

Design and Synthesis of a 1 α ,25-Dihydroxyvitamin D₃ Dimer as a Potential Chemical Inducer of Vitamin D Receptor Dimerization.

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Supporting Information

Compound 2: ¹H NMR (200 Mz, MeOD) δ 6.32 and 6.06 (AB system, d, $J = 11.3$ Hz, 4 H), 5.29 (s, 2 H), 4.90 (s, 2 H), 4.34 (t, $J = 5.3$ Hz, 2 H), 4.12 (m, 2 H), 2.91 (m, 2 H), 2.52 (m, 2 H), 1.16 (s, 12 H), 0.98 (d, $J = 5.8$ Hz, 6 H), 0.57 (s, 6 H). ¹³C NMR (50 Mz, MeOD) δ 149.8, 141.8, 135.8, 124.8, 118.9, 111.9, 71.4, 67.4, 57.8, 46.9, 46.1, 45.3, 43.7, 38.8, 37.7, 37.5, 37.1, 35.9, 30.8, 29.2, 28.1, 22.9, 21.9, 19.5, 13.2. FAB-MS (3-NBA) 938 (M⁺+Na, 3).

Compound 3: ¹H NMR (200 Mz, CD₂Cl₂) δ 6.27 and 6.02 (AB system, d, $J = 11.2$ Hz, 2 H), 5.83 (ddt, $J = 17.1, 13.2, 6.8$ Hz, 1 H), 5.18 (d, $J = 2.5$ Hz, 1 H), 5.01 (m, 1 H), 4.93 (m, 1 H), 4.38 (m, 1 H), 4.18 (m, 1 H), 2.88 (dd, $J = 13.2$ and 3.9 Hz, 1 H), 2.47 (dd, $J = 13.2$ and 3.9 Hz, 1 H), 1.18 (s, 6 H), 0.94 (t, $J = 8.3$ Hz, 9 H), 0.88 (s, 9 H), 0.56 (c, $J = 7.8$ Hz, 6 H), 0.52 (s, 3 H). ¹³C NMR (50 Mz, CD₂Cl₂) δ 148.8, 140.8, 139.7, 135.5, 123.4, 118.2, 114.2, 111.5, 73.9, 72.36, 67.9, 57.0, 56.8, 54.8, 54.3, 53.8, 53.2, 52.7, 37.2, 36.8, 36.5, 36.1, 34.6, 31.9, 30.1, 30.0, 28.4, 26.0, 22.3, 21.2, 19.1, 18.5, 18.4, 12.9, 7.3, 7.1, -4.6, -4.62, -4.7, -4.9. FAB-MS (3-NBA) 813 (M⁺, 2).

Compound 4: ¹H NMR (200 Mz, CDCl₃) δ 5.79 (ddt, $J = 17.1, 13.2, 6.8$ Hz, 1 H), 5.01 (d, $J = 17.1$ Hz, 1 H), 4.96 (d, $J = 13.2$ Hz, 1 H), 2.40 (m, 2 H), 1.19 (s, 3 H), 0.94 (t, $J = 6.8$ Hz, 9 H), 0.96 (d, $J = 6.4$ Hz, 3 H), 0.56 (c, $J = 8.3$ Hz, 6 H). ¹³C NMR (50 Mz, CD₂Cl₂) δ 211.4, 138.3, 114.7, 73.4, 61.9, 56.5, 49.1, 47.6, 46.2, 45.4, 36.5, 36.2, 36.0, 35.5, 31.3, 30.0, 29.8, 27.8, 27.0, 20.7, 18.9, 18.7, 13.8, 13.2, 7.1, 6.8. FAB-MS (3-NBA) 449 (M⁺, 6).

Compound 7: ¹H NMR (200 Mz, CDCl₃) δ 2.45 (dd, $J = 11.3, 7.6$ Hz, 1 H), 2.27 (dd, $J = 7.3, 3.6$ Hz, 1 H), 1.16 (s, 6 H), 0.93 (d, $J = 5.6$ Hz, 3 H), 0.91 (t, $J = 7.3$ Hz, 6 H), 0.61 (s, 3 H), 0.53 (c, $J = 7.9$ Hz, 9 H). ¹³C NMR (50 Mz, CDCl₃) δ 73.3, 61.9, 56.7, 49.9, 45.4, 40.9, 38.9, 36.2, 35.4, 29.9, 29.7, 27.4, 24.0, 20.6, 19.0, 18.6, 12.4, 7.1, 6.7. MS (EI, 70 eV) 379 (M⁺-Me, 5), 365 (M⁺-Et, 5), 336 (M⁺-2Et, 7).

Compound 8: ^1H NMR (200 Mz, CDCl_3) δ 6.75 (m, 1 H), 5.98 (dd, $J = 9.0, 2.9$ Hz, 1 H), 2.7–2.3 (m, 3 H), 1.19 (s, 6 H), 0.94 (t, $J = 7.5$ Hz, 9 H), 0.92 (d, $J = 6.8$ Hz, 3 H), 0.76 (s, 3 H), 0.56 (c, $J = 7.8$ Hz, 6 H). ^{13}C NMR (50 Mz, CDCl_3) δ 202.0, 147.6, 135.2, 129.5, 73.3, 63.5, 59.2, 56.7, 47.5, 45.4, 42.9, 36.1, 35.4, 35.0, 30.0, 29.8, 27.4, 20.6, 19.4, 18.4, 11.9, 7.35, 7.10, 6.8, 6.2. MS (EI, 70 eV) 363 ($\text{M}^+ - \text{Et}$, 30).

Compound 9: ^1H NMR (200 Mz, MeOD) δ 6.31 and 6.06 (AB system, d, $J = 11.3$ Hz, 4 H), 5.40 (m, 2 H), 5.29 (s, 2 H), 4.90 (s, 2 H), 4.34 (t, $J = 4.8$ Hz, 2 H), 4.13 (m, 2 H), 2.90 (m, 2 H), 2.54 (m, 2 H), 1.16 (s, 12 H), 0.98 (d, $J = 5.8$ Hz, 6 H), 0.56 (s, 6 H). ^{13}C NMR (50 Mz, MeOD) δ 150.0, 141.7, 135.7, 131.6, 130.9, 124.9, 118.9, 111.7, 71.5, 67.4, 57.8, 47.0, 46.1, 45.3, 43.7, 38.4, 37.7, 37.5, 37.0, 34.9, 31.8, 30.7, 29.3, 29.1, 23.1, 21.9, 19.5, 13.5.

Compound 10: ^1H NMR (200 Mz, CDCl_3) δ 2.40 (m, 4 H), 1.19 (s, 12 H), 0.96 (d, $J = 5.6$ Hz, 6 H), 0.95 (t, $J = 7.6$ Hz, 12 H), 0.64 (s, 6 H), 0.56 (c, $J = 7.9$ Hz, 18 H). ^{13}C NMR (50 Mz, CDCl_3) δ 211.7, 73.37, 61.92, 56.5, 49.2, 47.8, 46.4, 45.4, 37.5, 36.7, 36.2, 35.5, 30.0, 29.8, 29.7, 27.8, 27.2, 20.7, 18.9, 18.8, 13.2, 7.1, 6.8. FAB-MS (3-NBA) 842 ($\text{M}^+ - \text{Et}$).