

Supporting Information

An Ammonium Chloride Promoted Three Component Synthesis of 5-Aminooxazole and Four Component Synthesis of Pyrrolo[3,4-b]pyridin-5-one

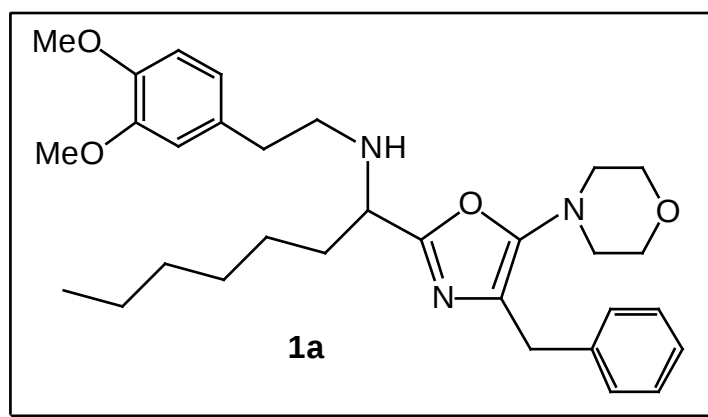
Pierre Janvier,^a Xiaowen Sun,^a Hugues Bienaymé,^b Jieping Zhu^{a,*}

^a Institut de Chimie des Substances Naturelles, CNRS, 91198 Gif-sur-Yvette, France

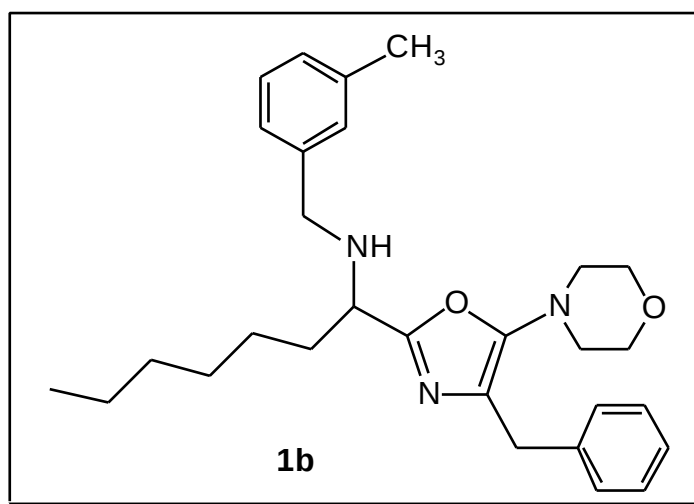
^b Chrysalon, 11 Avenue A. Einstein, 69100 Villeurbanne, France

Experimental Section

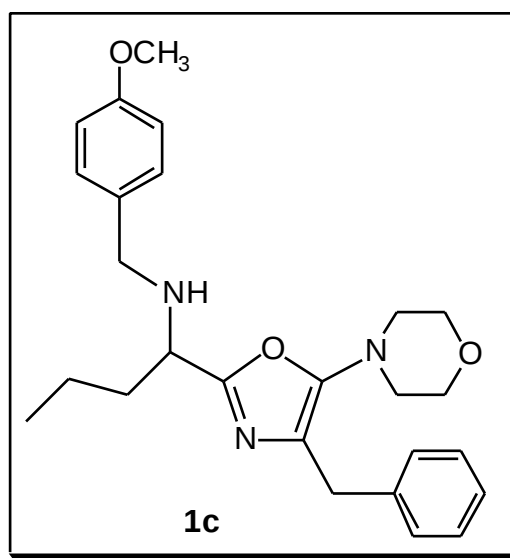
Melting points were determined with a Kofler apparatus and were uncorrected. Infrared (IR) spectra were recorded on a Nicolet-205 spectrometer. ¹H NMR spectra were measured on Bruker AC-200 (200MHz), Bruker AC-250 (250MHz), Bruker (300 MHz) and Bruker WM-400 (400MHz) spectrometers with tetramethylsilane as internal standard (δ ppm). Solvents and reagents were purified according to standard laboratory techniques. All reactions requiring anhydrous conditions or in an inert atmosphere were conducted under an atmosphere of Argon.



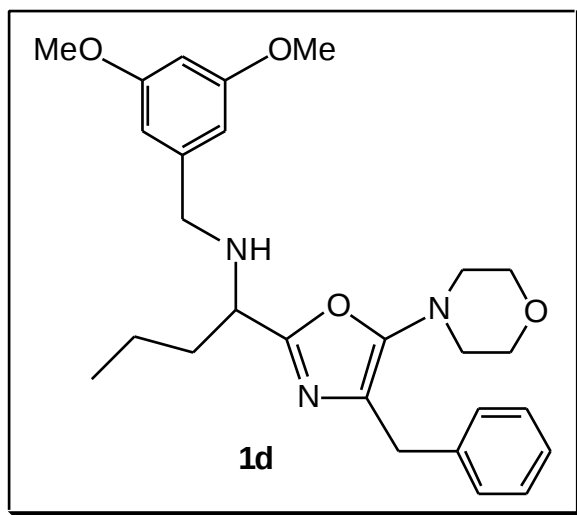
Pale yellow oil, yield: 68% ; IR (CHCl₃) ν 3316, 2932, 2859, 1666, 1592, 1516, 1454, 1263, 1114 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.13-7.30 (m, 5H), 6.65-6.80 (m, 3H), 3.85 (s, 3H), 3.82 (s, 3H), 3.81 (s, 2H), 3.71 (m, 5H), 2.93 (m, 4H), 2.60-2.82 (m, 4H), 1.74 (q, *J* = 7.0 Hz, 2H), 1.23 (m, 8H), 0.85 (t, *J* = 6.5 Hz, 3H); ¹³C NMR (62.5MHz, CDCl₃) δ 160.8, 151.8, 149.0, 147.5, 139.7, 132.6, 128.4, 126.2, 124.4, 120.6, 112.1, 111.4, 67.0, 57.2, 56.0, 55.9, 51.1, 49.1, 36.0, 34.5, 31.9, 31.7, 29.0, 25.9, 22.6, 14.1; MS (EI): *m/z* 521 (M⁺, 29%).



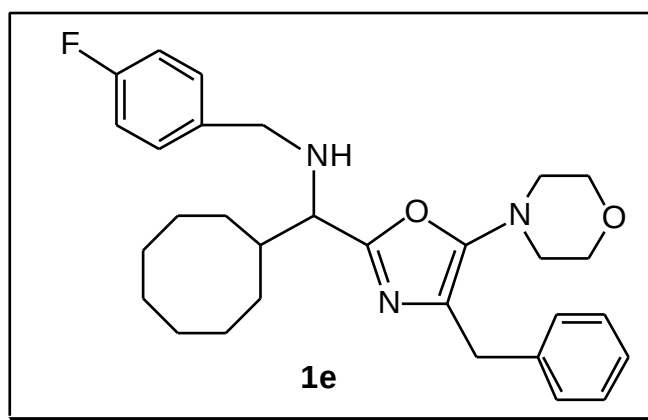
Pale yellow oil, yield: 65%; IR (CHCl₃) ν 3333, 2929, 2860, 1666, 1608, 1496, 1454, 1376, 1236, 1115 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.00-7.33 (m, 9H), 3.83 (s, 2H), 3.72 (m, 5H), 3.68 (d, J = 12.8 Hz, 1H), 3.60 (d, J = 12.8 Hz, 1H), 2.96 (m, 4H), 2.32 (s, 3H), 1.77 (q, J = 7.1 Hz, 2H), 1.23 (m, 8H), 0.85 (t, J = 6.4 Hz, 3H); ¹³C NMR (62.5MHz, CDCl₃) δ 161.0, 151.8, 140.0, 139.7, 138.0, 129.1, 128.5, 128.4, 128.3, 127.8, 126.2, 125.4, 124.6, 67.0, 56.6, 51.8, 51.2, 34.5, 31.9, 31.7, 29.0, 25.9, 22.6, 21.4, 14.1; MS (EI): m/z 461 (M⁺, 4%).



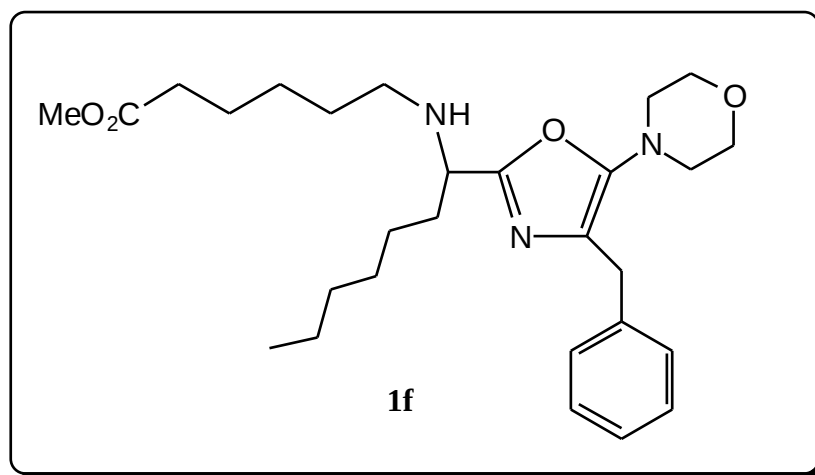
Pale yellow oil, yield: 60%; IR (CHCl₃) ν 3329, 2961, 2839, 1666, 1612, 1513, 1454, 1249, 1114 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.20-7.35 (m, 5H), 7.17 (d, J = 8.6 Hz, 2H), 6.81 (d, J = 8.6 Hz, 2H), 3.83 (s, 2H), 3.78 (s, 3H), 3.72 (m, 5H), 3.66 (d, J = 12.8 Hz, 1H), 3.57 (d, J = 12.8 Hz, 1H), 2.96 (m, 4H), 1.74 (q, J = 7.5 Hz, 2H), 1.32 (qt, J = 7.3, 7.5 Hz, 2H), 0.87 (t, J = 7.3 Hz, 3H); ¹³C NMR (62.5MHz, CDCl₃) δ 161.1, 158.7, 151.8, 139.8, 132.3, 129.5, 128.5, 128.5, 126.2, 124.6, 113.8, 67.0, 56.2, 55.3, 51.2, 36.8, 31.9, 19.3, 13.9; MS (ESI): m/z 436 (M+H⁺, 85%).



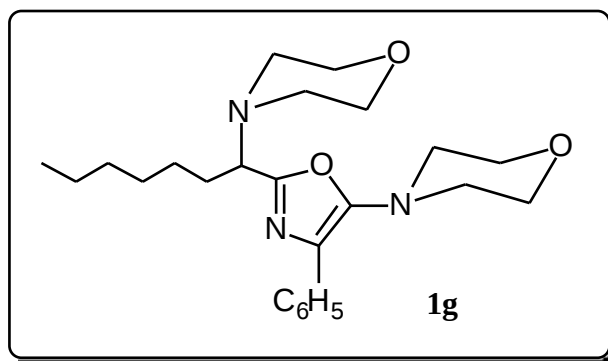
Pale yellow oil, yield: 75%; IR (CHCl₃) ν 3333, 2964, 2842, 1665, 1598, 1455, 1431, 1205, 1155, 1115 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.13-7.34 (m, 5H), 6.46 (d, J = 2.3 Hz, 2H), 6.34 (t, J = 2.3 Hz, 1H), 3.83 (s, 2H), 3.76 (s, 6H), 3.72 (m, 5H), 3.70 (d, J = 13.3 Hz, 1H), 3.59 (d, J = 13.3 Hz, 1H), 2.96 (m, 4H), 1.76 (q, J = 7.5 Hz, 2H), 1.35 (qt, J = 7.3, 7.5 Hz, 2H), 0.89 (t, J = 7.3 Hz, 3H); ¹³C NMR (62.5MHz, CDCl₃) δ 160.9, 160.8, 151.8, 142.6, 139.7, 128.4, 126.1, 124.4, 106.0, 99.1, 66.9, 56.1, 55.3, 51.8, 51.1, 36.7, 31.9, 19.3, 13.9; MS (EI): m/z 465 (M⁺, 16%).



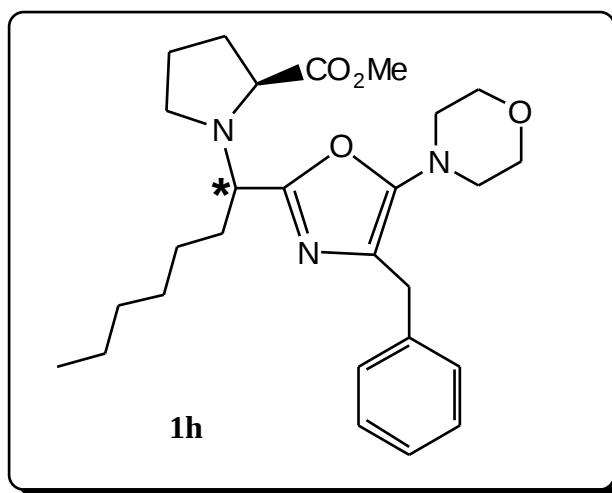
Pale yellow oil, yield: 60%; IR (CHCl₃) ν 3334, 2922, 2851, 1667, 1602, 1510, 1454, 1374, 1115 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.13-7.32 (m, 5H), 7.08 (m, 2H), 6.93 (m, 2H), 3.82 (s, 2H), 3.73 (m, 4H), 3.47 (d, J = 7.2 Hz, 1H), 2.93 (m, 4H), 2.58-2.82 (m, 4H), 1.18-2.00 (m, 15H); ¹³C NMR (62.5MHz, CDCl₃) δ 161.5 (d, J = 242.3 Hz), 160.7, 151.8, 139.8, 135.8, 130.1 (d, J = 7.5 Hz), 128.4, 126.1, 124.4, 115.1 (d, J = 21.1 Hz), 67.0, 63.1, 51.2, 49.6, 41.8, 35.6, 31.8, 29.5, 29.4, 27.0, 26.8, 26.6, 26.0, 25.7; MS (ESI): m/z 506 (M+H⁺, 85%).



Pale yellow oil, yield: 65%; IR (CHCl₃) ν 3019, 2954, 2931, 2860, 1731, 1661, 1643, 1586, 1495, 1454, 1438, 1375, 1302, 1264, 1222, 1212, 1160, 1114, 1000, 909 cm⁻¹; ¹H NMR (250 MHz, CDCl₃) δ 7.26 (m, 5H), 3.86 (s, 2H), 3.76 (m, 4H), 3.72 (t, J = 4.9 Hz, 1H), 3.69 (s, 3H), 3.01 (m, 4H), 2.64 (br. s, 1H), 2.54 (t, J = 7.4 Hz, 2H), 2.33 (t, J = 7.4 Hz, 2H), 1.80 (q, J = 6.9 Hz, 2H), 1.64 (quintet, J = 7.4 Hz, 2H), 1.20-1.50 (m, 12 H), 0.90 (t, J = 6.5 Hz, 3H); ¹³C NMR (62.5 MHz, CDCl₃) δ 174.1, 160.0, 152.0, 139.5, 128.5, 126.1, 124.3, 66.8, 57.0, 53.4, 51.4, 51.0, 47.2, 34.0, 33.9, 31.7, 31.6, 29.2, 28.9, 26.7, 25.6, 24.6, 22.4, 14.1; MS (ESI): m/z 486 (M+H)⁺

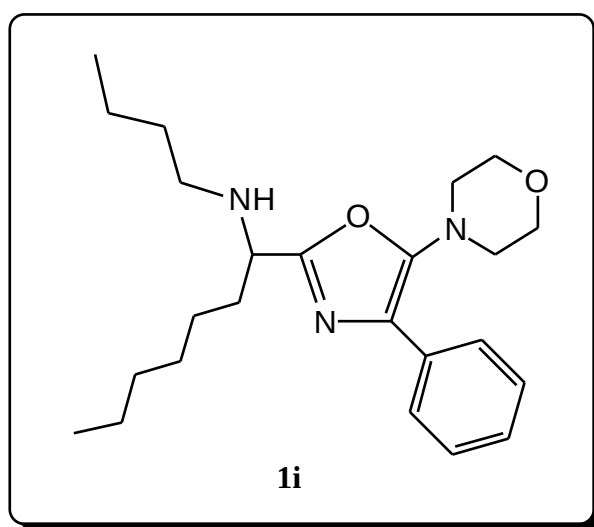


Yield 90%; IR (CHCl₃) ν 2961, 2860, 1637, 1497, 1453, 1374, 1263, 1194, 1115, 1070, 985, 911, 862 cm⁻¹; ¹H NMR (250 MHz, CDCl₃) δ 7.93 (m, 2H), 7.39 (m, 2H), 7.25 (m, 1H), 3.86 (m, 4H), 3.61-3.73 (m, 5H), 3.11 (m, 4H), 2.52-2.61 (m, 4H), 1.90 (m, 2H), 1.26-1.31 (m, 8H), 0.87 (t, J = 7.5 Hz, 3H); ¹³C NMR (62.5 MHz, CDCl₃) δ 157.8, 150.7, 131.8, 128.3, 126.8, 125.9, 123.5, 67.2, 66.8, 63.1, 50.3, 50.0, 31.5, 29.7, 28.9, 26.1, 22.5, 13.9; MS (EI): m/z 413 (M⁺, 3%), 328 (15), 184 (100), 126 (7).

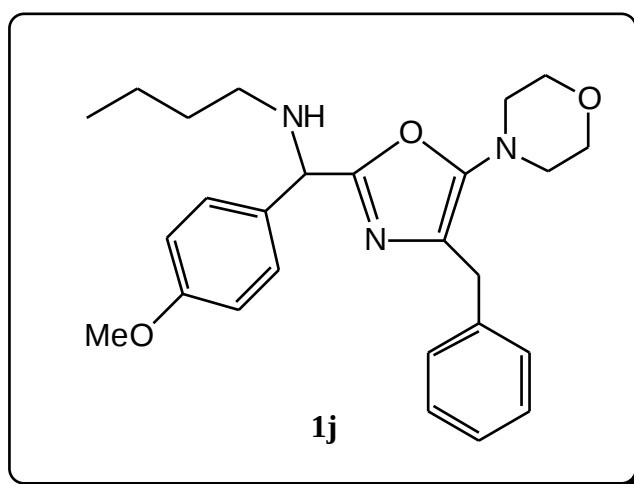


Diastereomer A: yellow oil, yield : 68%; $[\alpha]_D = -44.8^\circ$; IR (CHCl₃) ν 3025, 3019, 2956, 2929, 2860, 1736, 1665, 1635, 1495, 1454, 1437, 1375, 1262, 1221, 1204, 1177, 1114, 1030, 920 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.23 (m, 5H), 3.95 (t, $J = 7.3$ Hz, 1H), 3.81 (s, 2H), 3.71 (m, 4H), 3.69 (s, 3H), 3.44 (dd, $J = 8.1, 6.4$ Hz, 1H), 3.08 (m, 1H), 2.94 (m, 4H), 2.66 (m, 1H), 1.95-1.60 (m, 8H), 1.37-1.21 (m, 6H), 0.86 (m, 3H); ¹³C NMR (62 MHz, CDCl₃) δ 174.7, 158.7, 151.9, 139.8, 128.5, 128.4, 126.2, 124.2, 66.9, 62.8, 58.3, 51.8, 51.1, 47.4, 31.8, 31.7, 31.6, 29.4, 29.1, 26.3, 23.6, 22.7, 14.2; MS (ESI): m/z (M+Na)⁺ 492.

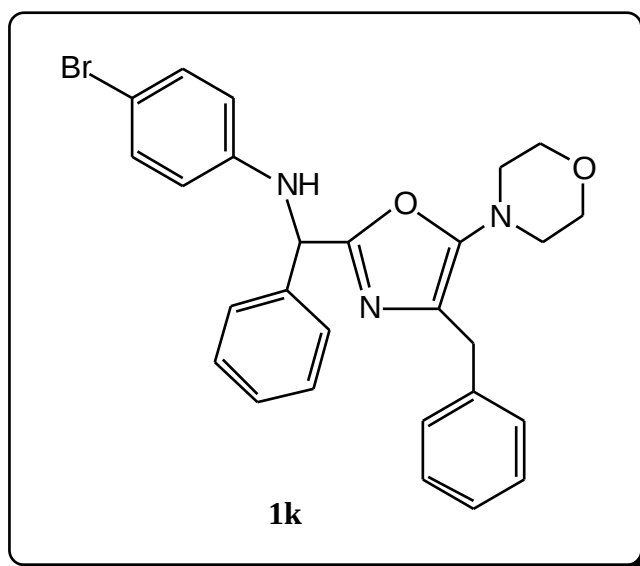
Diastereomer B: yellow oil, yield : 28%; $[\alpha]_D = -25.7^\circ$; IR (CHCl₃) ν 3026, 3016, 2957, 2929, 2859, 1736, 1664, 1635, 1495, 1454, 1436, 1375, 1262, 1228, 1205, 1174, 1114, 1030, 928 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.23 (m, 5H), 3.83-3.77 (m, 4H), 3.72 (m, 4H), 3.69 (s, 3H), 3.20 (m, 1H), 2.96 (m, 4H), 2.63 (m, 1H), 1.95-1.60 (m, 8H), 1.33-1.15 (m, 6H), 0.84 (m, 3H); ¹³C NMR (62 MHz, CDCl₃) δ 175.5, 158.7, 151.7, 139.5, 128.3, 128.2, 125.9, 124.0, 66.7, 60.9, 60.6, 52.2, 51.5, 50.9, 32.1, 31.6, 31.4, 30.4, 28.8, 26.0, 23.9, 22.3, 13.9; MS (ESI): m/z (M+Na)⁺ 492.



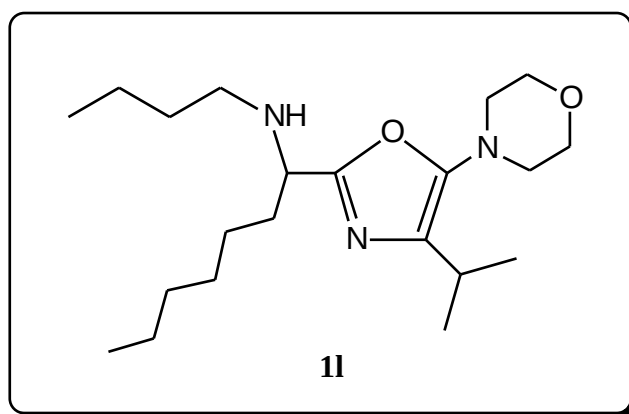
Pale yellow oil, yield: 53%; IR (CHCl₃) ν 2960, 2930, 2860, 1718, 1648, 1602, 1584, 1495, 1449, 1374, 1270, 1115, 1071, 981, 911 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.97 (br. d, J = 7.2 Hz, 2H), 7.41 (br. t, J = 7.3 Hz, 2H), 7.26 (tt, J = 7.3, 1.4 Hz, 1H), 3.87 (m, 4H), 3.77 (t, J = 7.1 Hz, 1H), 3.12 (m, 4H), 2.57 (t, J = 7.1 Hz, 2H), 1.82 (q, J = 7.1 Hz, 2H), 1.69 (br.s, 1H), 1.49-1.25 (m, 12H), 0.90 (t, J = 7.1 Hz, 3H), 0.87 (t, J = 6,6 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 160.9, 150.6, 132.0, 128.4, 126.9, 126.0, 123.8, 67.0, 57.2, 50.5, 47.4, 34.5, 32.2, 31.6, 29.0, 25.9, 22.5, 20.4, 14.0, 13.9; MS (EI): m/z (M)⁺ 399.



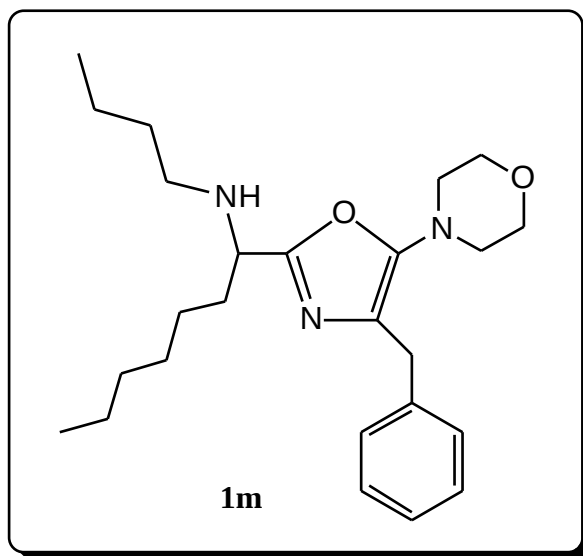
Pale yellow oil, yield : 60% ; IR (CHCl₃) ν 2962, 2931, 2861, 1721, 1640, 1611, 1512, 1495, 1454, 1376, 1303, 1250, 1178, 1114, 1071, 1033, 927 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.35 (d, J = 7.7 Hz, 2H), 7.24 (m, 5H), 6.88 (d, J = 7.7 Hz, 2H), 4.82 (s, 1H), 3.82 (s, 2H), 3.81 (s, 3H), 3.69 (m, 4H), 2.92 (m, 4H), 2.55 (t, J = 7.1 Hz, 2H), 1.84 (br.s, 1H), 1.50 (quintet, J = 7.3 Hz, 2H), 1.33 (quintet, J = 7.0 Hz, 2H), 0.88 (t, 7.3 Hz, 3H) ; ¹³C NMR (75 MHz, CDCl₃) δ 159.2, 152.0, 139.6, 133.4, 131.5, 128.8, 128.5, 128.3, 126.1, 124.7, 113.9, 66.9, 60.8, 55.3, 51.0, 47.4, 31.8, 29.7, 20.4, 13.9; MS (ESI): m/z (M+Na)⁺ 458.



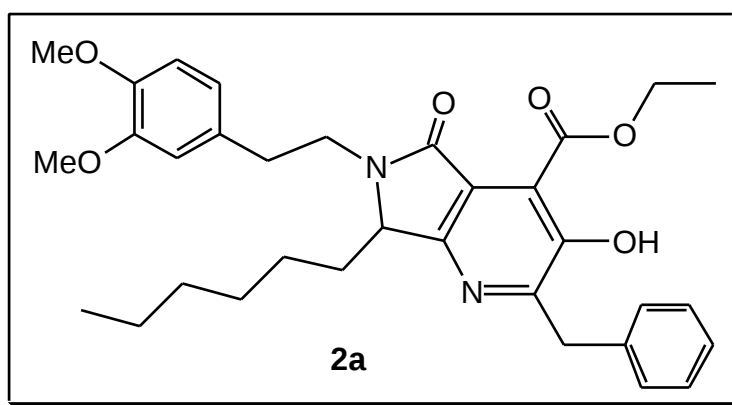
Yellow oil, yield : 67% ; IR (CHCl₃) ν 3413, 2968, 2914, 1861, 1664, 1636, 1596, 1560, 1495, 1454, 1398, 1375, 1314, 1263, 1220, 1114, 1073, 1002, 917 cm⁻¹ ; ¹H NMR (300 MHz, CDCl₃) δ 7.48-7.18 (m, 10H), 7.20 (d, *J* = 8.1 Hz, 2H), 6.50 (d, *J* = 8.1 Hz, 2H), 5.51 (s, 1H), 5.02 (br.s, 1H), 3.81 (s, 2H), 3.69 (m, 4H), 2.91 (m, 4H) ; ¹³C NMR (75 MHz, CDCl₃) δ 157.5, 152.4, 145.3, 139.2, 138.4, 131.8, 128.9, 128.4, 128.2, 127.0, 126.2, 124.7, 115.2, 109.9, 66.7, 56.8, 50.9, 31.7 ; MS (ESI): *m/z* (M+Na)⁺ 542.



Colorless oil, yield : 52% ; IR (CHCl₃) ν 2962, 2931, 2860, 1659, 1602, 1562, 1467, 1454, 1375, 1363, 1262, 1114, 1070, 1039, 917 cm⁻¹ ; ¹H NMR (300 MHz, CDCl₃) δ 3.77 (m, 4H), 3.72 (t, *J* = 7.1 Hz, 1H), 3.25 (br. s, 1H), 2.98 (m, 4H), 2.85 (hept, *J* = 7.1 Hz, 1H), 2.52 (m, 2H), 1.81 (q, *J* = 7.0 Hz, 2H), 1.46 (m, 2H), 1.35-1.17 (m, 16H), 0.85 (t, *J* = 7.1 Hz, 3H), 0.82 (t, *J* = 6.9 Hz, 3H) ; ¹³C NMR (75 MHz, CDCl₃) δ 159.7, 149.8, 132.0, 66.9, 57.1, 51.5, 47.0, 33.8, 31.5, 31.4, 28.8, 25.8, 25.3, 22.4, 21.9, 21.8, 20.2, 13.9, 13.7 ; MS (ESI): *m/z* (M+Na)⁺ 388.

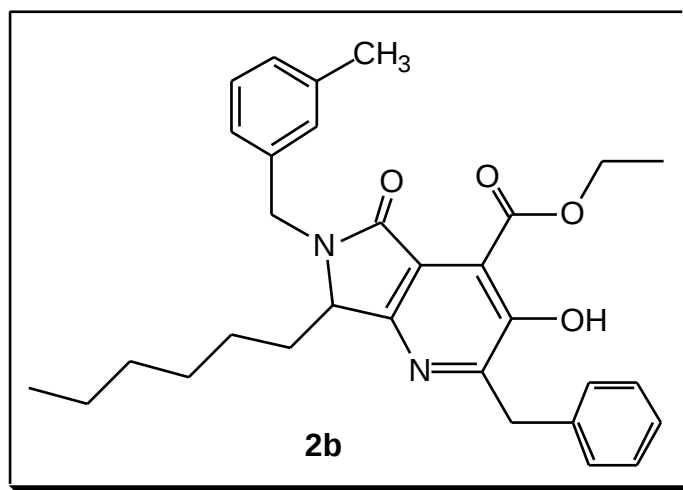


Pale yellow oil, yield : 73% ; IR (CHCl₃) ν 2960, 2930, 2860, 1716, 1682, 1604, 1573, 1573, 1495, 1454, 1376, 1263, 1115, 1071, 918 cm⁻¹ ; ¹H NMR (300 MHz, CDCl₃) δ 7.22 (m, 5H), 3.83 (s, 2H), 3.73 (m, 4H), 3.68 (t, J = 7.2 Hz, 1H), 2.97 (m, 4H), 2.50 (td, J = 7.2, 0.8 Hz, 2H), 1.80 (br.s, 1H), 1.76 (q, J = 7.2 Hz, 2H), 1.42 (sextet, J = 7.3 Hz, 2H), 1.35-1.23 (m, 10H), 0.87 (t, J = 7.2 Hz, 3H), 0.86 (t, 6.4 Hz, 3H) ; ¹³C NMR (75 MHz, CDCl₃) δ 167.1, 152.7, 139.1, 128.3, 128.2, 126.2, 124.3, 66.6, 56.0, 50.7, 46.0, 31.6, 31.5, 31.4, 28.9, 28.5, 25.8, 22.3, 20.0, 13.9, 13.5 ; MS (EI): m/z (M)⁺ 413.

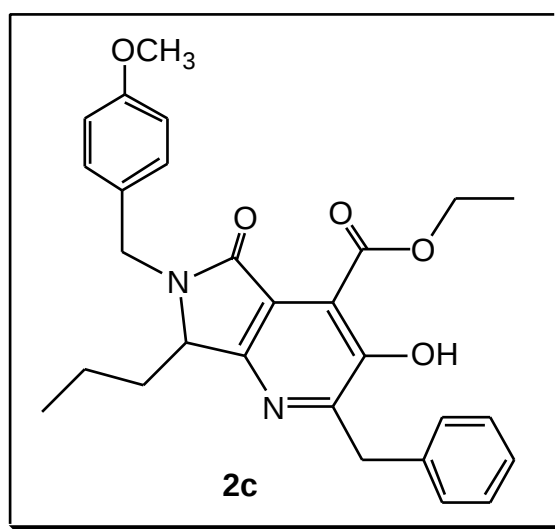


Pale yellow gum, yield: 65%; IR (CHCl₃) ν 3202, 3005, 2934, 1682, 1594, 1516, 1465, 1264, 1195 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 10.57 (s, 1H, OH), 7.15-7.41 (m, 5H), 6.77 (m, 3H), 4.55 (q, J = 7.1 Hz, 2H), 4.33 (d, J = 13.6 Hz, 1H), 4.24 (d, J = 13.6 Hz, 1H), 4.25 (m, 2H), 3.85 (s, 3H), 3.83 (s, 3H), 3.27 (m, 1H), 2.91 (m, 2H), 2.13 (m, 1H), 1.84 (m, 1H), 1.49 (t, J = 7.1 Hz, 3H), 1.15 (m, 6H), 1.06 (m, 1H), 0.83 (t, J = 6.7 Hz, 3H), 0.69 (m, 1H); ¹³C NMR (62.5MHz, CDCl₃): δ 168.8, 164.9, 156.4, 155.7, 153.2, 149.1, 147.8, 138.2, 131.4, 129.2, 128.4, 126.5, 121.2,

120.7, 115.8, 112.0, 111.5, 63.0, 59.4, 55.9, 42.1, 39.4, 34.2, 31.6, 29.5, 29.2, 22.5, 22.4, 14.2, 14.1; MS (EI): m/z 560 (M^+ , 19%).

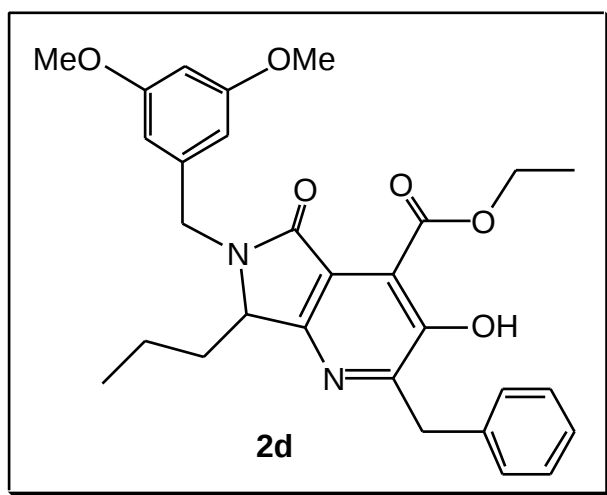


Pale yellow gum, yield: 58%; IR (CHCl_3) ν 3010, 2931, 2860, 1686, 1603, 1466, 1379, 1277, 1199 cm^{-1} ; ^1H NMR (250MHz, CDCl_3) δ 10.61 (s, 1H, OH), 7.00-7.38 (m, 9H), 5.35 (d, J = 15.0 Hz, 1H), 4.56 (q, J = 7.1 Hz, 2H), 4.28 (m, 3H), 4.08 (d, J = 15.0 Hz, 1H), 2.31 (s, 3H), 2.13 (m, 1H), 1.86 (m, 1H), 1.60 (m, 1H), 1.51 (t, J = 7.1 Hz, 3H), 1.15 (m, 5H), 1.05 (m, 1H), 0.84 (t, J = 6.7 Hz, 3H), 0.70 (m, 1H); ^{13}C NMR (62.5MHz, CDCl_3) δ 169.0, 165.1, 156.6, 155.9, 153.3, 138.6, 138.3, 137.0, 129.3, 129.0, 128.7, 128.5, 128.4, 126.5, 125.4, 121.1, 115.9, 63.1, 58.5, 44.1, 39.4, 31.7, 29.2, 22.6, 22.3, 21.5, 14.2, 14.1; MS (ESI): m/z 501 ($M+H^+$, 67%).

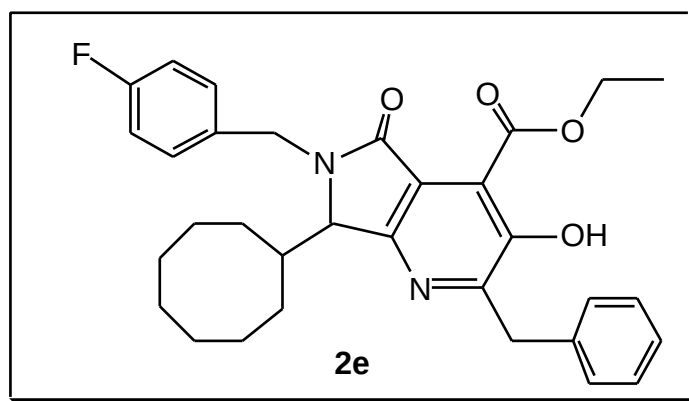


Pale yellow gum, yield: 57%; IR (CHCl_3) ν 3204, 2936, 2839, 1682, 1612, 1513, 1464, 1343, 1175 cm^{-1} ; ^1H NMR (250MHz, CDCl_3) δ 10.55 (br s, 1H, OH), 7.10-7.39 (m, 7H), 6.84 (d, J

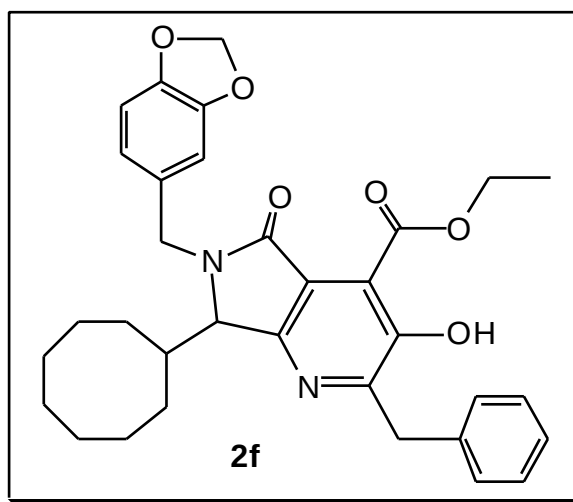
= 8.6 Hz, 2H), 5.34 (d, J = 14.9 Hz, 1H), 4.55 (q, J = 7.1 Hz, 2H), 4.27 (s, 2H), 4.26 (m, 1H), 4.06 (d, J = 14.9 Hz, 1H), 3.78 (s, 3H), 2.12 (m, 1H), 1.85 (m, 1H), 1.50 (t, J = 7.1 Hz, 3H), 1.14 (m, 1H), 0.82 (m, 4H); ^{13}C NMR (62.5MHz, CDCl_3) d 168.9, 165.0, 159.2, 156.7, 155.8, 153.3, 138.3, 129.6, 129.2, 128.4, 126.5, 121.1, 115.9, 114.2, 63.0, 58.3, 55.4, 43.6, 39.4, 31.5, 15.9, 14.2, 14.1; MS (ESI): m/z 475 ($\text{M}+\text{H}^+$, 100%).



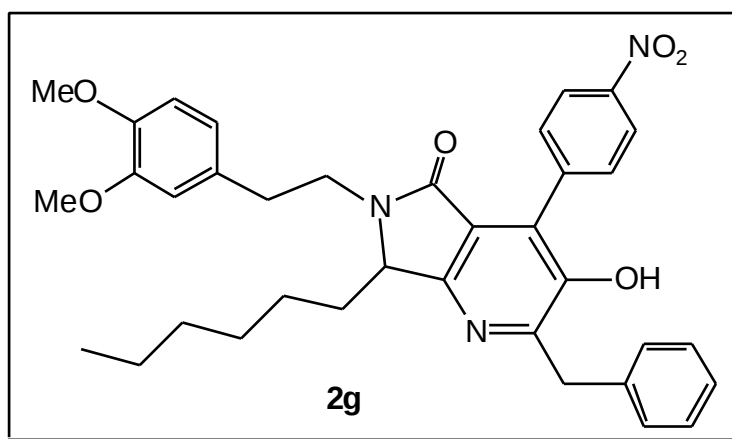
Pale yellow solid, mp 101-103°, yield: 68%; IR (CHCl_3) ν 3198, 2936, 2840, 1682, 1598, 1464, 1343, 1158 cm^{-1} ; ^1H NMR (250MHz, CDCl_3) d 10.57 (s, 1H, OH), 7.15-7.39 (m, 5H), 6.42 (d, J = 2.2 Hz, 2H), 6.36 (t, J = 2.2 Hz, 1H), 5.34 (d, J = 15.4 Hz, 1H), 4.55 (q, J = 7.1 Hz, 2H), 4.33 (dd, J = 3.3, 5.4 Hz, 1H), 4.28 (s, 2H), 4.04 (d, J = 15.4 Hz, 1H), 3.76 (s, 6H), 2.12 (m, 1H), 1.86 (m, 1H), 1.50 (t, J = 7.1 Hz, 3H), 1.15 (m, 1H), 0.82 (m, 4H); ^{13}C NMR (62.5MHz, CDCl_3) d 168.9, 165.0, 161.2, 156.6, 155.9, 153.2, 139.4, 138.2, 129.2, 128.4, 126.5, 120.9, 115.9, 106.3, 99.4, 63.0, 58.5, 55.4, 44.2, 39.4, 31.5, 15.9, 14.1, 14.1; MS (ESI): m/z 505 ($\text{M}+\text{H}^+$, 66%)



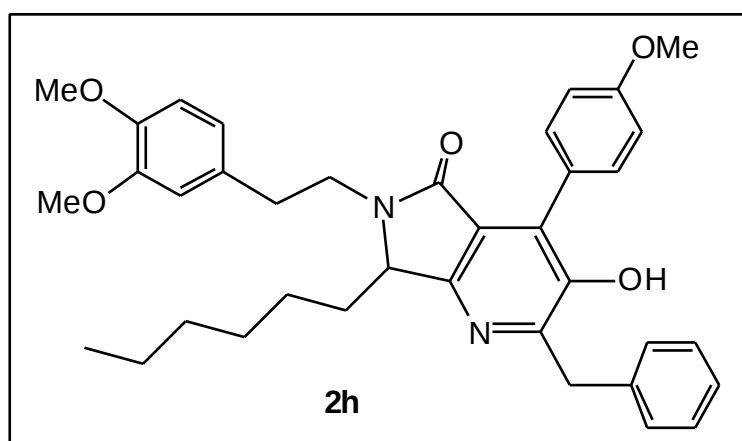
Pale yellow gum, yield: 66%; IR (CHCl₃) ν 3020, 2926, 1682, 1602, 1510, 1463, 1378, 1193 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 10.55 (s, 1H, OH), 7.12-7.40 (m, 7H), 6.98 (t, J = 8.6 Hz, 2H), 4.55 (q, J = 7.2 Hz, 2H), 4.38 (d, J = 13.4 Hz, 1H), 4.22 (m, 1H), 4.17 (d, J = 13.4 Hz, 1H), 4.15 (d, J = 3.1 Hz, 1H), 3.32 (m, 1H), 2.92 (m, 2H), 2.25 (m, 1H), 1.96 (m, 1H), 1.49 (t, J = 7.2 Hz, 3H), 1.07-1.80 (m, 12H), 0.84 (m, 1H); ¹³C NMR (62.5MHz, CDCl₃) δ 168.9, 165.3, 161.8 (d, J = 242.8 Hz), 156.0, 155.4, 153.1, 138.3, 134.6, 130.2 (d, J = 7.5 Hz), 129.4, 128.4, 126.5, 121.4, 115.7, 115.5 (d, J = 20.8 Hz), 65.5, 63.0, 42.4, 39.3, 38.9, 33.9, 30.1, 27.3, 26.65, 26.56, 26.50, 26.3, 26.1, 14.2; MS (ESI): m/z 545 (M+H⁺, 53%).



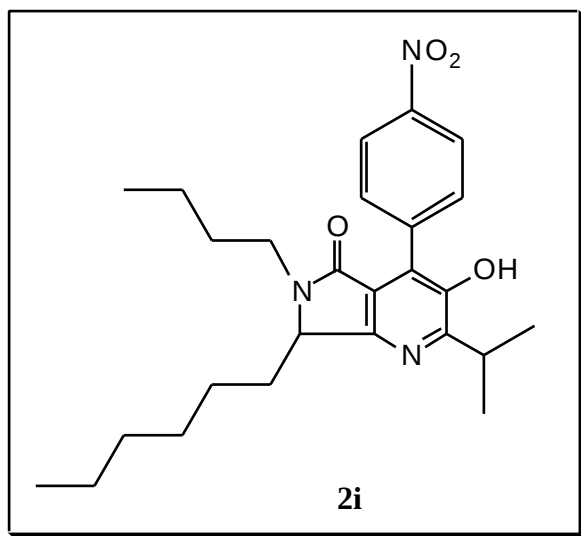
Pale yellow solid, mp 109-111°, yield: 58%; IR (CHCl₃) ν 3009, 2925, 1690, 1601, 1490, 1377, 1246, 1197 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 10.57 (s, 1H, OH), 7.12-7.40 (m, 5H), 6.75 (m, 3H), 5.92 (d, J = 1.8 Hz, 2H), 5.28 (d, J = 15.0 Hz, 1H), 4.55 (q, J = 7.1 Hz, 2H), 4.34 (d, J = 14.1 Hz, 1H), 4.17 (d, J = 14.1 Hz, 1H), 4.14 (d, J = 2.8 Hz, 1H), 4.08 (d, J = 15.0 Hz, 1H), 2.28 (m, 1H), 1.97 (m, 1H), 1.50 (t, J = 7.1 Hz, 3H), 1.05-1.80 (m, 12H), 0.83 (m, 1H); ¹³C NMR (62.5MHz, CDCl₃) δ 169.0, 165.5, 156.3, 155.5, 153.1, 148.2, 147.1, 138.3, 131.1, 129.4, 128.4, 126.5, 121.5, 121.2, 115.7, 108.7, 108.4, 101.2, 64.5, 63.0, 44.2, 39.3, 38.4, 30.3, 27.0, 26.6, 26.4, 26.3, 26.1, 14.2; MS (ESI): m/z 557 (M+H⁺, 100%).



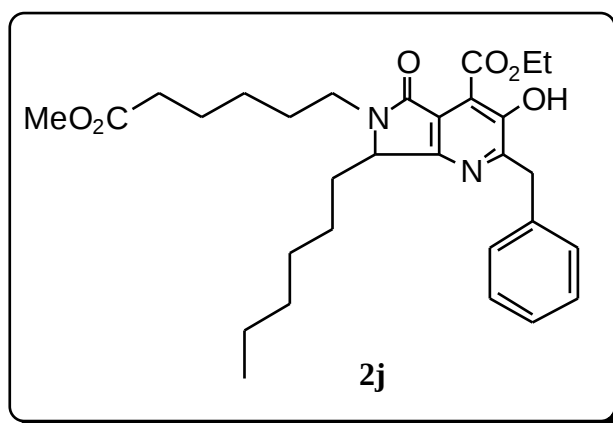
Yellow solid, mp 161-163°, yield: 63%; IR (CHCl₃) ν 3553, 3022, 2935, 2860, 1678, 1604, 1518, 1465, 1350, 1263, 1157 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 8.33 (d, *J* = 8.8 Hz, 2H), 7.62 (d, *J* = 8.8 Hz, 2H), 7.18-7.39 (m, 5H), 6.75 (m, 3H), 5.19 (s, 1H, OH), 4.42 (dd, *J* = 3.3, 5.3 Hz, 1H), 4.36 (d, *J* = 14.2 Hz, 1H), 4.29 (d, *J* = 14.2 Hz, 1H), 4.10 (m, 1H), 3.85 (s, 3H), 3.80 (s, 3H), 3.28 (m, 1H), 2.86 (m, 2H), 2.20 (m, 1H), 1.90 (m, 1H), 1.22 (m, 7H), 0.90 (m, 1H), 0.86 (t, *J* = 6.6 Hz, 3H); ¹³C NMR (62.5MHz, CDCl₃) δ 166.1, 157.6, 153.2, 149.1, 148.2, 147.8, 146.7, 138.0, 136.8, 131.4, 131.1, 130.3, 129.0, 128.7, 126.9, 123.6, 121.3, 120.7, 112.0, 111.5, 59.9, 56.0, 41.9, 40.1, 34.3, 31.7, 29.6, 29.2, 22.64, 22.6, 14.1; MS (ESI): *m/z* 610 (M+H⁺, 100%).



Pale yellow gum, yield: 55%; IR (CHCl₃) ν 3529, 3022, 2935, 2859, 1678, 1610, 1516, 1464, 1258, 1150 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.16-7.40 (m, 7H), 7.04 (d, *J* = 8.8 Hz, 2H), 6.75 (m, 3H), 5.50 (s, 1H, OH), 4.38 (dd, *J* = 3.3, 5.1 Hz, 1H), 4.33 (d, *J* = 13.7 Hz, 1H), 4.25 (d, *J* = 13.7 Hz, 1H), 4.11 (m, 1H), 3.85 (s, 6H), 3.80 (s, 3H), 3.26 (m, 1H), 2.86 (m, 2H), 2.19 (m, 1H), 1.88 (m, 1H), 1.19 (m, 7H), 0.85 (m + t, *J* = 6.6 Hz, 4H); ¹³C NMR (62.5MHz, CDCl₃): δ 166.7, 160.6, 157.3, 152.0, 149.1, 147.8, 147.1, 138.9, 131.9, 131.4, 131.2, 129.1, 128.4, 126.4, 121.5, 121.1, 120.7, 114.7, 112.0, 111.5, 59.8, 56.0, 55.4, 42.0, 39.9, 34.4, 31.7, 29.7, 29.3, 22.6, 14.1; MS (ESI): *m/z* 695 (M+H⁺, 100%).

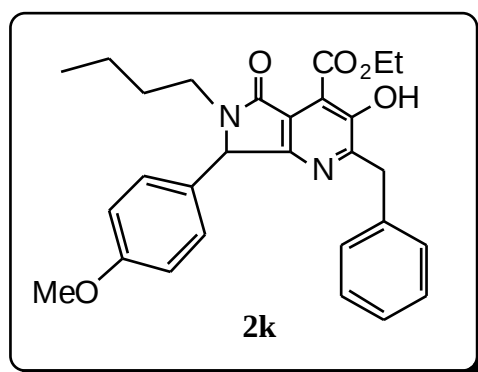


Pale yellow gum, yield : 59% ; IR (CHCl₃) ν 2964, 2932, 2873, 2400, 1675, 1603, 1525, 1456, 1414, 1156, 1109, 928 cm⁻¹ ; ¹H NMR (300 MHz, CDCl₃) δ 8.33 (d, *J* = 8.8 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 2H), 4.52 (dd, *J* = 5.8, 3.0 Hz, 1H), 3.87 (ddd, *J* = 14.0, 8.6, 7.6 Hz, 1H), 3.52 (hept, *J* = 7.0 Hz, 1H), 3.05 (ddd, *J* = 14.0, 8.6, 5.2 Hz, 1H), 2.22 (m, 1H), 1.85 (m, 1H), 1.57 (m, 2H), 1.35 (d, *J* = 6.9 Hz, 3H), 1.33 (d, *J* = 6.9 Hz, 3H), 1.35-1.24 (m, 10H), 0.91 (t, *J* = 7.1 Hz, 3H), 0.86 (t, *J* = 6.3 Hz, 3H) ; ¹³C NMR (75 MHz, CDCl₃) δ 166.2, 159.0, 157.4, 148.1, 145.3, 137.2, 131.3, 129.1, 123.7, 109.7, 59.3, 39.6, 31.5, 30.4, 30.1, 29.5, 29.0, 22.5, 22.4, 21.2, 21.0, 20.2, 14.0, 13.7 ; MS (CI): *m/z* (M+H)⁺ 454.

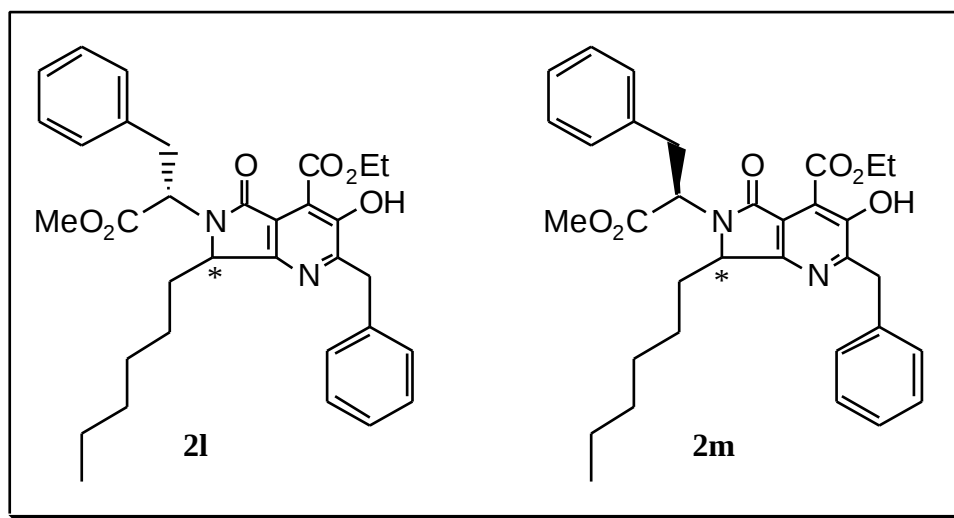


Yellow gum, yield : 50% ; IR (CHCl₃) ν 3697, 3030, 3012, 2933, 2861, 2400, 1724, 1700, 1683, 1602, 1465, 1438, 1376, 1337, 1233, 1219, 1199, 1018, 928 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 10.59 (broad s, 1H), 7.37-7.20 (m, 5H), 4.53 (q, *J* = 7.4 Hz, 2H), 4.47 (dd, *J* = 3.2, 5.1 Hz, 1H), 4.36-4.25 (AB, *J* = 13.4 Hz, 2H), 3.97 (ddd, *J* = 6.9, 8.2, 13.7 Hz, 1H), 3.67 (s, 3H), 3.09

(ddd, $J = 5.5, 8.8, 13.7$ Hz, 1H), 2.32 (t, $J = 7.4$ Hz, 2H), 2.22 (m, 1H), 1.88 (m, 1H), 1.80-1.00 (m, 13H), 1.49 (t, $J = 7.2$ Hz, 3H), 0.85 (t, $J = 7.4$ Hz, 3H), 0.71 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) d 174.0, 168.9, 164.9, 156.4, 155.6, 153.3, 138.3, 129.2, 128.4, 126.5, 121.3, 115.8, 63.0, 58.9, 51.6, 40.1, 39.4, 34.0, 31.6, 29.4, 29.2, 28.1, 26.6, 24.7, 22.6, 22.3, 14.1 ; MS (ESI): m/z ($\text{M}+\text{H}$) $^+$ 525, ($\text{M}+\text{Na}$) $^+$ 547



Yellow gum, yield : 55% ; IR (CHCl_3) n 2961, 2934, 2874, 1683, 1612, 1513, 1495, 1463, 1400, 1378, 1336, 1305, 1269, 1175, 1111, 1020, 959 cm^{-1} ; ^1H NMR (250 MHz, CDCl_3) d 10.60 (s, 1H), 7.27-7.26 (m, 5H), 7.09 (d, $J = 8.7$ Hz, 2H), 6.90 (d, $J = 8.5$ Hz, 2H), 5.34 (s, 1H), 4.56 (q, $J = 7.2$ Hz, 2H), 4.27 (d, $J = 13.8$ Hz, 1H), 4.14 Hz (d, $J = 13.8$ Hz, 1H), 3.94 (ddd, $J = 7.7, 7.7, 13.8$ Hz, 1H), 3.83 (s, 3H), 2.85 (ddd, $J = 6.2, 7.7, 13.8$ Hz, 1H), 1.59 (m, 2 H), 1.51 (t, $J = 7.2$ Hz, 3H), 1.32 (sext, $J = 7.2$ Hz, 1H), 0.89 (t, $J = 7.2$ Hz, 3H) ; ^{13}C NMR (75 MHz, CDCl_3) d 168.8, 164.4, 159.7, 156.8, 156.1, 153.1, 138.0, 129.0, 128.2, 127.6, 126.3, 120.5, 115.6, 114.3, 63.6, 62.9, 55.3, 40.2, 39.3, 30.2, 20.1, 14.0, 13.7 ; MS (CI): m/z ($\text{M}+\text{H}$) $^+$ 475



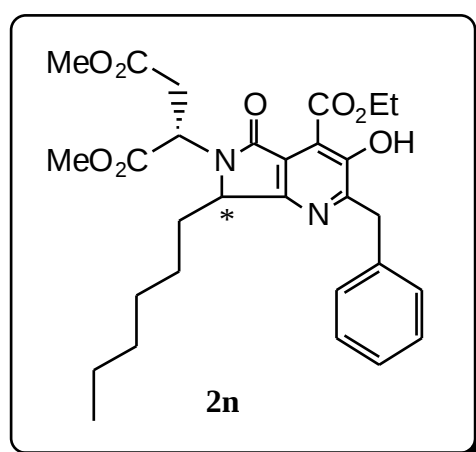
Yellow gum, yield : 54%

2l (diastereomer A)

IR (CHCl₃) ν 2956, 2930, 2858, 1741, 1695, 1603, 1495, 1455, 1402, 1378, 1341, 1302, 1276, 1195, 1112, 1012, 975, 863 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 10.50 (br. s, 1H), 7.32-7.15 (m, 10H), 4.53 (q, J = 7.4 Hz, 2H), 4.43 (t, J = 8.6 Hz, 1H), 4.33 (d, J = 14.4 Hz, 1H), 4.18 (d, J = 14.4 Hz, 1H), 3.75 (s, 3H), 3.66 (dd, J = 3.9, 5.5 Hz, 1H), 3.48 (d, J = 8.1 Hz, 2H), 2.02 (m, 1H), 1.70 (m, 1H), 1.48 (t, J = 7.2 Hz, 3H), 1.44-1.05 (m, 7H), 0.84 (t, J = 6.9 Hz, 3H), 0.70 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ 170.3, 168.6, 164.6, 156.6, 155.9, 153.0, 138.0, 137.8, 129.0, 128.9, 128.6, 128.2, 126.8, 126.3, 120.5, 115.6, 62.8, 61.0, 57.6, 52.4, 39.3, 35.1, 31.5, 30.2, 29.1, 22.5, 22.4, 14.0, 13.9 ; MS (CI): m/z (M+H)⁺ 559; [α]_D = -102.6 (c =0.3, CHCl₃) {enantiomer **2m** [α]_D = +101.8 (c =0.3, CHCl₃)}

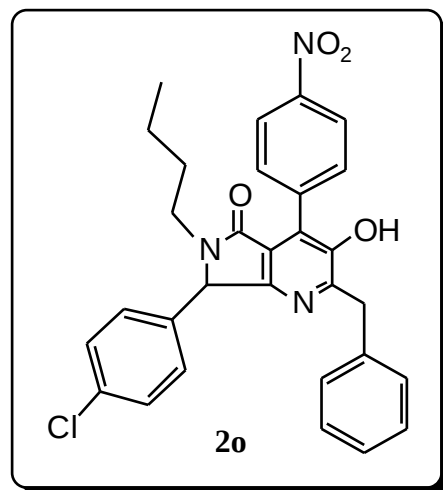
2l (diastereomer B)

IR (CHCl₃) ν 2956, 2930, 2858, 1741, 1698, 1602, 1495, 1456, 1400, 1378, 1340, 1271, 1195, 1018, 862 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 10.50 (br. s, 1H), 7.35-7.19 (m, 10H), 4.59 (dd, J = 9.8; 6.0 Hz, 1H), 4.53 (m, 2H), 4.38 (dd, J = 3.4; 5.1 Hz, 1H), 4.33 (d, J = 13.7 Hz, 1H), 4.18 (d, J = 13.7 Hz, 1H), 3.73 (s, 3H), 3.68 (dd, J = 9.8, 14.6 Hz, 1H), 3.54 (dd, J = 6.8, 14.6 Hz, 1H), 2.00-0.80 (m, 9H), 1.50 (t, J = 7.2 Hz, 3H), 0.85 (t, J = 6.9 Hz, 3H), 0.63 (m, 1H) ; ¹³C NMR (75 MHz, CDCl₃) δ 171.0, 168.5, 166.1, 156.6, 156.1, 153.1, 138.0, 137.9, 129.2, 129.1, 128.5, 128.3, 126.7, 126.4, 120.7, 115.5, 62.8, 60.9, 57.6, 52.5, 39.3, 35.1, 31.5, 30.3, 29.3, 22.8, 22.5, 14.0, 13.9 ; MS (CI): m/z (M+H)⁺ 559; [α]_D = -58.0 (c =0.2, CHCl₃) {enantiomer **2m** [α]_D = +57.9 (c =0.2, CHCl₃)}

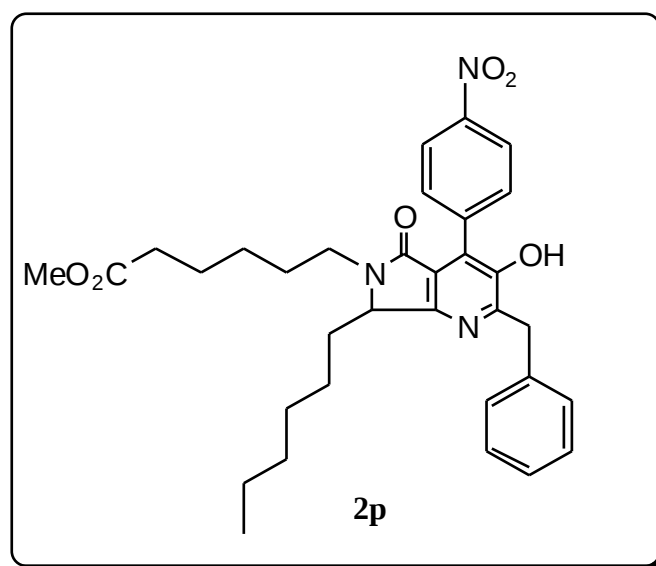


Two diastereomers were produced. They are not isolated. NMR signals corresponding to the same moiety for a different diastereomer are indicated in brackets. Pale yellow gum, yield : 40% ; IR (CHCl₃) ν 3008, 2955, 2930, 2858, 1738, 1698, 1601, 1568, 1494, 1464, 1438, 1402, 1378, 1277, 1199, 1113, 1017, 860 cm⁻¹; ¹H NMR (250 MHz, CDCl₃) δ 10.66 (s, 1H) [10.61], 7.38-7.20 (m, 5H), 4.83 (t, J = 6.8 Hz, 1H) [4.81], 4.57 (dd, J = 3.9, 5.8 Hz, 1H) [4.55], 4.51 (q, J = 7.1 Hz,

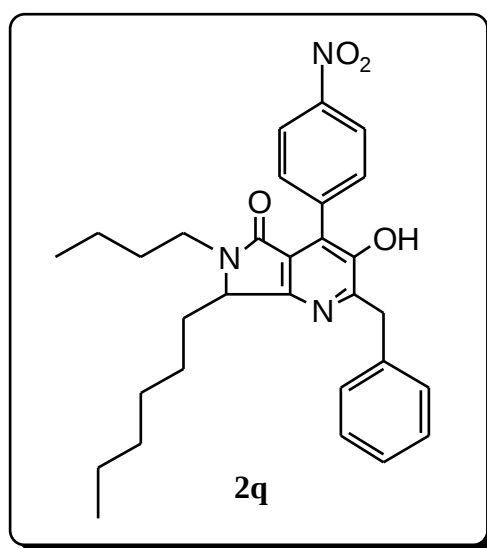
2H) [4.50], 4.38-4.23 (AB, $J = 13.7$ Hz, 2H), 3.75 (s, 3H) [3.73], 3.69 (s, 3H), 3.52 (dd, $J = 7.7$; 17.1 Hz, 1H) [3.33], 3.00 (dd, $J = 6.0$; 17.1 Hz, 1H) [2.95], 1.45 (t, $J = 7.3$ Hz, 3H) [1.44], 1.37-1.23 (m, 10H), 0.87 (t, $J = 6.8$ Hz, 3H) [0.86] ; MS (CI): m/z (M+H)⁺ 541



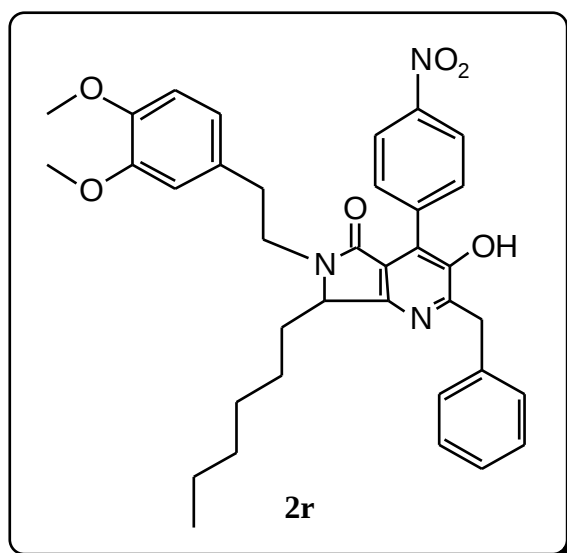
Yellow gum, yield : 32% ; IR (CHCl₃) ν 3017, 2962, 2931, 2860, 1682, 1651, 1602, 1524, 1414, 1355, 1348, 1114, 1092, 1015, 855 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.37 (d, $J = 8.6$ Hz, 2H), 7.68 (d, $J = 8.6$ Hz, 2H), 7.40 (d, $J = 8.5$ Hz, 2H), 7.45-7.26 (m, 5H), 7.18 (d, $J = 8.5$ Hz, 2H), 5.44 (s, 1H), 4.27 (d, $J = 14.5$ Hz, 1H), 4.14 (d, $J = 14.5$ Hz, 1H), 3.84 (ddd, $J = 7.1, 8.4, 14.2$ Hz, 1H), 2.84 (ddd, $J = 5.7, 7.6, 13.8$ Hz, 1H), 1.72-1.23 (m, 4H), 0.87 (t, $J = 7.2$ Hz, 3H), ; ¹³C NMR (75 MHz, CDCl₃) δ 173.9, 165.9, 157.3, 153.6, 148.2, 147.0, 137.5, 136.4, 134.6, 134.2, 130.4, 129.1, 129.0, 128.9, 128.7, 126.8, 123.6, 120.5, 63.7, 40.1, 30.2, 29.6, 20.1, 13.6 ; MS (CI): m/z (M+H)⁺ 528



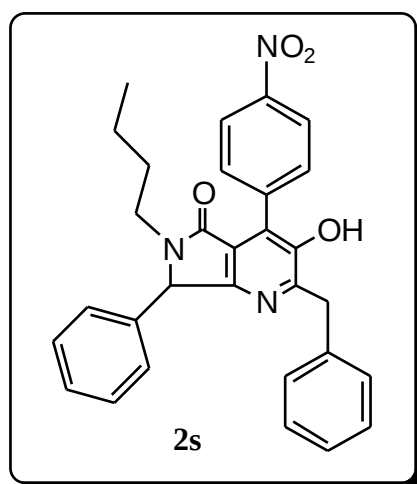
Yellow solid, yield : 75% ; mp 95°C ; IR (CHCl₃) ν 3551, 3009, 2931, 2859, 1732, 1677, 1603, 1571, 1524, 1494, 1458, 1411, 1349, 1265, 1237, 1175, 109, 1016, 843 cm⁻¹; ¹H NMR (250 MHz, CDCl₃) δ 8.32 (d, J = 8.3 Hz, 2H), 7.62 (d, J = 8.3 Hz, 2H), 7.33-7.20 (m, 5H), 5.11 (broad s, 1H), 4.53 (dd, J = 3.0, 5.5 Hz, 1H), 4.33 (s, 2H), 3.84 (ddd, J = 8.1, 8.1, 13.8 Hz, 1H), 3.63 (s, 3H), 3.06 (ddd, J = 5.7, 8.1, 13.8 Hz, 1H), 2.28 (t, J = 7.3 Hz, 2H), 2.00-1.11 (m, 16H), 0.86 (t, J = 6.8 Hz, 3H) ; ¹³C NMR (75 MHz, CDCl₃) δ 173.9, 165.9, 157.4, 153.0, 147.9, 146.6, 137.9, 136.8, 131.3, 130.3, 128.9, 128.6, 126.7, 123.4, 121.2, 59.3, 51.5, 39.9, 39.7, 33.8, 31.5, 29.4, 29.1, 27.9, 26.4, 24.5, 22.5, 22.4, 14.0; MS (CI): m/z (M+H)⁺ 574



Yellow gum, yield : 71% ; IR (CHCl₃) ν 3553, 3008, 2960, 2932, 2860, 1676, 1603, 1570, 1523, 1494, 1458, 1411, 1349, 1237, 1109, 1016, 843 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.30 (d, J = 8.5 Hz, 2H), 7.60 (d, J = 8.5 Hz, 2H), 7.32-7.22 (m, 5H), 4.50 (dd, J = 3.0, 5.1 Hz, 1H), 4.38-4.26 (AB, J = 14.1 Hz, 2H), 3.85 (ddd, J = 7.0, 8.6, 14.1 Hz, 1H), 3.05 (ddd, J = 5.5, 8.6, 14.1 Hz, 1H), 2.26 (m, 1H), 1.91 (m, 1H), 1.62-1.09 (m, 12H) 0.91 (t, J = 7.3 Hz, 3H), 0.86 (t, J = 6.8 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃) δ 165.9, 157.3, 153.0, 147.8, 146.7, 138.0, 136.9, 131.3, 130.3, 128.9, 128.5, 126.6, 123.3, 121.2, 59.3, 39.9, 38.8, 31.5, 30.3, 29.3, 29.1, 22.4, 22.3, 20.1, 14.0, 13.6 ; MS (CI): m/z (M+H)⁺ 502

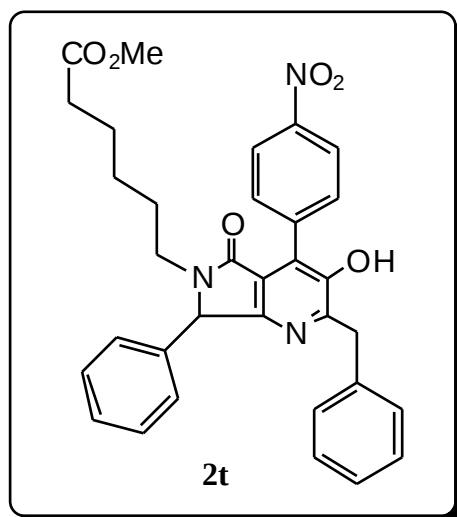


Pale yellow solid, yield : 70% ; mp 132°C ; IR (CHCl₃) ν 3550, 3009, 2933, 2859, 1678, 1603, 1574, 1516, 1494, 1464, 1410, 1349, 1264, 1239, 1221, 1208, 1148, 1028, 843 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 8.33 (d, J = 8.7 Hz, 2H), 7.62 (d, J = 8.7 Hz, 2H), 7.23-7.36 (m, 5H), 6.80-6.73 (m, 3H), 5.22 (br. s, 1H), 4.44 (dd, J = 3.7, 5.2 Hz, 1H), 4.38-4.27 (AB, J = 14.4 Hz, 2H), 4.11 (ddd, J = 6.7, 8.7, 13.9 Hz, 1H), 3.85 (s, 3H), 3.83 (m, 2H), 3.80 (s, 3H), 3.29 (ddd, J = 6.7, 8.4, 13.9 Hz, 1H), 2.24 (m, 1H), 1.95 (m, 1H), 1.74-0.77 (m, 11H) ; ¹³C NMR (75 MHz, CDCl₃) δ 166.0, 157.5, 152.9, 148.9, 148.1, 147.7, 146.5, 137.8, 136.5, 131.2, 130.9, 130.1, 128.9, 128.6, 126.8, 123.6, 121.2, 120.6, 111.8, 111.3, 59.8, 56.9, 55.8, 41.8, 40.1, 34.2, 31.5, 29.4, 29.1, 22.5, 22.4, 14.0 ; MS (CI): m/z (M+H)⁺ 610

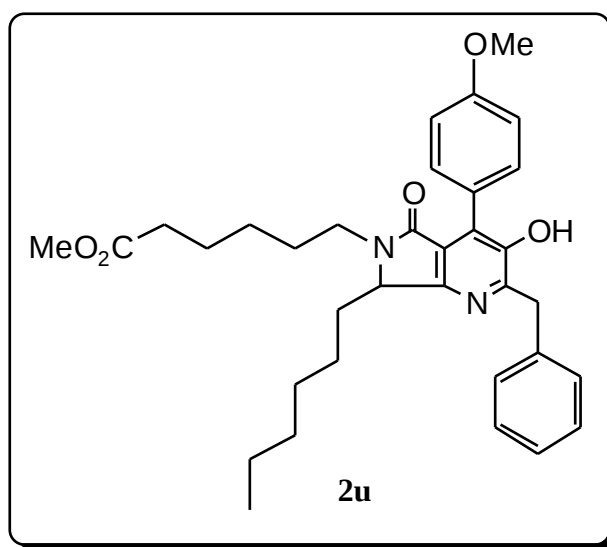


Yellow oil, yield : 70% ; IR (CHCl₃) ν 3550, 3009, 2962, 2932, 2874, 1682, 1603, 1523, 1494, 1455, 1381, 1349, 1314, 1237, 1120, 1064, 854 cm⁻¹; ¹H NMR (250 MHz, CDCl₃) δ 8.34 (d, J = 8.3 Hz, 2H), 7.65 (d, J = 8.3 Hz, 2H), 7.43-7.21 (m, 10H), 5.46 (s, 1H), 5.18 (br. s, 1H), 4.29-4.16 (AB, J = 14.1 Hz, 2H), 3.84 (ddd, J = 7.7, 7.7, 14.0 Hz, 1H), 3.29 (ddd, J = 6.0, 8.1, 14.0 Hz,

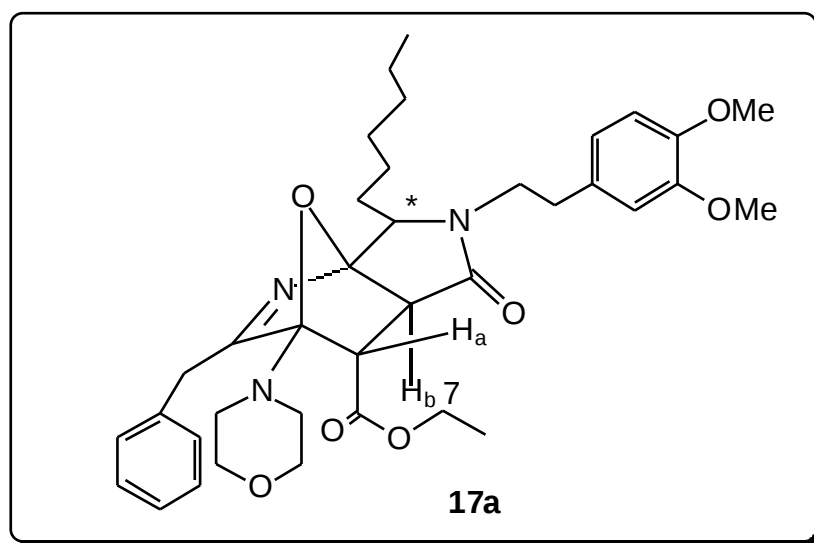
1H), 1.48 (m, 2H), 1.26 (sextet, $J = 7.3$ Hz, 2H), 0.85 (t, $J = 7.3$ Hz, 3H) ; ^{13}C NMR (75 MHz, CDCl_3) d 165.9, 157.8, 153.3, 148.1, 146.9, 137.5, 136.5, 135.6, 131.3, 130.3, 129.1, 129.0, 128.9, 128.7, 127.8, 126.8, 123.6, 120.6, 64.5, 40.1, 30.2, 20.1, 13.6 ; MS (CI): m/z ($\text{M}+\text{H}$) $^+$ 494



Yellow oil, yield : 35% ; IR (CHCl_3) ν 3008, 2952, 1730, 1684, 1603, 1523, 1494, 1456, 1349, 1237, 1121, 854 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) d 8.37 (d, $J = 9.0$ Hz, 2H), 7.69 (d, $J = 9.0$ Hz, 2H), 7.46-7.23 (m, 10H), 5.48 (s, 1H), 5.34 (br. s, 1H), 4.33-4.22 (AB, $J = 14.1$ Hz, 2H), 3.83 (ddd, $J = 7.5, 7.5, 13.9$ Hz, 1H), 3.66 (s, 3H), 2.90 (ddd, $J = 5.6, 7.8, 13.7$ Hz, 1H), 2.27 (t, $J = 7.7$ Hz, 2H), 1.69-1.26 (m, 6H) ; ^{13}C NMR (75 MHz, CDCl_3) d 173.9, 166.0, 157.8, 153.5, 148.2, 146.9, 137.5, 136.5, 135.6, 131.4, 130.3, 129.1, 128.9, 128.8, 128.7, 127.9, 126.8, 123.6, 120.6, 64.6, 51.5, 40.2, 33.8, 29.7, 27.8, 26.4, 24.5 ; MS (CI): m/z ($\text{M}+\text{H}$) $^+$ 566



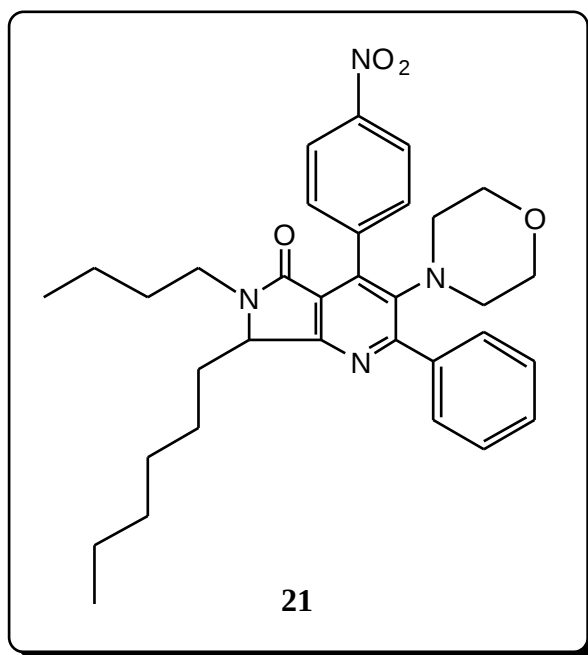
Deep yellow oil, yield : 65% ; IR (CHCl₃) ν 3528, 3009, 2934, 2860, 1731, 1677, 1605, 1579, 1514, 1494, 1458, 1440, 1402, 1377, 1289, 1251, 1176, 1134, 1112, 1031, 864, 830 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.37 (d, J = 8.3 Hz, 2H), 7.39-7.19 (m, 5H), 7.03 (d, J = 8.3 Hz, 2H), 5.53 (br. s, 1H), 4.50 (dd, J = 3.2, 4.9 Hz, 1H), 4.33-4.26 (AB, J = 13.7 Hz, 2H), 3.87 (m, 1H), 3.85 (s, 3H), 3.64 (s, 3H), 3.05 (ddd, J = 5.1, 8.5, 14.7 Hz, 1H), 2.29 (t, J = 7.2 Hz, 2H), 2.00-1.00 (m, 16H), 0.86 (t, J = 6.8 Hz, 3H) ; ¹³C NMR (75 MHz, CDCl₃) δ 173.9, 166.4, 160.3, 157.0, 151.8, 146.9, 138.8, 131.9, 131.1, 129.0, 128.3, 126.2, 121.4, 121.0, 114.4, 59.0, 55.2, 51.4, 39.8, 39.7, 33.8, 31.5, 29.4, 29.1, 28.0, 26.5, 24.6, 22.5, 22.3, 14.0 ; MS (CI): m/z (M+H)⁺ 559



Two diastereomers were produced and one of them was isolated in pure form (relative stereochemistry unknown): pale yellow gum, yield: 45%; IR (CHCl₃) ν 3011, 2934, 2859, 1732, 1681, 1594, 1516, 1455, 1263, 1119 cm⁻¹; ¹H NMR (250MHz, CDCl₃) δ 7.10-7.35 (m, 5H), 6.75 (m, 3H), 4.27 (dq, J = 7.1, 10.9 Hz, 1H), 4.11 (dq, J = 7.1, 10.9 Hz, 1H), 3.88 (s, 3H), 3.85 (s, 3H), 3.83 (m, 1H), 3.60-3.78 (m, 5H), 3.68 (d, J = 16.4 Hz, 1H), 3.54 (d, J = 16.4 Hz, 1H), 3.37 (d, J = 4.1 Hz, 1H, H_a), 3.21 (m, 1H), 3.01 (m, 2H), 2.98 (d, J = 4.1 Hz, 1H, H_b), 2.82 (m, 2H), 2.57 (m, 2H), 1.82 (m, 2H), 1.57 (m, 1H), 1.20-1.48 (m, 10H), 0.89 (t, J = 6.6 Hz, 3H); ¹³C NMR (62.5MHz, CDCl₃) δ 178.3, 171.4, 169.3, 149.0, 147.8, 135.5, 131.3, 129.4, 128.4, 126.9, 121.0, 112.1, 111.3, 108.1, 101.3, 67.0, 61.7, 60.8, 56.03, 55.98, 53.0, 48.8, 43.3, 35.5, 33.5, 31.7, 29.6, 29.4, 25.3, 22.6, 14.3, 14.2; MS (CI): m/z 648 (M+H⁺, 100%).



1216, 1210, 1118, 904 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.91 (d, J = 8.6 Hz, 2H), 7.55 (d, J = 8.6 Hz, 2H), 7.33-7.10 (m, 5H), 4.48 (dd, J = 11.0, 4.5 Hz, 1H)) [3.89 (dd, J = 8.2, 3.8 Hz, 1H)], 4.07 (d, J = 3.8 Hz, 1H), 3.77 (m, 1H), 3.68 (m, 4H), 3.15 (m, 2H), 3.06 (d, J = 3.8 Hz, 1H), 2.92 (m, 1H), 2.77 (m, 2H), 2.01 (m, 1H), 1.82 (m, 1H), 1.80-1.31 (m, 10H), 0.97 (t, J = 6.8 Hz, 3H), 0.96 (t, J = 6.8 Hz, 6H); MS (CI): m/z ($\text{M}+\text{H}$) $^+$ 575



Yellow gum, yield: 65%; IR (CHCl_3) ν 2930, 2860, 1678, 1602, 1560, 1521, 1456, 1387, 1348, 1110, 908 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 8.36 (d, J = 8.8 Hz, 2H), 7.60-7.46 (m, 7H), 4.59 (dd, J = 5.1, 3.6 Hz, 1H), 3.87 (ddd, J = 13.8, 9.2, 6.7 Hz, 1H), 3.21 (m, 4H), 3.05 (ddd, J = 13.8, 8.2, 5.1 Hz, 1H), 2.55 (m, 4H), 2.27 (m, 1H), 1.95 (m, 1H), 1.65-1.15 (m, 12H), 0.93 (t, J = 7.2 Hz, 3H), 0.86 (t, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 165.7, 160.7, 147.7, 143.1, 143.0, 141.0, 140.2, 130.9, 130.7, 129.0, 128.7, 128.4, 123.0, 122.4, 66.2, 59.5, 51.8, 39.8, 31.5, 30.3, 29.3, 29.2, 29.1, 22.5, 20.3, 14.0, 13.7; MS (CI): m/z ($\text{M}+\text{H}$) $^+$ 556