Supporting Information

for

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69451 Weinheim, Germany
Enantioselective Total Synthesis of the Cyclotryptamine Alkaloid Idiospermuline.

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Part A. Experimental Procedures for Key Transformations

(3S,3H,3aR,7aR)-1-Benzyl-1H-methyl-2,2-dimethyl-3a,4a,7a,7a-tetrahydrospiro[3H-indole-3,5(6H)-(1,3]benzodioxole-6(3H)-[3H]indole]-2,2(1H,1H)dione (10). A solution of 8 (1.03 g, 2.79 mmol), THF (35 mL) and HMPA (6 mL) was sparged for 45 min with Ar, cooled to –40 °C and a THF solution of LHMDS (9.3 mL, 0.7 M) was added by syringe pump at a rate of 0.20 mL/min. After 1 h, a THF solution of ditriflate 7 (15.0 mL, 0.2 M) was

1 The procedure we employed to purify THF, CH₂Cl₂, Et₂O, DME, and toluene has been described: Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. Organometallics, 1996, 15, 1518. Amine bases Et₃N and i-Pr₂EtN, DMF, were purified in a similar manner on slightly modified alumina columns. All reactions were conducted under nitrogen or argon and the molarities indicated for organolithium reagents were established by titration with BHT and phenanthroline. Other general experimental details have been described: Deng, W.; Overman, L. E.; J. Am. Chem. Soc. 1994, 116, 11241.

2 Due to the oxygen sensitivity of this reaction, all solvents and reaction solutions were sparged for 45 min with Ar prior to their addition to the reaction vessel.
added using a syringe pump at a rate of 0.20 mL/min. The resulting solution was warmed to −35 °C, maintained at −35 °C for 12 h (cryocool), then allowed to warm to rt over 1 h. The solution was partitioned between Et₂O (25 mL) and H₂O (15 mL). The layers were separated and the aqueous layer washed with Et₂O (1 ¥ 15 mL) and EtOAc (20 mL). The combined organic layers were dried (MgSO₄), filtered, and concentrated in vacuo. The foam residue was purified on silica gel (5:1 hexanes–EtOAc) to yield 1.03 g (75%) of 10 as a colorless foam: [α]₂₈⁴₀⁵ −698, [α]₂₈⁴₃₅ −528, [α]₂₈₅₄₆ −259, [α]₂₈₅₇₇ −224, [α]₂₈₅₂ −213 (c 0.94, MeOH); ¹H NMR (500 MHz, C₆D₆) δ 7.37 (dd, J = 7.2, 1.5 Hz, 1H), 7.34 (d, J = 7.6 Hz, 1H), 6.95 (t, J = 3.2 Hz, 3H), 6.90–6.86 (m, 2H), 6.70 (t, J = 7.7 Hz, 1H), 6.57–6.50 (m, 3H), 5.98 (d, J = 7.5 Hz, 1H), 5.84 (d, J = 7.8 Hz, 1H), 5.46–5.39 (m, 2H), 4.63 (d, J = 15.7 Hz, 1H), 4.29 (d, J = 15.7 Hz, 1H), 3.34 (m, 2H), 2.52 (s, 3H), 2.18 (dd, J = 12.7, 3.4 Hz, 1H), 2.09 (dd, J = 12.7, 3.4 Hz, 1H), 1.57 (s, 6H); ¹³C NMR (125 MHz, C₆D₆) δ 177.6, 177.3, 143.7, 143.0, 136.3, 129.5, 129.4, 129.3, 129.1, 128.9, 128.0, 127.9, 125.4, 125.1, 123.0, 122.8, 110.3, 109.3, 108.1, 74.7, 74.6, 53.7, 53.3, 43.9, 34.1, 33.6, 32.3, 28.0, 25.7, 23.4, 14.7; IR (film) 2980, 1698, 1610, 1370, 1073, 841, 752 cm⁻¹; HRMS (EI) m/z calcd for C₃₁H₃₀N₂O₄ (M⁺) 494.2206, found 494.2206.
Trifluoromethanesulfonic acid 2-(methyl{4-[methyl(toluene-4-sulfonyl)amino]-but-2-ynonyl}amino)phenyl ester (16). Diisopropylethylamine (5.0 mL, 28.7 mmol) and trimethylsilyl trifluoromethanesulfonate (TMSOTf) (5.0 mL, 26.1 mmol) were added sequentially to an Et₂O solution of N-methylpropargylamine (75 mL, 0.35 M) at 0 °C. A colorless precipitate formed immediately upon addition of TMSOTf. This mixture was stirred at 0 °C for 10 min, then allowed to warm to rt. The reaction mixture was filtered through a Schlenk filter to give a clear yellow solution of the crude N-methyl-N-trimethysilylpropargylamine. This solution was cooled to −78 °C and a hexane solution of n-BuLi (12.5 mL, 2.6 M) was added. After 10 min, 3-methylbenzoxazolinone (3.89 g, 26.1 mmol) was added as a solid under a positive flow of N₂, followed by THF (30 mL). The resulting solution was warmed to −20 °C, and maintained at this temperature for 2 h at which time N-phenylbis(trifluoromethanesulfonimide) (9.32 g, 26.1 mmol) was added as a solid under a positive flow of N₂. This solution was warmed to −10 °C, and after 30 min at −10 °C, p-toluenesulfonyl chloride (4.98 g, 26.1 mmol) and silica gel (3 g) were added. This mixture was allowed to warm to rt with stirring. After 6 h,
additional silica gel was added and the mixture was concentrated in vacuo and the residue was loaded onto a silica gel column and eluted with (3:1 hexanes–EtOAc–1:2 hexanes–EtOAc) to yield 4.66 g (41%) of 16 as a yellow oil: \(^1\)H NMR (500 MHz, CDCl\(_3\)) a mixture of rotamers (see accompanying spectra), only major peaks are listed: 7.75 (d, \(J = 8.3\) Hz, 2H), 7.47 (dt, \(J = 7.6, 1.8\) Hz, 1H), 7.44–7.40 (m, 2H), 7.37 (dd, \(J = 8.0, 1.5\) Hz, 1H), 7.32 (d, \(J = 8.0, 2\) H), 7.28 (dd, \(J = 6.0, 1.6\) Hz, 1H), 3.99 (d, \(J = 18.1\) Hz, 1H), 3.77 (d, \(J = 18.1\) Hz, 1H), 2.45 (s, 3H), 2.44 (s, 3H), 2.38 (s, 3H); \(^{13}\)C NMR (125 MHz, CDCl\(_3\)) a mixture of rotamers (see accompanying spectra), only major peaks are listed: 152.8, 145.2, 144.0, 135.42, 130.6, 130.3, 129.7, 129.3, 127.6, 122.6, 117.1, 84.8, 78.3, 39.6, 35.7, 33.9, 21.4; IR (film) 3069, 2247, 1659, 1498, 1351, 1212, 1162, 1139 cm\(^{-1}\); HRMS (ESI) \(m/z\) calcd for \(C_{20}H_{19}F_3N_2O_6S_2 (M + Na^+)\) 527.0534, found 527.0540.

![Diagram](image.png)

**Trifluoromethanesulfonic acid 2-{methyl[4-{methyl-(toluene-4-sulfonyl)amine]-2-(tributylstannyl)but-2E-enoyl]amino}phenyl ester (13).** Degassed (sparged with Ar for 1 h) THF (80 mL) was added to a flask charged with 16 (4.66 g, 9.24 mmol) and the resulting solution was cooled to 0 °C. Under positive Ar flow, Pd(PPh\(_3\))\(_4\) (0.39 g, 0.37 mmol) was added to this solution. A THF
solution of Bu₃SnH (10.0 mL, 0.93 M) then was added dropwise at 0 °C over 20 min using a syringe pump. After 2 h, the solution was concentrated and the residue was purified on silica gel using gradient elution (3:1 hexanes–EtOAc–2:1 hexanes–EtOAc) to yield 6.44 g (88%) of 13 as a colorless oil: ¹H NMR (500 MHz, CDCl₃) a ~1:1 mixture of rotamers (see accompanying spectra), only major peaks are listed [7.71 (dd, J = 8.2, 6.6 Hz), 7.46–7.33 (m), 7.30–7.27 (m), 5.74 (ddd, J_{Sn-H} = 21.5 Hz, J = 6.5, 6.5 Hz), 3.86 (m), 3.29 (s), 3.28 (s), 2.76 (s), 2.73 (s), 2.46 (s), 2.45 (s), 1.81 (s), 1.65–1.51 (m), 1.40 (t, J = 7.3 Hz), 1.35 (t, J = 7.3 Hz), 1.28 (t, J = 7.3 Hz), 1.24 (t, J = 7.3 Hz), 1.10 (tt, J_{Sn-H} = 26.2 Hz, J = 8.3 Hz), 0.94 (t, J = 7.3 Hz), 0.89 (t, J = 7.3 Hz); ¹³C NMR (125 MHz, CDCl₃) a ~1:1 mixture of rotamers (see accompanying spectra), only major peaks are listed [172.6, 172.3, 145.7, 143.2, 136.6, 134.6, 129.6, 127.4, 121.9, 119.6, 117.1, 53.4, 50.9, 38.4, 34.6, 34.4, 28.7(t), 28.5(t), 27.1(d), 21.3, 13.5, 10.7(d); IR (film) 2926, 1640, 1602, 1424, 1212, 1162 cm⁻¹; HRMS (ESI) m/z calcd for C₃₂H₄₂F₉N₂O₆SnS₂ (M + H⁺) 797.1932, found 797.1923, calcd for (M + Na⁺) 819.1751, found 819.1761.
Heck cyclization of Z-butenanilide 5 to form oxindoles 4 and 14. A sealed tube was charged with 5 (0.162 g, 0.188 mmol), Pd(OAc)$_2$ (0.004 g, 0.019 mmol) and (S)-Tol-BINAP (0.026 g, 0.038 mmol), followed by CH$_3$CN (2 mL, previously sparged with Ar for 3 h) and 1,2,2,6,6-pentamethylpiperidine (0.14 mL, 0.75 mmol). The solution was sparged with Ar for 15 min until the solution was a deep red color. The reaction vessel was sealed and heated to 80 °C for 18 h. After cooling to rt, the reaction solution was partitioned between saturated aqueous NaCN (10 mL) and EtOAc (15 mL). The layers were separated and the aqueous layer was extracted with EtOAc (2 ¥ 10 mL) and CH$_2$Cl$_2$ (2 ¥ 10 mL). The combined organic extracts were dried (MgSO$_4$), filtered, and concentrated in vacuo. The residue was purified on silica gel using gradient elution (10:1:0–10:1:0.05 CH$_2$Cl$_2$–MeOH–NH$_4$OH) to yield 0.130 g (97%) of 4 and 14 as a 6:1 mixture of diastereomers as determined by $^1$H NMR analysis. An analytical sample of 4 was obtained by reverse-phase HPLC: Zorbax Extend-C18 5 µm, 150 ¥ 21.2 mm, 70:30 CH$_3$CN–H$_2$O (1% NH$_4$OH), 16 mL/min, UV detection at 254 nm; [α]$_{254}$ $^27$405 –472, [α]$_{254}$ $^27$435 –353, [α]$_{254}$ $^27$546 –165,
$[\text{I}]^{27}_{577} -141, \; [\text{II}]^{27}_{0} -133 \; (c \; 0.64, \; \text{MeOH}); \; ^1\text{H} \; \text{NMR} \; (500 \; \text{MHz}, \; (\text{CD}_3)_2\text{SO}, \; 360 \; \text{K}) \; \delta \; 7.52 \; (d, \; J = 8.3 \; \text{Hz}, \; 2H), \; 7.46 \; (ddd, \; J = 7.7, 7.7, 1.3 \; \text{Hz}, \; 1H), \; 7.43 \; (d, \; J = 8.0 \; \text{Hz}, \; 2H), \; 7.21 \; (t, \; J = 7.5 \; \text{Hz}, \; 1H), \; 7.17 \; (d, \; J = 7.9 \; \text{Hz}, \; 1H), \; 7.01 \; (d, \; J = 7.9 \; \text{Hz}, \; 1H), \; 6.91 \; (d, \; J = 7.6 \; \text{Hz}, \; 1H), \; 6.87 \; (t, \; J = 7.6 \; \text{Hz}, \; 1H), \; 6.74 \; (br \; d, \; J = 7.2 \; \text{Hz}, \; 1H), \; 6.66 \; (d, \; J = 7.2 \; \text{Hz}, \; 1H), \; 6.60 \; (d, \; J = 14.3 \; \text{Hz}, \; 1H), \; 6.45 \; (t, \; J = 7.6 \; \text{Hz}, \; 1H), \; 6.34 \; (t, \; J = 7.4 \; \text{Hz}, \; 1H), \; 6.22 \; (d, \; J = 7.8 \; \text{Hz}, \; 1H), \; 5.37 \; (d, \; J = 14.3 \; \text{Hz}, \; 1H), \; 4.45 \; (br \; s, \; 1H), \; 4.35 \; (br \; s, \; 1H), \; 4.19 \; (br \; d, \; J = 3.6 \; \text{Hz}, \; 1H), \; 3.24 \; (s, \; 3H), \; 2.91 \; (s, \; 3H), \; 2.89 \; (s, \; 3H), \; 2.60-2.52 \; (m, \; 2H), \; 2.44 \; (s, \; 3H), \; 2.42-2.36 \; (m, \; 2H), \; 2.35 \; (s, \; 3H), \; 2.32-2.26 \; (m, \; 1H), \; 2.22-2.17 \; (m, \; 1H), \; 2.15 \; (s, \; 3H), \; 1.89-1.85 \; (m, \; 1H), \; 1.80 \; (ddd, \; J = 11.8, 5.6, 2.4 \; \text{Hz}, \; 1H); \; ^{13}\text{C} \; \text{NMR} \; (125 \; \text{MHz}, \; \text{(CD}_3)_2\text{SO}, \; 360 \; \text{K}) \; \delta \; 176.0, \; 152.3, \; 149.1, \; 143.5, \; 142.4, \; 134.8, \; 133.6, \; 132.6, \; 130.0, \; 129.41, \; 129.38, \; 128.1, \; 126.9, \; 126.1, \; 125.7, \; 124.3, \; 122.5, \; 122.1, \; 122.0, \; 117.2, \; 115.9, \; 110.1, \; 108.5, \; 104.9, \; 91.0, \; 82.8, \; 61.96, \; 61.92, \; 55.5, \; 51.1, \; 50.7, \; 36.9, \; 34.7, \; 34.63, \; 34.59, \; 31.8, \; 25.7, \; 20.4; \; \text{IR} \; (\text{film}) \; 3385, \; 2930, \; 1702, \; 1602, \; 1459, \; 1351, \; 1254, \; 1158 \; \text{cm}^{-1}; \; \text{HRMS} \; (\text{ESI}) \; m/z \; \text{calcd} \; \text{for} \; \text{C}_{42}\text{H}_{46}\text{N}_6\text{O}_3\text{S} \; (\text{M} + \text{H}^+) \; 715.3430, \; \text{found} \; 715.3421.

An analytical sample of 14 was obtained by reverse-phase HPLC: Zorbax Extend-C18 5 mm, 150 × 21.2 mm, 70:30 \text{CH}_3\text{CN:}\text{H}_2\text{O} (1\% \text{NH}_4\text{OH}), \; 16 \; \text{mL/min}, \; \text{UV} \; \text{detection} \; \text{at} \; 254 \; \text{nm}) \; [\text{I}]^{27}_{405} -587, \; [\text{II}]^{27}_{435} -438, \; [\text{III}]^{27}_{546} -203, \; [\text{IV}]^{27}_{577} -174, \; [\text{V}]^{27}_{577} -165 \; (c \; 0.51, \; \text{CH}_2\text{Cl}_2); \; ^1\text{H} \; \text{NMR} \; (500 \; \text{MHz}, \; \text{(CD}_3)_2\text{SO}, \; 360 \; \text{K}) \; \delta \; 7.57 \; (d, \; J = 8.3 \; \text{Hz}, \; 2H), \; 7.45
(d, J = 7.9 Hz, 2H), 7.43–7.38 (m, 2H), 7.20–7.13 (m, 3H), 7.05 (d, J = 7.2 Hz, 1H), 6.93 (t, J = 7.5 Hz, 1H), 6.64 (d, J = 14.3 Hz, 1H), 6.62 (d, J = 6.8 Hz, 1H), 6.48 (t, J = 7.5 Hz, 2H), 6.30 (d, J = 7.7 Hz, 1H), 5.35 (d, J = 14.3 Hz, 1H), 4.90 (s, 1H), 4.27 (s, 1H), 4.16 (s, 1H), 3.25 (s, 3H), 2.93 (s, 3H), 2.92 (s, 3H), 2.44 (s, 3H), 2.43–2.38 (m, 4H), 2.35–2.31 (m, 1H), 2.30 (s, 3H), 2.24–2.19 (m, 1H), 1.95 (s, 3H), 1.90–1.84 (m, 2H); \(^{13}\)C NMR (125 MHz, (CD\(_3\))\(_2\)SO, 360 K) \[ 176.1, 152.3, 149.0, 143.6, 142.3, 134.7, 133.7, 132.4, 129.45, 128.0, 127.2, 126.1, 125.8, 124.3, 123.1, 122.9, 122.0, 119.2, 116.9, 115.9, 110.3, 108.5, 105.1, 91.0, 84.0, 61.6, 55.8, 51.4, 51.3, 37.1, 35.4, 34.8, 34.5, 31.8, 25.8, 20.4; IR (film) 3385, 2930, 1702, 1602, 1459, 1351, 1254, 1158 cm\(^{-1}\); HRMS (ESI) m/z calcd for C\(_{42}\)H\(_{46}\)N\(_6\)O\(_3\)S (M + H\(^+\)) 715.3430, found 715.3428.
Part B. Scheme Showing Reagents and Yields for Each Step in the Elaboration of 10 to 11.

Scheme 5. Reaction conditions: a) (±)-10-camphorsulfonic acid, MeOH, CH₂Cl₂, rt; b) NaIO₄, THF, H₂O, rt; c) NaBH₄, MeOH, rt (85% 3 steps); d) Red-Al, THF, 67 °C; e) HN₃, DEAD, PPh₃, THF, 0 °C (75% 2 steps); f) PPh₃, THF, H₂O, rt; g) MeOH, 110 °C; h) CH₂O, NaBH(OAc)₃, MeOH, rt (76% 3 steps).
Part C. Characterization Data for Selected Compounds

8-Benzyl-1,8,1-trimethyl-2,3,8,8aR,2a3a8aR-octahydro-1H,1H-[3aS,3aS]bipyrrrolo[2,3-b]indole (11): ¹H NMR (500 MHz, (CD₃)₂SO, 353 K) δ 7.42–7.36 (m, 4H), 7.29 (t, J = 6.9 Hz, 1H), 7.06 (d, J = 7.2 Hz, 1H), 7.01 (d, J = 7.3 Hz, 1H), 6.95 (dt, J = 6.7, 1.1 Hz, 1H), 6.88 (dt, J = 6.8, 1.1 Hz, 1H), 6.55–6.47 (dd, J = 7.4, 7.4 Hz, 1H and dd, J = 7.4, 7.4 Hz, 1H), 6.29 (d, J = 7.8 Hz, 1H), 6.20 (d, J = 7.8 Hz, 1H), 4.57 (d, J = 16.3 Hz, 1H), 4.50 (s, 1H), 4.44 (d, J = 16.3 Hz, 1H), 4.39 (s, 1H), 2.95 (s, 3H), 2.68–2.60 (m, 2H), 2.57–2.45 (m, 3H), 2.41–2.35 (m, 1H and s, 3H), 2.23 (s, 3H), 2.01–1.90 (m, 2H); ¹³C NMR (125 MHz, (CD₃)₂SO, 353 K) δ 152.2, 151.6, 138.8, 132.3, 132.2, 127.7, 127.3, 127.1, 126.8, 126.1, 123.1, 123.0, 116.1, 115.8, 105.4, 104.9, 91.4, 91.3, 62.2, 61.9, 51.8, 51.7, 51.1, 38.6, 37.6, 34.7, 34.6, 33.9; IR (film) 3049, 2930, 2795, 1602, 1490, 1351, 1258, 1158, 1027 cm⁻¹; HRMS (ESI) m/z calcd for C₃₀H₄₄N₄ (M + H⁺) 451.2862, found 451.2869.
Z-butenanilide 5. $^1$H NMR (500 MHz, CD$_2$Cl$_2$, 200 K) a mixture of rotamers$^3$ (see accompanying spectra), only major peaks are listed [7.59 (t, $J = 9.7$ Hz, 2H), 7.37 (t, $J = 9.3$ Hz, 2H), 6.28 (d, $J = 7.6$ Hz, 0.5H), 6.12 (d, $J = 7.7$ Hz, 0.5H), 5.86 (t, $J = 7.6$ Hz, 0.3H), 5.62 (d, $J = 7.4$ Hz, 0.3H), 4.85 (br s, 1H), 4.77 (br s, 1H), 4.67 (br s, 0.5H), 4.61 (br s, 0.5H), 3.21 (s, 1.3H), 3.07 (s, 2H), 2.95 (s, 2H), 2.91 (s, 1H), 2.58 (s, 1H), 2.54 (s, 2H), 2.45–2.38 (m, 9H), 2.31 (s, 2H); $^{13}$C NMR (125 MHz, CD$_2$Cl$_2$) a mixture of rotamers (see attached spectra), only major peaks are listed [169.4, 147.1, 144.7, 143.9, 134.5, 131.2, 129.6, 127.4, 121.9, 117.2, 110.8, 110.8, 105.5, 90.5 (br), 84.5 (br), 62.1, 49.0, 36.9, 34.6, 21.4; IR (film) 3416, 2937, 1648, 1602, 1498, 1216, 1162, 1139 cm$^{-1}$; HRMS (ESI) m/z calcd for C$_{43}$H$_{47}$F$_3$N$_6$O$_6$S$_2$ (M + H$^+$) 887.2849, found 887.2876.

$^3$Attempts to resolve peaks in the $^1$H NMR at high temperature led to extensive decomposition.
Part D. Copies of $^1$H and $^{13}$C NMR Spectra for New Compounds
Part E. Copies of low temperature (233 K) $^1$H and $^{13}$C NMR Spectra for synthetic idiospermuline, 3a$\[ $8a$\]$-bis-
epi idiospermuline, and natural idiospermuline
Part F. Copies of CD Spectra for synthetic idiospermuline, 3a\[\text{H} \] 8a\[\text{H} \] bis-epiidiospermuline, and natural idiospermuline
Part G. Copies of HPLC traces for synthetic idiospermuline and natural idiospermuline, including co-injection
**synthetic idiospermuline, 80:20 MeOH:H2O (1%NH4OH), 1 mL/min**

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**Results obtained with enhanced integrator!**

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**End of Report**

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**Page 1 of 1**
natural idiospermuline, 80:20 MeOH:H2O (1%NH4OH), 1mL/m

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<td>3.6237</td>
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<td>7.578</td>
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<td>2.4659</td>
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<td>0.5636</td>
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<td>BB</td>
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<td>12.109</td>
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<td>0.5465</td>
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<td>1.72012</td>
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<td>VB</td>
<td>0.5714</td>
<td>3552.86743</td>
<td>91.10531</td>
<td>73.6393</td>
</tr>
</tbody>
</table>

Totals: 4284.69046 127.76098

Achiral 2/10/03 8:05:34 PM emily  Page 1 of 2
co-injection of synthetic and natural idioperumline, 8
0:20 MeOH:H2O (18NH4OH), 1 mL/min

Injection Date : 2/7/03 2:17:21 PM
Sample Name : SYN + NAT
Acq. Operator : emily
Inj Volume : 5 μl

Analysis Method : C:\HPchem2\METHODS\DAW4176.M
Last changed : 2/10/03 8:01:18 PM by emily
(modified after loading)

VWD1 A, Wavelength=254 nm (PETERTON\0207034.D)

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type Width Area Height Area
# [min] [min] mAU *s [mAU] |
---|-------|--------|--------|--------|
1 1.758 BV 0.0890 36.21000 5.45855 0.9131
2 1.909 VV 0.1171 107.07786 11.84978 2.7000
3 10.099 PV 0.7765 311.11539 5.25576 7.8449
4 15.426 VB 0.5721 3511.40845 89.05025 86.5420

Totals : 3965.81170 111.61434

Results obtained with enhanced integrator!

*** End of Report ***