Flow synthesis using gaseous ammonia in a Teflon AF-2400 tube-in-tube reactor: Paal-Knorr pyrrole formation and gas concentration measurement by inline flow titration.

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General Experimental:

All solvents and reagents were used as supplied from commercial suppliers (Sigma-Aldrich, Alfa-Aesar, Fisher Scientific) and used as supplied unless stated otherwise.
Reactions were monitored by analytical thin layer chromatography (TLC) on Merck silica gel 60 F_{254} pre-coated glass-backed plates and visualised by ultraviolet fluorescence (λ = 254 nm) and/or by staining with aqueous acidic ammonium molybdate, aqueous permanganate or ethanolic vanillin/AcOH/H_2SO_4. Flash column chromatography was performed on silica gel (Merck 9385 grade), using compressed air to elute the column. Petrol refers to petroleum ether distillate (b.p. 40–60 °C).

Infrared spectra were recorded as thin films on a Perkin–Elmer Spectrum One FT-IR spectrometer using universal ATR sampling accessories. Only selected peaks are reported.

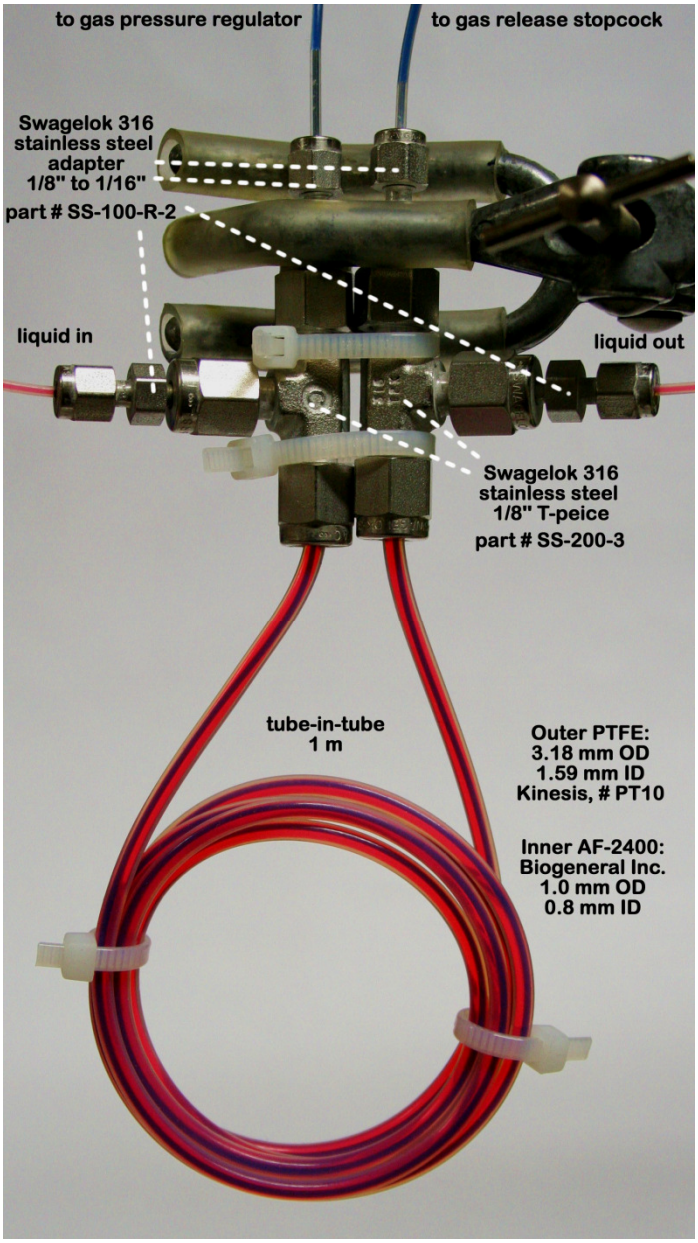
^{1}H NMR spectra were recorded on either a Bruker DRX-600 (600 MHz) operating at 600 MHz using incomplete deuteration of the solvent as the internal lock. Chemical shift data are given in units δ, with calibration based on residual monoprotic solvent (7.26 $CHCl_3$). Data is reported as follows: chemical shift δ/ppm (number of protons, multiplicity, coupling constant J/Hz, assignment). Coupling constants are reported to the nearest 0.1 ppm and the multiplicity of a signal is indicated as: br – broad, s – singlet, d – doublet, t – triplet, sept – septet, m – multiplet or multiples thereof. They are only denoted if the multiplicity is clear. Diastereotopic protons are denoted as HX and HX' where they give rise to separate signals or HX if they result in only one signal.

^{13}C NMR spetra were recorded at 150 MHz on a Bruker DRX-600 spectrometer with broadband proton decoupling using incomplete deuteration of the solvent as the internal lock. Chemical shift data are given in units δ, with calibration based on residual monoprotic

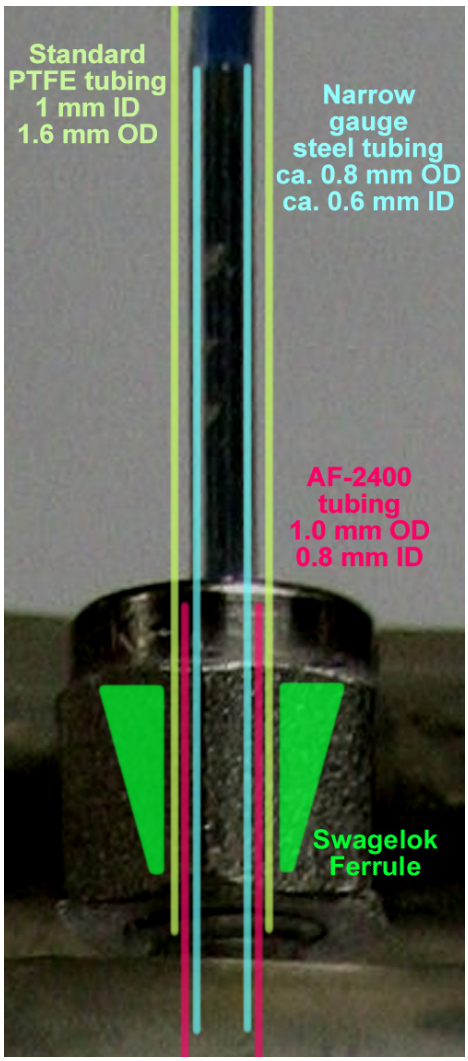
solvent (δ_c $CDCl_3$ = 77.0). Spectra are assigned as fully as possible using DEPT, COSY, HMQC, HMBC, TOCSY and nOe experiments.

High resolution mass spectrometry (HRMS) was performed on a Waters Micromass LCT Premier spectrometer using time of flight with positive electrospray ionisation (ESI^+) or negative electrospray ionisation (ESI^-), an ABI/MDS Sciex Q-STAR Pulsar with ESI^+, or a Bruker BioApex II 4.7e FTICR utilising either ESI^+ or a positive electron impact ionisation (EI^+) source equipped with a direct insertion probe. The mass reported is that containing the most abundant isotopes (^{35}Cl and ^{79}Br).

Parts used in the construction of the reactor:

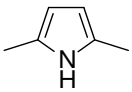


Schematic of the seal at the gas connection to the 1/16'' end of the Swagelok 1/8'' to 1/16'' reduction adapter (narrow gauge steel tubing is not essential for the operation of the reactor, but it makes construction easier):



Compound Data (arbitrary numbering):

2a



Trofimov, Tarasova, Mikhaleva, Kalinina, Sinegovskaya, Henkelmann, *Synthesis*, **2000**, 1585.

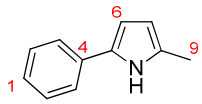
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.83 (1H, br. s, N*H*), 5.79 (2H, s, C*H*), 2.27 (6H, s, CH_3).

^{13}C NMR (150 MHz, $CDCl_3$): δ 126.2 (C), 105.7 (*C*H), 12.9 (*C*H_3).

IR (neat, cm^{-1}): 3359 (s, NH), 3106, 2925, 1594.

HRMS (+ESI) m/z 96.0808 [$(M+H)^+$ calculated for $C_6H_{10}N$ 96.0808].

2b



D. M. Barber, H. Sanganee, D. J. Dixon, *Chem. Commun.*, **2011**, *47*, 4379.
Wen, Zhang, Chen, Zhang, Yu, *J. Org. Chem.*, **2012,** 766.

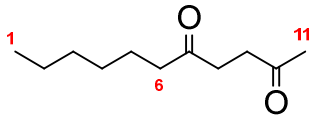
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.12 (1H, br. s, N*H*), 7.44 (2H, d, *J* = 7.6, H3), 7.35 (2H, app. t, *J* = 7.6 Hz, H2), 7.18 (1H, t, *J* = 7.6 Hz, H1), 6.42 (1H, app. t, *J* = 2.8 Hz, H6), 5.97 (1H, br. s, H7), 2.35 (3H, s, H9).

^{13}C NMR (150 MHz, $CDCl_3$): δ 132.9 (C4), 130.8 (C5), 129.0 (C8), 128.8 (C2), 125.6 (C1), 123.3 (C3), 107.9 (C7), 106.2 (C6), 13.2 (C9).

IR (neat, cm^{-1}): 3399 (s, NH), 3099, 3045, 2921, 1683, 1513.

HRMS (+ESI) m/z 158.0962 [$(M+H)^+$ calculated for $C_{11}H_{12}N$ 158.0964].

1c



Stetter, Kuhlmann, Haese, *Org. Synth. Col. Vol. 8*, **1993**, 620
Wedler, Schick, *Synthesis*, **1992**, 543

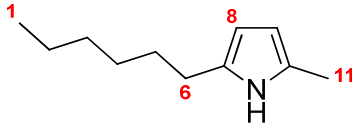
^{1}H NMR (600 MHz, $CDCl_3$): δ 2.68 (4H, m, H8 and H9), 2.44 (2H, t, *J* = 7.4 Hz, H6), 2.18 (3H, s, H11), 1.57 (2H, dt, *J* = 7.5, 7.5 Hz, H5), 1.27 (6H, m, H2, H3 and H4), 0.87 (3H, t, *J* = 7.1 Hz, H1).

^{13}C NMR (150 MHz, $CDCl_3$): δ 209.6 (C7), 207.3 (C10), 42.8 (C6), 36.9 (C9), 36.0 (C8), 31.6 (C3), 29.9 (C11), 28.8 (C4), 23.8 (C5), 22.4 (C2), 14.0 (C1).

IR (neat, cm^{-1}): 2930, 1697 (s, C=O), 1695 (s, C=O), 1414.

HRMS (+ESI) m/z 185.1531 [$(M+H)^+$ calculated for $C_{11}H_{21}O_2$ 185.1536].

2c

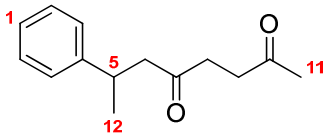


^{1}H NMR (600 MHz, $CDCl_3$): δ 7.57 (1H, br. s, N*H*), 5.78 (1H, s, H9), 5.78 (1H, s, H8), 2.6 (2H, t, *J* = 7.6 Hz, H6), 2.25 (3H, s, H11), 1.61 (2H, dt, *J* = 7.6, 7.6 Hz, H5), 1.38 (2H, m, H4), 1.32 (4H, m, H2 and H3), 0.91 (3H, t, *J* = 7.2 Hz, H1).

^{13}C NMR (150 MHz, $CDCl_3$): δ 131.6 (C7), 125.8 (C10), 105.6 (C9), 104.8 (C8), 31.7 (C2), 29.8 (C5), 29.1 (C4), 27.8 (C6), 22.6 (C3), 14.1 (C1), 13.0 (C11).

IR (neat, cm^{-1}): 3366 (s, NH), 2927, 2855, 1592, 1465, 1401.
HRMS (+ESI) m/z 166.1585 [$(M+H)^+$ calculated for $C_{11}H_{20}N$ 166.1590].

1d

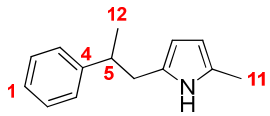


^{1}H NMR (600 MHz, $CDCl_3$): δ 7.28 (2H, app. t, *J* = 7.5 Hz, H2), 7.19 (3H, m, H1 and H3), 3.31 (2H, app. sextet, *J* = 7.1 Hz, H5), 2.78 (1H, dd, *J* = 16.3, 6.5 Hz, H6), 2.69–2.52 (5H, m, H6', H8 and H9), 2.15 (3H, s, H11), 1.26 (3H, d, *J* = 7.1 Hz, H12).

^{13}C NMR (150 MHz, $CDCl_3$): δ 208.2 (C7), 207.1 (C10), 126.2 (C4), 128.5 (C2), 126.7 (C3), 126.3 (C1), 51.1 (C6), 36.8 (C8 or C9), 36.7 (C8 or C9), 35.4 (C5) 29.9 (C11), 21.9 (C12).

IR (neat, cm^{-1}): 3028, 2963, 1709 (s, C=O), 1603, 1494.
HRMS (+ESI) m/z 219.1378 [$(M+H)^+$ calculated for $C_{14}H_{19}O_2$ 219.1380].

2d

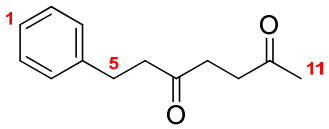


^{1}H NMR (600 MHz, $CDCl_3$): δ 7.34 (2H, dd, *J* = 7.7, 7.1 Hz, H2), 7.25 (4H, m, H1, H3 and N*H*), 5.77 (1H, m, H8), 5.75 (1H, br. s, H9), 3.00 (1H, app. sextet, *J* = 7.0 Hz, H5), 2.89 (1H, dd, *J* = 14.8, 7.0 Hz, H6), 2.80 (1H, dd, *J* = 14.8, 7.5 Hz, H6'), 2.16 (3H, s, H11), 1.31 (3H, d, *J* = 7.0 Hz, H12).

^{13}C NMR (150 MHz, $CDCl_3$): δ 147.0 (C4), 129.5 (C7), 128.5 (C2), 127.0 (C3), 126.2 (C1), 126.1 (C10), 106.3 (C8), 105.4 (C9), 40.9 (C5), 36.9 (C6), 21.6 (C12), 12.9 (C11).

IR (neat, cm^{-1}): 3370 (s, NH), 3027, 2961, 2927, 1593, 1493.
HRMS (+ESI) m/z 200.1427 [$(M+H)^+$ calculated for $C_{14}H_{18}N$ 200.1434].

1g



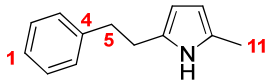
Xue, Li, Liu, Guo, *J. Org. Chem.*, **2006**, 215.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.27 (2H, app. t, *J* = 7.8 Hz, H2), 7.18 (3H, m, H1 and H3), 2.90 (2H, t, *J* = 7.2 Hz, H5), 2.78 (2H, t, *J*= 7.2 Hz, H6), 2.71 (2H, m, H8' and H9'), 2.65 (2H, m, H8' and H9'), 2.18 (3H, s, H11).

^{13}C NMR (150 MHz, $CDCl_3$): δ 208.4 (C7), 207.1 (C10), 141.0 (C4), 128.5 (C2), 128.3 (C3), 126.1 (C1), 44.2 (C6), 36.9 (C9), 36.2 (C8), 29.9 (C11), 29.7 (C5).

IR (neat, cm^{-1}): 3028, 2906, 1709 (s, C=O), 1604, 1496.
HRMS (+ESI) m/z 205.1217 [$(M+H)^+$ calculated for $C_{13}H_{17}O_2$ 205.1223].

2g

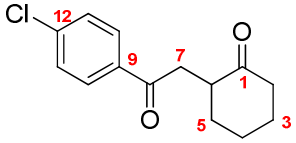


Mori, Akashi, Hori, Hori, Nishida, Sato, *Bull. Chem. Soc. Jap.* **2004**, 1655

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.49 (1H, br. s, N*H*), 7.33 (2H, app. t, *J* = 7.7 Hz, H2), 7.24 (3H, m, H1 and H3), 5.85 (1H, s, H9), 5.80 (1H, s, H8), 2.95 (2H, m, H5 and H6), 2.89 (2H, m, H5' and H6'), 2.23 (3H, s, H11).
^{13}C NMR (150 MHz, $CDCl_3$): δ 141.8 (C4), 130.6 (C7), 128.4 (C2), 128.4 (C3), 126.2 (C10), 126.1 (C1), 105.7 (C8), 105.2 (C9), 36.3 (C5), 29.8 (C6), 13.0 (C11).

IR (neat, cm^{-1}): 3366 (s, NH), 3027, 2925, 1593, 1496.
HRMS (+ESI) m/z 186.1270 [$(M+H)^+$ calculated for $C_{13}H_{16}N$ 186.1277].

1i



Xie, Huang, *Chem. Comm.* **2010**, 1947

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.91 (2H, d, *J* = 8.8 Hz, H10), 7.42 (2H, d, *J* = 8.8 Hz, H11), 3.54 (1H, dd, *J* = 17.5, 7.1 Hz, H7), 3.14 (1H, ddd, *J* = 12.2, 12.2, 5.8 Hz, H6), 2.60 (1H, dd,

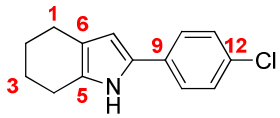
J = 17.5, 5.4 Hz, H7'), 2.42 (2H, m, H2), 2.18 (1H, m, H5), 2.13 (1H, m, H3), 1.89 (1H, m, H4), 1.76 (1H, qt, *J* = 12.9, 3.1 Hz, $H4_{ax}$), 1.67 (1H, m, H3'), 1.44 (1H, qd, *J* = 13.0, 3.5 Hz, $H5_{ax}$).

^{13}C NMR (150 MHz, $CDCl_3$): δ 211.1 (C1), 197.4 (C8), 139.4 (C9), 135.4 (C12), 129.5 (C10), 128.8 (C11), 46.5 (C6), 41.9 (C2), 38.3 (C7), 34.3 (C5), 27.9 (C3), 25.3 (C4).

IR (neat, cm^{-1}): 2934, 2861, 1708 (s, C=O), 1683 (s, C=O), 1588, 1572.

HRMS (+ESI) m/z 251.0825 [$(M+H)^+$ calculated for $C_{14}H_{16}O_2Cl$ 251.0833].

2i



^1H NMR (600 MHz, $CDCl_3$): δ 7.91 (1H, br. s, N*H*), 7.33 (2H, d, *J* = 8.3 Hz, H10), 7.28 (2H, d, *J* = 8.3 Hz, H11), 6.26 (1H, d, *J* = 2.4 Hz, H7), 2.63 (2H, app. t, *J* = 6.2 Hz, H4 or H1), 2.54 (2H, app t, *J* = 5.8 Hz, H4 or H1), 1.85 (2H, m, H2 or H3), 1,78 (2H, m, H2 or H3).

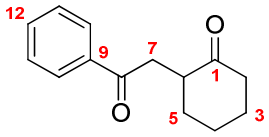
^{13}C NMR (150 MHz, $CDCl_3$): δ 131.7 (C9), 130.9 (C12), 129.1 (C5), 128.9 (C11), 128.7 (C8), 124.4 (C10), 119.2 (C6), 105.7 (C7), 23.7 (C2 or C3), 23.3 (C2 or C3), 22.8 (C1 or C4), 22.8 (C1 or C4).

IR (neat, cm^{-1}): 3444 (s, NH), 2934, 2856, 1587, 1513.

HRMS (+ESI) m/z 232.0884 [$(M+H)^+$ calculated for $C_{14}H_{15}NCl$ 232.0888].

m.p. 174–176 ºC

1j



Ma, Inokuchi, *Chem. Comm.* **2010**, 7073

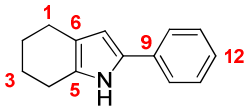
^1H NMR (600 MHz, $CDCl_3$): δ 7.98 (2H, d, *J* = 7.4 Hz, H10), 7.55 (1H, t, *J* = 7.4 Hz, H12), 7.45 (2H, app. t, *J* = 7.5 Hz, C11), 3.59 (1H, dd, *J* = 17.3, 6.0 Hz, H7), 3.16 (1H, ddd, *J* = 12.3, 12.3, 5.9 Hz, H6), 2.67 (1H, dd, *J* = 17.5, 6.1 Hz, H7'), 2.44 (2H, m, H2), 2.19 (1H, m, H5), 2.13 (1H, m, H3), 1.88 (1H, m, H4), 1.77 (1H, qt, *J* = 13.1, 3.5 Hz, $H4_{ax}$), 1.66 (1H, m, H3'), 1.44 (1H, qd, *J* = 13.1, 3.8 Hz, $H5_{ax}$).

^{13}C NMR (150 MHz, $CDCl_3$): δ 211.5 (C1), 198.6 (C8), 137.1 (C12), 133.0 (C9), 128.5 (C11), 128.1 (C10), 46.4 (C6), 42.0 (C2), 38.3 (C7), 34.3 (C5), 28.0 (C3), 25.4 (C4).

IR (neat, cm^{-1}): 2933, 2862, 1709 (s, C=O), 1684 (s, C=O), 1597, 1581, 1448.

HRMS (+ESI) m/z 217.1221 [$(M+H)^+$ calculated for $C_{14}H_{17}O_2$ 217.1223].

2j



Hiroya, Matsumoto, Ashikawa, Ogiwara, Sakamoto, *Org. Lett.* **2006**, 5349

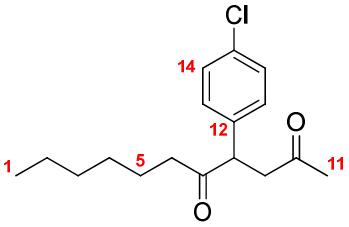
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.95 (1H, br. s, N*H*), 7.43 (2H, d, *J* = 8.1 Hz, H10), 7.34 (2H, app. t, *J* = 8.1 Hz, H11), 7.17 (1H, t, *J* = 8.1 Hz, H12), 6.30 (1H, d, *J* = 2.2 Hz, H7), 2.65 (2H, app. t, *J* = 5.9 Hz, H1), 2.57 (2H, app. t, *J* = 5.9 Hz, H4), 1.87 (2H, m, H2), 1.80 (2H, m, H3).

^{13}C NMR (150 MHz, $CDCl_3$): δ133.2 (C9), 130.2 (C8), 128.8 (C11), 128.5 (C6), 125.5 (C12), 123.4 (C10), 119.0 (C5), 105.2 (C7), 23.8 (C3), 23.4 (C2), 22.9 (C1), 22.0 (C4).

IR (neat, cm^{-1}): 3429 (s, NH), 2922, 2837, 1607, 1592, 1519.

HRMS (+ESI) m/z 298.1272 [$(M+H)^+$ calculated for $C_{14}H_{16}N$ 198.1277].

1h



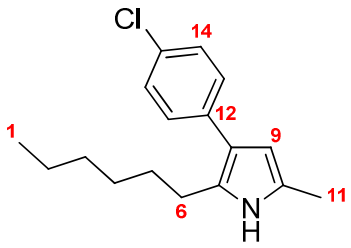
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.26 (2H, d, *J* = 8.6 Hz, H13), 7.11 (2H, d, *J* = 8.6 Hz, H14), 4.16 (1H, dd, *J* = 10.4, 3.5 Hz, H8), 3.39 (1H, dd, *J* = 18.1, 10.0 Hz, H9), 2.53 (1H, dd, *J* = 18.2, 4.1 Hz, H9')2.46 (1H, ddd, *J* = 17.3, 8.7, 6.1 Hz, H6), 2.34 (1H, ddd, *J* = 17.5, 8.3, 6.6 Hz, H6'), 2.13 (3H, s, H11), 1.48 (1H, m, H5), 1.44 (1H, m, H5'), 1.20 (2H, m, H2), 1.14 (4H, m, H3 and H4), 0.80 (3H, t, *J* = 7.2 Hz, H1).

^{13}C NMR (150 MHz, $CDCl_3$): δ 209.0 (C7), 206.4 (C10), 136.5 (C12), 133.4 (C15), 129.5 (C14), 129.2 (C13), 52.5 (C8), 46.5 (C9), 41.6 (C6), 31.4 (C11), 29.9 (C3), 28.6 (C4), 23.5 (C5), 22.4 (C2), 13.9 (C1).

IR (neat, cm^{-1}): 2930, 2858, 1711 (s, C=O), 1672, 1490.

HRMS (+ESI) m/z 295.1452 [$(M+H)^+$ calculated for $C_{17}H_{24}O_2Cl$ 295.1459].

2h



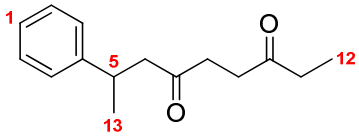
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.67 (1H, br. s, N*H*), 7.30 (4H, m, H13 and H14), 5.95 (1H, d, *J* = 1.9 Hz, H9), 2.69 (2H, t, *J* = 7.7 Hz, H6), 2.28 (3H, s, H11), 1.61 (2H, tt, *J* = 7.7, 7.7 Hz, H5), 1.35 (2H, tt, *J* = 7.7, 7.7 Hz, H4), 1.29 (4H, m, H2 and H3), 0.89 (3H, t, *J* = 7.3 Hz, H1).

^{13}C NMR (150 MHz, $CDCl_3$): δ 135.9 (C12), 130.6 (C15), 128.8 (C13), 128.3 (C14), 127.7 (C7), 125.9 (C10), 119.7 (C8), 106.3 (C9), 31.6 (C3), 30.1 (C5), 29.2 (C4), 26.5 (C6), 22.6 (C2), 14.0 (C1), 12.9 (C11).

IR (neat, cm^{-1}): 3434 (s, NH), 2924, 2855, 1601, 1525, 1488.

HRMS (+ESI) m/z 276.1505 [$(M+H)^+$ calculated for $C_{17}H_{23}NCl$ 276.1514].

1e



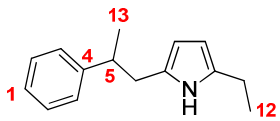
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.28 (2H, m, H2), 7.19 (3H, m, H1 and H3), 3.31 (1H, m, H5), 2.78 (1H, dd, *J* = 16.2, 6.1, H6), 2.64 (5H, m, H6', H8 and H9), 2.44 (2H, q, *J* = 7.3 Hz, H11), 1.25 (3H, d, *J* = 7.1 Hz, H13), 1.04 (3H, t, *J* = 7.3 Hz, H12).

^{13}C NMR (150 MHz, $CDCl_3$): δ 209.9 (C10), 208.4 (C7), 146.2 (C4), 128.5 (C2), 126.7 (C3), 126.2 (C1), 51.1 (C6), 36.8 (C9), 35.9 (C11), 35.5 (C8), 35.4 (C5), 21.9 (C13), 7.8 (C12).

IR (neat, cm^{-1}): 2966, 1708 (s, C=O), 1494, 1452, 1410, 1366.

HRMS (+ESI) m/z 233.1533 [$(M+H)^+$ calculated for $C_{15}H_{21}O_2$ 233.1536].

2e



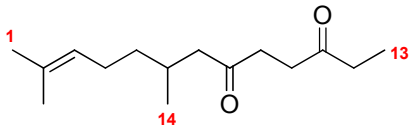
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.34 (2H, app. t, *J* = 7.6 Hz, H2), 7.24 (3H, m, H1 and H3), 7.18 (1H, br. s, N*H*), 5.79 (1H, m, H8), 5.76 (1H, m, H9), 3.01 (1H, qdd, *J* = 7.4, 7.4, 7.4 Hz, H5), 2.89 (1H, dd, *J* = 14.7, 7.1 Hz, H6), 2.82 (1H, dd, *J* = 14.9, 7.1 Hz, H6'), 2.49 (2H, q, *J* = 7.4 Hz, H11), 1.32 (3H, d, *J* = 7.0 Hz, H13), 1.14 (3H, t, *J* = 7.4 Hz, H12).

^{13}C NMR (150 MHz, $CDCl_3$): δ 147.0 (C4), 132.7 (C10), 129.3 (C7), 128.5 (C2), 127.1 (C3), 126.2 (C1), 106.0 (C8), 103.6 (C9), 40.9 (C5), 36.9 (C6), 21.6 (C13), 20.8 (C11), 13.5 (C12).

IR (neat, cm^{-1}): 3478 (s, NH), 3027, 2964, 2928, 1493, 1452.

HRMS (+ESI) m/z 214.1591 [$(M+H)^+$ calculated for $C_{15}H_{20}N$ 214.1590].

1f



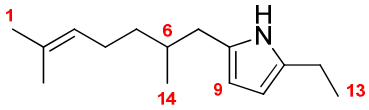
^{1}H NMR (600 MHz, $CDCl_3$): δ 5.07 (1H, t, *J* = 6.8 Hz, H3), 2.65 (4H, m, H9 and H10), 2.47 (2H, q, *J* = 7.4 Hz, H12), 2.43 (1H, dd, *J* = 15.8, 5.6 Hz, H7), 2.24 (1H, dd, *J* = 15.8, 8.4 Hz, H7'), 1.96 (3H, m, H4 and H6), 1.66 (3H, s, H1), 1.58 (3H, s, H1'), 1.29 (1H, m, H5), 1.17 (1H, m, H5'), 1.05 (3H, t, *J* = 7.4 Hz, H13), 0.88 (3H, d, *J* = 6.6 Hz, H14).

^{13}C NMR (150 MHz, $CDCl_3$): δ 210.0 (C11), 209.4 (C8), 131.4 (C2), 124.3 (C3), 50.2 (C7), 37.0 (C4), 36.7 (C9 or C10), 35.9 (C12), 35.5 (C9 or C10), 29.0 (C6), 25.7 (C1), 25.4 (C5), 19.6 (C14), 17.6 (C1'), 7.8 (C13).

IR (neat, cm^{-1}): 2915, 1710 (s, C=O), 1458, 1410, 1376.

HRMS (+ESI) m/z 239.2000 [$(M+H)^+$ calculated for $C_{15}H_{27}O_2$ 239.2006].

2f



^{1}H NMR (600 MHz, $CDCl_3$): δ 7.57 (1H, br. s, N*H*), 5.82 (1H, m, H10), 5.80 (1H, m, H9), 5.13 (1H, t, *J* = 6.7 Hz, H3), 2.62 (2H, q, *J* = 7.5 Hz, H12), 2.57 (1H, dd, *J* = 14.7, 6.0 Hz, H7), 2.38 (1H, dd, *J* = 14.7, 8.0 Hz, H7'), 2.07 (1H, m, H5), 2.00 (1H, m, H5'), 1.71 (3H, s, H1), 1.70 (1H, m, H6), 1.63 (3H, s, H1'), 1.43 (1H, m, H4), 1.26 (3H, t, *J* = 7.5 Hz, H13), 1.20 (1H, m, H4'), 0.94 (3H, d, *J* = 6.7 Hz, H14).

^{13}C NMR (150 MHz, $CDCl_3$): δ 132.5 (C11), 131.2 (C2), 129.9 (C8), 124.8 (C3), 105.8 (C9), 103.9 (C10), 36.8 (C4), 35.5 (C7), 33.6 (C6), 25.7 (C1), 25.7 (C5), 20.9 (C12), 19.7 (C14), 17.7 (C1'), 13.6 (C13).

IR (neat, cm^{-1}): 3379 (s, NH), 2965, 2913, 1590, 1455, 1376.

HRMS (+ESI) m/z 220.2055 [$(M+H)^+$ calculated for $C_{15}H_{26}N$ 220.2060].