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A general method for making bicyclic compounds with nitrogen at a bridgehead – application to the halichlorine spiro subunit

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Spectral data for final products. The symbols s, d, t, q in the ^{13}C NMR spectra refer to 0, 1, 2, or 3 attached hydrogens.

Compound 3: FTIR (CH_2Cl_2 cast) 1732, 1714, 1672 cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 0.91 (d, J = 6.3 Hz, 3 H), 1.07-1.15 (m, 1 H), 1.35-1.57 (m, 7 H), 1.64-1.79 (m, 3 H), 1.86-1.91 (m, 1 H), 1.97-2.17 (m, 4 H), 2.20-2.26 (m, 1 H), 2.29-2.35 (m, 1 H), 2.96-3.00 (m, 1 H), 3.28-3.32 (m, 1 H), 3.44 (d, J = 16.0 Hz, 1 H), 3.59 (s, 3 H), 3.72 (s, 3 H), 6.81-6.83 (m, 1 H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 20.1 (d), 21.7 (t), 23.6 (t), 29.1 (t), 31.6 (q), 32.61 (t), 32.63 (t), 36.1 (t), 41.1 (t), 47.6 (t), 51.1 (d), 51.5 (q), 52.9 (d), 57.1 (q), 67.2 (s), 129.0 (s), 136.6 (d), 166.5 (s), 174.3 (s); exact mass m/z calcd for $\text{C}_{20}\text{H}_{32}\text{NO}_4$ ($M + H$) 350.233134, found 350.233264.

Compound 21: FTIR (CH_2Cl_2 cast) 1716, 1650 cm^{-1} ; $[\alpha]_D^{22} + 195.9$ (c 1.1, CH_2Cl_2); ^1H NMR (CDCl_3 , 400 MHz) δ 1.38-1.47 (m, 1 H), 1.70-1.92 (m, 2 H), 1.95-2.03 (m, 1 H), 2.06-2.15 (m, 2 H), 2.19 (ABq, $\bullet\nu_{AB} = 18.0$ Hz, J = 9.2 Hz, 1 H), 2.38-2.47 (m, 1 H), 2.82-2.87 (m, 1 H), 3.21 (dt, J = 2.4, 8.8 Hz, 1 H), 3.71 (s, 3 H), 3.78-3.83 (m, 1 H), 6.97-7.00 (m, 1 H); ^{13}C NMR (CDCl_3 , 100 MHz)

δ 21.4 (t), 30.4 (t), 32.8 (t), 51.5 (t), 51.6 (q), 54.1 (t), 58.7 (d), 129.4 (s), 138.3 (d), 166.3 (s); exact mass m/z calcd for C₁₀H₁₅NO₂ 181.11028, found 181.11019.

Compound **26**: FTIR (CH₂Cl₂ cast) 1713, 1661 cm⁻¹; ¹H NMR (CDCl₃, 400 MHz) δ 1.17-1.35 (m, 2 H), 1.56-1.80 (m, 4 H), 2.00-2.16 (m, 3 H), 2.21-2.29 (m, 1 H), 2.76-2.82 (m, 1 H), 3.02 (d, J = 11.5 Hz, 1 H), 3.59 (d, J = 16.6 Hz, 1 H), 3.71 (s, 3 H), 6.91-6.93 (m, 1 H); ¹³C NMR (CDCl₃, 100 MHz) δ 24.2 (t), 25.8 (t), 33.2 (t), 34.0 (t), 51.5 (q), 53.4 (t), 55.9 (t), 56.4 (d), 128.1 (s), 137.1 (d), 166.1 (s); exact mass m/z calcd for C₁₁H₁₇NO₂ 195.12593, found 195.12585.

Compound **32**: FTIR (CH₂Cl₂ cast) 1716, 1660 cm⁻¹; ¹H NMR (CDCl₃, 300 MHZ) δ 1.21-1.80 (m, 8 H), 2.02-2.38 (m, 2 H), 2.38-2.50 (m, 1 H), 2.68-2.91 (m, 2 H), 3.20 (d, J = 16.5 Hz, 1 H), 3.57 (d, J = 16.5 Hz, 1 H) 3.72 (s, 3 H), 6.91-6.99 (m, 1 H); ¹³C NMR (CDCl₃, 100 MHz) δ 24.4 (t), 27.4 (t), 28.7 (t), 33.0 (t), 35.1 (t), 51.5 (d), 54.3 (t), 54.8 (t), 59.0 (q), 129.5 (s), 138.6 (d), 166.2 (s); exact mass m/z calcd for C₁₂H₁₉NO₂ 209.14159, found 209.14128.

Compound **38**: FTIR (CH₂Cl₂ cast) 1715, 1664 cm⁻¹; ¹H NMR (CDCl₃, 300 MHz) δ 1.32-1.84 (m, 10 H), 1.96-2.30 (m, 2 H), 2.61-2.80 (m, 2 H), 2.80-2.96 (m, 1 H), 3.32 (d, J = 17.0 Hz, 1 H), 3.49 (d, J = 17.0 Hz, 1 H) 3.70 (s, 3 H), 6.94-6.98 (m, 1 H); ¹³C NMR (CDCl₃, 100 MHz) δ 26.0 (t), 26.2 (t), 26.6 (t), 27.6 (t), 31.1 (t), 33.1 (t), 50.0 (t), 51.4 (d), 52.3 (t), 56.2 (q), 129.5 (s), 138.6 (d), 166.5 (s); exact mass m/z calcd for C₁₃H₂₁NO₂ 223.15723, found 223.15691.