (1:1 mixture of diastereomers). IR (CDCl₃) 3434, 1793, 1747, 1715 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.13 (s, 2H), 7.49 (d, *J* = 6.5 Hz, 1H), 7.38 (m, 3H), 7.16 (t, *J* = 7.6 Hz, 2H), 6.95 (t, *J* = 8.6 Hz, 2H), 5.88 (d, *J* = 9 Hz, 1H), 5.30 (d, *J* = 9 Hz, 1H), 5.08 (m, 2H), 2.99 (m, 1H), 2.85 (dd, *J* = 9.3, 4.2 Hz, 1H), 2.73 (t, *J* = 12.2 Hz, 1H), 2.51 (dd, *J* = 13.7, 5.6 Hz, 1H), 1.48 (s, 18H); ¹³C NMR (75 MHz, d⁸-THF) δ 176.7, 174.2, 156.2, 143.7, 143.1, 131.8, 131.7, 128.6, 128.0, 125.8, 125.4, 123.6, 123.5, 111.2, 111.0, 81.2, 80.5, 79.8, 50.9, 50.3, 37.9, 37.2, 28.6; APCI *m/z* (relative intensity) 336.0 (M + NH₄, 100%), 262.9 (M + H - *t*-Bu, 40%); HRMS calcd for C₁₅H₁₁N₄O₄S (M + NH₄) 336.1559, found 336.1526.

2-Phenylsulfanyl-1*H*-indole-3-carbaldehyde (20). Sodium hydride (1.88 g, 60% dispersion in mineral oil, 47.0 mmol) was added in small portions to a solution of benzenethiol (5.50 mL, 53.5 mmol) in dimethylacetamide (180 mL). Chloride **19** (8.50 g, 47.5 mmol) was added and the reaction solution was heated at 90 °C for 2.5 h. The reaction solution was added to ice, extracted with EtOAc (3 x 40 mL), dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 1/1 hexanes/Et₂O as the eluent to yield 9.95 g (83%) of **20** as a white solid: mp 166 – 169 °C; IR (CDCl₃) 3439, 1655 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 10.3 (s, 1H), 8.41 (s, 1H), 8.29 (m, 1H), 7.46 (m, 2H), 7.39 (m, 3H), 7.29 (m, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 185.6, 140.7, 136.5, 131.9, 131.6, 130.2, 128.9, 126.1, 124.7, 123.4, 121.3, 118.8, 111.1; ESI *m/z* (relative intensity) 254.0 (M + H, 100%); HRMS calcd for C₁₅H₁₁N₄O₄S (M + H) 254.0640, found 254.0631; Anal. Calcd for C₁₅H₁₁N₄O₄S: C, 71.12; H, 4.38; N, 5.53; S, 12.66; Found: C, 71.12; H, 4.42; N, 5.54; S, 12.89.

2-Azido-3-(*tert*-butyl-dimethyl-silanyloxy)-3-(2-phenylsulfanyl-1*H*-indol-3-yl)-propionic acid (21). *n*-Butyl lithium (9.1 mL, 2.3 M in hexanes, 21 mmol) was added to (carbomethoxy)triphenylphosphonium bromide (8.8 g, 21 mmol) in DME (100 mL). To this solution, **20** (4.89 g, 19.3 mmol) in DME (100 mL) was added and the reaction solution was brought to reflux and held there for 40 h. The reaction solution was added to ice, extracted with CH₂Cl₂ (3 x 50 mL), dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 33% ether in hexanes as the eluent to yield 4.84 g (81%) of the alkene as a white solid. mp 161 - 162 °C; IR (CDCl₃) 3450, 1698, 1627 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.18 (d, *J* = 16.1 Hz, 1H), 7.93 (m, 1H), 7.73 (m, 2H), 7.46 (m, 1H), 7.36 (m, 1H), 7.26 (m, 5H), 6.65 (d, *J* = 16.1 Hz, 1H), 3.79 (s, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 168.5, 137.3, 134.8, 132.0, 131.3, 129.4, 128.6, 126.9, 125.8, 124.1, 121.7, 120.4, 117.3, 115.0, 111.6, 51.5; APCI *m/z* (relative intensity) 310.2 (M + H, 100%); HRMS calcd for C₁₈H₁₅NO₂S (M + H) 310.0902, found 310.0892.

Sodium hydride (702 mg, 60% dispersion in mineral oil, 17.6 mmol) was added to a solution of the alkene (5.0 g, 16 mmol) in THF (100 mL) and stirred for 2 h. Benzyl

chloroformate (2.56 mL, 17.9 mmol) was added and stirring was continued for 15 h. The reaction solution was added to ice, extracted with CH₂Cl₂ (3 x 25 mL), dried over Na₂SO₄, filtered, and concentrated in vacuo to yield 7.4 g crude of the CBz-protected indole.

The crude CBz-protected indole (7.4 g) was suspended in *t*-BuOH/H₂O (90 mL, 1/1). A modified AD mix- α (potassium ferricyanide (15.80 g), potassium carbonate (6.67 g), (DHQ)₂PHAL (625 mg), and K₂OsO₂(OH)₄ (63 mg)) and methanesulfonamide (4.61 g, 48.5 mmol) were added in 4 equal portions at 1 hour intervals, and stirring was continued for 15 h. Sodium sulfite (25 g) was added, and the reaction solution was stirred for 30 min. The reaction mixture was then extracted with EtOAc (3 x 25 mL), and the combined organic fractions were washed with 1 M KOH (25 mL), water, and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 40% EtOAc in hexanes as the eluent to yield 6.45 g (85%, 2 steps, > 99% ee³) of the diol as a white solid. mp 142 – 144 °C; [α]_D²⁵ + 7.8 (c 1 CH₂Cl₂); IR (CDCl₃) 3531, 1737 cm⁻¹; ¹H NMR (360 MHz, CDCl₃) δ 8.16 (d, *J* = 8.4 Hz, 1H), 8.05 (d, *J* = 7.8 Hz, 1H), 7.34 (m, 7H), 7.18 (m, 3H), 7.03 (m, 2H), 5.60 (dd, *J* = 6, 4 Hz, 1H), 5.27 (d, *J* = 12 Hz, 1H), 5.22 (d, *J* = 12 Hz, 1H), 4.39 (dd, *J* = 6, 4 Hz, 1H), 3.75 (s, 3H), 3.60 (d, *J* = 6 Hz, 1H), 3.31 (d, *J* = 6 Hz, 1H); ¹³C NMR (75 MHz, CDCl₃) δ , 173.0, 150.9, 138.0, 136.8, 134.8, 130.3, 129.9, 129.3, 128.9, 128.8, 127.3, 126.7, 126.6, 126.2, 124.5, 123.6, 121.0, 116.1, 74.5, 70.9, 69.2, 53.3; ESI *m/z* (relative intensity) 460.2 (M + H – H₂O, 100%); Anal. Calcd for C₂₆H₂₃NO₆S: C, 65.39; H, 4.85; N, 2.93; S, 6.71; Found: C, 65.11; H, 4.90; N, 3.00; S, 6.53.

The diol (1.6 g, 3.4 mmol) was dissolved in CH₂Cl₂ (60 mL) and cooled to 0 °C. Triethylamine (966 μ L, 6.9 mmol) and 4-nitrobenzenesulfonyl chloride (772 mg, 3.5 mmol) were added and the reaction solution was stirred at 0 °C for 15 h. The reaction solution was added to 1 M HCl (20 mL), extracted with CH₂Cl₂ (3 x 20 mL), washed with water and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo to yield 2.4 g of the crude nosylate as a yellow solid.

Sodium azide (450 mg, 6.9 mmol) was added to a solution of the nosylate (2.4 g, crude) in acetone/water (50 mL, 1/1), and the reaction solution was heated for 15 h at 60 °C. The acetone was removed in vacuo, and the resulting water layer was extracted with CH₂Cl₂ (3 x 25 mL). The organic layers were washed with water and brine, dried over Na₂SO₄, filtered, and concentrated in vacuo to yield 1.78 g of the crude azide.

2,6-Lutidine (1.08 mL, 9.4 mmol) was added to the crude azide (1.78 g) in CH₂Cl₂ (60 mL) and stirred for 5 min. Tertbutyldimethylsilyl trifluoromethanesulfonate (1.08 mL, 4.7 mmol) was added and stirring was continued for 15 h. The reaction solution was added to water (30 mL), extracted with CH₂Cl₂ (3 x 30 mL), dried over Na₂SO₄, filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 33% ether in hexanes as the eluent, providing 1.40 g (67% over 3 steps) of the TBS ether as a yellow oil. IR (CDCl₃) 2116, 1739 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.23 (m, 2H), 7.45 (m, 7H), 7.21 (m, 3H), 7.03 (m, 2H), 6.03 (d, *J* = 3 Hz, 1H), 5.36 (d, *J* = 12 Hz, 1H), 5.29 (d, *J* = 12 Hz, 1H), 3.80 (s, 3H), 3.72 (d, *J* = 3 Hz, 1H), 0.90 (s, 9H), -

³ The enantiomeric excess was determined by HPLC using an OJ-H column. The retention times using 96:4 hexanes as the eluent: isopropylalcohol were 303.6 min for the (2S, 3S) enantiomer and 349.6 min for the (2R, 3R) enantiomer.

0.02 (s, 3H), -0.28 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 168.7, 150.8, 137.6, 136.9, 134.5, 131.0, 129.2, 128.7, 128.6, 128.0, 126.2, 126.1, 126.0, 125.8, 123.2, 122.2, 121.9, 115.5, 72.7, 69.1, 67.5, 52.7, 25.5, 18.0, -4.8, -6.0; ESI m/z (relative intensity) 639.2 ($\text{M} + \text{Na}$, 100%); HRMS calcd for $\text{C}_{32}\text{H}_{36}\text{N}_4\text{O}_5\text{SSi}$ ($\text{M} + \text{NH}_4$) 634.2519, found 634.2558.

The TBS ether (835 mg, 1.35 mmol) and $\text{LiOH}\cdot\text{H}_2\text{O}$ (192 mg, 4.57 mmol) were stirred for 15 h in $\text{THF}/\text{H}_2\text{O}$ (25 mL, 4/1). The reaction solution was acidified with 1 M HCl (10 mL), extracted with CH_2Cl_2 (3 x 10 mL), dried over Na_2SO_4 , and concentrated in vacuo, to yield 950 mg crude product of **21**, which was found to be unstable to chromatography, as a dark brown oil. IR (CDCl_3) 3455, 3063, 2116, 1722 cm^{-1} ; ^1H NMR (360 MHz, CDCl_3) δ 9.83 (s, 1H), 8.18 (s, 1H), 8.06 (d, $J = 7.9$ Hz, 1H), 7.29 (m, 8H), 5.86 (d, $J = 3.6$ Hz, 1H), 3.70 (d, $J = 3.6$ Hz, 1H), 0.85 (s, 9H), -0.01 (s, 3H), -0.30 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 173.6, 136.8, 135.8, 129.4, 127.8, 127.1, 126.6, 123.6, 122.2, 121.5, 120.8, 120.4, 110.9, 72.1, 67.7, 25.6, 18.1, -4.9, -5.8; ESI m/z (relative intensity) 467.1 ($\text{M} - \text{H}$), 100%), 503.1 ($\text{M} + \text{Cl}$, 10%); HRMS calcd for $\text{C}_{23}\text{H}_{28}\text{N}_4\text{O}_3\text{SSi}$ ($\text{M} - \text{H}$) 467.1573, found 467.1570.

Spiro Butyrolactone Thioimide 23. A solution of **21** (20 mg, 0.043 mmol) in CH_2Cl_2 (1 mL) was cooled to -78°C . 2, 6-Lutidine (15 μL , 0.13 mmol) followed by $\text{PhI}(\text{CN})\text{OTf}$ (41 mg, 0.11 mmol) were added and the reaction solution was slowly warmed to room temperature over 45 min and stirred there for 15 h. The reaction solution was added to water (3 mL), extracted with CH_2Cl_2 (3 x 5 mL), washed with water and brine, dried over Na_2SO_4 , filtered, and concentrated in vacuo. The crude products were purified by flash chromatography using 20% Et_2O in hexanes as the eluent to yield 8 mg (40%) of **23** as an orange oil. IR (CDCl_3) 2116, 1803, 1524 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.68 (m, 2H), 7.49 (m, 3H), 7.34 (m, 2H), 7.22 (m, 2H), 5.03 (d, $J = 9.5$ Hz, 1H), 4.56 (d, $J = 9.5$ Hz, 1H), 0.79 (s, 9H), 0.00 (s, 3H), -0.48 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 178.0, 169.8, 154.7, 134.5, 134.0, 132.0, 129.9, 129.6, 126.9, 125.7, 122.8, 120.2, 93.2, 79.2, 63.6, 25.3, 17.7, -5.2, -5.9; ESI m/z (relative intensity) 467.1 ($\text{M} + \text{H}$, 100%); HRMS calcd for $\text{C}_{23}\text{H}_{26}\text{N}_4\text{O}_3\text{SSi}$ ($\text{M} + \text{H}$) 467.1573, found 467.1591.

Oxindole 24. Cerium (IV) ammonium nitrate (42 mg, 0.077 mmol) was added to a solution of **17** (30 mg, 0.06 mmol) in $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ (4.5 mL, 5/1) and stirred for 15 h. The reaction solution was extracted with EtOAc (3 x 5 mL), dried over Na_2SO_4 , filtered, and concentrated in vacuo. The crude product was purified by flash chromatography using 1/1 hexanes/ Et_2O as the eluent to yield 12 mg (50%) of **25**, as a white solid: mp $128 - 130^\circ\text{C}$; IR (CDCl_3) 3432, 2117, 1807, 1752 cm^{-1} ; ^1H NMR (360 MHz, CDCl_3) δ 7.50 (m, 2H), 7.41 (m, 1H), 7.15 (m, 1H), 6.91 (d, $J = 7.7$ Hz, 1H), 4.93 (d, $J = 5.1$ Hz, 1H), 4.51 (d, $J = 5.1$ Hz), 0.87 (s, 9H), -0.06 (s, 3H), -0.36 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 173.4, 170.1, 142.4, 132.1, 125.2, 123.9, 123.6, 110.8, 83.1, 79.9, 62.7, 25.4, 17.8, 0.2, -5.5; ESI m/z (relative intensity) 392.2 ($\text{M} + \text{NH}_4$, 100%) 375.2 ($\text{M} + \text{H}$, 16%); HRMS calcd for $\text{C}_{17}\text{H}_{22}\text{N}_4\text{O}_4\text{Si}$ ($\text{M} + \text{H}$) 375.1489, found 375.1454; Anal. Calcd for $\text{C}_{17}\text{H}_{22}\text{N}_4\text{O}_4\text{Si}$: C, 54.53; H, 5.92; N, 14.96; Found: C, 54.32; H, 5.94; N, 14.73.

AGK111 75

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AGKY 107

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AKT11 137 fraction 2 reduced

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AKK111 137 fraction 2

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D1: 1.00000000 sec
D11

===== D1NAME: f2 =====
NUC1: 1H
P1: 15.70 usec
PL1: 0.00 dB
SFO1: 299.8718518 MHz

F2 - Processing parameters
SI: 32768
SF: 299.8718518 MHz
WDW: no
SSB: 0
GB: 0.00 Hz
PC: 1.00

TD: 65536
FIDRES: 0.0944589 Hz
AQ: 5.3064650 sec
RG: 2728.1
INJ: 1
DE: 6.10000 usec
DS: 5.00 usec
TE: 300.0 K
D1: 1.00000000 sec
D11

155.221

143.723
-143.120
131.770
-131.675
-128.554
-127.948
125.799
125.369
-123.634
123.512
111.233
-111.003

84.452
~~82.480~~
79.781
67.984
67.690
67.397
67.104
66.811

50.936
50.336

37.881
37.154
28.597
25.819
25.552
25.287
25.021
24.755

[illegible]

Current Data Parameters:
NAME: A04-3-5-04
EXPNO: 1
PROCNO: 1

F2 - Acquisition Parameters:
Date_: 20040605
Time: 13.45
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 6
DS: 2
SWH: 8172.1539 Hz
FIDRES: 0.0945350 Hz
AQ: 3.201660 sec
RG: 61.000
DQ: 0.000000
DE: 5.00 usec
TE: 300.0 K
D1: 1.00000000 sec

===== CHANNEL f1 =====
NUC1: 1H
P1: 11.70 usec
PL1: 0.00 dB
SFO1: 299.8718518 MHz

F2 - Processing parameters:
SI: 32768
SF: 299.8700100 MHz
WDW: EM
SSB: 0
LB: 0.30 Hz
GB: 0
PC: 1.00

10 MHz 2D parameters:
CX: 20.00 cm
F1F2: 12.000 ppm
F1: 3098.44 Hz
F2: -1.000 ppm
F2F1: -299.87 Hz
FREQ: 0.65000 GHz/cm
H2O: 194.91550 kHz/cm

Current Data Parameters
Name: A01-75-02
ExpNo: 2
ProcNo: 1

F2 - Acquisition Parameters
Date_: 20040506
Time: 15.07
Instr: spect
Prgram: 5 m Dip 1H/1
PulProg: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 880
DS: 4
AQ: 18196.992 Hz
RG: 0.280819 Hz
FIDRES: 1.743076 sec
AQ: 512
SC: 65.600 dBdc
SW: 5.00 dBdc
F2: 100.625000 kHz
C1: 0.0000000 sec
D11: 0.0000000 sec
D12: 0.0000000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 5.00 dBdc
PL1 -6.00 dB
SFO1 75.4186567 MHz

===== CHANNEL f2 =====
DPR2 2 waltz16
NUC2 1H
P2 115.00 dBdc
PL2 0.00 dB
PL12 20.00 dB
PL13 20.00 dB
SFO2 295.8711950 MHz

F2 - Processing parameters
SI 32768
SF 75.463072 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

===== CHANNEL f3 =====
F3 20.00 cm
FIP 210.000 dBm
F1 45834.50 Hz
F2 15.000 dBm
F3 754.02 Hz
F4 11.750 dBm
F5 803.45250 Hz

ADKIV 191

Current Data Parameters
Name ADK-9-17-03
ExptNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080917
Time 12:03
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 18
DS 2
SWH 7462.697 Hz
FIDRES 0.113872 Hz
AQ 4.2505621 sec
RG 64
DQ 67.000 usec
ZG 59.72 usec
TE 300.2 K
NUC1 13C
D1 12.00 usec
P1 1.0000000 sec
SF01 360.1361820 MHz
NUC1EL1 13C

F2 - Processing parameters
S1 32768
SF 360.1361820 MHz
RG 64
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F9 12.000 gain
F1 4231.61 Hz
F2 -1.000 ppm
F2 360.13 Hz
PNUC1 0.63100 ppm/cen
HZCM 234.09710 Hz/cen

AGXIV 22a-fraction 1

ASKE 33.2

Current Data Parameters
NAME AOK-1-B-14
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040108
Time 11:56:42
INSTRUM spect
PROBHD 5 mm DNP-11
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 7452.89742
FIDRES 0.11387242
AQ 4.3809671 sec
RG 1024
OR 67.000 usec
DE 95.71 usec
TE 300.2 K
NUC1 0.61
NUC2 1.00000000 usec
PC 12.00 usec
PC1 366.136650 MHz
NUCLEO 11
F2 - Processing parameters
SI 32768
SF 300.1339836 MHz
WDW HC
SSB 0
GB 0.00 Hz
PC 1.00
F2 - NMR list parameters
C1 20.00 cm
C2 1.00000000
F1 4331.61 MHz
F2 1.00000000
PC 360.13 Hz
PCHCK 0.00000000
HCHK 234.08/08 Hz/cm

Current Data Parameters

NAME	Value
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Parameter	Value
DATE_	20080505
TIME	00.00
INSTRUM	1H
PROBHD	5 mm BBO-13
PULPROG	zgpg30
TD	32768
SOLVENT	CDCl3
NS	6322
DS	2
SWH	33333.332 Hz
FIDRES	1.017223 Hz
AQ	0.4915700 sec
RG	32768
DM	15.000 usec
DE	21.43 usec
TE	300.0 K
DELTA	0.030000 sec
CHRGPG	WALTZ16
PC1	115.00 usec
S2	21 dB
HL1	21 dB
D1	10.0000000 sec
P1	6.00 usec
SFO1	125.7703415 MHz
NULBUS	13C

F2 - Processing parameters

Parameter	Value
SF	16384
FF	125.757256 MHz
MD	EM
SSB	0
GB	0.30 Hz
GC	0
SC	1.00

13 NMR plot parameters

Parameter	Value
CX	20.00 CF
F1P	210.000 ppm
F1	26405.13 Hz
F2	-10.000 ppm
F2	-1257.59 Hz
PP4CM	11.00000 ppm/cm
HZCM	1303.3345 Hz/cm