Supporting Informations

Total Synthesis of Batzelladine D

Takanori Ishiwata,³ Tomoyuki Hino,¹ Yuichi Hashimoto,¹ Tadashi Nakata^{2,3} and
Kazuo Nagasawa^{1*}

¹*Institute of Molecular and Cellular Biosciences, The University of Tokyo
1-1-1 Yayoi, Bunkyo-ku, Tokyo 113-0032, Japan*

²*RIKEN (The Institute of Physical and Chemical Research)
2-1 Hirosawa, Wako, Saitama 351-0198, Japan*

³*Graduate School of Science and Engineering, Saitama University
255 Shimookubo, Saitama, Saitama 338-8570, Japan*

General

IR spectra were measured with a JASCO VALOR-III FT-IR spectrophotometer. ¹H and ¹³C NMR spectra were recorded on JEOL GSX-500 instruments. Mass spectra were recorded on JEOL JMA-HX110 spectrometers.

Spectral data

Compound **5**: IR (neat) 3331, 2979, 1722, 1642 1415 cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 11.47 (brs, 1H), 8.37 (brs, 1H), 3.69 (t, *J* = 6.1 Hz, 2H), 2.33 (q, *J* = 7.0 Hz, 2H), 1.72-1.60 (m, 4H), 1.49 (s, 9H), 1.48 (s, 9H); ¹³C NMR (CDCl₃, 125 MHz) 163.5, 156.2, 153.3, 83.1, 79.3, 62.2, 40.3, 29.5, 28.3, 28.0, 25.6 ppm; HRMS (FAB, MH⁺) calcd for C₁₅H₃₀N₃O₅ 332.2185, found 332.2179.

14

Compound **14**: IR (neat) 2926, 2855, 1465 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 3.97 (m, 1H), 3.70 (n, 1H), 3.10 (m, 2H), 2.03-1.90 (m, 4H), 1.84-1.20 (m, 18H) 0.83 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) 76.4, 64.8, 57.0, 42.5, 33.9, 31.8, 31.7, 29.62, 29.57, 29.45, 29.21, 26.4, 24.3, 22.6, 14.0 ppm; HRMS (FAB, MH^+) calcd for $\text{C}_{15}\text{H}_{30}\text{NO}$ 240.2327, found 240.2383.

17

Compound **17**: IR (neat) 3361, 2925, 2853, 1736, 1437 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 4.30 (m, 1H), 4.08 (q, $J = 6.8$ Hz, 1H), 3.92 (m, 1H), 3.70 (s, 3H), 3.37 (m, 1H), 3.04 (t, $J = 9.8$ Hz, 1H), 1.96-1.84 (m, 2H), 1.74 (ddd, $J = 4.6, 10.1, 10.1$ Hz, 1H), 1.59-1.24 (m, 19H), 1.32 (d, $J = 6.8$ Hz, 3H), 0.86 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) 170.3, 72.1, 69.1, 65.43, 65.35, 57.3, 51.8, 39.2, 37.6, 31.9, 29.7, 29.6, 29.5, 29.3, 28.9, 27.3, 25.7, 22.6, 16.6, 14.1 ppm; HRMS (FAB, MH^+) calcd for $\text{C}_{20}\text{H}_{38}\text{NO}_4$ 355.2801, found 355.2840.

Compound **19**: IR (neat) 3360, 2954, 2928, 2856, 1470 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 3.97, (m, 1H), 3.75 (m, 1H), 3.67 (m, 1H), 3.62 (dd, $J = 4.0, 10.4$ Hz, 1H), 3.51 (dd, $J = 4.6, 10.4$ Hz, 1H), 3.36 (m, 1H), 2.04 (m, 1H), 1.84 (m, 1H), 1.78 (m, 1H), 1.71 (m, 1H), 1.62-1.20 (m, 19H), 1.17 (d, $J = 6.1$ Hz, 3H), 0.88 (t, $J = 7.0$ Hz, 3H), 0.87 (s, 9H), 0.86 (s, 9H), 0.040 (s, 3H), 0.035 (s, 3H), 0.02 (s, 6H); ^{13}C NMR (CDCl_3 , 125 MHz) 71.1, 66.7, 62.7, 58.9, 53.6, 48.7, 41.9, 36.7, 33.0, 31.9, 29.7, 29.6, 29.5, 29.3, 26.2, 25.9, 25.8, 25.5, 22.6, 21.9, 18.1, 18.0, 14.1 –4.47, -4.52, -5.56, -5.71 ppm; HRMS (FAB, MH^+) calcd for $\text{C}_{31}\text{H}_{68}\text{NO}_3\text{Si}_2$ 558.4738, found 558.4733.

Compound **24**: IR (neat) 3332, 2929, 2856, 1724, 1614, 1455, 1416 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 8.20 (brs, 1H), 7.25-7.12 (m, 10H), 5.05 (d, $J = 11.5$ Hz, 1H), 4.87 (d, $J = 12.5$ Hz, 1H), 4.80 (d, $J = 12.5$ Hz, 1H), 4.77 (d, $J = 11.5$ Hz, 1H), 4.39 (m, 1H), 3.99 (m, 3H), 3.76 (m, 2H), 3.31 (m, 2H), 3.11 (dd, $J = 4.9, 7.3$ Hz, 1H), 2.17 (m, 2H), 1.80-1.18 (m, 25H), 1.40 (s, 9H), 1.39 (s, 9H), 1.28 (d, $J = 6.7$ Hz, 3H), 0.78 (t, $J = 7.0$ Hz, 3H), 0.77 (s, 9H), -0.048 (s, 3H), -0.056 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) 169.8, 163.6, 160.0, 156.1, 153.3, 152.9, 150.0, 137.2, 135.6, 128.3-128.0 (12 carbons), 127.4, 83.1, 79.2, 71.1, 68.1, 66.8, 64.8, 58.5, 56.2, 52.2, 50.1, 40.7, 40.5, 40.4, 40.2, 37.4, 31.9, 29.9, 29.6, 29.3, 28.8, 28.3, 28.02, 27.98, 27.5, 25.9, 25.8, 25.6, 24.4, 22.7, 18.0, 16.4, 14.1, -4.32, -4.40 ppm; HRMS (FAB, MH^+) calcd for $\text{C}_{57}\text{H}_{91}\text{N}_6\text{O}_{11}\text{Si}$ 1063.6515, found 1063.6475. NOEs were observed from H-7 to H-8 and H-15.

Compound **26**: IR (neat) 2929, 1720, 1615, 1457, 1416 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 4.19 (t, $J = 6.4$ Hz, 2H), 3.96 (m, 1H), 3.85 (m, 1H), 3.52 (m, 2H), 3.38 (m, 2H), 3.15 (dd, $J = 4.4, 4.8$ Hz, 1H), 2.35 (dd, $J = 2.0, 4.9, 12.7$ Hz, 1H), 2.22 (m, 2H), 1.75-1.27 (m, 23H), 1.52 (s, 9H), 1.47 (s, 9H), 1.28 (d, $J = 7.0$ Hz, 3H), 0.89 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) 179.5, 164.5, 157.7, 154.2, 151.4, 84.6, 80.6, 65.8, 57.7, 57.3, 53.2, 49.8, 45.8, 41.4, 37.1, 34.3, 33.0, 31.4, 30.6, 30.5, 29.4, 28.6, 28.2, 27.0, 26.3, 23.7, 18.5, 14.4 ppm; HRMS (FAB, MH^+) calcd for $\text{C}_{35}\text{H}_{63}\text{N}_6\text{O}_6$ 663.4809, found 663.4835.