Supporting Information

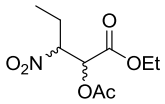
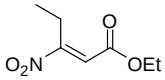
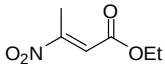
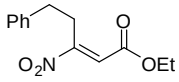
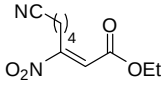
General. ^{1}H NMR were recorded at 400 MHz on a Varian Mercury Plus 400. ^{13}C NMR were recorded at 100 MHz. Microanalyses were performed with a CHNS-O analyser Model EA 1108 from Fisons Instruments. Mass spectra were performed with a GC/MS system Agilent Technologies 6850 II/5973 Inert by means of the EI technique (70 eV). IR spectra were recorded with a Perkin-Elmer Paragon 500 FT-IR.

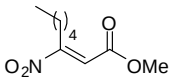
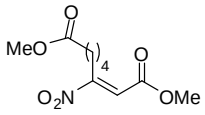
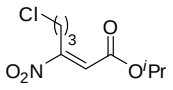
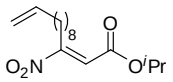
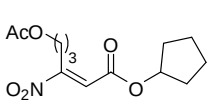
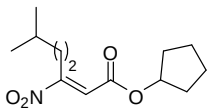
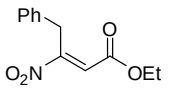
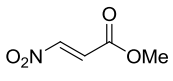
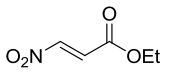
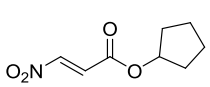
General procedure for the synthesis of compounds 4a-k.

Amberlyst 15 (0.125 g) was added to a stirred solution of acetic anhydride (6.25 mmol, 0.638 g) and nitroalcohols **2a-k** (5 mmol). The resulting mixture was stirred for 2 hours, then, the excess of acetic anhydride and the acetic acid (generated) were evaporated under vacuum followed by filtration of the Amberlyst 15 and washing the resin with EtOAc (3 mL). The resulting solution was treated with KF/Al_2O_3 (5 mmol, 0.910 g) and stirred for further 24 hours. At the end, the heterogeneous mixture was filtered through a pad of SiO_2/EtOAc (1 g of system), washing with fresh EtOAc (8 mL). Finally, the evaporation of the solvent under vacuum leads to isolate the products **4a-k**.

General flow procedure for the synthesis of compounds 4l-n.

Amberlyst 15 (0.050 g) was added to a stirred solution of acetic anhydride (2.5 mmol, 0.255 g) and nitroalcohol **2l-n** (2 mmol). The resulting mixture was stirred for 2 hours then, the excess of acetic anhydride and the acetic acid (generated) were evaporated under vacuum and, successively, the Amberlyst 15 was filtered off washing the resin with EtOAc (10 mL). The resulting solution was diluted with EtOAc in order to obtain a 0.07M solution that was fluxed with a flow rate of 0.3 mL/min, through two columns packed with KF/Al_2O_3 (12 mmol, 2.181 g), and Florisil® (6 g), respectively. Finally, the evaporation of the solvent under vacuum leads to isolate the pure products **4l-n**.

3a		Colourless oil. IR (neat) ν: 1041, 1212, 1361, 1555, 1701, 1731 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$) δ: 0.99-1.06 (m, 3H), 1.26-1.33 (m, 3H), 1.78-1.97 (m, 1.5H), 2.08-2.32 (m, 0.5H), 2.14 (s, 1.5H), 2.16 (s, 1.5H), 4.21-4.31 (m, 2H), 4.75-4.85 (m, 1H), 5.36 (d, 0.5H, *J* = 6.4 Hz), 5.60 (d, 0.5H, *J* = 4.3 Hz). ^{13}C NMR (100MHz, $CDCl_3$) δ: 10.3, 10.7, 14.2, 20.5, 20.6, 22.7, 23.0, 62.7, 62.8, 71.3, 71.7, 87.8, 88.2, 166.4, 166.7, 169.6, 169.8. Anal. Calcd for $C_9H_{15}NO_6$ (233.22): C, 46.35; H, 6.48; N, 6.01. Found: C, 46.42; H, 6.53; N, 5.98.
4a		Yellow oil. IR (neat) ν: 1037, 1205, 1333, 1537, 1674, 1732, 3080 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$) δ: 1.17 (t, 3H, *J* = 7.3 Hz), 1.32 (t, 3H, *J* = 7.3 Hz), 3.06 (q, 2H, *J* = 7.3 Hz), 4.27 (q, 2H, *J* = 7.3 Hz), 6.95 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 12.6, 14.3, 21.3, 62.0, 120.8, 164.9, 165.5. GC-MS (EI) m/z: 127, 99(100), 81, 71, 53, 43, 38, 29. Anal. Calcd for $C_7H_{11}NO_4$ (173.17): C, 48.55; H, 6.40; N, 8.09. Found: C, 48.60; H, 6.43; N, 8.5.
4b		Yellow oil. IR (neat) ν: 1039, 1207, 1330, 1538, 1676, 1731, 3082 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$) δ: 1.33 (t, 3H, *J* = 7.3 Hz), 2.58 (s, 3H), 4.28 (q, 2H, *J* = 7.3 Hz), 7.06 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 14.2, 14.3, 62.0, 121.6, 160.0, 164.4. GC-MS (EI) m/z: 114, 113, 85, 67(100), 41, 38, 29. Anal. Calcd for $C_6H_9NO_4$ (159.14): C, 45.28; H, 5.70; N, 8.80. Found: C, 45.33; H, 5.73; N, 8.77.
4c		Yellow oil. IR (neat) ν: 700, 751, 766, 860, 1174, 1327, 1373, 1538, 1668, 1727, 3029 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$) δ: 1.30 (t, 3H, *J* = 7.3 Hz), 2.84-2.90 (m, 2H), 3.32-3.39 (m, 2H), 4.22 (q, 2H, *J* = 7.3 Hz), 7.02 (s, 1H), 7.18-7.33 (m, 5H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 14.2, 29.6, 34.3, 62.0, 122.1, 126.8, 128.7, 128.8, 139.8, 162.9, 164.0. GC-MS (EI) m/z: 249(M^+), 203, 173, 129, 91(100), 65. Anal. Calcd for $C_{13}H_{15}NO_4$ (249.26): C, 62.64; H, 6.07; N, 5.62. Found: C, 62.59; H, 6.02; N, 5.66.
4d		Yellow oil. IR (neat) ν: 1204, 1341, 1373, 1537, 1671, 1726, 2247, 3086 cm^{-1}. ^{1}H NMR (400MHz, $CDCl_3$) δ: 1.33 (t, 3H, *J* = 7.3 Hz), 1.69-1.79 (m, 4H), 2.40 (t, 2H, *J* = 6.4 Hz), 3.07-3.13 (m, 2H), 4.28 (q, 2H, J = 7.3 Hz), 7.04 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 14.3, 17.0, 25.0, 26.5, 27.1, 62.2, 119.4, 122.1, 162.0, 164.1. GC-MS (EI) m/z: 181, 180, 134(100), 106, 79, 67, 53, 41, 29. Anal. Calcd for $C_{10}H_{14}N_2O_4$ (226.23): C, 53.09; H, 6.24; N, 12.38. Found: C, 53.14; H, 6.28; N, 13.33.

4e		Yellow oil. IR (neat) ν: 1130, 1180, 1337, 1541, 1672, 1735, 3086 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 0.88 (t, 3H, *J* = 7.3 Hz), 1.29-1.39 (m, 4H), 1.48-1.59 (m, 2H), 3.02-3.08 (m, 2H), 3.82 (s, 3H), 6.97 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 14.1, 22.4, 27.4, 27.8, 31.5, 52.8, 120.5, 164.7, 164.9. GC-MS (EI) m/z: 184, 170, 155, 123, 111, 99, 95(100), 91, 59, 41, 29. Anal. Calcd for $C_9H_{15}NO_4$ (201.22): C, 53.72; H, 7.51; N, 6.96. Found: C, 53.77; H, 7.55; N, 6.92.
4f		Yellow oil. IR (neat) ν: 1209, 1338, 1538, 1673, 1731, 1737, 3079 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.54-1.64 (m, 2H), 1.66-1.75 (m, 2H), 2.34 (t, 2H, J = 7.3 Hz), 3.03-3.11 (m, 2H), 3.66 (s, 3H), 3.82 (s, 3H), 7.00 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 22.5, 27.1, 27.5, 33.7, 51.8, 52.8, 121.1, 164.1, 164.6, 173.8. GC-MS (EI) m/z: 214, 199, 167, 135, 107, 79, 59(100), 41. Anal. Calcd for $C_{10}H_{15}NO_6$ (245.23): C, 48.98; H, 6.17; N, 5.71. Found: C, 49.04; H, 6.20; N, 5.68.
4g		Yellow oil. IR (neat) ν: 1106, 1338, 1540, 1670, 1723, 3086 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.31 (d, 6H, *J* = 6.0 Hz), 2.00-2.10 (m, 2H), 3.16-3.22 (m, 2H), 3.60 (t, 2H, *J* = 6.4 Hz), 5.07-5.20 (m, 1H), 7.05 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 21.9, 25.5, 30.9, 44.2, 70.2, 123.0, 162.2, 163.5. GC-MS (EI) m/z: 193, 176, 147, 129, 65, 41(100). Anal. Calcd for $C_9H_{14}ClNO_4$ (235.66): C, 45.87; H, 5.99; N, 5.94. Found: C, 45.91; H, 6.03; N, 5.90.
4h		Yellow oil. IR (neat) ν: 1107, 1204, 1338, 1374, 1538, 1668, 1725, 3079 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.21-1.41 (m, 16), 1.47-1.57 (m, 2H), 1.97-2.06 (m, 2H), 2.99-3.05 (m, 2H), 4.88-5.00 (m, 2H), 5.06-5.17 (m, 1H), 5.72-5.86 (m, 1H), 6.93 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 21.9, 27.5, 28.1, 29.1, 29.2, 29.3, 29.5, 34.0, 69.8, 114.4, 121.6, 139.4, 163.8, 164.1. GC-MS (EI) m/z: 238, 209, 192, 176, 161, 147, 121, 107, 93, 81, 67, 55, 43(100), 41. Anal. Calcd for $C_{16}H_{27}NO_4$ (297.39): C, 64.62; H, 9.15; N, 4.71. Found: C, 64.57; H, 9.11; N, 4.74.
4i		Yellow oil. IR (neat) ν: 1193, 1237, 1339, 1368, 1540, 1670, 1722, 1742, 3076 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.56-1.79 (m, 6H), 1.82-1.97 (m, 4H), 2.03 (s, 3H), 3.11-3.19 (m, 2H), 4.12 (t, 2H, *J* = 6.8 Hz), 5.21-5.31 (m, 1H), 7.00 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 21.1, 23.9, 24.6, 27.2, 32.9, 63.6, 79.2, 122.5, 162.9, 163.8, 171.2. GC-MS (EI) m/z: 239, 171, 158, 153, 140, 129, 111, 69, 43(100), 41. Anal. Calcd for $C_{13}H_{19}NO_6$ (285.29): C, 54.73; H, 6.71; N, 4.91. Found: C, 54.76; H, 6.74; N, 4.88.
4j		Yellow oil. IR (neat) ν: 1196, 1338, 1370, 1540, 1669, 1724, 3071 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 0.93 (d, 6H, *J* = 6.8 Hz), 1.36-1.45 (m, 2H), 1.56-1.80 (m, 7H), 1.84-1.98 (m, 2H), 3.00-3.06 (m, 2H), 5.25-5.32 (m, 1H), 6.92 (s, 1H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 22.4, 23.9, 25.8, 28.5, 32.9, 37.0, 79.0, 121.3, 164.1, 164.5. GC-MS (EI) m/z: 187, 169, 141, 131, 95, 81, 69(100), 67, 55, 41, 29. Anal. Calcd for $C_{13}H_{21}NO_4$ (255.31): C, 61.16; H, 8.29; N, 5.49. Found: C, 61.20; H, 8.32; N, 5.47.
4k		Yellow oil. IR (neat) ν: 698, 856, 1183, 1228, 1337, 1372, 1540, 1602, 1671, 1726, 3031 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.36 (t, 3H, *J* = 7.3 Hz), 4.33 (q, 2H, *J* = 7.3 Hz), 4.47 (s, 2H), 7.12 (s, 1H), 7.21-7.33 (m, 5H). ^{13}C NMR (100MHz, $CDCl_3$) δ: 14.3, 32.7, 62.3, 121.9, 127.6, 129.0, 129.1, 135.1, 162.2, 164.3. GC-MS (EI) m/z: 235(M^+), 188, 144, 133, 115(100), 105, 91, 65, 29. Anal. Calcd for $C_{12}H_{13}NO_4$ (235.24): C, 61.27; H, 5.57; N, 5.95. Found: C, 61.30; H, 5.60; N, 5.92.
4l		Yellow oil. IR (neat) ν: 1539, 1637, 1730 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 3.88 (s, 3H), 7.08 (d, 1H, *J* = 13.5 Hz), 7.69 (d, 1H, *J* = 13.5 Hz). ^{13}C NMR (100MHz, $CDCl_3$) δ: 53.3, 127.4, 149.3, 163.3. GC-MS (EI) m/z: 131(M^+), 100, 85, 59(100), 53, 46, 41, 30. Anal. Calcd for $C_4H_5NO_4$ (131.09): C, 36.65; H, 3.84; N, 10.69. Found: C, 36.68; H, 3.86; N, 10.66.
4m		Waxy yellow solid. IR (neat) ν: 1541, 1638, 1732 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.32 (t, 3H, *J* = 7.3 Hz), 4.29 (q, 2H, *J* = 7.3 Hz), 7.05 (d, 1H, *J* = 13.5 Hz), 7.64 (d, 1H, *J* = 13.5 Hz). ^{13}C NMR (100MHz, $CDCl_3$) δ: 14.1, 62.6, 127.8, 149.1, 162.8. GC-MS (EI) m/z: 146($M+1^+$), 100, 85, 71, 53, 43, 29(100). Anal. Calcd for $C_5H_7NO_4$ (145.11): C, 41.38; H, 4.86; N, 9.65. Found: C, 41.42; H, 4.89; N, 9.61
4n		Waxy yellow solid. IR (neat) ν: 1542, 1641, 1730 cm^{-1}. ^1H NMR (400MHz, $CDCl_3$) δ: 1.56-1.82 (m, 6H), 1.84-1.99 (m, 2H), 5.28-5.34 (m, 1H), 7.05 (d, 1H, *J* = 13.5 Hz), 7.63 (d, 1H, *J* = 13.5 Hz). ^{13}C NMR (100MHz, $CDCl_3$) δ: 23.9, 32.8, 79.9, 128.4, 149.0, 162.6. GC-MS (EI) m/z: 186($M+1^+$), 101, 85, 68(100), 53, 41, 30. Anal. Calcd for $C_8H_{11}NO_4$ (185.18): C, 51.89; H, 5.99; N, 7.56. Found: C, 51.93; H, 6.03; N, 7.51.