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Esterase Screening Using Whole Cells of Brazilian Soil Microorganisms

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*Synthesis of the alcohol **8** and hydrolytic substrates **5**, **6** and **7***

*Alcohol **8***

To a round bottom flask containing acetophenone (1.063 g, 8.71 mmol) in MeOH (3.0 mL), $NaBH_4$ (440 mg, 9.5 mmol) was added and the resulting suspension was stirred for 20 min. The reaction mixture was treated with 3 portions of 5 mL of brine and the organic phase was dried over anhydrous Na_2SO_4 and evaporated. Purification of the crude product by flash column chromatography (hexanes/EtOAc, 3:4) afforded **8** as a colorless oil, in 87% yield. MM: 122 g mol^{-1} ($C_8H_{10}O$). EI-MS *m/z* 122 ($M^{\cdot+}$, 34%), 107 (100), 79 (90), 77 (51), 51 (14), 43 (18). ^{1}H NMR (300.01 MHz, $CDCl_3$) δ 1.45 (d, 3H, *J* 6.6 Hz, H-8); 2.10 (sl, 1H, OH); 4.90 (q, 1H, *J* 6.6 Hz, H-7); 7.40 (m, 5H, H-2, H-3, H-4, H-5, H-6). ^{13}C NMR (75.5 MHz, $CDCl_3$, δ_{CDCl3} 77.0) d 25.0 (CH_3, C-8); 69.8 (CH, C-7); 125.3 (2 CH, C-3 and C-5); 127.0 (CH, C-4); 128.0 (2 CH, C-2 and C-6).

*Syntheses of esters **5**, **6** and **7**, general procedure:*

To a solution of alcohol **8** (1.617 g; 13.2 mmol) in methylene chloride (25 mL) at 0 °C, acyl chloride (21.2 mmol of acetyl chloride or propanoyl chloride or octanoyl chloride) and DMAP (1.58 g, 16.8 mmol) were added. The reaction was stirred for 10 h at room temperature. After washing with a saturated solution of $NaHCO_3$, the organic phase was dried over Na_2SO_4 and evaporated under reduced pressure to afford the expected products **5**, **6** or **7**.

*Ester **5**:* Purification of the crude product by flash column chromatography (hexanes/EtOAc, 9:1) afforded **5** as a colorless oil, in 85% yield. MM: 164 g mol^{-1} ($C_{10}H_{12}O_2$). EI-MS *m/z* 164 ($M^{\cdot+}$, 22%), 122 (100), 107 (37), 105 (74), 104 (90), 77 (34), 43 (42). ^{1}H NMR (300.01 MHz, $CDCl_3$) δ 1.55 (d, 3H, *J* 6.6 Hz, H-8); 2.10 (s, 3H, H-10); 5.90 (q, 1H, *J* 6.6 Hz, H-7); 7.40 (m, 5H, H-2, H-3, H-4, H-5, H-6). ^{13}C NMR (75.5 MHz, $CDCl_3$, δ_{CDCl3} 77.0) δ 21.3 (CH_3, C-10); 22.2 (CH_3, C-8); 72.3 (CH, C-7); 126.0 (2 CH, C-3 and C-5); 127.8 (CH, C-4); 128.5 (2 CH, C-2 and C-5); 141.67 (C_0, C-1); 170.3 (C_0, C-9).

*Ester **6**:* Purification of the crude product by flash column chromatography (hexanes/EtOAc, 5:1) afforded **6** as a colorless oil, in 80% yield. MM: 178 g mol^{-1} ($C_{11}H_{14}O_2$). EI-MS *m/z* 178 ($M^{\cdot+}$, 25%), 122 (95), 105 (100), 104 (78), 77 (26), 57 (31). ^{1}H NMR (300.01 MHz, $CDCl_3$) δ 1.17 (t, 3H, *J* 7.7 Hz, H-11); 1.50 (d, 3H, *J* 6.7 Hz, H-8); 2.35 (q, 2H, *J* 7.7 Hz, H-10); 5.90 (q, 1H, *J* 6.7 Hz, H-7); 7.40 (m, 5H, H-2, H-3, H-4, H-5, H-6). ^{13}C NMR (75.5 MHz, $CDCl_3$, δ_{CDCl3} 77.0) δ 9.06 (CH_3, C-11); 22.2 (CH_3, C-8); 27.8 (CH_2, C-10); 72.0 (CH, C-7); 126.0 (2 CH, C-3 and C-5); 127.7 (CH, C-4); 128.4 (2 CH, C-2 and C-5); 141.8 (C_0, C-1); 173.6 (C_0, C-9).

*Ester **7**:* Purification of the crude product by flash column chromatography (hexanes/EtOAc, 5:1) afforded **7** as colorless oil, in 79% yield. MM: 248 g mol^{-1} ($C_{16}H_{24}O_2$) EI-MS *m/z* 248 ($M^{\cdot+}$, 2%), 143 (5), 122 (100), 105 (87), 57 (11). ^{1}H NMR (300.01 MHz, $CDCl_3$) δ 0.86 (t, 3H, *J* 6.0 Hz, H-16); 1.20-1.35 (m, 8H, H-12, H-13, H-14 and H-15), 1.52 (d, *J* 6.0 Hz, 3H, H-8); 1.62 (t, 2H, *J* 6.0 Hz, H-11); 2.32 (t, 2H, *J* 6.0 Hz, H-10); 5.90 (q, 1H, *J* 6.0 Hz, H-7); 7.22-7.40 (m, 5H, H-2, H-3, H-4, H-5, H-6). ^{13}C NMR (75.5 MHz, $CDCl_3$, δ_{CDCl3} 77.0) δ 13.9 (CH_3, C-16), 22.2 (CH_3, C-8); 22.5 (CH_2, C-15), 24.9 (CH_2, C-14), 28.8 (CH_2, C-13); 28.9 (CH_2, C-12); 31.6 (CH_2, C-11); 35.5 (CH_2, C-10); 71.9 (CH, C-7); 125.9 (2 CH, C-3 and C-5); 127.7 (CH, C-4); 128.4 (2 CH, C-2 and C-5); 141.8 (C_0, C-1); 173.0 (C_0, C-9).

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