

Concise Synthesis of the (\pm)- N_b -Desmethyl-*meso*-Chimonanthine

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N_b -Desmethyl-*meso*-chimonanthine (3): White foam; $R_f = 0.30$ ($\text{CH}_2\text{Cl}_2/\text{MeOH}/\text{NH}_4\text{OH}$ 9/1/1); IR (neat) 1602, 1483, 737 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 6.98-6.88 (m, 3H), 6.60-6.42 (m, 3H), 6.41 (d, $J = 7.9$ Hz, 1H), 6.40 (d, $J = 7.7$ Hz, 1H), 4.97 (s, 1H), 4.46 (s, 1H), 3.85 (s, 1H), 3.02 (dd, $J = 10.5, 6.6$ Hz, 1H), 2.74-2.70 (m, 1H), 2.66-2.63 (m, 1H), 2.44-2.38 (m, 1H), 2.30 (s, 3H), 2.30-2.20 (m, 2H), 2.10 (dd, $J = 11.7, 5.1$ Hz, 1H), 2.01 (dd, $J = 10.0, 1.8$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ 151.6 (2C), 131.5, 131.4, 128.1 (2C), 124.7, 124.3, 118.8, 118.3, 108.8, 108.4, 83.4, 80.1, 64.4, 63.6, 52.2, 45.6, 38.0, 36.5, 35.7 (CH_3); MS (CH_4) m/z : 333 ($\text{M}^+ + 1$, 100), 173 (65), 159 (25); HRMS m/z : 333.2087 [$\text{M}^+ + 1$] (calcd for $\text{C}_{21}\text{H}_{24}\text{N}_4$, 332.2001).

Compound 12: Orange foam; $R_f = 0.21$ (pentane/ethyl acetate 2/1); IR (neat) 2094, 1703, 1617, 739 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 8.85 (s, 1H), 7.99 (s, 1H), 7.37 (d, $J = 7.5$ Hz, 1H), 7.34 (d, $J = 8.0$ Hz, 1H), 7.25 (td, $J = 7.5, 1.0$ Hz, 1H), 7.18 (td, $J = 7.5, 1.0$ Hz, 1H), 7.17 (d, $J = 7.5$ Hz, 1H), 7.08 (td, $J = 6.5, 1.0$ Hz, 1H), 7.04 (td, $J = 7.5, 1.0$ Hz, 1H), 6.93 (d, $J = 2.0$ Hz, 1H), 6.88 (d, $J = 7.5$ Hz, 1H), 3.26-3.14 (m, 2H), 3.00-2.72 (m, 4H), 2.58 (s, 3H), 2.38-2.20 (m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 179.9, 140.7, 136.2, 129.7, 129.0, 127.5, 124.6, 122.8, 121.9, 121.7, 119.2, 118.8, 114.1, 111.1, 110.4, 68.8, 52.4, 46.6, 35.5, 34.4, 24.8; MS (NH_3) m/z : 375 ($\text{M}^+ + 1$, 100), 304 (10), 188 (20), 175 (43).