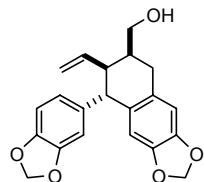


Novel, Stereoselective and Convergent Synthesis of Aryltetralins

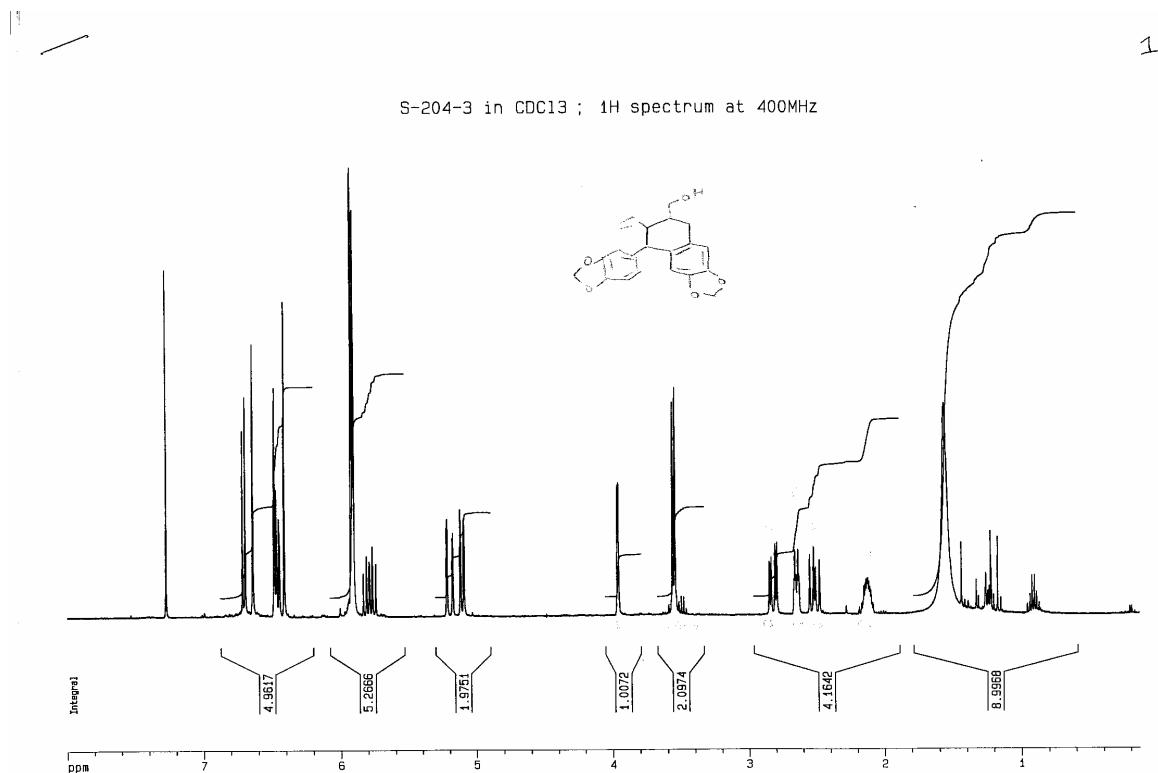
Steven M. Miles, Stephen P. Marsden^{*} Robin J. Leatherbarrow and William J. Coates

Supplementary information: Compound data and nmr spectra for compounds **7**

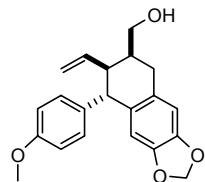
[(6*S*,7*RS*,8*SR*)-8-Benzo[1,3]dioxol-5-yl-7-vinyl-5,6,7,8-tetrahydro-naphtho[2,3-*d*][1,3]dioxol-6-yl]-methanol (Table 1, entry 1)



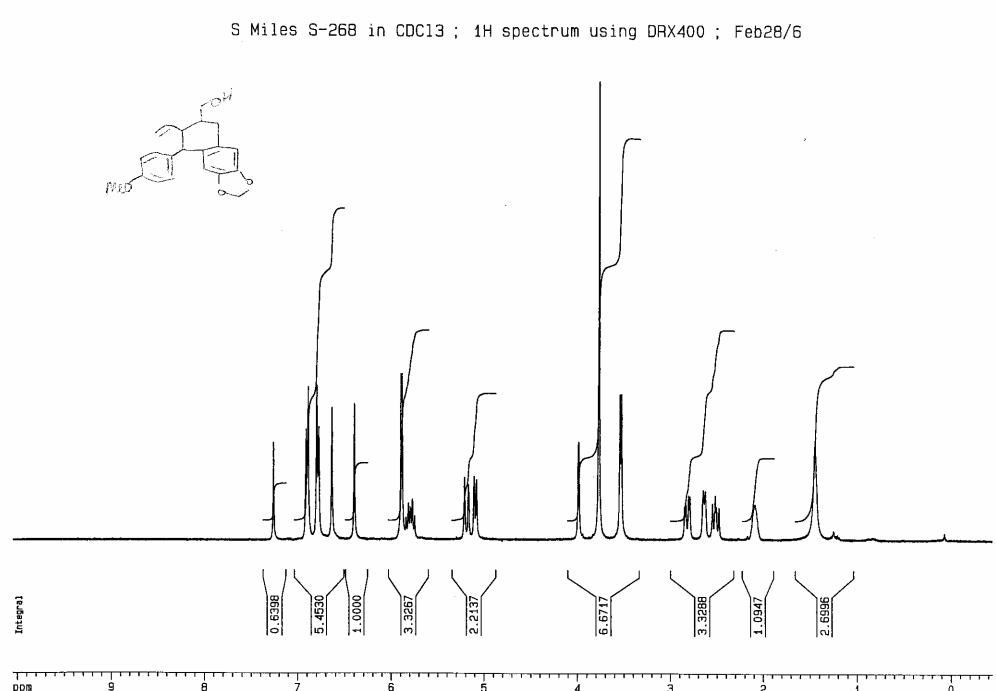
mp 60-61 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.13 (1H, m, 6-H), 2.51 (1H, dd, J 17.0, 11.5, 5-H_aH), 2.65 (1H, ddd, J 9.5, 3.0, 2.5, 7-H), 2.82 (1H, dd, J 17.0, 5.5, 5-HH_b), 3.55 (2H, d, J 7.0, CH_2OH), 3.96 (1H, d, J 2.5, 8-H), 5.10 (1H, dd, J 10.0, 2.0, $\text{CH}=\text{CH}_a\text{H}$, *cis* coupled), 5.20 (1H, ddd, J 17.0, 2.0, 0.5, $\text{CH}=\text{CH}\text{H}_b$, *trans* coupled), 5.78 (1H, ddd, J 17.0, 10.0, 9.5, $\text{CH}=\text{CH}_2$), 5.90 (1H, d, J 1.5, OCH_aHO), 5.91 (1H, d, J 1.5, OCHH_bO), 5.91 (1H, d, J 1.5, 2'-H_aH), 5.92 (1H, d, J 1.5, 2'-HH_b), 6.41 (1H, s, 1-H), 6.45 (1H, dd, J 8.0, 1.5, 6'-H), 6.48 (1H, d, J 1.5, 4'-H), 6.64 (1H, s, 4-H), 6.70 (1H, d, J 8.0, 7'-H); ^{13}C NMR (100 MHz, CDCl_3) δ 28.9 (t), 34.3 (d), 47.8 (d), 50.3 (d), 65.6 (t), 100.7 (t), 100.9 (t), 107.8 (d), 108.2 (d), 109.4 (d), 110.2 (d), 116.8 (t), 122.0 (d), 129.0 (s), 129.3 (s), 138.2 (d), 140.3 (s), 145.8 (s), 146.1 (s), 146.4 (s), 147.4 (s); Anal. found C 71.51, H 5.60, $\text{C}_{21}\text{H}_{20}\text{O}_5$ requires C 71.58, H 5.72.



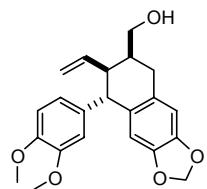
[(6SR,7RS,8SR)-8-(4-Methoxy-phenyl)-7-vinyl-5,6,7,8-tetrahydro-naphtho[2,3-d][1,3]dioxol-6-yl]-methanol (Table 1, Entry 2)



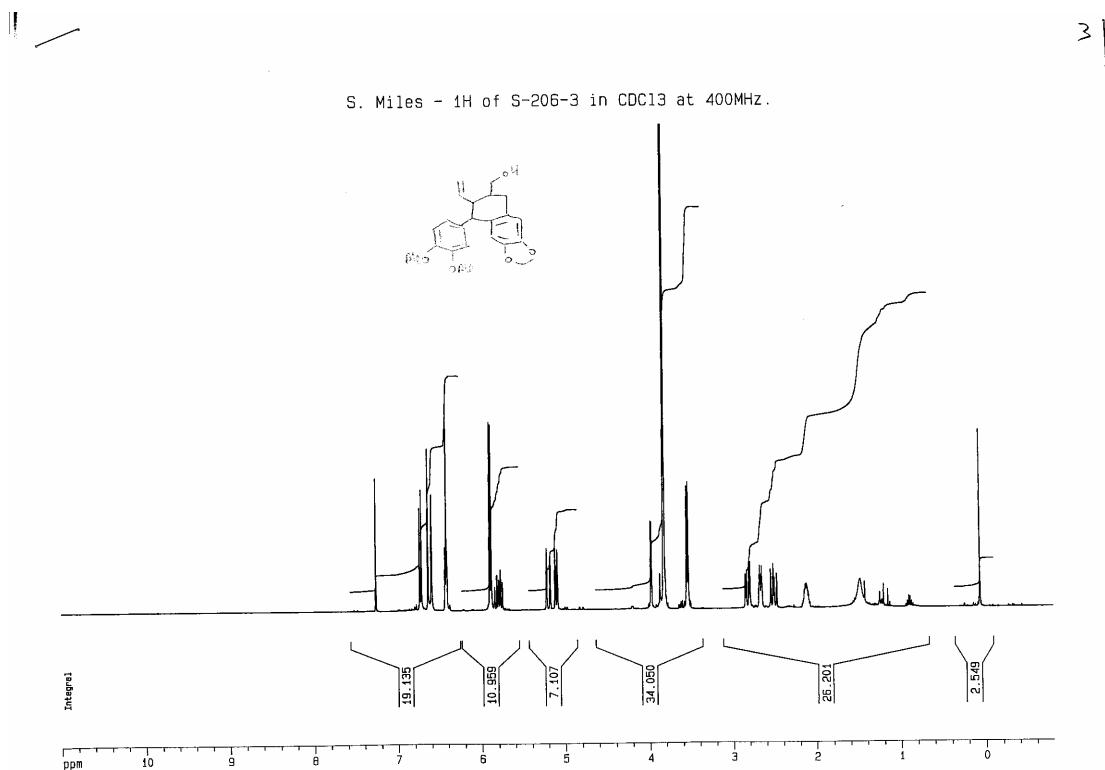
mp 96-97 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.10 (1H, m, 6-H), 2.52 (1H, dd, J 17.0, 11.5, 5-H_a), 2.64 (1H, ddd, J 9.0, 3.0, 2.5, 7-H), 2.82 (1H, dd, J 17.0, 5.5, 5-H_b), 3.53 (2H, d, J 6.5, $\text{CH}_2\text{-OH}$), 3.77 (3H, s, OCH_3), 3.98 (1H, d, J 2.5, 8-H), 5.10 (1H, dd, J 10.0, 1.5, $\text{CH}=\text{CH}_a\text{H}$, *cis* to $\text{CH}=\text{CH}_2$), 5.19 (1H, dd, J 17.0, 1.5, $\text{CH}=\text{CH}\text{H}_b$, *trans* to $\text{CH}=\text{CH}_2$), 5.79 (1H, ddd, J 17.0, 10.0, 9.0, $\text{CH}=\text{CH}_2$), 5.89 (1H, s, OCH_aHO), 5.89 (1H, s, $\text{OCH}\text{H}_b\text{O}$), 6.40 (1H, s, 1-H), 6.64 (1H, s, 4-H), 6.79 (2H, d, J 8.5, 5'-H & 3'-H), 6.90 (2H, d, J 8.5, 2'-H & 6'-H); ^{13}C NMR (125 MHz, CDCl_3) δ 29.1 (t), 34.4 (d), 47.9 (d), 50.0 (d), 55.3 (q), 65.8 (t), 100.8 (t), 108.3 (d), 110.3 (d), 113.6 (d), 116.8 (t), 129.1 (s), 129.6 (s), 129.9 (d), 138.5 (s), 138.5 (d), 146.2 (s), 146.4 (s), 158.0 (s); Anal. found C 74.42, H 6.58, $\text{C}_{21}\text{H}_{22}\text{O}_4$ requires C 74.54, H, 6.55.



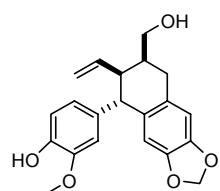
[(6*S*,7*RS*,8*SR*)-8-(3,4-Dimethoxy-phenyl)-7-vinyl-5,6,7,8-tetrahydro-naphtho[2,3-*d*][1,3]dioxol-6-yl]-methanol (Table 1, Entry 3)



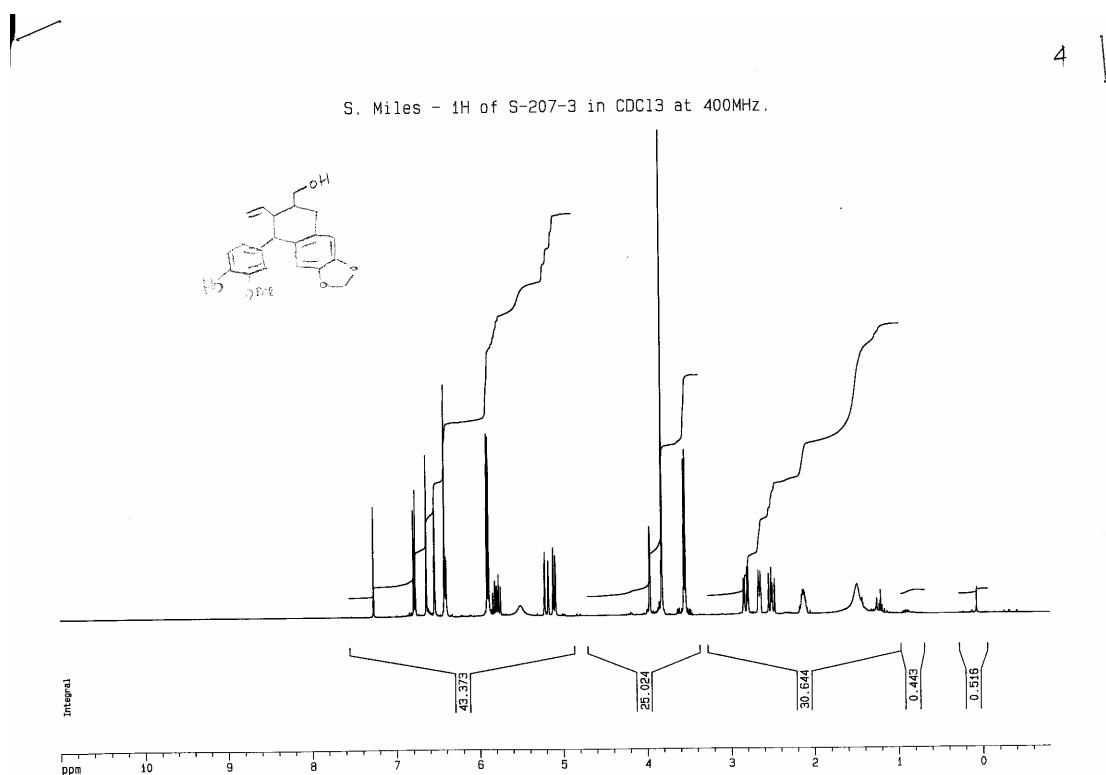
mp 64-66 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.12 (1H, m, 6-H), 2.51 (1H, dd, J 17.0, 11.5, 5-H_aH), 2.67 (1H, ddd, J 9.5, 3.0, 2.5, 7-H), 2.82 (1H, dd, J 17.0, 5.5, 5-HH_b), 3.54 (2H, d, J 7.0, CH_2OH), 3.82 (3H, s, OCH₃), 3.83 (3H, s, OCH₃), 3.97 (1H, d, J 2.5, 8-H), 5.10 (1H, dd, J 10.0, 2.0, CH=CH_aH, *cis* coupled), 5.20 (1H, dd, J 17.0, 1.5, CH=CHH_b, *trans* coupled), 5.79 (1H, ddd, J 17.0, 10.0, 9.5, CH=CH₂), 5.88 (1H, d, J 1.0, OCH_aHO), 5.90 (1H, d, J 1.0, OCHH_bO), 6.41 (1H, s, 1-H), 6.42 (1H, dd, J 8.0, 2.0, 6'-H), 6.59 (1H, d, J 2.0, 2'-H), 6.63 (1H, s, 4-H), 6.72 (1H, d, J 8.0, 5'-H); ^{13}C NMR (75 MHz, CDCl_3) δ 29.0 (t), 34.5 (d), 47.6 (d), 50.2 (d), 55.9 (q), 56.0 (q), 65.6 (t), 100.7 (t), 108.2 (d), 110.3 (d), 110.8 (d), 112.2 (d), 116.8 (t), 121.2 (d), 129.1 (s), 129.5 (s), 138.4 (d), 138.9 (s), 146.1 (s), 146.3 (s), 147.4 (s), 148.7 (s); Anal. found C 71.63, H 6.42, C₂₂H₂₄O₅ requires C 71.72, H 6.57.



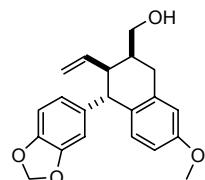
4-[(5SR,6RS,7SR)-7-Hydroxymethyl-6-vinyl-5,6,7,8-tetrahydro-naphtho[2,3-d][1,3]dioxol-5-yl]-2-methoxy-phenol (Table 1, Entry 4)



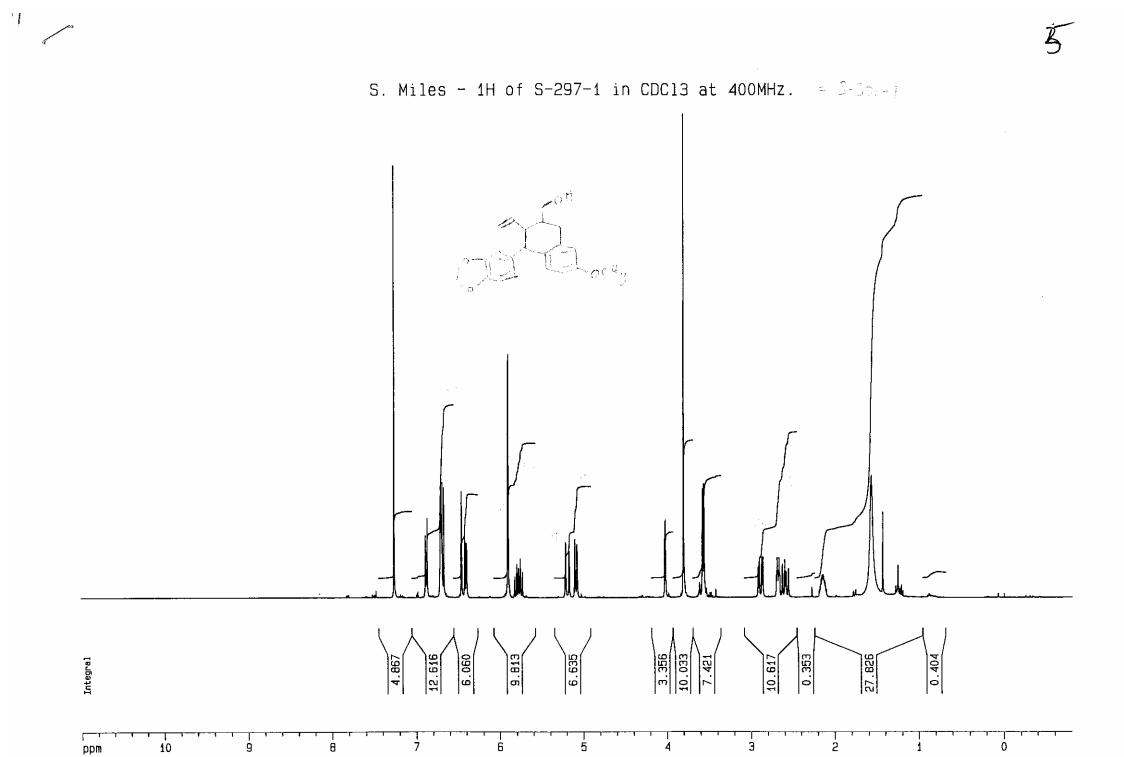
mp 170-171 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.13 (1H, m, 7'-H), 2.51 (1H, dd, J 17.0, 11.5, 8'- H_a H), 2.66 (1H, ddd, J 9.5, 3.0, 2.5, 6'-H), 2.82 (1H, dd, J 17.0, 5.5, 8'- H_b), 3.54 (2H, d, J 7.0, CH_2OH), 3.82 (3H, s, OCH_3), 3.96 (1H, d, J 2.5, 5'-H), 5.10 (1H, dd, J 10.5, 2.0, $\text{CH}=\text{CH}_\text{a}$ H, *cis* coupled), 5.19 (1H, dd, J 17.0, 1.5, $\text{CH}=\text{CH}_\text{b}$, *trans* coupled), 5.51 (1H, br s, Ar-OH), 5.79 (1H, ddd, J 17.0, 10.0, 9.5, $\text{CH}=\text{CH}_2$), 5.88 (1H, d, J 1.5, OCH_a HO), 5.90 (1H, d, J 1.5, OCH_b O), 6.41 (1H, dd, J 8.0, 2.0, 5-H), 6.41 (1H, s, 4'-H), 6.53 (1H, d, J 2.0, 3-H), 6.63 (1H, s, 1'-H), 6.78 (1H, d, J 8.0, 6-H); ^{13}C NMR (100 MHz, CDCl_3) δ 29.0 (t), 34.4 (d), 47.7 (d), 50.3 (d), 55.9 (q), 65.6 (t), 100.7 (t), 108.2 (d), 110.2 (d), 111.3 (d), 113.8 (d), 116.8 (t), 121.8 (d), 129.0 (s), 129.4 (s), 138.2 (s), 138.4 (d), 143.9 (s), 146.1 (s), 146.2 (s), 146.3 (s); Anal. found C 70.98, H 6.13, $\text{C}_{21}\text{H}_{22}\text{O}_5$ requires C 71.17, H 6.26.



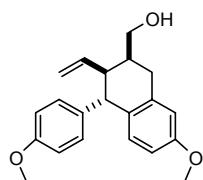
[(2S_R,3R_S,4S_R)-4-Benzo[1,3]dioxol-5-yl-7-methoxy-3-vinyl-1,2,3,4-tetrahydro-naphthalen-2-yl]-methanol (Table 1, Entry 5)



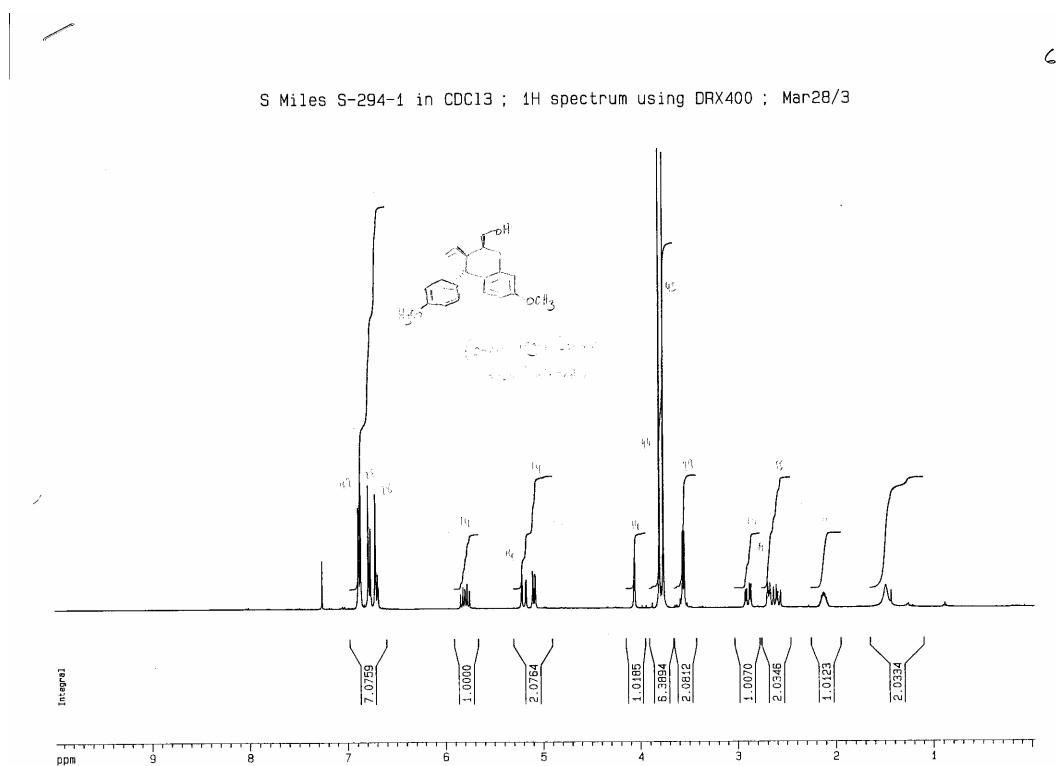
mp 43–45 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.14 (1H, m, 2-H), 2.59 (1H, dd, J 17.0, 11.5, 1-H_aH), 2.68 (1H, ddd, J 9.5, 3.0, 2.5, 3-H), 2.89 (1H, dd, J 17.0, 5.5, 1-HH_b), 3.57 (2H, dd, J 6.5, 1.5, CH₂OH), 3.80 (3H, s, OCH₃), 4.02 (1H, d, J 2.5, 4-H), 5.09 (1H, dd, J 10.0, 2.0, CH=CH_aH, *cis* coupled), 5.19 (1H, dd, J 17.0, 2.0, CH=CHH_b, *trans* coupled), 5.78 (1H, ddd, J 17.0, 10.0, 9.5, CH=CH₂), 5.90 (1H, d, J 1.5, 2'-H_aH), 5.91 (1H, d, J 1.5, 2'-HH_b), 6.41 (1H, dd, J 8.0, 1.5, 6'-H), 6.46 (1H, d, J 1.5, 4'-H), 6.68 (1H, d, J 8.0, 7'-H), 6.71 (1H, dd, J 9.0, 1.5, 6-H), 6.71 (1H, d, J 1.5, 8-H), 6.88 (1H, d, J 9.0, 5-H); ^{13}C NMR (75 MHz, CDCl_3) δ 29.2 (t), 34.4 (d), 48.1 (d), 49.5 (d), 55.3 (q), 65.7 (t), 100.9 (t), 107.9 (d), 109.4 (d), 112.9 (d), 113.2 (d), 116.8 (t), 122.0 (d), 128.8 (s), 132.1 (d), 137.2 (s), 138.4 (d), 140.8 (s), 145.8 (s), 147.5 (s), 158.2 (s); Anal. found C 74.47, H 6.67, $\text{C}_{21}\text{H}_{22}\text{O}_4$ requires C 74.54, H 6.55.



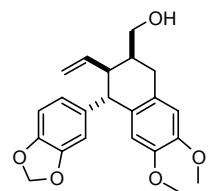
[(2SR,3RS,4SR)-7-Methoxy-4-(4-methoxy-phenyl)-3-vinyl-1,2,3,4-tetrahydro-naphthalen-2-yl]-methanol (Table 1, Entry 6)



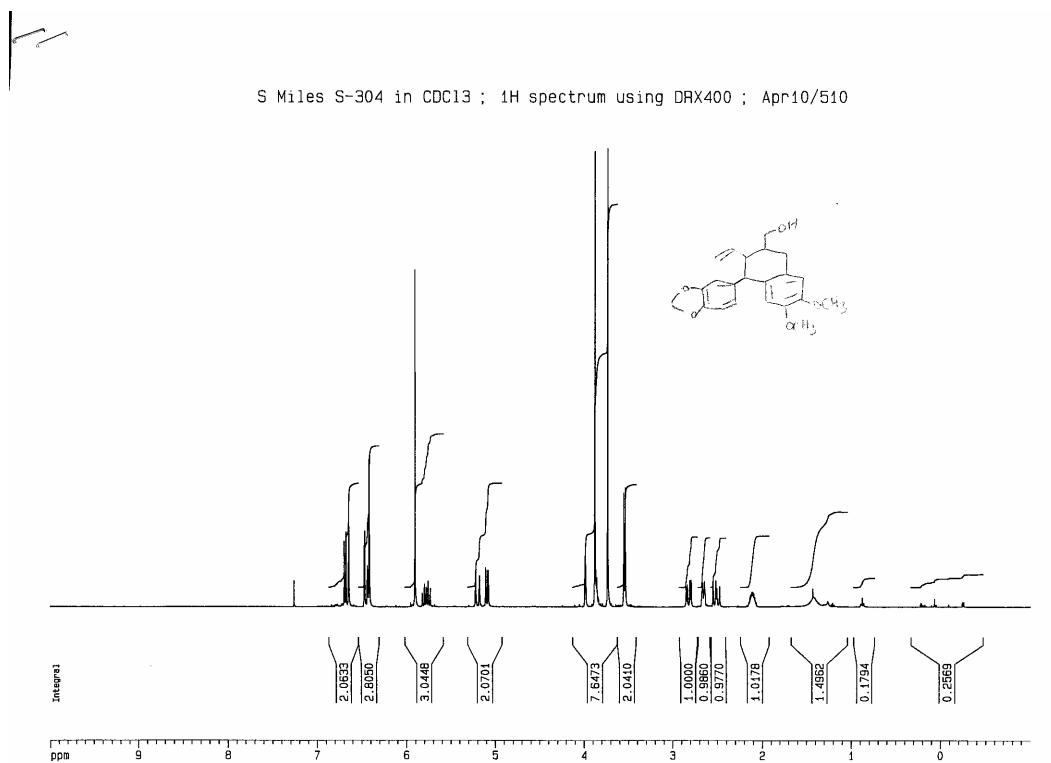
¹H NMR (400 MHz, CDCl₃) δ 2.12 (1H, m, 2-H), 2.60 (1H, dd, *J* 17.5, 11.5, 1-H_aH), 2.68 (1H, apparent dt, *J* 9.5, 3.0, 3-H), 2.89 (1H, dd, *J* 17.5, 5.5, 1-HH_b), 3.55 (2H, dd, *J* 7.0, 1.0, CH₂OH), 3.76 (3H, s, 4'-OCH₃), 3.80 (3H, s, 7-OCH₃), 4.06 (1H, d, *J* 3.0, 4-H), 5.09 (1H, dd, *J* 10.0, 2.0, CH=CH_aH *cis* coupled), 5.19 (1H, ddd, *J* 17.0, 2.0, 0.5, CH=CHH_b, *trans* coupled), 5.80 (1H, ddd, *J* 17.0, 10.0, 9.5, CH=CH₂), 6.70 (1H, dd, *J* 10.0, 2.5, 6-H), 6.72 (1H, d, *J* 2.5, 8-H), 6.77 (2H, d, *J* 9.0, 3'-H & 5'-H), 6.88 (2H, d, *J* 9.0, 2'-H & 6'-H), 6.88 (1H, d, *J* 10.0, 5-H); ¹³C NMR (75 MHz, CDCl₃) δ 29.2 (t), 34.3 (d), 48.0 (d), 49.0 (d), 55.2 (q), 55.3 (q), 65.6 (t), 112.8 (d), 113.2 (d), 113.5 (d), 116.7 (t), 129.0 (s), 129.8 (d), 132.0 (d), 137.2 (s), 138.5 (d), 138.9 (s), 157.8 (s), 157.9 (s); Anal. found C 78.00, H 7.36, C₂₁H₂₄O₃ requires C 77.75, H 7.46.



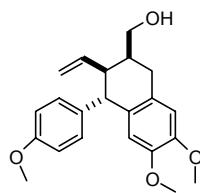
[(2SR,3RS,4SR)-4-Benzo[1,3]dioxol-5-yl-6,7-dimethoxy-3-vinyl-1,2,3,4-tetrahydro-naphthalen-2-yl]-methanol (Table 1, Entry 7)



mp 58-60 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.11 (1H, m, 2-H), 2.51 (1H, dd, J 17.0, 11.5, 1-H_aH), 2.65 (1H, ddd, J 9.5, 3.0, 2.5, 3-H), 2.82 (1H, dd, J 17.0, 5.5, 1-HH_b), 3.54 (2H, d, J 7.0, CH_2OH), 3.73 (3H, s, OCH₃), 3.88 (3H, s, OCH₃), 3.99 (1H, d, J 2.5, 4-H), 5.09 (1H, dd, J 10.0, 2.0, CH=CH_aH, *cis* coupled), 5.20 (1H, ddd, J 17.0, 2.0, 0.5, CH=CHH_b, *trans* coupled), 5.78 (1H, ddd, J 17.0, 10.0, 9.5, CH=CH₂), 5.90 (2H, s, 2'-H₂), 6.42 (1H, s, 5-H), 6.42 (1H, apparent ddd, J 8.0, 2.0, 0.5, 6'-H), 6.47 (1H, d, J 2.0, 4'-H), 6.64 (1H, s, 8-H), 6.68 (1H, d, J 8.0, 7'-H); ^{13}C NMR (100 MHz, CDCl_3) δ 28.4 (t), 34.3 (d), 48.1 (d), 50.1 (d), 55.8 (q, 2 coincident peaks), 65.8 (t), 100.9 (t), 107.9 (d), 109.5 (d), 111.1 (d), 113.1 (d), 116.9 (t), 122.0 (d), 128.0 (s), 128.1 (s), 138.4 (d), 140.5 (s), 145.8 (s), 147.5 (s), 147.6 (s), 147.7 (s) [22C expected, 21C seen]; Anal. found C 71.79, H 6.43, $\text{C}_{22}\text{H}_{24}\text{O}_5$ requires C 71.72, H 6.57.



[(2SR,3RS,4SR)-6,7-Dimethoxy-4-(4-methoxy-phenyl)-3-vinyl-1,2,3,4-tetrahydro-naphthalen-2-yl]-methanol (Table 1, Entry 8)



mp 54–56 °C; ^1H NMR (400 MHz, CDCl_3) δ 2.09 (1H, m, 2-H), 2.53 (1H, dd, J 17.0, 11.5, 1-H_aH), 2.67 (1H, ddd, J 9.5, 3.0, 2.5, 3-H), 2.83 (1H, dd, J 17.0, 5.5, 1-HH_b), 3.53 (2H, d, J 7.0, CH_2OH), 3.72 (3H, s, OCH₃), 3.76 (3H, s, OCH₃), 3.88 (3H, s, OCH₃), 4.02 (1H, d, J 2.5, 4-H), 5.09 (1H, dd, J 10.0, 2.0, CH=CH_aH, *cis* coupled), 5.20 (1H, ddd, J 17.0, 2.0, CH=CHH_b, *trans* coupled), 5.80 (1H, ddd, J 17.0, 10.0, 9.5, CH=CH₂), 6.42 (1H, s, 5-H), 6.66 (1H, s, 8-H), 6.78 (2H, d, J 8.5, 3'-H & 5'-H), 6.89 (2H, d, J 8.0, 2'-H & 6'-H); ^{13}C NMR (75 MHz, CDCl_3) δ 28.5 (t), 34.2 (d), 48.0 (d), 49.6 (d), 55.3 (q), 55.8 (q, 2 coincident peaks), 65.9 (t), 111.1 (d), 113.1 (d), 113.5 (d), 116.8 (t), 128.0 (s), 128.3 (s), 129.8 (d), 138.5 (d & s, 2 coincident peaks), 147.6 (s), 147.6 (s), 157.8 (s) [20C expected, 18C seen]; Anal. found C 74.57, H 7.40, $\text{C}_{22}\text{H}_{26}\text{O}_4$ requires C 74.55, H 7.39.

