Microwave-assisted synthesis of near-infrared fluorescent sphingosine derivatives

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SUPPORTING INFORMATION

All chemicals and solvents were of American Chemical Society or high-performance liquid chromatography (HPLC) purity and were used as received. HPLC grade acetonitrile was purchased from VWR International (West Chester, PA, USA). HPLC grade water was from American Bioanalytic (Natick, MA, USA). All other chemicals were purchased from Fisher Scientific (Pittsburgh, PA, USA) and Sigma-Aldrich (Saint Louis, MO, USA).

NMR: All NMR measurements were performed on the Varian Unity INOVA 600 MHz vertical bore spectrometer using either 5 mm high-resolution probe. Samples were placed in 5 mm NMR tubes.

NMR and LCMS:

*Compound **2a**:* ^{1}H NMR (CD_3OD, 600 MHz): δ*ppm* 8.54 (s, 1H, NH), 8.05 (d, J=13.2 Hz, 2H, 2xCH), 7.37 (d, J=7.2 Hz, 2H, Ar-H), 7.30 (t, J=7.6 Hz, 2H, Ar-H), 7.09 (t, J=8.8 Hz, 4H, Ar-H), 5.87 (d, J=13.2 Hz, 2H, 2xCH), 5.75-5.81 (m, 2H, 2xCH), 5.45-5.57 (m, 2H, 2xCH), 4.25 (t, J=6.4Hz, 1H, CH), 4.12 (t, J=5.2 Hz, 1H, CH), 3.95 (m, 4H, 2xCH_2), 2.63 (t, J=5.4Hz, 1H, CH), 2.43 (q, J=8.0 Hz, 2H, CH_2), 2.07 (m, 3H, NH & CH_2), 1.83 (m, 4H, 2xCH_2), 1.69 (t, J=6.9 Hz, 10H, 2xCH_3 & 2xCH_2), 1.23- 1.28 (s, 28H, 11xCH_2 & 2xCH_3), 1.03 (t, J=7.2 Hz, 3H, CH_3), 0.89 (t, J=7.2 Hz, 6H, 2xCH_3); LC-MS (ES-TOF$^+$) *m/z*: 802.65 (M^+).

*Compound **2b**:* ^{1}H NMR (CD_3OD, 600 MHz): δ*ppm* 8.51 (s, 1H, NH), 8.00 (d, J=13.2 Hz, 2H, 2xCH), 7.36 (d, J=7.2 Hz, 2H, Ar-H), 7.32 (t, J=7.6 Hz, 2H, Ar-H), 7.07 (t, J=8.8 Hz, 4H, Ar-H), 5.83 (d, J=13.2 Hz, 2H, 2xCH), 5.75-5.79 (m, 2H, 2xCH), 5.49-5.56 (m, 2H, 2xCH), 4.56 (s, 3H, CH_3), 4.25 (t, J=6.4Hz, 1H, CH), 4.14 (t, J=5.2 Hz, 1H, CH), 3.99 (t, J=6.8 Hz, 2H, CH_2), 3.46 (s, 3H, CH_3), 2.60 (t, J=5.4Hz, 1H, CH), 2.42 (q, J=8.0 Hz, 2H, CH_2), 2.06 (m, 3H, NH & CH_2), 1.85 (t, J=6.8 Hz, 2H, CH_2), 1.65 (t, J=6.9 Hz, 6H, 2xCH_3), 1.28- 1.35 (s, 28H, 11xCH_2 & 2xCH_3), 0.94 (t, J=7.2 Hz, 3H, CH_3); LC-MS (ES-TOF$^+$) *m/z*: 746.56 (M^+).