

Supporting Information

Synthesis: All reagents were used as purchased from Sigma Aldrich or other commercial chemical sales companies, unless otherwise noted. Purification by silica gel chromatography was carried out using silica gel 60 (230–240 Mesh). NMR spectra were recorded using a Varian 500MHz instrument. High-resolution mass spectra were recorded using an ESI Quadrupole Time of Flight Mass Spectrometer.

Synthesis of 1: Cholic acid (20 g, 0.05 mol), benzyl chloride (24.7 g, 0.196 mol), and potassium carbonate (27.1 g, 0.196 mol) were dissolved in DMF and stirred overnight at room temperature. The reaction mixture was put under an air stream for 24 h to remove DMF and excess BnCl. The resulting material was dissolved in dichloromethane (50 mL) and washed with brine (2 x 50 mL). Silica gel chromatography (ethyl acetate, then 5% methanol/dichloromethane, then 10% methanol/dichloromethane) gave **1** (86% yield) as a clear oil. ¹H NMR (500 MHz, CDCl₃) δ 5.09 (m, 2H), 3.91 (s, 1H), 3.8 (s, 1H), 3.4 (s, 2H), 2.91 (d, 2H), 2.85 (d, 2H), 2.41 (m, 1H), 2.25 (m, 3H), 1.84 (m, 5H), 1.72 (m, 3H), 1.64 (d, 1H), 1.57 (d, 2H), 1.48 (m, 4H), 1.35 (s, 4H), 1.23 (m, 2H), 1 (m, 5H), 0.85 (d, 3H), 0.62 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 174.17, 162.68, 136.1, 128.5, 128.15, 128.12, 73, 71.79, 68.33, 66.02, 60.39, 46.86, 46.36, 41.54, 41.44, 39.45, 39.32, 36.54, 35.35, 35.31, 34.82, 34.77, 31.47, 31.33, 30.92, 30.36, 28.16, 27.56, 26.16, 23.26, 22.47, 21.04, 17.27, 14.21, 12.44. HRMS (ESI) m/z: [M+NH₄]⁺ 516.3731; calculated: 516.368.

Synthesis of 2: Compound **1** (21.2 g, 0.043 mol), Boc β alanine (48.3 g, 0.255 mol), DCC (52.6 g, 0.255 mol), DMAP (1.04 g, 0.009 mol) were dissolved in dichloromethane (100 mL). The mixture was stirred overnight at room temperature. The resulting mixture was repeatedly filtered and concentrated in vacuo until clear of dicyclohexylurea. Silica gel chromatography (25% ethyl acetate/toluene) yielded **2** (31.5 g, 73%) as a clear oil. ¹H NMR (500 MHz, CDCl₃) δ 7.25 (m, 1H), 7.2 (m, 2H), 7.1 (t, 2H), 5.01 (m, 4H), 4.88 (d, 1H), 4.52 (m, 1H), 3.82 (t, 1H), 3.32 (m, 5H), 2.6 (t, 1H), 2.54 (t, 2H), 2.44 (m, 3H), 2.27 (s, 2H), 2.18 (m, 1H), 1.91 (m, 3H), 1.77 (m, 3H), 1.6 (m, 3H), 1.54 (d, 1H), 1.49 (t, 5H), 1.44 (s, 1H), 1.34 (m, 32H), 1.16 (m, 3H), 0.99 (m, 2H), 0.85 (s, 2H), 0.72 (d, 2H), 0.62 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 173.74, 172.04, 171.72, 171.61, 169.01, 159.72, 155.84, 152.06, 137.89, 136, 129.05, 128.58, 128.36, 128.27, 128.24, 125.31, 83.59, 79.34, 75.53, 74.09, 70.98, 66.19, 47.6, 45.13, 43.38, 42.31, 40.83, 37.76, 36.32, 36.19, 35.4, 35.03, 34.9, 34.76, 34.58, 34.52, 34.32, 34.1, 32.74, 31.34, 31.26, 30.75, 28.83, 28.42, 28.07, 27.5, 27.07, 26.85, 26.3, 25.59, 24.73, 22.86, 22.52, 21.49, 17.59. HRMS (ESI) m/z: [M+NH₄]⁺ 1012.6047; calculated: 1012.6104

Synthesis of 3: Compound **2** (5.0 g, 0.00495 mol), Pd-C 10% (0.524 g, 0.000495 mol) were dissolved in 50 ml of methanol and stirred at 500 psi of H₂ overnight. The mixture was centrifuged and 4.63 g of compound **3** (92%) was collected. ¹H NMR (500 MHz, CDCl₃) δ 5.12 (t, 3H), 4.95 (s, 1H), 4.58 (m, 1H), 3.43 (m, 7H), 2.65 (t, 3H), 2.51 (m, 4H), 2.37 (m, 1H), 2.21 (m, 1H), 1.99 (m, 3H), 1.86 (m, 2H), 1.77 (m, 3H), 1.66 (m, 4H), 1.59 (s, 1H), 1.56 (m, 1H), 1.53 (d, 3H), 1.51 (s, 1H), 1.43 (d, 32H), 1.31 (t, 1H), 1.12 (m, 1H), 0.93 (s, 2H), 0.82 (d, 2H), 0.73 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 178.19, 174.21, 172.01, 171.59, 156.23, 155.96, 155.87, 153.65, 83.24, 80.02, 79.46, 79.35, 79.17, 75.5, 74.11, 71.01, 51.78, 47.13, 45.09, 45, 44.57, 43.4, 41, 40.8, 37.74, 36.36, 36.19, 36.08, 35.07, 34.96, 34.88, 34.74, 34.56, 34.51, 34.41, 34.29, 32.67, 31.32, 30.81, 30.7, 30.47, 28.8, 28.42, 28.39, 28.22, 28.1, 28, 27.12, 26.83, 26.23, 25.54, 25.28, 24.72, 23.56, 22.85, 22.49, 21.6, 17.57, 12.15, 1.02. HRMS (ESI) m/z: [M+NH₄]⁺ 922.5538; calculated: 922.5635

Synthesis of 5: Compound **3** (4 g, 0.00434 mol), monocaprin (2.14 g, 0.00869 mol), EDCI (0.995 g, 0.00521 mol), and DMAP (0.106 g, 0.000869 mol) were dissolved in dichloromethane (100 ml) and was stirred for 3 hours. Silica gel chromatography (40% ethyl acetate/hexanes) yielded compound **5** (85%) as an oil. ¹H NMR (500 MHz, CDCl₃) δ 5.13 (t, 3H), 4.95 (s, 1H), 4.59 (m, 1H), 4.13 (m, 5H), 3.39 (s, 6H), 2.92 (s, 1H), 2.64 (s, 3H), 2.51 (m, 3H), 2.37 (m, 3H), 2.25 (m, 1H), 2 (m, 4H), 1.88 (m, 1H), 1.8 (d, 1H), 1.7 (t, 1H), 1.68 (d, 1H), 1.65 (d, 1H), 1.62 (t, 3H), 1.58 (s, 1H), 1.56 (d, 1H), 1.54 (s, 1H), 1.53 (s, 1H), 1.43 (s, 31H), 1.27 (m, 16H), 1.12 (m, 1H), 0.93 (s, 3H), 0.88 (t, 3H), 0.81 (d, 3H), 0.73 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 173.98, 173.89, 171.99, 171.64, 171.56, 171.19, 155.84, 83.14, 79.35, 75.49, 74.07, 70.96, 68.11, 65.16, 64.97, 60.41, 47.38, 45.16, 45.12, 43.35, 42.12, 40.8, 37.73, 36.34, 36.18, 35.01, 34.95, 34.88, 34.73, 34.55, 34.5, 34.3, 34.08, 31.83, 31.31, 30.87, 30.53, 29.39, 29.23, 29.1, 28.81, 28.42, 28.39, 28.06, 27.52, 27.07, 26.82, 25.56, 24.86, 22.84, 22.65, 22.49, 21.06, 17.58, 14.19, 14.12, 12.22, 9.87. HRMS (ESI) m/z: [M+NH₄]⁺ 1150.7401; calculated 1150.7360

Synthesis of 4: Compound **3** (0.02 g, 2.17×10^{-5} mol), 1-octanoyl-*rac*-glycerol (0.00947 g, 4.34×10^{-5} mol), DCC (0.00447 g, 2.17×10^{-5} mol), and DMAP (0.00053 g, 4.34×10^{-6} mol) were combined and dissolved in DCM (100 ml). This mixture was stirred overnight at room temperature. Silica gel chromatography (40% ethyl acetate/hexanes) was performed. Compound **4** (0.0163 g, 65%) was collected. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.1735 (m, 4H), 5.8824 (s, 0H), 5.1116 (m, 1H), 4.8795 (m, 1H), 4.6918 (t, 0H), 4.6473 (s, 0H), 4.4832 (m, 1H), 4.3466 (d, 0H), 4.2745 (t, 0H), 4.1507 (d, 1H), 4.03 (m, 3H), 3.8632 (m, 1H), 3.7691 (m, 0H), 3.6975 (m, 5H), 3.6351 (s, 4H), 3.5829 (m, 4H), 3.5119 (m, 1H), 3.3482 (s, 3H), 2.9697 (s, 3H), 2.2643 (t, 3H), 2.0362 (m, 1H), 1.8669 (d, 3H), 1.693 (d, 5H), 1.5488 (d, 4H), 1.427 (m, 3H), 1.224 (m, 18H), 1.0056 (m, 2H), 0.8777 (m, 2H), 0.7764 (d, 6H), 0.6516 (s, 2H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 173.3174, 172.9298, 172.8926, 159.8675, 156.6579, 105.1073, 104.5352, 95.0089, 94.5776, 89.3275, 75.0773, 74.2916, 72.6071, 72.5201, 72.296, 71.2313, 70.3206, 70.292, 70.2697, 70.0946, 69.5084, 69.4757, 69.223, 68.6602, 67.8404, 66.9595, 66.065, 65.8248, 65.1298, 64.9187, 64.1146, 64.0177, 63.7715, 63.4754, 62.9768, 62.3053, 61.8114, 61.3615, 61.0592, 60.7581, 60.6829, 60.4666, 58.4303, 49.4526, 44.9599, 43.8917, 41.9037, 41.6647, 41.5751, 41.0922, 39.5027, 36.8906, 33.5927, 33.3352, 33.2263, 33.1348, 33.0693, 31.9468, 30.9045, 30.6441, 30.6159, 30.1158, 28.6803, 28.6383, 28.3455, 28.0826, 28.059, 28.0314, 27.9211, 27.8877, 27.8717, 27.2016, 25.8281, 24.1671, 23.8831, 23.861, 23.4634, 21.676, 21.5905, 21.5721, 21.1892, 16.6942, 13.0824, 13.0503, 10.9471. HRMS (ESI) m/z : $[\text{M}+\text{NH}_4]^+$ 1122.6972; calculated 1122.7047

Synthesis of 6: Compound **3** (0.02 g, 2.17×10^{-5} mol), 1-lauroyl-*rac*-glycerol (0.0119 g, 4.34×10^{-5} mol), DCC (0.00447 g, 2.17×10^{-5} mol), and DMAP (0.00053 g, 4.34×10^{-6} mol) were dissolved in DCM (100 ml) and was stirred at room temperature overnight. Silica gel chromatography (40% ethyl acetate/hexanes) produced 0.014 g of compound **6** (54%). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 8.2657 (d, 2H), 8.0269 (s, 0H), 7.8813 (s, 1H), 5.8863 (s, 0H), 5.1056 (s, 1H), 4.9997 (m, 0H), 4.8877 (m, 1H), 4.8179 (t, 0H), 4.7356 (d, 0H), 4.6974 (d, 0H), 4.6447 (s, 0H), 4.5568 (m, 0H), 4.473 (m, 1H), 4.3776 (s, 0H), 4.347 (d, 0H), 4.3153 (s, 0H), 4.2757 (t, 0H), 4.2511 (m, 0H), 4.239 (d, 0H), 4.2195 (d, 0H), 4.201 (s, 0H), 4.1554 (d, 1H), 4.1211 (d, 0H), 4.0702 (m, 1H), 3.9732 (m, 1H), 3.8676 (m, 1H), 3.7865 (m, 1H), 3.7039 (m, 6H), 3.6338 (s, 3H), 3.5987 (d, 2H), 3.5689 (t, 4H), 3.3246 (s, 3H), 2.9528 (d, 3H), 2.4039 (m, 1H), 2.2259 (t, 3H), 2.0429 (m, 1H), 1.8668 (m, 3H), 1.7014 (m, 4H), 1.5557 (d, 5H), 1.4158 (m, 3H), 1.2103 (s, 28H), 0.996 (s, 1H), 0.8806 (m, 2H), 0.7778 (m, 6H), 0.6663 (s, 1H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 173.3285, 172.9356, 172.9006, 159.8663, 156.5459, 105.1073, 104.5348, 95.0089, 94.5772, 89.3263, 72.6075, 72.5203, 72.2962, 71.2293, 70.8607, 70.2918, 70.269, 70.0922, 69.5083, 69.2231, 68.6653, 67.8397, 67.7707, 66.9315, 66.0651, 65.8245, 65.1294, 64.9172, 64.1155, 64.018, 63.6836, 63.4747, 62.976, 62.2939, 61.8095, 61.4125, 61.0652, 60.7667, 60.6872, 60.4667, 58.4291, 49.2526, 44.9563, 43.8779, 41.8946, 41.6632, 41.573, 39.5114, 36.8836, 33.2272, 33.1325, 33.1038, 33.0724, 32.0554, 30.898, 30.8817, 28.6806, 28.6095, 28.5757, 28.4841, 28.4286, 28.3352, 28.3101, 28.2319, 28.1529, 28.109, 28.0807, 28.0439, 27.9972, 25.8284, 24.2141, 23.8871, 23.8638, 23.5087, 21.6647, 21.1723, 16.6631, 13.1249, 13.1081, 10.9072, 6.7866. HRMS (ESI) m/z : $[\text{M}+\text{NH}_4]^+$ 1178.7593; calculated: 1178.7673

Synthesis of 7: Compound **3** (0.02 g, 2.17×10^{-5} mol), 1-decanoyl-*rac*-glycerol (0.0131 g, 4.34×10^{-5} mol), DCC (0.00447 g, 2.17×10^{-5} mol), DMAP (0.00053 g, 4.34×10^{-6} mol) were added together and dissolved in DCM (100 ml). The mixture was stirred overnight at room temperature. Silica gel chromatography (40% ethyl acetate/hexanes) yielded compound **7** (0.11 g, 42%) as a clear oil. $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 4.7876 (s, 55H), 4.0066 (m, 1H), 3.6549 (m, 1H), 3.5677 (m, 2H), 3.4857 (m, 1H), 3.2099 (s, 3H), 3.1305 (m, 2H), 2.7406 (m, 1H), 2.2813 (m, 1H), 1.8329 (m, 1H), 1.7251 (m, 2H), 1.639 (t, 1H), 1.6035 (t, 1H), 1.5728 (s, 1H), 1.5132 (m, 2H), 1.3599 (m, 1H), 1.2033 (m, 13H), 1.1201 (m, 1H), 1.0146 (m, 1H), 0.8945 (s, 1H), 0.8003 (t, 2H), 0.753 (d, 1H), 0.7067 (s, 1H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) δ 174.2191, 174.1831, 173.9492, 173.913, 170.4707, 170.2758, 170.2523, 158.4494, 95.2219, 76.4818, 74.9456, 73.6033, 73.5707, 72.1528, 72.0854, 71.0457, 70.0796, 69.7338, 68.8563, 66.9654, 66.7352, 66.6798, 65.0863, 64.7517, 64.6217, 64.5626, 62.6386, 60.763, 60.3205, 60.2789, 45.1031, 45.0535, 43.3476, 42.4542, 42.3718, 40.7473, 37.5211, 35.1269, 35.0492, 34.999, 34.5468, 34.5162, 34.1665, 34.0554, 33.5478, 33.5088, 33.4708, 33.322, 31.6812, 31.1461, 31.1024, 31.0229, 30.8001, 30.491, 29.3876, 29.3618, 29.3268, 29.3076, 29.2249, 29.2079, 29.0964, 29.084, 29.0332, 29.0213, 28.9972, 28.9779, 28.8051, 28.7644, 28.7158, 26.8195, 26.4723, 25.3297, 25.2388, 24.6676, 24.5996, 22.4084, 22.3485, 21.4301, 16.757, 13.0859, 13.0701, 11.2053. HRMS (ESI) m/z : $[\text{M}+\text{NH}_4]^+$ 1206.7926; calculated: 1206.7986

Synthesis of 8: Compound **3** (0.02 g, 2.17×10^{-5} mol), 1,2-dihexanoyl-*sn*-glycerol (0.0125 g, 4.34×10^{-5} mol), DCC (0.00447 g, 2.17×10^{-5} mol), and DMAP (0.00053 g, 4.34×10^{-6} mol) were dissolved in DCM (100 ml) and was stirred overnight at room temperature. Compound **8** (0.012g) was isolated using silica gel chromatography (45% ethyl acetate/hexanes). $^1\text{H NMR}$ (500 MHz, CDCl_3) δ 5.1976 (m, 1H), 5.0351 (m, 3H), 4.88 (d, 1H), 4.7117 (s, 0H), 4.5195 (m, 1H), 4.364 (s, 0H), 4.268 (d, 0H), 4.2345 (t, 1H), 4.198 (t, 1H), 4.1598 (d, 0H), 4.1333 (d, 0H), 4.1093 (d, 0H),

4.0626 (m, 3H), 3.7388 (s, 0H), 3.668 (d, 0H), 3.3304 (m, 5H), 2.5647 (s, 3H), 2.4466 (m, 3H), 2.2984 (m, 1H), 2.2596 (d, 1H), 2.2429 (d, 2H), 2.2247 (d, 1H), 2.1446 (m, 1H), 2.0744 (d, 0H), 2.0097 (d, 0H), 1.9735 (m, 1H), 1.9038 (m, 3H), 1.8067 (m, 1H), 1.7575 (d, 0H), 1.7107 (t, 2H), 1.6633 (s, 1H), 1.632 (s, 1H), 1.6047 (d, 1H), 1.5833 (m, 1H), 1.521 (m, 7H), 1.3844 (s, 27H), 1.247 (m, 9H), 1.2054 (s, 1H), 1.1847 (t, 2H), 1.1423 (m, 1H), 1.0238 (m, 2H), 0.8348 (m, 9H), 0.7246 (d, 3H), 0.6553 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 172.3635, 172.2952, 171.8713, 154.8032, 78.2989, 74.4768, 69.9455, 67.8229, 61.179, 61.0639, 59.385, 46.6583, 44.1215, 42.3148, 39.7933, 36.738, 35.2962, 35.1575, 33.9436, 33.8523, 33.7348, 33.5449, 33.2945, 33.1276, 32.9744, 30.3015, 30.2013, 30.1512, 29.9944, 29.5586, 27.7882, 27.395, 27.3774, 26.0435, 25.8148, 24.5489, 23.5269, 23.4998, 21.8278, 21.4782, 21.2683, 20.0502, 16.5675, 13.1805, 12.8963, 12.88, 11.2379. HRMS (ESI) m/z: [M+NH₄]⁺ 1209.7728; calculated: 1209.7781

Synthesis of 9: Compound **3** (0.03 g, 3.36x10⁻⁵ mol), 1,2-dioctanoyl-sn-glycerol (0.0224 g, 6.51x10⁻⁵ mol), EDCI (0.00747 g, 3.91x10⁻⁵ mol), and DMAP (0.000795 g, 6.51x10⁻⁶ mol) were dissolved in DCM. The mixture was stirred overnight at room temperature. Silica gel chromatography (40% ethyl acetate/hexanes) yielded compound **9** (30%). ¹H NMR (500 MHz, CDCl₃) δ 5.1859 (m, 1H), 5.0383 (m, 3H), 4.8869 (d, 1H), 4.5209 (m, 1H), 4.2332 (t, 1H), 4.2008 (t, 1H), 4.0611 (m, 2H), 3.7975 (m, 1H), 3.6107 (t, 1H), 3.3299 (m, 5H), 2.5668 (m, 3H), 2.432 (d, 2H), 2.2988 (m, 1H), 2.2586 (d, 1H), 2.2414 (d, 1H), 2.2251 (d, 1H), 2.1443 (m, 1H), 1.8971 (d, 3H), 1.8153 (m, 2H), 1.7239 (m, 4H), 1.6487 (m, 2H), 1.543 (d, 11H), 1.3738 (s, 29H), 1.1885 (m, 21H), 1.0269 (m, 3H), 0.8646 (s, 2H), 0.8109 (t, 5H), 0.7311 (d, 2H), 0.6663 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 172.3656, 172.3016, 171.8756, 154.8061, 152.6364, 78.3332, 74.481, 67.8175, 61.1719, 61.0646, 46.6673, 44.1215, 42.2998, 39.7903, 36.7381, 35.299, 35.1502, 33.9511, 33.8357, 33.732, 33.5445, 33.2922, 33.1691, 33.0168, 31.7045, 30.6244, 30.3013, 29.9969, 29.795, 29.5519, 28.6799, 28.0298, 27.983, 27.889, 27.7869, 27.394, 27.3732, 26.0426, 25.812, 25.254, 24.545, 24.4502, 24.2543, 23.8581, 23.8233, 23.6975, 21.8311, 21.5761, 21.4745, 16.5684, 13.0566, 11.2364. HRMS (ESI) m/z: [M+NH₄]⁺ 1265.8456; calculated 1265.8357

Synthesis of 10: Compound **3** (0.03 g, 3.26x10⁻⁵ mol), 1,2-dibutanoyl-sn-glycerol (0.0260 g, 6.515x10⁻⁵ mol), DCC (0.00671 g, 3.26x10⁻⁵ mol), and DMAP (0.000795 g, 4.34x10⁻⁶ mol) were dissolved in DCM and stirred overnight at room temperature. Silica gel chromatography (40% ethyl acetate/hexanes) yielded compound **10** (45%). ¹H NMR (500 MHz, CDCl₃) δ 5.1862 (m, 1H), 5.0313 (m, 3H), 4.8927 (d, 1H), 4.517 (m, 1H), 4.2143 (m, 2H), 4.0622 (m, 2H), 3.3147 (m, 6H), 2.5706 (s, 3H), 2.4862 (d, 1H), 2.4294 (s, 2H), 2.2963 (m, 1H), 2.2554 (d, 1H), 2.2398 (d, 2H), 2.2226 (d, 1H), 2.1464 (m, 1H), 1.9414 (d, 1H), 1.915 (s, 1H), 1.8807 (m, 1H), 1.8086 (m, 1H), 1.7589 (d, 1H), 1.7125 (t, 2H), 1.6349 (s, 1H), 1.6036 (d, 1H), 1.5808 (d, 1H), 1.5191 (d, 8H), 1.4475 (s, 1H), 1.3534 (s, 29H), 1.1886 (s, 28H), 1.0186 (m, 2H), 0.8539 (s, 3H), 0.8087 (t, 6H), 0.7278 (d, 3H), 0.6608 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 172.3548, 172.2918, 171.867, 154.7961, 78.2958, 74.4704, 73.0438, 69.929, 67.8145, 61.168, 61.0663, 59.3794, 46.6658, 44.1197, 42.3091, 39.7918, 36.7406, 35.1504, 33.7343, 33.543, 33.2926, 33.1738, 33.0215, 30.8429, 30.8358, 30.3009, 29.9855, 29.55, 28.4206, 28.4023, 28.2628, 28.2471, 28.0855, 28.0429, 27.7838, 27.3949, 27.3765, 26.0427, 25.815, 24.5446, 23.8652, 23.8305, 21.8309, 21.6464, 21.4753, 16.5697, 13.0987, 11.2365. HRMS (ESI) m/z: [M+NH₄]⁺ 1321.9030; calculated 1321.8983

Synthesis of 11: Compound **4** (16 mg) was added to hydrochloric acid in dioxane (5 mL). The mixture was evaporated under reduced pressure and compound **11** (8.2 mg, 69%) was collected. ¹H NMR (500 MHz, CDCl₃) δ 5.12 (s, 1H), 4.53 (s, 11H), 3.98 (m, 3H), 3.57 (m, 1H), 3.22 (s, 8H), 3.12 (m, 4H), 2.74 (m, 4H), 2.25 (m, 3H), 2 (m, 1H), 1.93 (d, 1H), 1.84 (m, 2H), 1.7 (m, 4H), 1.55 (m, 5H), 1.44 (m, 1H), 1.36 (m, 2H), 1.23 (s, 13H), 1.03 (t, 1H), 0.89 (s, 2H), 0.81 (t, 4H), 0.75 (d, 2H), 0.7 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 174.2, 173.95, 170.47, 170.29, 170.27, 76.5, 74.96, 72.1, 69.73, 66.97, 65.08, 64.62, 64.56, 62.63, 62.21, 45.05, 43.36, 40.75, 37.5, 35.13, 35.06, 35, 34.56, 34.51, 34.15, 34.05, 33.54, 33.5, 31.47, 31.14, 31.1, 31.02, 30.79, 30.49, 28.8, 28.76, 28.73, 28.7, 26.82, 26.47, 25.24, 24.61, 22.39, 22.31, 22.29, 22.27, 21.41, 16.74, 13.08, 13.05. HRMS (ESI) m/z: [M+H]⁺ 822.5422; calculated 822.5474

Synthesis of 12: Compound **5** (1.2g) was added to hydrochloric acid in dioxane (5 mL) and stirred. The product was concentrated *in vacuo* until clear of dioxane. Lyophilization yielded compound **12** (). ¹H NMR (500 MHz, CDCl₃) δ 8.2 (m, 5H), 7.92 (s, 3H), 5.11 (m, 1H), 5.02 (m, 1H), 4.87 (s, 1H), 4.47 (s, 1H), 4.13 (m, 3H), 3.66 (m, 1H), 3.35 (d, 5H), 2.97 (m, 7H), 2.37 (m, 1H), 2.21 (m, 3H), 2.03 (m, 2H), 1.86 (m, 3H), 1.63 (m, 10H), 1.38 (m, 5H), 1.19 (m, 13H), 0.99 (s, 2H), 0.87 (m, 3H), 0.77 (s, 6H), 0.66 (m, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 174.52, 174.5, 173.94, 173.93, 170.07, 168.9, 168.63, 76.11, 75.2, 71.94, 67.93, 67.11, 65.28, 64.72, 44.93, 42.92, 40.51, 37.92, 36.05, 35.75, 34.59, 34.32, 34.26, 34.14, 32.9, 32.7, 31.89, 31.1, 30.37, 30.12, 29.47, 29.45, 29.32, 29.31, 29.29, 29.18, 29.14, 28.21, 26.47, 24.9, 23, 22.7, 22.21, 17.7, 14.16, 11.96, [M+H]⁺ 850.5838; calculated 850.5787

Synthesis of 13: Compound **6** (14 mg) was added to hydrochloric acid in dioxane (5 mL). The mixture was concentrated *in vacuo* until clear of dioxane. Lyophilization yielded compound **13** (7.3 mg, 69%). ¹H NMR (500 MHz, CDCl₃) δ 5.13 (s, 1H), 4.94 (s, 1H), 4.54 (s, 11H), 4.02 (m, 3H), 3.57 (m, 1H), 3.46 (m, 1H), 3.22 (s, 10H), 3.13 (m, 3H), 2.73 (m, 3H), 2.25 (m, 3H), 2 (d, 1H), 1.84 (m, 1H), 1.7 (m, 3H), 1.59 (d, 1H), 1.52 (t, 4H), 1.44 (d, 1H), 1.36 (m, 2H), 1.21 (s, 23H), 1.11 (m, 1H), 0.9 (s, 1H), 0.81 (t, 4H), 0.75 (d, 2H), 0.7 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 174.23, 174.19, 173.96, 173.92, 170.48, 170.3, 170.29, 76.51, 74.97, 72.11, 69.73, 68.85, 66.97, 65.08, 64.75, 64.62, 64.56, 62.63, 45.06, 43.37, 40.75, 37.51, 35.13, 35.05, 35, 34.55, 34.51, 34.16, 34.05, 33.54, 33.5, 33.46, 31.67, 31.13, 31.09, 31, 30.8, 30.49, 29.35, 29.33, 29.22, 29.2, 29.08, 29.07, 29.03, 29.02, 28.8, 26.82, 26.47, 25.25, 24.6, 22.39, 22.35, 22.34, 21.42, 16.75, 13.08, 13.06, 11.19. HRMS (ESI) m/z: [M+H]⁺ 878.6039; calculated 878.6100

Synthesis of 14: Compound **7** (11 mg) was added to hydrochloric acid in dioxane (5 mL). The mixture was then concentrated *in vacuo* until clear of dioxane. Lyophilization of mixture yielded compound **14** (6.7 mg, 81%). ¹H NMR (500 MHz, CDCl₃) δ 5.12 (s, 1H), 4.92 (m, 1H), 4.55 (m, 1H), 4.04 (m, 1H), 4 (m, 1H), 3.9 (m, 1H), 3.71 (m, 1H), 3.56 (m, 1H), 3.21 (m, 13H), 3.13 (m, 3H), 2.74 (m, 3H), 2.28 (m, 3H), 2.21 (m, 1H), 2.01 (m, 1H), 1.93 (d, 1H), 1.83 (m, 1H), 1.73 (m, 3H), 1.64 (m, 1H), 1.55 (m, 5H), 1.44 (d, 1H), 1.36 (s, 2H), 1.23 (s, 28H), 1.11 (m, 1H), 1.06 (s, 1H), 1.03 (d, 1H), 0.9 (s, 2H), 0.81 (t, 4H), 0.75 (d, 2H), 0.7 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 76.48, 74.93, 72.46, 72.07, 69.74, 68.87, 66.98, 65.06, 64.73, 64.61, 64.55, 63, 62.65, 60.28, 45.11, 45.06, 43.36, 40.76, 37.51, 35.14, 35.06, 35.01, 34.54, 34.16, 34.06, 33.53, 33.49, 33.45, 33.4, 31.68, 31.13, 31.08, 31, 30.8, 30.48, 29.39, 29.37, 29.33, 29.31, 29.3, 29.23, 29.21, 29.19, 29.09, 29.04, 29.03, 29, 28.99, 28.81, 26.82, 26.48, 25.3, 25.25, 24.63, 24.61, 24.6, 22.35, 21.42, 16.75, 13.06, 13.05, 11.19. HRMS (ESI) m/z: [M+CHOO₂]⁻ 950.5999; calculated 950.6323

Synthesis of 15: Compound **8** (20 mg) was added to hydrochloric acid in dioxane (5 mL). The mixture was concentrated *in vacuo* until clear of dioxane. Lyophilization of mixture yielded compound **15** (12.8 mg, 85%). ¹H NMR (500 MHz, CDCl₃) δ 8.23 (d, 4H), 7.89 (s, 2H), 5.2 (s, 1H), 5.1 (s, 1H), 4.87 (s, 1H), 4.48 (s, 1H), 4.25 (d, 2H), 4.04 (m, 2H), 3.7 (m, 3H), 3.35 (s, 5H), 2.97 (s, 5H), 2.28 (m, 5H), 2.04 (d, 2H), 1.87 (s, 3H), 1.64 (m, 11H), 1.42 (s, 3H), 1.21 (d, 13H), 1 (s, 2H), 0.82 (m, 12H), 0.63 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 172.92, 172.57, 172.33, 171.9, 169.07, 167.74, 167.54, 75.11, 74.25, 71.22, 70.93, 70.32, 70.09, 69.51, 69.47, 69.43, 67.83, 67.75, 67.37, 64.92, 64.01, 61.22, 61.17, 61.04, 60.96, 60.68, 46.83, 44, 41.8, 41.62, 39.5, 36.98, 35.06, 34.74, 33.58, 33.31, 33.15, 33.04, 32.99, 31.76, 30.9, 30.34, 30.21, 30.16, 30.1, 29.41, 28.68, 28.64, 28.35, 28.3, 27.07, 26.12, 25.46, 25.39, 23.78, 23.54, 23.51, 22.06, 21.68, 21.28, 21.19, 16.65, 13.12, 12.92, 12.9, 12.88, 11.07.

Synthesis of 16: Compound **9** (16 mg) was added to hydrochloric acid in dioxane (5 mL). The mixture was then concentrated *in vacuo* until clear of dioxane. Lyophilization yielded **16** (16 mg, 84% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.1735 (m, 4H), 5.1116 (m, 1H), 4.8795 (m, 1H), 4.4832 (m, 1H), 4.1507 (d, 1H), 4.03 (m, 3H), 3.8632 (m, 1H), 3.6975 (m, 5H), 3.6351 (s, 4H), 3.5829 (m, 4H), 3.5119 (m, 1H), 3.3482 (s, 3H), 2.9697 (s, 3H), 2.2643 (t, 3H), 2.0362 (m, 1H), 1.8669 (d, 3H), 1.693 (d, 5H), 1.5488 (d, 4H), 1.427 (m, 3H), 1.224 (m, 18H), 1.0056 (m, 2H), 0.8777 (m, 2H), 0.7764 (d, 6H), 0.6516 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 173.3174, 172.9298, 172.8926, 159.8675, 156.6579, 105.1073, 104.5352, 95.0089, 94.5776, 89.3275, 75.0773, 74.2916, 72.6071, 72.5201, 72.296, 71.2313, 70.3206, 70.292, 70.2697, 70.0946, 69.5084, 69.4757, 69.223, 68.6602, 67.8404, 66.9595, 66.065, 65.8248, 65.1298, 64.9187, 64.1146, 64.0177, 63.7715, 63.4754, 62.9768, 62.3053, 61.8114, 61.3615, 61.0592, 60.7581, 60.6829, 60.4666, 58.4303, 49.4526, 44.9599, 43.8917, 41.9037, 41.6647, 41.5751, 41.0922, 39.5027, 36.8906, 33.5927, 33.3352, 33.2263, 33.1348, 33.0693, 31.9468, 30.9045, 30.6441, 30.6159, 30.1158, 28.6803, 28.6383, 28.3455, 28.0826, 28.059, 28.0314, 27.9211, 27.8877, 27.8717, 27.2016, 25.8281, 24.1671, 23.8831, 23.861, 23.4634, 21.676, 21.5905, 21.5721, 21.1892, 16.6942, 13.0824, 13.0503, 10.9471. [M+H]⁺ 948.6455; calculated 948.6514

Synthesis of 17: Compound **10** (30 mg) was added to hydrochloric acid in dioxane (5 mL). The mixture was then concentrated *in vacuo* until clear of dioxane. Lyophilization yielded **17** (21 mg, 90% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.29 (d, 5H), 7.9 (s, 3H), 5.16 (d, 2H), 4.88 (s, 1H), 4.46 (s, 1H), 4.23 (s, 2H), 4.06 (m, 2H), 3.35 (s, 5H), 2.97 (s, 6H), 2.27 (m, 6H), 2.05 (s, 2H), 1.88 (s, 3H), 1.69 (d, 6H), 1.54 (d, 5H), 1.42 (s, 3H), 1.24 (s, 28H), 1.02 (s, 2H), 0.82 (m, 11H), 0.64 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 172.6, 172.33, 171.89, 67.82, 61.2, 44, 33.58, 33.31, 33.19, 33.04, 30.85, 30.07, 28.44, 28.42, 28.28, 28.26, 28.1, 28.06, 23.88, 23.84, 21.65, 21.17, 16.66, 13.12, 13.11, 11.06. HRMS (ESI) m/z: [M+H]⁺ 1004.7125; calculated 1004.7145

Synthesis of 18: Compound **3** (0.3 g, 0.000326 mol), solketal (0.086 g, 0.000651 mol), EDCI (0.0747 g, 0.000391 mol), and DMAP (0.00795 g, 6.51x10⁻⁵ mol) were dissolved in DCM and stirred for 3 hours at room temperature. Silica gel chromatography (35% E:H) yielded compound **18** (0.189 g, 56% yield). ¹H NMR (500 MHz, CDCl₃) δ 5.17 (d, 4H), 4.86 (s, 1H), 4.54 (m, 1H), 4.26 (m, 1H), 4.08 (m, 1H), 4 (m, 2H), 3.66 (m, 1H), 3.32 (d, 5H), 2.58 (s, 3H), 2.45 (m, 3H), 2.31 (m, 1H), 2.18 (m, 1H), 1.93 (m, 4H), 1.75 (m, 4H), 1.61 (m, 3H), 1.52 (m, 4H), 1.36 (m, 35H), 1.18 (m, 4H), 1.02 (m, 2H), 0.84 (s, 3H), 0.74 (d, 3H), 0.65 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 173.6, 171.86, 171.52, 171.43, 171.05, 155.78, 109.73, 109.2, 79.19, 76.14, 75.41, 73.96, 73.54, 70.87, 66.21, 65.88, 64.62, 62.96, 60.3, 47.48, 45.04, 43.28, 40.73, 37.67, 36.27, 36.15, 34.92, 34.83, 34.66, 34.49, 34.46, 34.43, 34.23, 31.24, 30.91, 30.87, 30.57, 29.59, 28.72, 28.36, 27.92, 27, 26.74, 26.64, 25.47, 25.3, 25.24, 22.77, 22.42, 20.97, 17.52, 14.14.

Synthesis of 19: Compound **18** (0.17 g, 8.85x10⁻⁶ mol) was dissolved in prepared solution of H₂O and AcOH (2:8 H₂O:AcOH). The mixture was stirred overnight at 55 °C. Silica gel chromatography (80% E:H) was performed and yielded compound **19** (0.1 g, 60% yield). ¹H NMR (500 MHz, CDCl₃) δ 5.1 (d, 3H), 4.89 (s, 1H), 4.52 (m, 1H), 4.08 (m, 3H), 3.86 (t, 1H), 3.61 (m, 1H), 3.53 (m, 1H), 3.32 (m, 6H), 2.58 (d, 3H), 2.45 (d, 3H), 2.31 (m, 1H), 2.18 (m, 1H), 1.98 (s, 3H), 1.93 (d, 2H), 1.88 (s, 1H), 1.81 (m, 1H), 1.72 (d, 2H), 1.66 (s, 1H), 1.56 (m, 6H), 1.38 (s, 28H), 1.18 (t, 4H), 1.12 (d, 1H), 1.01 (m, 2H), 0.86 (s, 3H), 0.74 (d, 3H), 0.66 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 174.31, 172, 171.66, 171.57, 171.25, 155.94, 155.87, 79.42, 79.34, 75.5, 74.98, 74.08, 70.99, 70.12, 65.27, 65.26, 63.36, 62, 60.43, 47.26, 45.11, 43.36, 40.78, 37.71, 36.34, 36.18, 34.94, 34.87, 34.72, 34.54, 34.48, 34.29, 31.29, 30.85, 30.53, 29.67, 28.81, 28.73, 27.04, 26.81, 25.56, 22.82, 22.48, 21.06, 17.58, 14.18, 14.13.

Synthesis of 20: Compound **19** (0.1 g, 0.000101 mol), butanoic acid (0.02 g, 0.000241 mol), EDCI (0.0461 g, 0.000241 mol), and DMAP (0.00245 g, 2.01x10⁻⁵ mol) were dissolved in DCM (100 ml). The mixture was stirred for 3 hours at room temperature. Silica gel chromatography (40% E:H) produced compound **20** (80 mg, 70% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.16 (d, 8H), 5.15 (d, 2H), 4.86 (s, 2H), 4.54 (s, 3H), 4.15 (d, 5H), 3.7 (m, 11H), 3.11 (d, 11H), 2.25 (s, 7H), 2.02 (d, 3H), 1.87 (s, 3H), 1.65 (m, 13H), 1.43 (m, 4H), 1.23 (d, 6H), 1.01 (s, 2H), 0.87 (s, 10H), 0.76 (s, 3H), 0.63 (s, 4H). ¹³C NMR (125 MHz, CDCl₃) δ 173.64, 173.18, 172.77, 170.32, 169.01, 168.75, 76.1, 75.08, 72.28, 71.96, 71.12, 68.87, 67.08, 64.99, 62.26, 62.19, 62.02, 61.61, 47.83, 45.02, 42.66, 40.49, 37.95, 36.28, 36.07, 35.91, 34.62, 34.31, 32.87, 31.17, 30.44, 29.7, 28.08, 27.18, 26.56, 25.09, 24.87, 24.4, 23.05, 22.23, 18.39, 18.35, 17.69, 14.14, 13.65, 13.61, 12.11, 1.02.

Synthesis of 21: Compound **20** (25 mg) was added to hydrochloric acid in dioxane (5 ml). Mixture was concentrated in vacuo and lyophilized without further purification. Process yielded compound **21** (17 mg, 92% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.14 (d, 7H), 5.18 (d, 2H), 4.89 (s, 1H), 4.51 (s, 1H), 4.15 (d, 4H), 3.67 (m, 1H), 3.13 (d, 10H), 2.29 (d, 6H), 2.05 (m, 2H), 1.72 (m, 14H), 1.43 (s, 3H), 1.29 (m, 3H), 1.16 (s, 2H), 1.01 (s, 2H), 0.87 (m, 9H), 0.76 (s, 3H), 0.64 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 172.61, 172.16, 171.75, 169.19, 167.86, 167.61, 75.12, 74.18, 70.96, 67.84, 61.24, 61.16, 44, 41.65, 39.47, 36.95, 35.05, 34.89, 33.59, 33.3, 31.96, 30.12, 29.44, 28.68, 27.07, 26.09, 21.2, 17.37, 17.33, 16.67, 12.63, 12.58, 11.08.

Synthesis of 22: Solketal R (0.4 g, 0.00303 mol), capric acid (0.521 g, 0.00303 mol), EDCI (0.564 g, 0.00363 mol), and DMAP (0.0739 g, 0.000606 mol) were dissolved in DCM (100 ml) and was stirred overnight at room temperature. The mixture was extracted twice with hydrochloric acid and brine followed by extracting once with NaHCO₃. Silica gel chromatography (40% E:H) yielded compound **22** (0.6 g, 69%) as a slightly yellow liquid.

Synthesis of 23: Compound **22** (0.6 g) was dissolved in a 1:1 mixture of TFA and MeOH and stirred for 30 minutes at room temperature. Silica gel chromatography (70% E:H) was performed and yielded compound **23** (0.455 g, 88%). ¹H NMR (500 MHz, CDCl₃) δ 4.1073 (d, 2H), 3.8774 (s, 1H), 3.6593 (d, 1H), 3.4496 (m, 3H), 2.2997 (t, 2H), 1.582 (t, 2H), 1.1927 (d, 12H), 0.8334 (t, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 174.3554, 74.7215, 70.1912, 69.2976, 68.2359, 64.9692, 63.3755, 62.9776, 61.6698, 34.1073, 31.8091, 29.3771, 29.2206, 29.0995, 24.8409, 22.6142, 14.047.

Synthesis of 24: Compound **3** (0.26 g, 0.000282 mol), compound **23** (0.139 g, 0.000565 mol), EDCI (0.065 g, 0.000339 mol), and DMAP (0.00689 g, 5.65x10⁻⁵ mol) were dissolved in DCM (100 ml). The mixture was stirred for 3 hours at room temperature. Silica gel chromatography (40% E:T) produced compound **24** (0.32 g, 98%). ¹H NMR (500 MHz, CDCl₃) δ 5.0837 (m, 2H), 4.9858 (s, 2H), 4.8787 (m, 1H), 4.5171 (m, 1H), 4.11 (m, 2H), 4.056 (m, 1H), 4.0061 (m, 1H), 3.3349 (d, 6H), 2.5716 (t, 3H), 2.4468 (m, 3H), 2.3041 (m, 3H), 2.1882 (m, 1H), 1.9171 (m, 3H), 1.7495 (m, 5H), 1.5586 (m, 8H), 1.3752 (m, 31H), 1.1888 (d, 16H), 1.0211 (m, 2H), 0.8583 (s, 3H), 0.8043 (t, 3H), 0.7366 (d, 3H),

0.6542 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 172.9838, 172.8926, 170.9781, 170.5432, 154.8252, 129.0444, 113.0951, 78.3612, 74.4794, 73.0583, 69.939, 67.2393, 64.1765, 63.9783, 60.4028, 54.2954, 46.3488, 44.1114, 42.3501, 39.7891, 36.717, 35.1659, 33.9405, 33.7277, 33.5378, 33.29, 33.0719, 30.8246, 30.2997, 29.8453, 29.511, 28.6804, 28.3782, 28.2266, 28.0936, 27.8063, 27.4023, 27.3758, 26.0527, 25.8174, 24.5553, 23.8549, 21.8266, 21.6413, 21.4766, 16.5655, 13.0987, 11.2113.

Synthesis of 25: Compound **24** (20 mg) was added to hydrochloric acid in dioxane (5 mL) and stirred at room temperature. The mixture was then concentrated *in vacuo* until clear of dioxane. Lyophilization yielded compound **25** (14 mg, 94%). ¹H NMR (500 MHz, CDCl₃) δ 8.0375 (d, 6H), 5.0703 (s, 1H), 4.8802 (s, 1H), 4.4874 (s, 1H), 4.2497 (d, 1H), 4.0667 (m, 3H), 3.6448 (s, 2H), 3.3543 (s, 5H), 2.9236 (s, 5H), 2.3054 (m, 4H), 1.9504 (m, 5H), 1.6353 (m, 9H), 1.4199 (s, 4H), 1.2245 (s, 19H), 1.0211 (s, 2H), 0.8178 (m, 9H), 0.6458 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 34.139, 33.7651, 32.9451, 32.7728, 31.942, 31.8896, 31.092, 29.7188, 29.6777, 29.6419, 29.5911, 29.5478, 29.4679, 29.3834, 29.3199, 29.3041, 29.183, 29.1339, 28.2023, 27.2666, 26.8405, 24.897, 24.7662, 22.6958, 22.2129, 17.7001, 14.1576, 12.115, 11.9758.

Synthesis of 26: Solketal L (0.4 g, 0.00303 mol), capric acid (0.521 g, 0.00303 mol), EDCI (0.564 g, 0.00363 mol), and DMAP (0.0739 g, 0.000606 mol) were dissolved in DCM and stirred overnight at room temperature. The mixture was extracted twice in HCl and brine followed by extracting once with NaHCO₃. Silica gel chromatography (40% E:H) yielded compound **26** (0.6 g, 69%) as a slightly yellow liquid.

Synthesis of 27: Compound **26** (0.6 g) was dissolved in a 1:1 mixture of TFA and MeOH and stirred for 30 minutes at room temperature. Silica gel chromatography (70% E:H) produced compound **27** (0.455 g, 88%). ¹H NMR (500 MHz, CDCl₃) δ 4.0512 (d, 2H), 3.8525 (m, 3H), 3.5921 (d, 1H), 3.4979 (m, 1H), 2.2668 (t, 2H), 1.5392 (t, 2H), 1.2086 (d, 12H), 0.8057 (t, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 174.336, 74.6513, 70.1541, 64.922, 63.3842, 61.3929, 34.0979, 31.8231, 29.4013, 29.2523, 29.238, 29.1208, 24.837, 22.6252, 14.0545.

Synthesis of 28: Compound **3** (0.15 g, 0.000163 mol), compound **27** (0.0801 g, 0.000326 mol), EDCI (0.03 g, 0.000195 mol), and DMAP (0.00397 g, 3.26x10⁻⁵ mol) were dissolved in DCM. The mixture stirred for 3 hours at room temperature. Silica gel chromatography (40% E:T) was performed and yielded compound **28** (0.18 g, 96% yield). (¹H NMR (500 MHz, CDCl₃) δ 5.0216 (m, 3H), 4.8891 (s, 1H), 4.5172 (m, 1H), 4.0777 (m, 4H), 3.3335 (m, 5H), 2.5726 (t, 3H), 2.452 (t, 3H), 2.3037 (m, 4H), 2.1869 (m, 1H), 1.9185 (m, 3H), 1.7478 (m, 4H), 1.5592 (m, 8H), 1.3771 (s, 27H), 1.1911 (d, 21H), 1.0204 (m, 2H), 0.8586 (s, 3H), 0.8051 (t, 4H), 0.7388 (d, 3H), 0.6662 (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 173.329, 172.9889, 172.8977, 170.6463, 154.842, 78.3619, 74.4819, 73.0538, 69.9471, 69.2243, 67.2412, 64.1791, 64.141, 63.9923, 62.2928, 61.5335, 61.0316, 60.9223, 60.3753, 46.3484, 44.1113, 42.3465, 39.7883, 36.7167, 35.3113, 35.157, 33.9457, 33.7275, 33.5374, 33.2895, 33.1262, 33.0725, 30.8243, 30.2985, 29.8444, 29.5109, 28.6778, 28.3774, 28.2238, 28.0938, 27.8057, 27.4021, 27.3759, 26.0544, 25.817, 24.5554, 23.8862, 23.8548, 21.8266, 21.6412, 21.4759, 16.5662, 13.0977, 11.2112.

Synthesis of 29: Compound **28** (19 mg) was added to hydrochloric acid in dioxane (5 mL) and stirred for 3 hours at room temperature. The product was concentrated *in vacuo* until clear of dioxane. Lyophilization yielded compound **29** (12 mg, 85% yield). ¹H NMR (500 MHz, CDCl₃) δ 8.2025 (d, 5H), 7.8527 (s, 3H), 5.097 (t, 1H), 4.8763 (s, 1H), 4.4942 (s, 1H), 4.1497 (m, 4H), 3.6288 (m, 3H), 3.3356 (s, 6H), 2.9737 (s, 5H), 2.3838 (s, 1H), 2.2209 (m, 3H), 2.0408 (s, 2H), 1.8693 (s, 3H), 1.6704 (d, 7H), 1.5263 (t, 3H), 1.4045 (d, 4H), 1.2056 (s, 15H), 0.9947 (s, 2H), 0.8832 (s, 2H), 0.8089 (t, 4H), 0.7668 (s, 2H), 0.6626 (s, 2H). ¹³C NMR (125 MHz, CDCl₃) δ 173.4533, 172.8842, 169.0912, 167.9143, 167.6504, 74.0919, 70.9066, 66.8734, 64.2432, 63.7141, 43.897, 39.4576, 36.868, 33.5149, 33.2175, 33.0982, 31.8565, 30.8514, 28.6811, 28.4292, 28.2811, 28.2658, 28.1438, 23.8585, 21.658, 21.1729, 16.6578, 13.1203, 10.9301.

Bacterial and Fungal Culture: Bacteria were acquired from the Antibacterial Resistance Leadership Group (ARLG, Durham, North Carolina) and the American Type Culture Collection, (ATCC, Manassas, Virginia), with the ARLG sourcing *Klebsiella pneumoniae* 1360 and the ATCC sourcing *Staphylococcus aureus* 25923, *Staphylococcus aureus* 6538, *Staphylococcus aureus* 27734, *Staphylococcus pseudintermedius* 49444, *Streptococcus uberis* 27958, *Staphylococcus aureus* BAA-42, *Proteus mirabilis* 29906, *Klebsiella pneumoniae* BAA-1706, *Klebsiella pneumoniae* 13883, *Pseudomonas aeruginosa* 47085, *Pseudomonas aeruginosa* 27853, *Acinetobacter baumannii* 19606, and *Escherichia coli* 25922. *Candida albicans* 90028 was also acquired from the ATCC while *Candida auris* strains 381-390 were acquired from the Center for Disease Control and Prevention (Atlanta, Georgia). Tryptic Soy Broth (TSB,

Millipore, Burlington, Massachusetts) and Sabouraud Dextrose Medium (SDM, made in house) were used to culture bacterial and fungal strains, respectively and culture plates were made by adding agar (Thermo Fisher Scientific, Waltham, Massachusetts) to media.

Antimicrobial Assays: Overnight cultures were prepared by removing a colony from freshly spread culture plates and adding the colony to a flask with culture media placed on a shaker plate (Thermo Fisher Scientific) at 37°C. Cultures were pelleted in a Sorvall ST16R centrifuge (Thermo Fisher Scientific) and rinsed three times with phosphate buffered saline (Thermo Fisher Scientific). Cells were resuspended and absorbance taken on a Genesys 30 visible spectrophotometer (Thermo Fisher Scientific) at 600 nm. To make inoculum, cell suspension was diluted to 10⁶ colony forming units (CFU)/mL in TSB for bacterial strains and 10⁵ CFU/ml SDM for fungal strains. Minimum Inhibitory Concentrations (MICs) were determined using a broth microdilution method in 96-well plates. Briefly, in triplicate, a series of two-fold dilutions of tested compounds were made from 1-128 µg/mL and inoculum, made as described above, was added 1:1 resulting in final test concentrations of 0.5-64 µg/mL. Plates were incubated at 37°C for 24 hours then visually checked for turbidity. The reported MIC is the lowest concentration at which no visible growth was observed. Minimum Bactericidal Concentrations (MBC) and Minimum Fungicidal Concentration (MFC) assays were run under identical conditions, but inoculum was diluted in PBS and plated to determine an exact initial CFU/mL. After 24 hour incubation, wells with no visible growth had 100 µL removed and placed into Dey/Engley Neutralizing Broth (Alpha Biosciences, Baltimore, Maryland). The resulting mixture was spread on agar plates, incubated for 24 hours (for bacteria) or 48 hours (for fungi) at 37°C and counted to determine a final CFU/mL. The reported MBC and MFC were the most dilute concentration at which at least two of the triplicates had ≤99.9% reduction in CFUs relative to controls.

Preparation of buffer solutions for stability assays:

3.6: Formic acid was added to deionized water (0.3 M) and titrated using NaOH to pH 3.6 as measured by pH meter.
7.2: Imidazole was added to deionized water (0.3 M) and titrated using aqueous HCl to pH 7.2 as measured by pH meter.
10: Triethylamine was added to deionized water (0.3 M) and titrated using aqueous HCl to pH 10.2 as measured by pH meter.

General preparation of 12 dissolved in various buffer solutions: 10 mg of 12 and 10 mg of CSA-131 was dissolved in 2 mL of a selected buffer solution and 2 mL of deionized water.

Preparation of 12 and 131 dissolved in lipase solution:

20 mg of 12 and 20 mg of 131 were added to a 500 ml volumetric flask and filled to marked level with 7.2 buffer solution. This solution was then added to a 100 ml volumetric flask that contained 3 mg of bovine esterase and filled to the marked level.

Preparation of reagent solutions for derivatization procedure:

Counter-buffer: Triethylamine was added to deionized water (0.3 M) and titrated using aqueous HCl to pH 10.4 as measured by pH meter.

Fmoc-Cl: 18 mg of Fmoc-Cl was added to 4 mL of acetone and prepared fresh on each day used.

Adamantan-1-amine: 10 mg of adamantan-1-amine was added to 4 mL of acetone and prepared fresh on a weekly basis.

General derivatization procedure using Fmoc-Cl: To a plastic HPLC vial, 100 µL of solution of interest is added into 200 µL of counter-buffer solution, 100 µL of Fmoc-Cl solution, and 900 µL of acetone. Mixture is then allowed to react for 20 minutes at room temperature and then stopped using 100 µL of Adamantan-1-amine solution. Mixture is then analysed as soon as possible by HPLC to avoid sample degradation.

HPLC method:

Detector: PDA, analyzing at 254 nm.

Pump A: Water, unbuffered

Pump B: Acetonitrile

Column: ZORBAX RRHT StableBond C3, 3.0 x 100 mm, 1.8 µm, 600 bar. 80Å, 600 bar pressure limit (Sourced from Agilent, Part Number: 828975-309)

Starting flow rate: 0.5 mL/min

Pump B concentration at start: 75%

Column Oven: 30 degrees Celsius



Time	Module	Command	Value
1	Pumps	Total Flow	0.5
5.01	Pumps	Pump B Conc.	100
5.99	Pumps	Total Flow	0.85
19.99	Pumps	Pump B Conc.	100
19.99	Pumps	Total Flow	0.85
20	Pumps	Pump B Conc.	75
20.01	Pumps	Total Flow	0.5
35	Controller	Stop	

CSA-131 elutes at 8.2 minutes

CSA-12 elutes at 4.7 minutes

Critical Micelle Concentration Assay Protocol: Orange OT solution was prepared by adding 2 mg of orange OT to 100 mL of water, mixing vigorously, and allowing any solids to settle to the bottom. The various analytes of interest (CSA-131, CSA-12, SDS, CSA-44) were diluted to the appropriate concentration and 1.2 mL of orange OT solution was added to the analyte solution, with the final volume being 2.0 mL and the final concentration being plotted. Absorbance of each concentration was then measured, compared to blank, and the difference plotted.

Measurement of Membrane Depolarization: Assay buffer was prepared with 250 mM sucrose (Mallinckrodt Baker, Phillipsburg, NJ), 5 mM MgSO₄ (Mallinckrodt Baker), and 10 mM KH₂PO₄ (Thermo Fisher). A MRSA ATCC BAA-42 culture was grown overnight in TSB. Cells were harvested by centrifugation and washed with assay buffer three times. The pellet was resuspended in assay buffer and adjusted to obtain an optical density of 0.085 measured at 600 nm. 3,3'-diethylthiadicarbocyanine iodide (Sigma-Aldrich, St. Louis, MO) was added to the cell suspension to obtain a 1 μM concentration and the dye was allowed to incorporate for 15 minutes at room temperature. Measurements were taken on a spectrofluorometer (Photon Technology International, Lawrenceville, NJ) with excitation at 600 nm and emissions measured at 660 nm. Samples were stirred throughout data collection and measurements were taken every 20 seconds for 1,000 seconds. CSAs were added after t=140s to achieve a final concentration of 8 μg/mL. Baseline correction was performed using the average fluorescence of the first 7 data points.