Supporting Information for

"Total Synthesis of (±)-Herbindole B and (±)-*cis*-Triketrin B"

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General

Melting points were measured on a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were recorded as thin films on NaCl plates. ^1H NMR spectra were obtained at either 400MHz or 600MHz (100MHz or 150MHz for ^{13}C) on Varian Mercury or Varian Inova spectrometers. Spectra recorded in CDCl_3 were referenced to residual CHCl_3 at 7.26 ppm for ^1H or 77.0 ppm for ^{13}C . Spectra recorded in $d_6\text{-DMSO}$ were referenced to residual DMSO at 2.49 ppm for ^1H or 40.0 ppm for ^{13}C . EI mass spectra were obtained at an ionizing voltage of 70 eV. Solvents were purified via a solvent purification system SPS-400-5 (Innovative Technology, Spectra Research Corp.) or distilled prior to use according to standard procedures.¹ Alternatively, solvents were used as obtained, with the addition of 4-Å molecular sieves. All other reagents were used as purchased from Aldrich without purification. Reactions were checked for completion by TLC (Macherey-Nagel, silica gel 0.25mm, UV₂₅₄) and/or ^1H NMR. Flash chromatography was performed using silica gel purchased from Silicycle Chemical Division Inc. (230-400 Mesh).

Compound 10

p-aminophenol **9** (2.10 g, 10.6 mmol) was dissolved in dry pyridine (20 mL) and cooled to 0 °C. Tosyl chloride (2.44 g, 12.8 mmol) was added portion-wise, the flask was purged with argon and the mixture was warmed to room temperature, stirring under argon until complete consumption of the starting material. The reaction was diluted with Et_2O and washed successively with water, 5 % HCl(aq) , water and brine. The organic layer was dried with MgSO_4 , filtered and concentrated to yield the crude sulfonamide. Typically the product was used crude directly in the next step; however it could be isolated via FC with a gradient elution (0, 5, 10,... % EtOAc/hexane) affording 2.50 g (7.1 mmol, 67 % yield) of the sulfonamide as a white solid. ^1H NMR (400 MHz, CDCl_3) δ = 7.96 (s, 1H), 7.53 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 8.6 Hz, 1H), 7.19 (d, J = 8.4 Hz, 2H), 6.69 (dd, J = 8.6, 2.9 Hz, 1H), 6.52 (d, J = 2.9 Hz, 1H), 5.17 (s, 1H), 4.29 (t, J = 5.4 Hz, 1H), 3.30 (s, 6H), 2.38 (s, 3H), 2.22 (d, J = 5.4 Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ = 154.2, 143.4, 137.2, 132.7, 129.5, 128.4, 128.3, 126.8, 117.7, 114.8, 106.1, 54.1, 36.1, 21.5; IR (thin film) ν = 3275, 1159 cm^{-1} . The sulfonamide (2.30 g, 6.6 mmol) was dissolved in dry CH_2Cl_2 (160 mL) in a 250-mL round bottomed flask, and cooled to 0 °C. PIBA (2.11 g, 6.6 mmol) was then added portion-wise and the flask was purged with argon. The mixture was stirred under argon until complete consumption of starting material (approximately 3 h). The mixture was then diluted with CH_2Cl_2 and washed with water (5 times), brine, dried with MgSO_4 , filtered and concentrated. The crude product was purified via FC with the gradient elution (0, 10, 20,... % EtOAc/hexane) to afford 1.86 g (5.33 mmol, ca. 81 % yield) of **10** as an orange solid. ^1H NMR (400 MHz, CDCl_3) δ = 8.19 (d, J = 10.6, 1H), 7.89 (d, J = 8.0 Hz, 2H), 7.38 (d, J = 8.0 Hz, 2H), 6.66-6.62 (m, 2H), 4.43 (t, J = 5.5 Hz, 1H), 3.22

¹Armarego, W. L. F.; Perrin, D. D. *Purification of Laboratory Chemicals 4th ed.*; Butterworth-Heinemann: Oxford, 1982.

(s, 6H), 2.72 (dd, $J = 5.5, 1.0$ Hz, 2H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 186.1, 163.9, 146.6, 144.7, 137.1, 134.9, 134.6, 130.6, 129.7, 127.4, 102.6, 53.5, 34.3, 21.6$; IR (thin film) $\nu = 2938, 1657, 1324\text{ cm}^{-1}$.

Compound 11

Dienophile **10** (1.79 g, 5.1 mmol) was dissolved in dry CH_2Cl_2 (25 mL) and cooled to 0°C . Cyclopentadiene (1.25 g, 18.4 mmol) was then added drop-wise under argon. The mixture was stirred at 0°C until complete consumption of the starting material (approximately 50 min) and then concentrated to yield the crude cycloadduct. The cycloadduct was then taken up in THF (25 mL) and HCl (concentrated) was added to effect indolization. Since the product and the starting material had the same R_f , the reaction was stirred overnight to ensure completion. Solid NaHCO_3 was added, and stirred for 10 min, and then MgSO_4 was added, with an additional 10 min of stirring. The reaction mixture was filtered and concentrated, yielding the crude product. The product was purified via FC (30% EtOAc/hexane) to yield 1.62 g (4.6 mmol, 90% yield) of the 5-hydroxyindole **11** as a white solid. ^1H NMR (400 MHz, d_6 -DMSO) $\delta = 9.14$ (s, 1H), 7.59 (d, $J = 8.5$ Hz, 2H), 7.54 (d, $J = 3.7$ Hz, 1H), 7.35 (d, $J = 8.5$ Hz, 2H), 6.80 (dd, $J = 5.3, 3.1$ Hz, 1H), 6.64 (d, $J = 3.7$ Hz, 1H), 6.55 (s, 1H), 6.48 (dd, $J = 5.3, 3.1$ Hz, 1H), 4.78 (br s, 1H), 4.12 (br s, 1H), 2.30 (s, 3H), 2.11 (d, $J = 6.8$ Hz, 1H), 1.98 (d, $J = 6.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 146.8, 144.5, 143.7, 142.9, 138.9, 136.4, 135.9, 131.3, 129.7, 128.8, 126.6, 125.8, 109.2, 103.7, 70.0, 50.4, 46.4, 21.5$; IR (thin film) $\nu = 3474, 1169\text{ cm}^{-1}$; MS m/z (relative intensity) 352 ($M+1$, 18), 351 (M^+ , 75), 196 (100); HRMS (EI 70 eV) - calcd for $\text{C}_{20}\text{H}_{17}\text{NO}_3\text{S}$: 351.0929, found: 351.0922.

Compound 12

5-Hydroxyindole **11** (1.62 g, 4.6 mmol) was dissolved in dry CH_2Cl_2 (50 mL) and cooled to -25°C . Collidine was then added under argon, followed by the drop-wise addition of Tf_2O . The mixture was stirred at -25°C until complete consumption of the starting material. The mixture was then warmed to room temperature, diluted with CH_2Cl_2 , washed with water, brine, dried with MgSO_4 , filtered and concentrated. The crude product was purified via FC (15 % EtOAc/hexane) to afford 1.66 g (3.4 mmol, 75 % yield) of the 5-trifloxyindole as a white solid. ^1H NMR (400 MHz, CDCl_3) $\delta = 7.69$ (d, $J = 3.9$ Hz, 1H), 7.60 (d, $J = 8.4$ Hz, 2H), 7.27 (d, $J = 8.4$ Hz, 2H), 7.04 (s, 1H), 6.79 (dd, $J = 5.1, 3.1$ Hz, 1H), 6.64 (d, $J = 3.9$ Hz, 1H), 6.38 (dd, $J = 5.1, 3.1$ Hz, 1H), 4.91 (br s, 1H), 4.19 (br s, 1H), 2.39 (s, 3H), 2.27 (d, $J = 7.3$ Hz, 1H), 2.21 (d, $J = 7.3$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) $\delta = 145.1, 143.7, 142.9, 142.4,$

140.9, 140.1, 135.8, 131.6, 130.1, 130.0, 126.6, 118.0 (q, $J = 321.4$ Hz, 1C), 117.1, 109.4, 108.6, 69.8, 50.8, 48.1, 21.5. The 5-trifloxyindole (0.242 g, 0.50 mmol) was suspended in a mixture of THF (3 mL) and H₂O (2 mL). A crystal of osmium tetroxide (< 1 mg) was added, and the mixture stirred for 5 min giving a black solution, after which NMO (0.070 g, 0.60 mmol) was added. The solution was stirred until complete consumption of the starting material (approximately 1 h), at which point Na₂SO₃ (0.756 g, 6 mmol) was added and the mixture was stirred for an additional 10 min. The mixture was then extracted with EtOAc (3 times), and the combined organic layers were then washed with brine, dried with MgSO₄, filtered and concentrated to yield the crude diol. Purification of this diol was performed via FC with gradient elution (0,10,20... % EtOAc/hexane) to afford 0.232 g (0.45 mmol, ca. 90 % yield) of **12** as a white solid. ¹H NMR (400 MHz, CDCl₃) δ = 7.79 (d, $J = 3.7$ Hz, 1H), 7.68 (d, $J = 8.6$ Hz, 2H), 7.32 (d, $J = 8.6$ Hz, 2H), 7.24 (s, 1H), 6.69 (d, $J = 3.7$ Hz, 1H), 4.13 (br s, 1H), 3.81-3.78 (m, 1H), 3.48 (br s, 1H), 3.37-3.34 (m, 1H), 2.92 (d, $J = 5.2$ Hz, 1H), 2.82 (d, $J = 5.2$ Hz, 1H), 2.41 (s, 3H), 2.25 (ddd, $J = 10.1, 1.5, 1.5$ Hz, 1H), 1.89 (ddd, $J = 10.1, 1.5, 1.5$ Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ = 145.5, 140.4, 135.8, 135.0, 132.5, 132.2, 130.2, 130.1, 128.7, 126.8, 111.7, 108.2, 70.4, 70.1, 50.6, 47.8, 43.2, 21.6; IR (thin film) ν = 3391, 1139 cm⁻¹; MS m/z (relative intensity) 518 (M+1, 2), 517 M⁺, 6), 457 (100), 362 (20), 155 (32), 91 (31); HRMS (EI 70 eV) - calcd for C₂₁H₁₈F₃NO₇S₂: 517.0477, found: 517.0428.

Compound 13

The Stille precursor **12** (1.29 g, 2.5 mmol) was dissolved in DMF (8 mL) in a tube. The organotin reagent **15** (1.38 g, 4.0 mmol) was added along with Pd(PPh₃)₂Cl₂ (0.088 g, 0.125 mmol), LiCl (0.530, 12.5 mmol) and a few crystals of BHT. The sides of the tube were then rinsed with an additional 1 mL of DMF before sealing the tube under an argon atmosphere. The reaction was heated at 140 °C for a period of 6 h, after which time the reaction was cooled to room temperature. The mixture was diluted with EtOAc, which was followed by washing with water (5 times), brine, dried with MgSO₄, filtered and concentrated. The crude product was purified via FC with gradient elution (0,3,5,8,10... % EtOAc/hexane) to afford 0.760 g (1.79 mmol, 72 % yield) of **13** as a white solid. ¹H NMR (600 MHz, CDCl₃) δ = 7.64-7.62 (m, 3H), 7.42 (s, 1H), 7.27-7.25 (m, 2H), 6.63 (d, $J = 3.7$ Hz, 1H), 6.57 (d, $J = 15.8$ Hz, 1H), 6.18 (dt, $J = 15.8, 6.6$ Hz, 1H), 4.07 (br s, 1H), 3.68-3.65 (m, 1H), 3.48 (br s, 1H), 3.38-3.35 (m, 1H), 3.06 (d, $J = 5.3$ Hz, 1H), 2.56 (d, $J = 5.3$ Hz, 1H), 2.37 (s, 3H), 2.28-2.20 (m, 2H), 2.18 (ddd, $J = 9.8, 1.6, 1.6$ Hz, 1H), 1.78 (ddd, $J = 9.8, 1.6, 1.6$ Hz, 1H), 1.09 (t, $J = 7.4$ Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 171.7, 144.8, 141.1, 133.9, 131.6, 130.0, 129.1, 128.4, 127.7, 126.6, 125.5, 115.5, 108.8, 99.5, 70.9, 70.6, 49.8, 48.3, 42.1, 29.7, 26.3, 21.6, 13.7 (one extra signal due to tin contamination); IR (thin film) ν = 3443, 1645 cm⁻¹; MS m/z (relative intensity) 424 (M+1, 4), 423 (M⁺, 13), 363 (100), 269 (37), 91 (10); HRMS (EI 70 eV) - calcd for C₂₄H₂₅NO₄S: 423.1504, found: 423.1494.

Compound 14

Diol **13** (0.379 g, 0.895 mmol) was dissolved in THF / H₂O (27: 7 mL, respectively). NaIO₄ (2.30 g, 10.7 mmol) was added and the reaction was vigorously stirred for 7 min. When the reaction was complete, the mixture was diluted with a further 60 mL of THF and cooled to 0 °C. NaBH₄ (1.02 g, 26.9 mmol) was added *very cautiously* in a portion-wise manner and the reaction was complete in 10 min. The mixture was then quenched with the addition of saturated NH₄Cl (aq) in a drop-wise manner. The reaction was then diluted with EtOAc, washed with water (5 times), brine, dried with MgSO₄, filtered and concentrated to give the crude diol. The crude diol was immediately dissolved in dry CH₂Cl₂ and cooled to 0 °C. TEA (0.50 mL, 3.6 mmol) and MsCl (0.15 mL, 1.97 mmol) were then added and the mixture was stirred overnight. The reaction was then diluted with CH₂Cl₂ and washed with water (2 times), 5 % HCl (aq), water, brine, dried with MgSO₄, filtered and concentrated. The crude bis-mesylate was purified via FC (40 % EtOAc/hexane) to afford 0.506 g (0.87 mmol, 97 % yield over 3 steps) of **14** as a gummy solid. ¹H NMR (600 MHz, CDCl₃) δ = 7.58 (d, J = 3.5 Hz, 1H), 7.50 (s, 1H), 7.47 (d, J = 8.4 Hz, 2H), 7.16 (d, J = 8.4 Hz, 2H), 6.67 (d, J = 3.5 Hz, 1H), 6.48 (d, J = 15.5 Hz, 1H), 6.25 (dt, J = 15.5, 6.4 Hz, 1H), 4.74 (dd, J = 9.4, 3.5 Hz, 1H), 4.57 (dd, J = 10.0, 3.5 Hz, 1H), 4.55-4.51 (m, 1H), 4.30 (dd, J = 9.4, 9.4 Hz, 1H), 4.06 (dd, J = 10.0, 10.0 Hz, 1H), 3.83-3.79 (m, 1H), 3.02 (s, 3H), 2.96 (s, 3H), 2.54-2.49 (m, 1H), 2.38 (d, J = 14.6 Hz, 1H), 2.32 (s, 3H), 2.29-2.24 (m, 2H), 1.10 (t, J = 7.3 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 145.1, 137.6, 135.6, 134.6, 134.0, 132.2, 131.5, 130.4, 129.9, 127.9, 126.4, 125.2, 118.4, 111.6, 72.5, 72.2, 43.6, 43.2, 37.2, 36.8, 29.8, 26.2, 21.5, 13.5; IR (thin film) ν = 2963, 1354, 1173 cm⁻¹; MS *m/z* (relative intensity) 582 (M+1, 6) 581 (M⁺, 17), 485 (18), 427 (29), 318 (28), 91 (73); HRMS (EI 70 eV) - calcd for C₂₆H₃₁NO₈S₃: 581.1211, found: 581.1187.

(±)-*cis*-Trikentrin B

The bis-mesylate **14** (0.1084 g, 0.186 mmol) was dissolved in DME (3.2 mL) and NaI (0.4497 g, 3 mmol) was then added. The mixture was heated in a sealed tube at 90 °C overnight. The tube was then cooled and opened. Zn (0.7844 g, 12 mmol) was then added, and the reaction mixture was then heated at 90 °C for an additional 8 h. The mixture was then cooled and diluted with EtOAc, washed with water (5 times), brine, dried with MgSO₄, filtered and concentrated. The crude product was purified via FC (5 % EtOAc / hexane) to afford 0.0364 g (0.092 mmol, ca. 50 % yield) of *N*-Tosyl-*cis*-trikentrin B as yellow oil. ¹H NMR (400 MHz, CDCl₃) δ = 7.56 (d, J = 3.7 Hz, 1H), 7.50 (d, J = 8.4 Hz, 2H), 7.45 (s, 1H), 7.12 (d, J = 8.4 Hz, 2H), 6.64 (d, J = 3.7 Hz, 1H), 6.53 (d, J = 15.6 Hz, 1H), 6.21 (dt, J = 15.6, 6.4 Hz, 1H), 4.19-4.11 (m, 1H), 3.45-3.37 (m, 1H), 2.57-2.50 (m, 1H), 2.30 (s, 3H), 2.29-2.21 (m, 1H), 1.39 (d, J = 7.0 Hz, 3H), 1.33 (d, J = 7.2 Hz, 3H), 1.10 (t, J = 4.7 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 144.3, 144.2, 134.8, 132.9, 132.6, 131.5, 131.0, 129.6, 129.0, 126.7, 126.4, 126.3, 116.1, 111.1, 40.5, 38.7, 37.7, 26.3, 23.5, 23.4, 21.5, 13.8; IR (thin film) ν = 2964, 1175 cm⁻¹. *N*-tosyl-*cis*-trikentrin B (0.067 g, 0.17 mmol) was dissolved in dry THF (10 mL) in a 25-mL round-bottomed flask equipped with a reflux condenser. TBAF (1.19 mL of a 1.0M solution in THF) was added to the reaction mixture and the flask was purged with argon. The mixture was heated to reflux, and stirred under argon until complete consumption of starting material (approximately 16 h). The mixture was then cooled to room temperature and diluted with water. The aqueous layer was extracted with EtOAc (5 times). The combined organic extracts were washed successively with water (3 times), brine, dried with MgSO₄, filtered and concentrated. The crude *cis*-trikentrin B was then purified via FC (10 % EtOAc/hexane) to afford 0.0354 g (0.15 mmol, 87 % yield) of *cis*-trikentrin B as a green-yellow oil. ¹H NMR (400 MHz, CDCl₃) δ = 7.92 (br s, 1H), 7.62 (s, 1H), 7.13 (dd, J = 3.0, 2.4 Hz, 1H), 6.60 (d, J = 15.7 Hz, 1H), 6.53 (dd, J = 3.0, 1.9 Hz, 1H), 6.18 (dt, J = 15.7, 6.6 Hz, 1H), 3.54-3.43 (m, 2H), 2.71 (ddd, J = 13.0, 9.2, 9.2 Hz, 1H), 2.30-2.23 (m, 2H), 1.55 (ddd, J = 13.0, 2.3, 2.3 Hz, 1H), 1.45 (d, J = 7.2 Hz, 3H), 1.35 (d, J = 7.0 Hz, 3H), 1.12 (t, J = 7.5 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ = 140.6, 131.9, 131.0, 129.1, 127.7, 127.4, 127.3, 123.7, 115.6, 103.1, 41.8, 38.4, 37.0, 26.3, 24.2, 22.5, 14.0; IR (thin film) ν = 3427, 2957, 1459 cm⁻¹; MS *m/z* (relative intensity) 240 (M+1, 19), 239 (M⁺, 84), 224 (100), 210 (16), 182 (31); HRMS (EI 70 eV) - calcd for C₁₇H₂₁N: 239.1674, found 239.1674.

Compound 7

To a chilled (0°C) solution of *p*-aminophenol (50.0g, 458mmol) in 500ml pyridine was added portionwise TsCl (96.1g, 504mmol). The reaction mixture was then warmed to room temperature and stirred mechanically for 6hrs or until complete by TLC. Pyridine was evaporated to near dryness under reduced pressure, diluted with EtOAc, washed copiously with 10% HCl (>5x) and saturated NaHCO₃ (3x) followed by drying over MgSO₄ and evaporation to a red oil which slowly solidified to a pink solid. Recrystallization from EtOAc/hexanes afforded 110g of *N*-tosyl-*p*-aminophenol (92%) as an off white solid. m.p. = 144-146 °C. ¹H-NMR (400MHz, CDCl₃): δ = 9.65 (br. s., 1H), 9.29 (br. s., 1H), 7.52 (d, J = 8.0Hz, 2H), 7.29 (d, J = 8.0, 2H), 6.83 (d, J = 8.8Hz, 2H), 6.58 (d, J = 8.8Hz, 2H), 2.30 (s, 3H); ¹³C-NMR (100MHz, CDCl₃): δ = 154.8, 142.8, 136.7, 129.5, 128.6, 126.8, 123.9, 115.5, 21.0; IR (thin film): ν_{max} = 3357, 3244, 1600, 1512, 1469, 1331, 1157, 1091; HRMS calc. for C₁₃H₁₃NO₃S = 263.0616, found 263.0619. To a solution of *N*-tosyl-*p*-aminophenol (18.3g, 70.0mmol) in 700ml DCM was added silica supported NaIO₄ (175.0g, 119mmol; 0.68mmol/g loading). The solution was mechanically stirred for 4 hours, or until complete by TLC and filtered. The filtrate was evaporated in vacuo yielding 14.26g (78%) crude product **7** as an orange solid, which could be recrystallized from EtOAc/hexanes if desired. m.p. = 124-126 °C ; ¹H-NMR (400MHz, CDCl₃): δ = 8.20 (dd, J = 10.0, 2.4Hz, 1H), 7.89 (d, 8.0Hz, 2H), 7.37 (d, J = 8.0Hz, 2H), 6.97 (dd, J = 10.0, 2.4Hz, 1H), 6.68 (adt, J = 10.0, 2.8Hz, 2H), 2.45 (s, 3H); ¹³C-NMR (100MHz, CDCl₃): δ = 186.1, 163.9, 145.2, 140.7, 136.9, 135.8, 135.4, 130.5, 130.1, 127.9, 21.0; IR (thin film) ν_{max} = 3068, 1655, 1624, 1556, 1540, 1323, 1155, 1090; HRMS calc. for C₁₃H₁₁NO₃S = 261.0460, found 261.0460.

Compound 21

To a tube charged with butadiene (7.48g, 138mmol) at -78°C was added a solution of tosyl-imine quinone **7** (10.0g, 38.4mmol) in 30ml DCM. The tube was sealed, brought to room temperature and stirred for 48 hours. The tube was then cooled to -78°C, opened and the mixture transferred to a round bottom flask and warmed to 0 °C. 5 drops of DBU were added (exothermic) and the reaction was stirred for an additional 30 minutes until precipitate formed. The solvent was evaporated in vacuo and the product triturated with hexanes affording 12.11g (quant.) pure dihydronaphthalene **21** as a tan coloured solid. m.p. = 190-194 °C. ¹H-NMR (600MHz, d₆-DMSO): δ = 9.40 (br. s., 1H), 9.08 (br. s., 1H), 7.52 (d, J = 8.4Hz, 2H), 7.34 (d, J = 8.4Hz, 2H), 6.48 (d, J = 8.7Hz, 1H), 6.45 (d, J = 8.7, 1H), 5.79-5.70 (m, 2H), 3.07 (br. s, 4H), 2.36 (s, 3H); ¹³C-NMR (100MHz, d₆-DMSO): δ = 153.5, 142.7, 138.1, 133.0, 129.5, 126.7, 125.8, 125.0, 123.6, 123.4, 121.5, 111.6, 25.6, 24.2, 21.0; IR (thin film) ν_{max} = 3423, 3268, 1595, 1467, 1305, 1154, 1092; HRMS calc. for C₁₇H₁₇NO₃S = 315.0929, found 315.0921.

Compound 22

To a suspension of dihydronaphthalene **21** (6.20g, 19.7mmol) in 400ml DCM was added SiO₂ supported NaIO₄ (49.2g, 33.4mmol; 0.68mmol/g loading). The reaction mixture was stirred mechanically for 4 hours or until complete by TLC. The silica was removed by filtration and the solvent evaporated in vacuo to afford 5.90 g (97%) crude product **22** as an orange solid which could be recrystallized from EtOAc/hexanes if desired. m.p. = 131-133°C. ¹H-NMR (400MHz, CDCl₃) δ = 8.15 (d, J = 10.4Hz, 1H), 7.88 (d, J = 8.4Hz, 2H), 7.36 (d, J = 8.4Hz, 2H), 6.62 (d, J = 10.4Hz, 1H), 5.79-5.70 (m, 2H), 3.09-3.01 (m, 4H), 2.45 (s, 3H); ¹³C-NMR (100MHz, CDCl₃): δ = 185.8, 164.1, 144.7, 141.5, 139.8, 137.7, 134.6, 130.6, 129.9, 127.6, 122.8, 122.6, 25.4, 24.5, 21.9; IR (thin film) ν_{max} = 3421, 3273, 3065, 1665, 1620, 1596, 1544, 1470, 1304, 1153, 1089; HRMS calc. for C₁₇H₁₅NO₃S = 313.0773, found 313.0776.

Compound 6

To a chilled (0 °C) solution of dienophile **22** (3.10g, 9.80mmol) in 32ml DCM was added freshly distilled cyclopentadiene (2.02ml, 24.5mmol). The mixture was stirred for 45 minutes, the solvent was evaporated and the residue taken up in THF. The solution was then cooled to -78 °C and MeLi (1.20ml of 1.6M solution in THF, 1.92mmol) was added dropwise under argon. The reaction mixture was stirred for an additional 3 hours, quenched with the addition of H₂O and poured into EtOAc. The organic phase was washed with 5% HCl (3x), H₂O (1x), NaHCO₃ (1x), brine (1x), dried over MgSO₄ and concentrated in vacuo. The crude product was triturated with Et₂O to afford 3.10g (83%) tetrahydroanthracene **6** as greenish/yellow solid. mp = 233-235 °C (d); ¹H-NMR (400MHz, d₆-DMSO): δ = 9.06 (br. s, 1H), 8.73 (br s., 1H), 7.50 (d, J = 8.0Hz, 2H), 7.32 (d, J = 8.0Hz, 2H), 6.56 (dd, J = 5.2, 3.2Hz, 1H), 6.08 (dd, J = 5.2, 3.2Hz, 1H), 5.69 (add, J = 29.2, 10.0Hz, 2H), 4.10 (s, 1H), 3.30 (s, 1H), 3.17-2.91 (m, 4H), 2.33 (s, 3H), 1.76 (d, J = 6.8Hz, 1H), 1.63 (d, J = 6.8Hz, 1H); ¹³C-NMR (100MHz, d₆-DMSO): δ = 149.9, 147.7, 143.3, 143.0, 142.8, 139.2, 133.2, 130.4, 130.1, 127.5, 124.8, 124.1, 120.9, 120.4, 68.14, 49.3, 46.7, 26.7, 25.5, 21.6.; ν_{max} = 3442, 3270, 3031, 2974, 2938, 2871, 1629, 1596, 1457, 1305, 1157, 1091.; HRMS calc. for C₂₂H₂₁NO₃S = 379.1242, found 379.1239.

Compound 26

To a solution of NaH (1.16g, 28.9mmol) in 5ml THF cooled to 0 °C was added via cannula a solution of tetrahydroanthracene **6** (3.60g, 9.60mmol) in 25ml THF. The mixture was stirred for 30 minutes and a solution of N-phenylbistrifluoromethanesulfonimide (4.10g, 11.6mmol) in 10ml THF was added via cannula. The reaction was stirred under argon and warmed to room temperature overnight. The reaction was quenched with H₂O at 0°C and the mixture was poured into EtOAc, washed with 10% HCl (3x), NaHCO₃ (1x), brine (1x), dried over MgSO₄ and evaporated in vacuo. The crude product was purified by silica gel flash chromatography (30% EtOAc/hexanes) affording 3.90g (79%) triflated product as a yellow oil which solidified under vacuum to a pale yellow solid. mp = 127-130 °C; . ¹H-NMR (600MHz, CDCl₃) δ = 7.65 (d, J=7.8Hz, 2H), 7.31 (d, J= 7.8Hz, 2H), 6.80 (dd, J=5.4, 3.0Hz, 1H), 6.71 (br. s, 1H), 6.66 (dd, J=5.4, 3.0Hz, 1H), 5.83 (m, 1H), 5.72 (m, 1H), 4.21 (s, 1H), 3.96 (s, 1H), 3.33 (s, 2H), 3.19 (d, J=19.5Hz, 1H), 2.88 (d, J=19.5Hz, 1H), 2.47 (3H), 2.19 (d, J=7.2Hz, 1H), 2.07 (d, J=7.19Hz, 1H); (100MHz, CDCl₃): δ = 151.9, 144.0, 143.3, 142.9, 141.7, 140.2, 136.8, 131.9, 129.7, 127.2, 126.9, 125.0, 123.4, 122.4, 118.5 (q, J = 349Hz, 1C), 68.0, 49.7, 48.7, 26.2, 24.9, 21.5; ν_{max} = 3260, 3042, 2981, 2940, 2872, 2362, 2339, 1599, 1495, 1380; HRMS calc. for C₂₃H₂₀F₃NO₅S₂ = 511.0735, found 511.0745.

To a solution of the triflated product (3.90g, 7.60mmol) in 15ml THF/H₂O (3:1) was added NMO (890mg, 7.60mmol) and a few crystals of OsO₄. The reaction was stirred overnight and Na₂SO₄ (9.58g, 76.0mmol) was added with additional stirring for 30 minutes. The mixture was poured into EtOAc and the organic fraction was washed with H₂O (3x), brine (1x), dried with MgSO₄ and evaporated in vacuo. The crude product was purified by silica flash chromatography (55% EtOAc/hexanes) yielding 2.53g (61%) of diol **26** as white solid. m.p. = 78-82 °C. ¹H-NMR (600MHz, CDCl₃): δ = 7.61 (d, J = 7.8Hz, 2H), 7.28 (d, J = 7.8Hz, 2H), 6.77 (br s., 1H), 5.82-5.70 (m, 2H), 3.90 (t, J = 5.4Hz, 1H), 3.78 (at, J = 5.4Hz, 1H), 3.43-3.01 (m, 8H), 2.43 (s, 3H), 2.11 (d, J = 10.2Hz, 1H), 1.64 (d, J = 10.2Hz, 1H); ¹³C-NMR (100MHz, CDCl₃): δ = 145.2, 144.6, 140.7, 136.9, 136.3, 135.3, 130.1, 128.0, 127.4, 127.5, 123.9, 122.6, 118.8 (q, J = 319Hz, 1C), 70.4, 70.3, 49.8, 49.0, 43.0, 26.8, 25.3, 21.8; IR (thin film) ν_{max} = 3475, 2997, 1728, 1597, 1406, 1324, 1215, 1137, 1050; HRMS calc. for [C₂₃H₂₀F₃NO₇S₂]⁻² = 543.0633, found = 543.0629.

Compound 27

To a solution of diol **26** (1.96g, 3.59mmol) and 2-methoxypropene (0.378ml, 35.9mmol) in 10ml DMF was added p-TsOH (137mg, 0.72mmol). The reaction was stirred until complete by TLC (2-3 hours)

and poured into Et₂O/H₂O. The ethereal layer was washed with saturated NaHCO₃ (3x), brine (1x), dried over MgSO₄ and evaporated in vacuo. The crude product was purified by silica gel flash chromatography (30% EtOAc/Hexanes) affording 1.90g (90%) of acetonide **27** as a white solid. mp = 77-80 °C; ¹H NMR (600MHz, CDCl₃): δ = 7.67 (d, J = 8.1Hz, 2H), 7.31 (d, J = 8.1Hz, 2H), 6.80 (br. s., 1H), 5.81-5.78 (m, 1H), 5.74-5.72 (m, 1H), 4.26 (d, J = 5.4Hz, 1H), 3.94 (d, J = 5.4Hz, 1H), 3.50 (s, 1H), 3.31 (br. s., 2H), 3.23-3.11 (m, 3H), 2.43 (s, 3H), 2.14 (d, J = 9.9Hz, 1H), 1.71 (d, J = 9.9Hz, 1H), 1.46 (s, 3H), 1.24 (s, 3H); ¹³C-NMR (150MHz, CDCl₃): δ = 144.7, 144.3, 140.9, 136.9, 135.8, 135.3, 129.9, 128.3, 127.9, 127.2, 123.7, 122.3, 118.5 (q, J = 317Hz, 1C), 112.7, 80.3, 90.1, 46.7, 46.0, 43.1, 26.6, 25.7, 25.1, 24.3, 21.6; IR (thin film) ν_{max} = 3567, 3261, 3043, 2991, 2255, 1920, 1631, 1494, 1211; HRMS calc. for C₂₆H₂₆F₃NO₇S₂ = 585.1103, found = 585.1117

Compound 28

To a solution of acetonide **27** (1.88g, 3.21mmol) and NMO (451mg, 3.85mmol) in 15ml THF/H₂O (3:1) was added a few crystals of OsO₄. The reaction was stirred for 24 hours or until TLC indicated complete consumption of starting material. Na₂SO₃ (4.04g, 32.1mmol) was then added to the reaction mixture and stirred for an additional 30 minutes. The reaction mixture was poured into EtOAc/H₂O and the aqueous fraction was extracted twice with EtOAc. The combined organic fractions were washed with H₂O (3x), brine (1x), dried with MgSO₄ and evaporated in vacuo affording the crude diol as a mixture of diastereomers. The crude product was dissolved in DCM and silica supported NaIO₄ (7.00g, 4.75mmol; 0.68mmol/g loading) was added. The reaction was stirred for 1 hour, the silica removed by filtration and the solvent removed in vacuo. The crude product was then dissolved in 30ml THF and 50 drops concentrated sulfuric acid were added. The reaction mixture was then stirred for 6 hours and poured into EtOAc/H₂O. The organic fraction was washed with H₂O (1x), saturated NaHCO₃ (3x), brine (1x), dried over MgSO₄ and evaporated in vacuo. The crude product was purified by silica gel flash chromatography (gradient elution, 25-30% EtOAc/hexanes) affording 1.54g (80%) indole **28** as a white solid. mp = 62-64 °C; ¹H NMR (600MHz, CDCl₃): δ = 9.71 (s, 1H), 7.84 (d, J = 3.6, 1H), 7.67 (d, J = 8.4Hz, 2H), 7.33 (d, J = 8.4Hz, 2H), 6.61 (d, J = 3.6Hz, 1H), 4.19-4.20 (m, 2H), 3.98-3.99 (m, 2H), 3.57 (s, 1H), 3.54 (d, J = 5.4Hz, 1H), 2.41 (s, 3H), 2.28 (d, J = 9.6, 1H), 1.94 (d, J = 9.6Hz, 1H), 1.52 (s, 3H), 1.21 (s, 3H); ¹³C-NMR (150MHz, CDCl₃): δ = 196.5, 145.4, 138.0, 135.8, 135.7, 133.2, 131.5, 130.5, 130.4, 129.0, 126.9, 118.5 (q, J = 317Hz, 1C), 116.3, 113.1, 106.2, 80.5 (2C), 47.8, 45.8, 43.6, 42.3, 25.8, 24.2, 21.6; IR (thin film) ν_{max} = 3154, 3054, 2991, 2940, 2849, 2361, 2340, 1728, 1597, 1457, 1418, 1405, 1212; HRMS calc. for C₂₆H₂₄F₃NO₈S₂ = 599.0895, found = 599.0860.

Compound 29

To a chilled (0°C) solution of indole **28** (1.49g, 2.49mmol) in 30ml MeOH was added portion wise NaBH₄ (113mg, 2.99mmol). The reaction mixture was stirred for 1 hour and quenched with saturated NH₄Cl. The solution was extracted with EtOAc (3x) and the combined organic fractions were washed with H₂O (3x), brine (1x), dried over MgSO₄ and evaporated in vacuo. The crude alcohol was then dissolved in DCM and triethylamine (0.585ml, 4.2mmol) was added. The solution was then cooled to 0°C and MsCl (0.196ml, 2.52mmol) was added. The reaction was then stirred for 4 hours, or until complete by TLC, the solvent evaporated in vacuo and the residue taken up in EtOAc. The solution was washed with H₂O (1x), 5% aqueous HCl (3x), saturated NaHCO₃ (1x), brine (1x), dried over MgSO₄ and evaporated in vacuo. The crude mesylate was purified by silica gel flash chromatography affording 981mg (58%, 2 steps) mesylate **29**, which solidified under vacuum to an off white solid. mp = 112-114°C; ¹H NMR (600MHz, CDCl₃): δ = 7.85 (d, J = 4.2Hz, 1H), 7.65 (d, J = 8.4Hz, 2H), 7.33 (d, J = 8.4Hz, 2H), 6.83 (d, J = 4.2Hz, 1H), 4.43-4.36 (m, 2H), 4.18 (br. s., 1H), 4.16 (d, J = 5.1Hz, 1H), 3.56 (br. s., 1H), 3.49 (d, J = 5.1Hz, 1H), 3.41-3.32 (m, 2H), 2.93 (s, 3H), 2.40 (s, 3H), 2.26 (d, J = 10.2Hz, 1H), 1.91 (d, J = 10.2Hz, 1H), 1.51 (s, 3H), 1.21 (s, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 145.7, 138.4, 136.0, 135.9, 133.4, 131.5, 130.7 (2C), 129.4, 127.2, 120.1, 118.8 (q, J = 320.31 Hz, 1C), 113.4, 106.6, 80.8, 80.7, 68.0, 48.1, 46.1, 43.9, 37.7, 28.1, 26.1, 24.5, 21.9; IR (thin film) ν_{max} = 3152, 2993, 2942, 2361, 2258, 1620, 1453, 1263; HRMS calc. for C₂₇H₂₈F₃NO₁₀S₃ = 679.0827, found = 679.0837.

Compound 30

To a solution of mesylate **29** (867mg, 1.27mmol) in 20ml DME was added NaI (1.90g, 12.7mmol) and activated Zn dust (833mg, 12.7mmol). (Zn dust was activated as follows: 20g Zn was stirred in 75 ml of 20% aqueous HCl for 15 minutes; Zn was filtered, washed with 50 ml H₂O followed by 50 ml acetone and dried in vacuo). The reaction mixture was brought to reflux for 3.5 hours until TLC indicated consumption of starting material. The Zn was removed by filtration and the filtrate was poured into EtOAc/H₂O. The organic fraction was washed with H₂O (3x), brine (1x), dried over MgSO₄ and evaporated in vacuo affording 707mg (95%) of product **30** as a white solid requiring no further purification. mp = 168-170°C; ¹H NMR (600MHz, CDCl₃): δ = 7.81 (d, J = 4.2Hz, 1H), 7.66 (d, J = 8.4Hz, 2H), 7.32 (d, J = 8.4Hz, 2H), 6.75 (d, J = 4.2Hz, 1H), 4.18 (d, J = 4.8, 1H), 4.15 (s, 1H), 3.55 (s, 1H), 3.51 (d, J = 4.8Hz, 1H), 2.96-2.85 (m, 2H), 2.40 (s, 3H), 2.25 (d, J = 9.6Hz, 1H), 1.90 (d, J = 9.6Hz, 1H), 1.51 (s, 3H), 1.24 (t, J = 7.8Hz, 3H), 1.21 (s, 3H); ¹³C NMR (100MHz, CDCl₃): δ = 145.13, 137.8, 135.9, 135.4, 132.1, 130.3, 129.5, 129.3, 129.0, 128.0, 126.9, 118.6 (q, J = 319.7Hz, 1C), 112.94, 106.54, 80.64, 80.59, 47.6, 45.8, 43.4, 25.8, 24.2, 21.6, 20.9, 14.4; IR (thin film) ν_{max} = 3152, 2984, 2938, 2881, 2360, 1617, 1494, 1455, 1406, 1374; HRMS calc. for C₂₆H₂₆F₃NO₇S₂ = 585.1103, found = 585.1239.

Compound 31

To a solution of **30** (696mg, 1.19 mmol), Pd(PPh₃)₄ (185mg, 0.119 mmol) and LiCl (252mg, 5.95 mmol) in 10ml DMF was added SnMe₄ (0.330ml, 2.38 mmol). The reaction flask was fitted with a condenser, purged with argon and brought to reflux for 3 hours. The reaction mixture was cooled, filtered through a pad of Celite and rinsed with Et₂O. The solution was diluted with Et₂O/H₂O and the aqueous layer was extracted with Et₂O (2x). The combined ethereal fractions were washed with H₂O (3x), brine (1x), dried with MgSO₄ and evaporated in vacuo affording 528mg (98%) of pure product as an off white solid. mp = 158-162 °C; ¹H NMR (400 MHz, CDCl₃): δ = 7.66 (d, J = 4.0 Hz, 1H), 7.64 (d, J = 8.4 Hz, 2H), 7.25 (d, J = 8.4 Hz, 2H), 6.68 (d, J = 4.0, 1H), 4.11 (s, 1H), 4.01 (d, J = 5.2 Hz, 1H), 3.53 (d, J = 5.2 Hz, 1H), 3.40 (s, 1H), 2.80 (q, J = 7.6 Hz, 2H), 2.36 (s, 3H), 2.28 (s, 3H), 2.18 (d, J = 9.2 Hz, 1H), 1.79 (d, J = 9.2 Hz, 1H), 1.52 (s, 3H), 1.20 (s, 3H), 1.16 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ = 144.7, 142.6, 136.7, 133.4, 130.4, 130.3, 128.7, 127.6, 127.1, 127.0, 126.0, 125.2, 112.8, 106.8, 81.6,

81.1, 47.2, 46.3, 42.5, 26.2, 24.4, 23.3, 21.8, 14.8 (2C); IR (thin film) ν_{max} = 3152, 3058, 2974, 2934, 2875, 2361, 2337, 1916, 1597, 1456, 1373; HRMS calc. for $\text{C}_{26}\text{H}_{29}\text{F}_3\text{NO}_4\text{S}$ = 451.1817, found = 451.1818.

Compound 33

Compound **31** (528 mg, 1.17 mmol) was dissolved in 30ml 70% aqueous acetic acid and brought to reflux for 6 hours or until TLC indicated consumption of starting material. The reaction was then cooled to 0°C and neutralized with saturated NaHCO_3 . The solution was poured into EtOAc and the organic fraction was washed several times (>5x) with saturated NaHCO_3 , brine (1x), dried over MgSO_4 and evaporated in vacuo affording 500mg (quant.) crude diol. The residue was taken up in 20ml THF/ H_2O (2:1) and NaIO_4 (1.30g, 6.06mmol) was added. The reaction was stirred for 1 hour, cooled to 0°C and NaBH_4 (680mg, 18.0mmol) was added portion wise (CAUTION: VIGOROUS REACTION). The reaction mixture was stirred vigorously for an additional 30 minutes and quenched by the addition of saturated NH_4Cl . The solution was poured into EtOAc/ H_2O , the aqueous layer extracted with EtOAc (3x) and the combined organic fractions washed with saturated NH_4Cl (1x), H_2O (3x), brine (1x), dried over MgSO_4 and evaporated. The residue was taken up in 20ml DCM and TEA (0.715ml, 5.13mmol) was added. To this solution was added MsCl (0.199ml, 2.56mmol) and the reaction mixture was stirred overnight. The reaction mixture was diluted with DCM and washed with 5% HCl (3x), saturated NaHCO_3 (1x) and brine (1x). The crude product was pre-absorbed onto silica (2.5g) and purified by silica gel flash chromatography (40% EtOAc/hexanes) affording 309mg (46% from **31**) of the bis-mesylate **33** as a white solid. mp = 153-155°C; ^1H NMR (600MHz, CDCl_3): 7.60 (d, J = 4.0 Hz, 1H), 7.48 (d, J = 8.4, 2H), 7.18 (d, J = 8.4 Hz, 2H), 6.73 (d, J = 4.0 Hz, 1H), 4.76 (dd, J = 9.0, 3.6 Hz, 1H), 4.55 (dd, J = 9.6, 3.6 Hz, 1H), 4.46 (dt, J = 9.0, 3.6 Hz, 1H), 4.23 (at, J = 9.0 Hz, 1H), 4.09 (at, J = 9.6 Hz, 1H), 3.71 (dt, J = 9.0, 3.6 Hz, 1H), 3.05 (s, 3H), 2.99 (s, 3H), 2.80-2.76 (m, 2H), 2.48-2.37 (m, 2H), 2.33 (s, 6H), 1.12 (t, J = 7.8, 3H); ^{13}C NMR (100MHz, CDCl_3): 145.2, 139.6, 136.4, 135.2, 133.0, 131.1, 130.2, 129.7, 128.5, 126.6, 125.2, 109.7, 73.1, 72.1, 44.3, 44.0, 37.5, 37.0, 30.2, 23.2, 21.8, 15.6, 14.4; IR (thin film) ν_{max} = 3152, 3030, 2696, 2941, 2879, 2362, 2338, 1596, 1458, 1354, 1234; HRMS calc. for $\text{C}_{25}\text{H}_{31}\text{NO}_8\text{S}_3$ = 569.1212, found = 569.1167.

Herbindole B

To a solution of *bis*-mesylate **33** (296mg, 0.52mmol) in 5ml DME was added NaI (1.56g, 10.39mmol) and activated Zn dust (679mg, 10.39mmol; see preparation of **30**). The reaction mixture was brought to reflux and stirred overnight. The solution was cooled to room temperature, filtered and the filtrate diluted with EtOAc/H₂O. The organic phase was washed with H₂O (3x), saturated NaHCO₃ (3x), brine (1x), dried over MgSO₄ and evaporated. The residue was taken up in 5 ml THF and TBAF (5.2ml of 1M solution in THF, 5.2mmol) was added. The reaction mixture was brought to reflux and stirred overnight. Following standard workup (EtOAc/H₂O), the product was purified by silica gel flash chromatography (5% EtOAc/hexanes) affording 70 mg (60%) of herbindole B. ¹H NMR (400MHz, C₆D₆): δ = 6.78 (b.s., 1H), 6.66 (dd, J = 3.2, 2.4 Hz, 1H), 6.61 (dd, J = 3.2, 2.0, 1H), 3.36-3.32 (m, 1H), 3.18-3.15 (m, 1H), 2.97 (q, J = 7.6 Hz, 2H), 2.59 (adt, J = 13.0, 9.1 Hz, 1H), 2.34 (s, 3H), 1.46 (adt, J = 13.0, 2.0 Hz), 1.35 (d, J = 6.8 Hz, 3H), 1.27 (t, J = 7.6 Hz, 3H), 1.21 (d, J = 7.2 Hz); ¹³C NMR (100MHz, CDCl₃): δ = 142.3, 132.7, 130.71, 126.8, 126.5, 122.8, 122.3, 101.4, 41.8, 39.2, 37.1, 23.7, 23.3, 22.7, 14.6, 14.1; IR (thin film) ν_{max} = 3416, 2960, 2930, 1870, 1456, 1401, 1125, 726; HRMS calc. for C₁₆H₁₁N = 227.1674, found = 227.1662.

10

[S8-11-207] in CDCl₃, Mercury
STANDARD IN OBSERVE

Archive directory: /export/home/vnr1/vmarsys/data
Sample directory: auto_23oct2003
Pulse Sequence: szpu1

10

[S8-12-129] In CDCl₃, Mercury
STANDARD IN OBSERVE

Archive directory: /export/hoae/vnari/vnarsys/data
Sample directory: auto_24Jul2004

Pulse Sequence: zgpg30

11

[S0-12-17] in DMSO, Inova 400
Archive directory: /export/home/kerr/vmarsys/data
Sample directory: sb1217_28oct2003-14:59:07
File: PROTON
Pulse Sequence: szpu1

11

[S0-12-17] in CDCl₃, Mercury
STANDARD IN OBSERVE

Archive directory: /export/home/vnmr1/vnmr/sys/data
Sample directory: auto_21Jul2004
Pulse Sequence: s2pul

12

[S8-11-119] in CDCl₃, Inova 400
Archive directory: /export/home/kerr/vmarsys/data
Sample directory: sb11119_01Aug2003
File: PROTON
Pulse Sequence: s2pu1

[58-12-187] In CDCl₃, Mercury
Archive directory: /export/home/vnari/vnarsys/data
Sample directory: auto_21Jul2884
Pulse Sequence: szpul

[58-12-77] in CDCl₃, Inova 600
 Data Collected on: mar3c888-inova600
 Archive directory: /export/inova/terr/vnmr/sys/data
 Sample ID: 58-12-77-01
 File: PR0TON_01
 Pulse Sequence: zgpg30
 Solvent: CDCl₃

13

[SR-12-77] in CDCl₃, Mercury
STANDARD IN OBSERVE

Archive directory: /export/home/vnari/vnarsys/data
Sample directory: auto_85Aug2004
Pulse Sequence: zgpg30

[SB-13-17] in CDCl₃, Inova 600

Data Collected on:
 nar3c68-inova600
 Archive directory:
 /usr/local/chem/vmarsys/data
 Sample directory:
 sb1317-2004-07-24
 File: PR0TON_01

Pulse Sequence: szpul
 Solvent: CDCl₃

14

[SB-13-17] in COCl₂, Mercury
STANDARD IN OBSERVE

Archive directory: /export/home/vnarl/vnmrsys/data
Sample directory: auto_25Jul2004

Pulse Sequence: s2pu1

cis-trikentrin B

[S8-13-13C] in CDCl₃, Inova 400
Archive directory: /export/home/kerr/vnarsys/data
Sample directory: sb1313c_31mar2004
File: PROTON
Pulse Sequence: t2pul

cis-trikentrin B

[SP-13-13C] in CDCl₃, Mercury
STANDARD IN OBSERVE

Archive directory: /export/home/vmr1/vmr1sys/data
Sample directory: auto_31Mar2004
Pulse Sequence: s2pu1

5-methylphenol-tertulate
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data

Sample directory: auto_16Aug2004

Pulse Sequence: szpul

Solvent: dmsc

Ambient temperature

File: PROTON

Mercury-400B8 "nmr400"

Relax: delay 1.000 sec

Pul: 45.0 degrees

Acq: time 4.000 sec

Width 6402.0 Hz

8 repetitions

Observed F1 400.0873771 MHz

DATA PROCESSING

FT size 65536

Total time 0 min, 43 sec

4-toluenesulfonyl-tosylate
STANDARD IN OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_16Aug2004

Pulse Sequence: szpul

Solvent: dmsd

Ambient temperature

File: 0209

Mercury-400BB "nmr400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

256 repetitions

Observed F1 400.1463637 MHz

Observed F2 100.6263637 MHz

DECOUPLE H1 400.0653637 MHz

Power 40 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 16 min, 21 sec

sj-Iminequinone
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vnmr1/vnmrSYS/data
 Sample directory: auto_16Aug2004
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 File: 0112
 Mercury-40008 "nmr400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 4.000 sec
 Frequency 400.146 MHz
 Channels 1
 8 repetitions
 OBSERVE H1, 400.0854794 MHz
 DATA PROCESSING
 17 slices 183360
 Total time 8 min, 43 sec

sj-aminomunone
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_16aug2004

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

File: 0113

Mercury-400008 "nmr400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

Temperature 300.2 K

OBSERVE C13 100.6015492 MHz

DECOUPLE H1 400.0674633 MHz

Power 40 dB

Continuously on

Contrast: 1.00000000

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 16 min, 21 sec

21

sj-4-137
Data Collected on:
nmr3c600-inova800
Archive directory:
S:\report\hms\kerr\vnarsys\data
Sj-4-137-2004-10-22
File: PROTON_01
Pulse Sequence: s2pul
Solvent: DMSO
Temp: 25.0 C / 298.1 K
Operator: kerr
Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 1.000 sec
Width: 9592.3 Hz
8 repetitions
OBSERVE: H1, 599.5252192 MHz
DATA PROCESSING
F1: 166531
Total time 0 min

21

Si-de-adduct
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_17Aug2004

Pulse Sequence: s2pu1

Solvent: dms

Ambient temperature

File: 0111

Mercury-40005 "nmr400"

Relax. delay: 1.000 sec

Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25125.6 Hz

Resolution 0.1100

OBSERVE F1, 400.6020037 MHz

DECOUPLE H1, 400.6020037 MHz

Power 40 dB

Continuously on

Watergate gated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 3 hr, 10 min, 49 sec

22

sj-4-13
STANDARD IN OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory: auto_19Aug2004-19:19:39

Pulse Sequence: zgpg30

Solvent: cdcl3

Ambient temperature

File: PROTON

Mercury-400BB "nmr400"

Relax. delay 1.000 sec

Acq. time 4.000 sec

Width 6402.0 Hz

8 repetitions

OBSERVE F1 400.0854792 MHz

Processing

File size 65536

Total time 0 min, 43 sec

22

sj-4-19
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vnari/vnarsys/data
 Sample directory: auto_19Aug2004-19:19:39
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Acquisition Temperature
 File: CARBON
 Mercury-400BB "nrm400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.180 sec
 Width 23125.6 Hz
 192 repetitions
 OBSERVE C13, 100.601500 MHz
 DECOUPLE H1, 499.8074633 MHz
 Preset: zgpg30
 Continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line processing 1.0 Hz
 File: sj-4-19
 Total time 1 hr, 16 min, 21 sec

6

sj-3-95
 Archive directory: /export/home/kerr/vnmrSYS/data
 Sample directory: sj-3-95_12Jul2004
 Pulse Sequence: szpu1
 Solvent: DMSO
 Ambient temperature
 Frequency: 400 MHz
 INOVA-400 "nuc3c400"
 Relax. delay 1.000 sec
 pulse 45.0 degrees
 Acq. time 3.744 sec
 Width 8396.8 Hz
 FID size 65536
 OBSERVE H1 399.7616429 MHz
 DATA PROCESSING
 FT size 65536
 Total time 1 min, 16 sec

SJ-4-23
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrsys/data
Sample directory: auto_31Sep2004

Pulse Sequence: s2pul

Solvent: dmso

Ambient temperature

File: 0104

Mercury-400B5 "nrm400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.18 sec

Width 25125.6 Hz

1000 repetitions

OBSERVE C13, 100.602030 MHz

DECOUPLE H1, 400.0053637 MHz

Post-acq. delay 1.000 sec

continuously on

VALT2-16 modulated

DATA PROCESSING

Frequency offset 1.0 Hz

FT size 65536

Total time 38 min, 10 sec

sj-4-161a
 Data Collected on:
 nmr3698-nov0800
 Archive directory:
 /export/home/kerr/vmarsys/data
 Sample directory:
 sj-4-161a-11-02
 File: PROTON_01
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 Operator: kerr
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 F2 (MHz) 500.136
 A repetitions
 OBSERVE H1, 599.5223884 MHz
 DATA PROCESSING
 F1 size 65536
 Total time 8 min

sj-4-27b-f
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: auto_07Sep2004
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 File: 0105
 Mercury-400BB "hnm400"
 Relax. delay 1.000 sec
 Pulse program: zgpg30
 Acq. time 1.139 sec
 Width 25125.6 Hz
 192 repetitions
 DECOUPLE CH1, 400.601580 MHz
 DECOUPLE CH1, 400.0074693 MHz
 Power 40 dB,
 Continuously on
 WALTZ-16 modulated
 Gamma Recoupling
 Line broadening 1.0 Hz
 FT size 65536
 Total time 38 min, 10 sec

26

SJ-4-1656
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmr1/data
Sample directory: auto_09Nov2004

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: PROTON
Mercury-400BB "nmr400"

Relax. delay 1.000 sec
Acq. time 4.000 sec
Width 6402.0 Hz
8 repetitions
OBSERVE FREQ 400.0854164 MHz
F1 size 85536
Total time 0 min, 43 sec

26

SJ-4-165B
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vmar1/vnmr/sv/data
 Sample directory: auto_09Nov2004
 Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
 Frequency: 500.135060
 Mercury-40088 "nrra400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.199 sec
 Obs. time 1.199 sec
 256 repetitions
 OBSERVE C13, 100.6015753 MHz
 DECOUPLE H1, 400.0874633 MHz
 Power 48 dB
 Cont. decoupling on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 65536
 Total time 9 min, 46 sec

27

SJ-4-171A
STANDARD 1H OBSERVE

Archive directory: /export/home/vmar1/vmarsys/data
Sample directory: auto_09Nov2004

Pulse Sequence: s2pul

Solvent: cdc13
Spectrum name: sj-4-171A
File: 8216
Mercury-400BB "nmr400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 25125.6 Hz
258 repetitions

OBSERVE C13, 100.6015745 MHz
DECOUPLE H1, 400.0074633 MHz
Pulse program continuously on

WALTZ-16 modulated

DATA PROCESSING
F1 frequency 1.0 Hz
F1 resolution 0.555 Hz
Total time 5 min, 46 sec

SJ-4-181a
 Data Collected on:
 nmr3ce00-inova600
 Archive directory:
 Sample directory: /vnmr3sys/data
 SJ-4-181a_2004-11-11
 File: PROTON_01
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 Operator: Kerr
 Relax. delay 1.000 sec
 Acq. time 1.832 sec
 Width 9592.3 Hz
 8 repetitions
 OBSERVED F1 500.1362722 MHz
 PROCESSED F1 500.1362722 MHz
 FT size 85536
 Total time 0 min

SJ-4-181a
STANDARD 1H OBSERVE
Archive directory: /support/home/vmari/vmarsys/data
Sample directory: auto_11Nov2004
Pulse Sequence: zgpg30
Solvent: cdcl3
Ambient temperature
File: C:\MSDCHEM\11NOV2004\11NOV2004.D
Mercury-4000B "nuc400b"
Relax: delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.199 sec
Width 23125.6 Hz
VFO 125.130 MHz
OBSERVE C13, 100.6015745 MHz
DECOUPLE H1, 400.0074633 MHz
Power 49 dB
Continuously on
VFO locked
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 16 min, 21 sec

SJ-4-09a
 Data Collected on:
 Bruker Avance 500
 Archive directory:
 /export/home/kerr/vnmrsys/data
 Sample directory:
 SJ-4-09a-09-27
 File: PROTON_01
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 Relax. delay: 1.000 sec
 Pulse: 45.0 degrees
 Acq. time: 1.892 sec
 Width: 8592.3 Hz
 SFO: 500.136 MHz
 QNP: 125.761 MHz
 QNP2: 598.5354663 MHz
 DATA PROCESSING
 FT size: 65536
 Total time: 0 min

SJ-4-89a
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vmr1/vmr1sys/data
 Sample directory: auto_27Sep2004
 Pulse Sequence: szpul
 Solvent: cdcl3
 Ambient temperature
 F1: 400.136360
 Mercury-400BB "nuc400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.199 sec
 Width 25123.6 Hz
 NS 1000
 OBSERVE C13 100.6015436 MHz
 DECOUPLE H1 400.0074633 MHz
 Power 40 dB
 Continuously on
 Locking on
 WALTZ-16 simulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 85536
 Total time 30 min, 10 sec

sj-3-137a
 Data Collected on:
 spectrometer: spect
 Archive directory:
 /export/home/kerr/vmarsys/data
 Sample directory:
 sj-3-137a/2004-07-15
 File: PRODM_01
 Pulse Sequence: szpul
 Solvent: CDCl3
 Temp: 25.0 C / 298.1 K
 Operator: Kerr
 Relax. delay: 1.000 sec
 Pulse: 45.0 degrees
 Acq. time: 1.882 sec
 Width: 9592.3 Hz
 Frequency: 500.136 MHz
 OBSERVE: 1H 599.5354130 MHz
 DATA PROCESSING
 FT size: 65536
 Total time: 0 min

SJ-3-1374
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vnmr1/vnmrsys/data
 Sample directory: auto_15Jul2004
 Pulse Sequence: s2pu1
 Solvent: cdcl3
 Ambient temperature
 File: CARBON
 Mercury-40000 "nmr400"
 Relax delay 1.000 sec
 Acq. time 1.199 sec
 Width 25125.6 Hz
 1888 repetitions
 OBSERVE CH3, 100.601560 MHz
 DECOUPLE CH3, 499.007453 MHz
 Power 40 dB,
 continuously on
 VAlT2-16 modulated
 D1 time 0.000 sec
 Line broadening 1.0 Hz
 FT size 65536
 Total time 1 hr, 16 min, 21 sec

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SJ-3-149a
 Archive directory: /export/home/kerr/vmarsys/data
 Sample directory: sj-3-149a_18Jul2004
 Pulse Sequence: szpul
 Solvent: CDCl₃
 Ambient temperature
 File: PROTON
 INOVA-400 "nuc3cd00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acquisition time 0.300 sec
 Width 6385.3 Hz
 8 repetitions
 OBSERVE H1, 399.7597478 MHz
 F1 A1, 65536.000 MHz
 F2 size 65536
 Total time 0 min, 39 sec

31

Filename: Gradient_12July04_AuHexp

sj-3-149a
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vnari/vnarsys/data
 Sample directory: auto_18Jul12004-20:47:11
 Pulse Sequence: s2pu1
 Solvent: cdc13
 Ambient temperature
 110.000
 Mercury-40000 "nmr400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.193 sec
 400.130 MHz
 2000 r.f. 1000 Hz
 OBSERVE C13, 100.615500 MHz
 DECOUPLE H1, 400.0074633 MHz
 Power 49 dB
 Contrast 1.000
 CONTINUOUS WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FI size 85536
 Total time 1 hr, 16 min, 21 sec

33

sj-3-207a
 Data Collected on:
 nar3c688-inova600
 Archive directory:
 /export/home/kerr/vmarsys/data
 Sample directory:
 Sample: 3-207-2004-06-08
 File: PROTON_01
 Pulse Sequence: zgpg30
 Solvent: CDCl3
 Temp: 23.0 C / 298.1 K
 Operator: Kerr
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. Time: 1.582 sec
 F1: 500.136 MHz
 F2: 500.136 MHz
 8 repetitions
 OBSERVE H1, 500.1354133 MHz
 DATA PROCESSING
 File size 15336
 Total time 8 min

33

SJ-3-207a
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vmar1/vmar/sys/data
 Sample directory: auto_08Aug2004
 Pulse Sequence: szpu1
 Solvent: cdcl3
 Acq. temperature: 300.2 K
 F1: CDCl3
 Mercury-400B8 "nmr400"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.139 sec
 Valtz 1.000 sec
 5000 repetitions
 OBSERVE c13, 100.601500 MHz
 DECOUPLE H1, 400.0874633 MHz
 Power 40.0 dB
 Valtz 1.000 sec
 VALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 F2 size 32768
 Total time 3 hr, 10 min, 49 sec

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SJ-4-13a
 STANDARD 1H OBSERVE
 Archive directory: /export/home/vmar1/vmarsys/data
 Sample directory: auto_09Aug2004-16:41:55
 Pulse Sequence: zgpg30
 Solvent: CDCl3
 Ambient Temperature
 F1 Size 6553a
 Mercury-1000B "nmr400b"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.199 sec
 Aft. 1.352 sec
 500Mhz 100Mhz
 OBSERVE C13 100.6015500 MHz
 DECOUPLE H1 400.0874833 MHz
 Power 40 dB
 Locking on
 Contin. Solvent
 VOLTAGE modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 6553a
 Total time 3 hr, 10 min, 49 sec