**Synthesis of 2-Alkylidenepyrrolidines, Pyrroles and Indoles by
Condensation of Silyl Enol Ethers and 1,3-Bis-Silyl Enol Ethers with
1-Azido-2,2-dimethoxyethane and subsequent Reductive Cyclization**

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Supplementary Material

Table of Contents:

S2	General Comments
S2	Experimental Section
S51	Crystal Structure Analyses
S98	Copies of NMR spectra

General Comments. All solvents were dried by standard methods and all reactions were carried out under an inert atmosphere. For the ^1H and ^{13}C NMR spectra the deuterated solvents indicated were used. Mass spectral data (MS) were obtained by electron ionization (70 eV), chemical ionization (CI, H_2O) or the electrospray ionization (ESI). For preparative scale chromatography silica gel (60-200 mesh) was used. Melting points are uncorrected.

Experimental Section

1-Azido-2,2-dimethoxyethane (2):¹⁶ Sodium azide (19.503 g, 300 mmol) and potassium iodide (3.320 g, 20 mmol) were added to a solution of 1-bromo-2,2-dimethoxyethane (823.64 mL, 200 mmol) in DMSO (140 mL) at room temperature. The reaction mixture was heated to 90 °C and stirred for 5 days at 90 °C. After cooling to room temperature, water (200 mL) and diethylether (200 mL) were added, the organic layer was separated and the aqueous layer was repeatedly extracted with diethylether (4 × 200 mL). The combined organic extracts were dried over Na_2SO_4 , filtered, and the filtrate was concentrated to dryness in vacuo. Product **2** was isolated without further purification as slightly yellow oil (22.31 g, 85%). For safety reasons, it is recommended to carry out the reaction on a small scale (2.0 g sodium azide, no decrease of the yield was observed) and to use a safety shield. Although synthetic applications of **2** have been previously reported,¹⁶ a procedure for its preparation was not provided. ^1H NMR (CDCl_3 , 300 MHz): δ = 3.27 (d, J = 5.3 Hz, 2 H, CH_2N_3), 3.43 (s, 6 H, $2\times\text{OCH}_3$), 4.48 (t, J = 5.3 Hz, 1 H, CH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 51.66 (CH_2N_3), 54.25 (OCH_3), 103.08 (CH). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2995 (w), 2939 (m), 2837 (w, C–H), 2102

(s, N₃), 1444 (m), 1311 (w), 1275 (s), 1196 (m), 1129 (s), 1089 (s), 1064 (s), 1035 (m), 979 (w), 922 (w). MS (EI, 70 eV): m/z (%) = 75 (M⁺-CH₂N₃, 100).

CAUTION: The handling of low-molecular weight azides is dangerous, due to their potentially explosive character. Although, in our hands, neat **2** did not appear to be shock sensitive, the compound should be handled with great care. Neat azides must not be heated or distilled and all reactions should be carried out on a small scale. The use of a safety shield is highly recommended.

General procedure for the reaction of 1,3-bis-silyl enol ethers (1) or silyl enol ethers (10) with 1-azido-2,2-dimethoxyethane (2): To a CH₂Cl₂-solution (10 mL/mmol) of **1** or **10** (1 equiv.) and **2** (1.3 equiv.) was added Me₃SiOTf (0.5 equiv.) at -78 °C and the solution was stirred for 2 h at -78 °C. The temperature of the reaction mixture was allowed to rise 20 °C during 14 h and the mixture was stirred for 3 h at 20 °C. To this solution a saturated aqueous solution of NaHCO₃ was added, the organic layer was separated and the aqueous layer was repeatedly extracted with dichloromethane. The combined organic extracts were dried over Na₂SO₄, filtered and the solvent of the filtrate was removed *in vacuo*. The residue was purified by column chromatography (silica gel, *n*-hexane/EtOAc) to give **3** or **11**.

6-Azido-5-methoxy-3-oxo-hexanoic acid methyl ester (3a): Starting with **2** (1.705 g, 13 mmol), 1-methoxy-1,3-bis-trimethylsilyloxy-buta-1,3-diene (**1a**) (2.605 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3a** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (1.025 g, 48%). ¹H NMR (CDCl₃, 300 Hz): δ = 2.72 (dd, *J* = 17.1, 5.7 Hz, 1 H, CH₂), 2.89 (dd, *J* = 17.1, 6.9 Hz, 1 H, CH₂), 3.26 (dd, *J* = 12.9, 5.1 Hz, 1 H, CH₂N₃), 3.41 (s, 3 H, OCH₃), 3.46 (dd, *J* = 12.9, 4.2 Hz, 1 H, CH₂N₃), 3.50 (s, 2 H, CH₂), 3.75 (s, 3 H, OCH₃), 3.83 – 3.88 (m, 1 H, CH). ¹³C NMR

(CDCl₃, 75 MHz): δ_c = 44.7, 49.5, 52.1, 52.5, 57.4, 75.7, 167.1, 200.4. IR (neat, cm⁻¹): $\tilde{\nu}$ = 2955 (w), 2938 (w), 2104 (s), 1747 (s), 1276 (s), 1194 (m), 1181 (m), 1151 (m), 1114 (m), 1093 (m), 1042 (w). MS (EI, 70 eV): m/z (%) = 201 (M⁺ - Me, 7), 169 (39), 159 (M⁺ - CH₂N₃, 65), 153 (5), 127 (95), 115 (14), 101 (97), 85 (100). Anal. calcd. for C₈H₁₃O₃N₃ (215.209): C 44.65, H 6.09, N 19.53; found C 45.46, H 5.88, N 19.53.

6-Azido-5-methoxy-3-oxo-hexanoic acid ethyl ester (3b): Starting with **2** (1.705 g, 13 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-buta-1,3-diene (**1b**) (2.745 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3b** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (1.163 g, 51%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.29 (t, *J* = 7.2 Hz, 3 H, CH₃), 2.72 (dd, *J* = 17.1, 5.7 Hz, 1 H, CH₂), 2.89 (dd, *J* = 17.1, 6.6 Hz, 1 H, CH₂), 3.26 (dd, *J* = 12.9, 5.1 Hz, 1 H, CH₂N₃), 3.41 (s, 3 H, OCH₃), 3.47 (dd, *J* = 12.9, 3.9 Hz, 1 H, CH₂N₃), 3.48 (s, 2 H, CH₂), 3.87 (quint, *J* = 5.3 Hz, 1 H, CH), 4.21 (q, *J* = 7.2 Hz, 2 H, OCH₂). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 14.0, 44.7, 49.8, 52.6, 57.4, 61.3, 75.7, 166.7, 200.5. IR (neat, cm⁻¹): $\tilde{\nu}$ = 2985 (w), 2938 (w), 2103 (s), 1743 (s), 1716 (s), 1463 (w), 1446 (w), 1410 (w), 1369 (m), 1353 (w), 1296 (m), 1252 (m), 1190 (m), 1152 (w), 1113 (m), 1097 (m), 1028 (m). MS (EI, 70 eV): m/z (%) = 229 (M⁺, 8), 197 (7), 183 (79), 173 (47), 1410 (100). Anal. calcd. for C₉H₁₅O₄N₃ (229.236): C 47.16, H 6.60, N 18.33; found C 47.50, H 6.38, N 18.03.

6-Azido-5-methoxy-3-oxo-hexanoic acid isopropyl ester (3c): Starting with **2** (1.705 g, 13 mmol), 1-isopropoxy-1,3-bis-trimethylsilanyloxy-buta-1,3-diene (**1c**) (2.885 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3c** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (1.133 g, 47%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.26 (d, *J* = 8.5 Hz, 6 H, 2×CH₃), 2.72 (dd, *J* = 17.2, 5.7 Hz, 1 H, CH₂), 2.89 (dd, *J* = 17.2, 6.6 Hz, 1 H, CH₂), 3.26 (dd, *J* = 12.9, 5.0 Hz, 1 H, CH₂N₃), 3.40 (s, 3 H, OCH₃),

3.45 (s, 2 H, CH₂), 3.46 (dd, $J = 29.1, 4.1$ Hz, 1 H, CH₂N₃), 3.87 (quint, $J = 4.2$ Hz, 1 H, CH), 5.04 (sept, $J = 6.2$ Hz, 1 H, CH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 21.5, 44.7, 50.2, 52.7, 57.5, 69.0, 75.7, 166.2, 200.6$. IR (neat, cm⁻¹): $\tilde{\nu} = 2984$ (m), 2937 (m), 2103 (s), 1739 (s), 1716 (s), 1646 (w), 1467 (m), 1457 (m), 1409 (m), 1376 (m), 1355 (m), 1338 (m), 1293 (s), 1275 (s), 1255 (s), 1182 (m), 1147 (m), 1106 (s), 1040 (w), 967 (w). MS (EI, 70 eV): m/z (%) = 187 (M⁺ - CH₂N₃, 45), 155 (4), 145 (100). Anal. calcd. for C₁₀H₁₇O₄N₃ (243.263): C 49.38, H 7.04, N 17.27; found C 49.58, H 7.09, N 17.11.

6-Azido-5-methoxy-3-oxo-hexanoic acid 2-methoxy-ethyl ester (3d): Starting with **2** (1.705 g, 13 mmol), 1-(2-methoxy-ethoxy)-1,3-bis-trimethylsilanyloxy-buta-1,3-diene (**1d**) (3.085 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3d** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (1.145 g, 44%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 2.72$ (dd, $J = 12.9, 5.7$ Hz, 1 H, CH₂), 2.90 (dd, $J = 17.4, 6.9$ Hz, 1 H, CH), 3.25 (dd, $J = 12.9, 5.1$ Hz, 1 H, CH₂N₃), 3.39 (s, 3 H, OCH₃), 3.41 (s, 3 H, OCH₃), 3.47 (dd, $J = 12.9, 3.9$ Hz, 1 H, CH₂N₃), 3.53 (s, 2 H, CH₂), 3.62 (t, $J = 6.5$ Hz, 2 H, OCH₂CH₂OCH₃), 3.87 (quint, $J = 6.6$ Hz, 1 H, CH), 4.31 (t, $J = 6.9$ Hz, 2 H, OCH₂CH₂OCH₃). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 44.4, 49.7, 52.6, 57.4, 58.7, 64.1, 70.0, 75.7, 166.7, 200.3$. IR (neat, cm⁻¹): $\tilde{\nu} = 2935$ (w), 2103 (s), 1744 (s), 1717 (s), 1453 (w), 1411 (w), 1370 (m), 1316 (m), 1293 (s), 1253 (s), 1200 (m), 1182 (m), 1151 (m), 1129 (s), 1104 (m), 1037 (m). MS (EI, 70 eV): m/z (%) = 203 (M⁺ - CH₂N₃, 100), 184 (3), 145 (21), 127 (55), 112 (37).

6-Azido-5-methoxy-1-phenyl-hexane-1,3-dione (3e): Starting with **2** (1.705 g, 13 mmol), (1,3-bis-trimethylsilanyloxy-buta-1,3-dienyl)-benzene (**1e**) (3.066 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3e** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.486 g, 95%). ¹H NMR (CDCl₃, 300 MHz): $\delta =$

2.60 (dd, $J = 15$, 3.0 Hz, 1 H, CH₂), 2.74 (dd, $J = 15$, 6.9 Hz, 1 H, CH₂), 3.30 (dd, $J = 12.9$, 5.4 Hz, 1 H, CH₂N₃), 3.44 (s, 3 H, OCH₃), 3.46 (dd, $J = 12.9$, 3.6 Hz, 1 H, CH₂N₃), 3.85 – 3.92 (m, 1 H, CH), 6.22 (s, 1 H, CH from enol form), 7.42 (d, $J = 8.7$ Hz, 2 H, 2×CH from Ph), 7.52 (t, $J = 8.0$ Hz, 1 H, CH from Ph), 7.88 (d, $J = 8.7$ Hz, 2 H, 2×CH from Ph), 16.11 (broad s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 41.6, 53.1, 57.6, 77.2, 97.2, 126.9, 128.5, 132.4, 134.3, 183.1, 193.1$. IR (neat, cm⁻¹): $\tilde{\nu} = 2983$ (w), 2934 (w), 2831 (w), 2102 (s), 1604 (s), 1574 (s), 1492 (m), 1459 (m), 1440 (m), 1363 (w), 1354 (w), 1291 (s), 1182 (w), 1150 (w), 1109 (m), 1083 (m), 766 (m), 698 (w, Ph). MS (EI, 70 eV): m/z (%) = 205 (M⁺ - CH₂N₃, 22), 174 (11), 161 (8), 147 (98), 105 (100).

6-Azido-5-methoxy-4-methyl-3-oxo-hexanoic acid methyl ester (3f): Starting with **2** (1.705 g, 13 mmol), 1-methoxy-1,3-bis-trimethylsilanyloxy-penta-1,3-diene (**1f**) (2.745 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3f** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.177 g, 95%). ¹H NMR (CDCl₃, 300MHz): $\delta = 1.12$ (dd, $J = 40.2$, 6.9 Hz, 3 H, CH₃), 2.97 – 3.11 (m, 1 H, CH), 3.15 – 3.65 (m, 1 H, CH₂N₃), 3.39 (s, 3 H, OCH₃), 3.49 (s, 2 H, CH₂), 3.50 – 3.65 (m, 1 H from CH₂N₃, and 1 H from CH), 3.77 (s, 3 H, OCH₃). IR (neat, cm⁻¹): $\tilde{\nu} = 2983$ (w), 2952 (m), 2940 (m), 2102 (s), 1748 (s), 1715 (s), 1653(w), 1632 (w), 1454 (m), 1439 (m), 1406 (w), 1379 (w), 1346 (w), 1285 (m), 1261 (s), 1196(m), 1180 (m), 1151 (m), 1098 (s), 1053 (m), 1017 (m). MS (EI, 70 eV): m/z (%) = 173 (M⁺ - CH₂N₃, 43), 99 (100). Anal. calcd. for C₉H₁₅O₄N₃ (229.236): C 47.16, H 6.60, N 18.33; found C 46.55, H 6.67, N 18.09.

6-Azido-4-ethyl-5-methoxy-3-oxo-hexanoic acid ethyl ester (3g): Starting with **2** (1.705 g, 13 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-hexa-1,3-diene (**1g**) (3.026 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100mL), **3g** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.512 g, 98%). ¹H NMR (CDCl₃,

300MHz): δ = 9.3 (t, J = 7.4 Hz, 3 H, CH₃), 1.28 (t, J = 7.2 Hz, 3 H, OCH₂CH₃), 1.62 (quint, J = 7.4 Hz, 2 H, CH₂), 2.52 (t, J = 7.5 Hz, 1 H, CH), 2.91 – 3.06 (m, 1 H, CH₂N₃), 3.15 – 3.20 (m, 1 H, CH₂N₃), 3.40 (ds, J = 19.8 Hz, 3 H, OCH₃), 3.38 – 3.49 (m, 1 H, CH), 3.51 (ds, J = 17.1 Hz, 2 H, CH₂), 4.20 (q, J = 7.2 Hz, 2 H, OCH₂).

6-Azido-4,5-dimethoxy-3-oxo-hexanoic acid methyl ester (3h): Starting with **2** (1.705 g, 13 mmol), 1,4-dimethoxy-1,3-bis-trimethylsilanyloxy-buta-1,3-diene (**1h**) (2.905 g, 10 mmol), and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3h** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (1.550 g, 63%). ¹H NMR (CDCl₃, 300 MHz): δ = 3.42 (d, J = 2.4 Hz, 3 H, OCH₃), 3.46 (d, J = 2.4 Hz, 3 H, OCH₃), 3.49 (d, J = 1.8 Hz, 2 H, CH₂), 3.55 (dd, J = 16.2, 3.0 Hz, 1 H, CH), 3.63 – 3.68 (m, 2 H, CH₂N₃), 3.75 (d, J = 1.5 Hz, 3 H, OCH₃), 3.88 (dd, J = 21.9, 3.9 Hz, 1 H, CH). MS (EI, 70 eV): m/z (%) = 200 (M⁺ - 3×Me, 10), 153 (10), 144 (55), 131 (51), 116 (100), 101 (23).

4-(2-Azido-1-methoxy-ethyl)-3-oxo-hept-6-enoic acid ethyl ester (3i): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-hepta-1,3,6-triene (**1i**) (3.146 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3i** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish oil (1.614 g, 60%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.28 (t, J = 7.2 Hz, 3 H, CH₃), 2.28 – 2.39 (m, 2 H, CH₂), 2.63 – 2.68 (m, 1 H, CH), 3.09 – 3.22 (m, 1 H, CH₂N₃), 3.37 (s, 3 H, OCH₃), 3.38 – 3.46 (m, 1 H, CH₂N₃), 3.48 (s, 2 H, CH₂), 3.49 – 3.57 (m, 1 H, CH), 4.10 – 4.24 (m, 1 H, OCH₂), 4.98 – 5.12 (m, 2 H, CH₂=CH), 5.63 – 5.84 (m, 1 H, CH=CH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2982 (w), 2936 (w), 2102 (s), 1742 (s), 1717 (s), 1643 (w), 1444 (w), 1370 (w), 1306 (m), 1249 (s), 1180 (s), 1156 (s), 1100 (m), 1031 (m), 853 (w). MS (EI, 70 eV): m/z (%) = 224 (M⁺ - OEt, 3), 213 (M⁺ - CH₂N₃, 100), 194 (4), 170 (16), 168 (3), 141 (13), 125 (85).

4-(2-Azido-1-methoxy-ethyl)-3-oxo-heptanoic acid ethyl ester (3j): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-hepta-1,3-diene (**1j**) (3.166 g, 10 mmol), and Me₃SiOTf (1.111 g, 5 mmol), in CH₂Cl₂ (100 mL), **3j** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.227 g, 82%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.88 (t, *J* = 6.9 Hz, 3 H, CH₃), 1.28 (t, *J* = 7.2 Hz, 3 H, OCHCH₃), 1.22 – 1.35 (m, 2 H, CH₂), 1.56 – 1.62 (m, 2 H, CH₂), 3.01 (dt, *J* = 3.0, 8.4 Hz, 1 H, CH), 3.19 (dd, *J* = 13.5, 4.5 Hz, 1 H, CH₂N₃), 3.36 (s, 3 H, OCH₃), 3.43 – 3.50 (m, 1 H, CH₂N₃), 3.54 (s, 2 H, CH₂), 3.57 (dd, *J* = 13.5, 3.0 Hz, 1 H, CH), 4.20 (q, *J* = 7.2 Hz, 2 H, OCH₂). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 13.9, 14.0, 20.2, 30.2, 50.3, 51.6, 53.2, 58.0, 61.0, 82.0, 166.6, 205.3. IR (neat, cm⁻¹): $\tilde{\nu}$ = 2962 (s), 2936 (s), 2874 (w), 2101 (s), 1745 (s), 1715 (s), 1647 (m), 1463 (m), 1411 (w), 1369 (m), 1301 (s), 1275 (s), 1238 (s), 1179 (m), 1156 (m), 1100 (s), 1034 (m). MS (EI, 70 eV): *m/z* (%) = 226 (M⁺ - OEt, 4), 215 (M⁺ - CH₂N₃, 55), 184 (4), 142 (3), 127 (100).

4-(2-Azido-1-methoxy-ethyl)-3-oxo-octanoic acid ethyl ester (3k): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-octa-1,3-diene (**1k**) (3.306 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3k** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.354 g, 83%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.92 – 1.00 (m, 3 H, CH₃), 1.35 (t, *J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.27 – 1.48 (m, 4 H, 2×CH₂), 1.55 – 1.88 (m, 2 H, CH₂), 2.55 – 2.71 (m, 1 H, CH), 3.03 – 3.28 (m, 1 H, CH₂-N₃), 3.42 (s, 3 H, OCH₃), 3.49 – 3.54 (m, 1 H, CH₂-N₃), 3.55 (s, 2 H, CH₂), 3.60 – 3.62 (m, 1 H, CH), 4.27 (q, *J* = 7.2 Hz, 2 H, OCH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2959 (m), 2934 (m), 2870 (w), 2102 (s), 1745 (s), 1715 (s), 1647 (m), 1463 (w), 1449 (w), 1370 (w), 1303 (m), 1249 (s), 1176 (w), 1155 (m), 1100 (m), 1033 (w). MS (EI, 70 eV): *m/z* (%) = 240 (M⁺ - OEt, 3), 229 (M⁺ - CH₂N₃, 53), 198 (2), 156 (2), 141 (100).

4-(2-Azido-1-methoxy-ethyl)-3-oxo-decanoic acid ethyl ester (3l): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-deca-1,3-diene (**1l**) (3.587 g, 10 mmol), and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3l** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.678 g, 86%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.87 (t, *J* = 6.9 Hz, 3 H, CH₃), 1.28 (t, *J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.19 – 1.35 (m, 8 H, 4×CH₂), 1.50 – 1.68 (m, 2 H, CH₂), 2.99 (dt, *J* = 3.7, 8.7 Hz, 1 H, CH), 3.18 (dd, *J* = 13.4, 4.4 Hz, 1 H, CH₂N₃), 3.36 (s, 3 H, OCH₃), 3.44 – 3.49 (m, 1 H, CH₂N₃), 3.54 (s, 2 H, CH₂), 3.57 (dd, *J* = 13.2, 2.7 Hz, 1 H, CH), 4.20 (q, *J* = 7.2 Hz, 2 H, OCH₂). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 13.9, 14.0, 22.4, 26.9, 28.1, 29.3, 31.4, 50.4, 51.7, 53.40, 58.1, 61.1, 82.1, 166.7, 205.4. IR (neat, cm⁻¹): $\tilde{\nu}$ = 2931 (s), 2859 (m), 2100 (s), 1746 (s), 1715 (s), 1649 (m), 1630 (m), 1424 (w), 1411 (w), 1369 (m), 1350 (w), 1300 (s), 1237 (s), 1179 (w), 1154 (m), 1102 (s), 1033 (m). MS (EI, 70 eV): *m/z* (%) = 313 (M⁺, 4), 268 (2), 257 (76), 225 (6), 184 (2), 169 (100). HRMS (ESI): calcd. for C₁₅H₂₇O₄N₃Na ([M+Na]⁺) = 336.18993; found = 336.18923.

4-(2-Azido-1-methoxy-ethyl)-3-oxo-dodecanoic acid ethyl ester (3m): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-dodeca-1,3-diene (**1m**) (3.862 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3m** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish oil (2.794 g, 82%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.88 (t, *J* = 6.9 Hz, CH₃), 1.23 – 1.33 (m, 15 H, 6×CH₂, CH₃), 1.56 – 1.61 (m, 2 H, CH₂), 2.54 – 2.62 (m, 1 H, CH), 3.15 – 3.32 (m, 1 H, CH₂N₃), 3.33 – 3.35 (ds, 3 H, OCH₃), 3.43 – 3.49 (ds, 2 H, CH₂), 3.54 – 3.68 (m, 1 H, CH₂N₃), 4.01 – 4.07 (m, 1 H, CH), 4.15 – 4.25 (m, 2 H, OCH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2928 (s), 2856 (m), 2101 (s), 1745 (s), 1716 (s), 1648 (w), 1463 (m), 1371 (w), 1351 (w), 1296 (m), 1237 (m), 1177 (m),

1156 (m), 1100 (s), 1032 (m). MS (EI, 70 eV): m/z (%) = 285 ($M^+ - CH_2N_3$, 66), 213 (4), 197 (100).

4-(2-Azido-1-methoxy-ethyl)-3-oxo-tridecanoic acid ethyl ester (3n): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-trideca-1,3-diene (**1n**) (4.008 g, 10 mmol) and Me_3SiOTf (1.111 g, 5 mmol) in CH_2Cl_2 (100 mL), **3n** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish oil (2.854 g, 80%). 1H NMR $CDCl_3$, 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.19 – 1.33 (m, 19 H, $8 \times CH_2$, OCH_2CH_3), 2.97 – 3.09 (m, 1 H, CH), 3.14 – 3.21 (m, 1 H, CH_2N_3), 3.36 (s, 3 H, OCH_3), 3.43 – 3.44 (m, 1 H, CH_2N_3), 3.49 (s, 2 H, CH_2), 3.54 – 3.55 (m, 1 H, CH), 4.20 (q, J = 7.2 Hz, 2 H, OCH_2). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2927 (s), 2855 (s), 2100 (s), 1746 (s), 1715 (s), 1649 (m), 1632 (m), 1463 (m), 1423 (w), 1370 (m), 1350 (w), 1299 (s), 1235 (s), 1176 (w), 1153 (m), 1103 (s), 1034 (m), 844 (w). MS (EI, 70 eV): m/z (%) = 310 ($M^+ - OEt$, 3), 299 ($M^+ - CH_2N_3$, 82), 226 (3), 211 (100).

4-(2-Azido-1-methoxy-ethyl)-3-oxo-tetradecanoic acid ethyl ester (3o): Starting with **2** (1.967 g, 15 mmol), 1-ethoxy-1,3-bis-trimethylsilanyloxy-tetradeca-1,3-diene (**1o**) (4.148, 10 mmol), and Me_3SiOTf (1.111 g, 5 mmol), in CH_2Cl_2 (100 mL), **3o** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a yellow oil (3.405 g, 92%). 1H NMR ($CDCl_3$, 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.19 – 1.34 (m, 21 H, $9 \times CH_2$, OCH_2CH_3), 2.95 – 3.09 (m, 1 H, CH), 3.14 – 3.21 (m, 1 H, CH_2N_3), 3.36 (s, 3 H, OCH_3), 3.42 – 3.44 (m, 1 H, CH_2N_3), 3.49 (s, 2 H, CH_2), 3.53 – 3.55 (m, 1 H, CH), 4.20 (q, J = 7.2 Hz, 2 H, OCH_2). IR (neat, cm^{-1}): $\tilde{\nu}$ = 2927 (s), 2856 (s), 2101 (s), 1746 (s), 1716 (s), 1649 (w), 1631 (w), 1463 (m), 1369 (w), 1300 (m), 1235 (s), 1177 (w), 1153 (m), 1102 (m), 1034 (m). MS (EI, 70 eV): m/z (%) = 324 ($M^+ - OEt$, 3), 313 ($M^+ - CH_2N_3$, 84), 225 (100), 196 (3).

3-(2-Azido-1-methoxy-ethyl)-2-oxo-cyclododecanecarboxylic acid ethyl ester (3p):

Starting with **2** (1.967 g, 15 mmol), 12-(ethoxy-trimethylsilanyloxy-methylene)-1-trimethylsilanyloxy-cyclododecene (**1p**) (3.967 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100mL), **3p** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (2.065 g, 58%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.28 (t, *J* = 7.2 Hz, 3 H, CH₃), 1.29 – 1.35 (m, 12 H, 6×CH₂), 3.05 – 3.13 (m, 1 H, CH), 3.21 – 3.32 (m, 1 H, CH), 3.33, 3.47 (ds, 3H, OCH₃), 3.59 (dt, *J* = 13.2, 3.3 Hz, 1 H, CH₂N₃), 3.64 – 3.73 (m, 1 H, CH₂N₃), 3.95 – 4.03 (m, 1 H, CH), 4.17 (q, *J* = 7.2 Hz, 2 H, OCH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2933 (s), 2867 (w), 2100 (s), 1743 (s), 1711 (s), 1469 (w), 1443 (w), 1291 (m), 1271 (m), 1250 (m), 1179 (m), 1154 (w), 1114 (m), 1099 (m), 1027 (m). MS (EI, 70 eV): *m/z* (%) = 298 (M⁺ - CH₂N₃, 100), 266 (5), 252 (95), 224 (9). Anal. calcd. for C₁₈H₃₁O₄N₃ (353.462): C 61.17, H 8.84, N 11.89; found C 61.33, H 8.96, N 11.64.

3-(2-Azido-1-methoxy-ethyl)-2-oxo-cyclohexanecarboxylic acid ethyl ester (3q):

Starting with **2** (1.705 g, 13 mmol), 6-(ethoxy-trimethylsilanyloxy-methylene)-1-trimethylsilanyloxy-cyclohexene (**1q**) (3.146 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3q** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow oil (1.280 g, 48%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.30 (dt, *J* = 7.5, 7.5 Hz, 3 H, CH₃), 1.40 – 1.54 (m, 1 H, CH₂), 1.70 – 1.91 (m, 2 H, CH₂), 1.98 – 2.08 (m, 1 H, CH₂), 2.11 – 2.25 (m, 1 H, CH₂), 2.28 – 2.51 (m, 1 H, CH₂), 2.58 – 2.88 (m, 1 H, CH), 3.12 – 3.28 (m, 1 H, CH₂N₃), 3.33 – 3.42 (m, ½ H, CH), 3.93 – 4.03 (m, ½ H, CH), 4.21 (q, *J* = 7.4 Hz, 2 H, OCH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2979 (s), 2939 (s), 2870 (m), 2834 (w), 2100 (s), 1743 (s), 1713 (s), 1648 (m), 1616 (m), 1449 (m), 1400 (m), 1377 (m), 1355 (w), 1297 (s), 1258 (s), 1225 (s), 1178 (m), 1115 (w), 1096 (s), 1024 (m), 841 (w). MS (EI, 70 eV): *m/z* (%) = 269 (M⁺, 3), 237 (9), 224 (59), 213 (74), 182 (11), 181 (9), 167 (100), 139 (19).

3-(2-Azido-1-methoxy-ethyl)-2-oxo-cyclooctanecarboxylic acid ethyl ester (3r): Starting with **2** (0.984 g, 7.5 mmol), 8-(ethoxy-trimethylsilanyloxy-methylene)-1-trimethylsilanyloxy-cyclooctene (**1r**) (1.713 g, 5.0 mmol) and Me₃SiOTf (0.556 g, 2.5 mmol) in CH₂Cl₂ (50 mL), **3r** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish oil (0.960 g, 65%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.19 – 1.29 (m, 3 H, CH₃), 1.30 – 1.81 (m, 6 H, 3×CH₂), 1.93 – 2.12 (m, 1 H, CH₂), 2.25 – 2.34 (m, 2 H, CH₂), 2.41 – 2.51 (m, 1 H, CH₂), 2.55 – 2.61 (m, 1 H, CH), 3.22 – 3.49 (m, 2 H, CH₂N₃), 3.58 (s, 3 H, OCH₃), 3.64 – 3.69 (dd, 1 H, CH), 3.87 (dd, 1 H, CH), 4.12 – 4.23 (m, 2 H, OCH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2933 (s), 2860 (m), 2099 (s), 1743 (s), 1707 (s), 1642 (m), 1618 (w), 1463 (m), 1447 (m), 1371 (m), 1290 (s), 1184 (s), 1098 (s), 1036 (m), 853 (m). MS (EI, 70 eV): *m/z* (%) = 297 (M⁺, 1), 241 (M⁺ - CH₂N₃, 100), 209 (2), 195 (24), 181 (5), 168 (3), 137 (2).

3-(2-Azido-1-methoxy-ethyl)-4-methyl-2-oxo-cyclohexanecarboxylic acid ethyl ester (3s): Starting with **2** (0.984 g, 7.5 mmol), 6-(ethoxy-trimethylsilanyloxy-methylene)-3-methyl-1-trimethylsilanyloxy-cyclohexene (**1s**) (1.643 g, 5.0 mmol) and Me₃SiOTf (0.556 g, 2.5 mmol) in CH₂Cl₂ (50 mL), **3s** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1), as a brownish oil (1.359 g, 96%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.00 – 1.07 (dd, 3 H, CH₃), 1.25 – 1.34 (m, 3 H, OCH₂CH₃), 1.61 – 1.98 (m, 2 H, CH₂), 1.99 – 2.27 (m, 2 H, CH₂), 2.29 – 2.39 (m, 1 H, CH), 3.42 – 3.47 (m, 2 H, CH₂N₃), 3.48 (s, 3 H, OCH₃), 3.49 – 3.58 (m, 1 H, CH), 3.65 – 3.70 (m, 1 H, CH), 3.82 – 3.87 (m, 1 H, CH), 4.17 – 4.26 (m, 2 H, OCH₂). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2956 (s), 2933 (s), 2874 (m), 2835 (w), 2100 (s), 1742 (s), 1714 (s), 1651 (s), 1616 (s), 1460 (m), 1401 (m), 1376 (s), 1351 (m), 1306 (s), 1276 (s), 1233 (s), 1212 (s), 1186 (s), 1158 (m), 1098 (s), 1029 (m), 834 (w). MS (EI, 70 eV): *m/z* (%) = 283 (M⁺, 4), 251 (7), 227 (M⁺ - CH₂N₃, 98), 2123 (14), 184 (100), 181 (97), 155 (20).

3-(2-Azido-1-methoxy-ethyl)-2-oxo-5-phenyl-cyclohexanecarboxylic acid methyl ester

(3t): Starting with **2** (1.967 g, 12 mmol), [5-(methoxy-trimethylsilanyloxy-methylene)-4-trimethylsilanyloxy-cyclohex-3-enyl]-benzene (**1t**) (3.777 g, 10 mmol) and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3t** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 10:1) as a brownish oil (2.366 g, 71%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.71 – 1.81 (m, 1 H, CH₂), 1.85 – 1.88 (m, 1 H, CH₂), 2.11 – 2.21 (m, 1 H, CH₂), 2.29 – 2.34 (m, 1 H, CH₂), 2.51 (dd, *J* = 15.6, 5.1 Hz, 1 H, CH), 2.62 – 2.72 (m, 1 H, CH), 3.14 – 3.27 (m, 1 H, CH), 3.30 (s, 3 H, OCH₃), 3.31 – 3.62 (m, 2 H, CH₂-N₃), 3.64 (s, 3 H, OCH₃), 3.65 – 3.69 (m, 1 H, CH), 7.09 – 7.22 (m, 5 H, 5×CH from Ph). IR (neat, cm⁻¹): $\tilde{\nu}$ = 2951 (m), 2102 (s), 1743 (m), 1718 (m), 1658 (s), 1495 (w), 1443 (s), 1361 (m), 1260 (s), 1228 (s), 1203 (m), 1159 (w), 1128 (w), 1091 (m), 1032 (w), 1012 (w), 845 (s), 761 (w), 701 (m). HRMS (ESI): calcd. for C₁₇H₂₁O₄N₃Na ([M+Na]⁺) = 354.14298; found = 354.14226.

7-Azido-6-methoxy-heptane-2,4-dione (3u)

(3u): Starting with **2** (1.705 g, 13 mmol), 2,4-bis-trimethylsilanyloxy-penta-1,3-diene (**1u**) (2.445 g, 10 mmol), and Me₃SiOTf (1.111 g, 5 mmol) in CH₂Cl₂ (100 mL), **3u** and **8** were isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as brownish oils (0.536 g, 27%; 0.582 g, 24%). ¹H NMR (CDCl₃, 300 MHz): δ = 2.07 (s, 3 H, CH₃), 2.46 (dd, *J* = 14.7, 6.0 Hz, 1 H, CH₂), 2.59 (dd, *J* = 14.7, 6.6 Hz, 1 H, CH₂), 3.28 (dd, *J* = 12.9, 5.4 Hz, 1 H, CH₂), 3.42 (s, 3 H, OCH₃), 3.43 (dd, *J* = 12.9, 3.9 Hz, 1 H, CH₂-N₃), 3.78 – 3.86 (m, 1 H, CH), 5.55 (s, 1 H, CH=C), 15.39 (broad s, 1 H, OH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 24.5, 40.6, 53.0, 57.5, 77.0, 101.0, 190.6, 191.0. IR (neat, cm⁻¹): $\tilde{\nu}$ = 2935 (m), 2102 (s), 1724 (w), 1708 (m), 1685 (w), 1618 (s), 1613 (s), 1562 (w), 1536 (w), 1458 (m), 1439 (m), 1426 (m), 1361 (m), 1293 (s), 1254 (m), 1195 (w), 1181 (w), 1152 (w), 1109 (m), 1036 (w). MS (EI, 70 eV): *m/z* (%) = 184 (M⁺ - Me, 98), 143 (M⁺ - CH₂N₃, 80), 128 (92), 99 (100).

1,9-Diazido-2,8-dimethoxy-nonane-4,6-dione (8): ^1H NMR (CDCl_3 , 300 MHz): δ = 2.49 (ddd, J = 15, 6, 0.9 Hz, 2 H, CH_2), 2.61 (dd, J = 15, 6 Hz, 2 H, CH_2), 3.28 (dd, J = 12.9, 5.7 Hz, 2 H, $\text{CH}_2\text{-N}_3$), 3.43 (s, 6 H, $2\times\text{OCH}_3$), 3.41 – 3.50 (m, 2 H, $2\times\text{CH}$), 5.60 (s, 1 H, CH), 15.29 (broad s, 1 H, OH). ^{13}C NMR (CDCl_3 , 75 MHz): δ = 40.7, 53.0, 57.6, 77.0, 101.8, 190.5. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3333 (br), 2935 (s), 2832 (w), 2101 (s), 1711 (s), 1668 (s), 1613 (s), 1442 (s), 1355 (s), 1291 (s), 1254 (s), 1195 (m), 1153 (m), 1039 (m), 931 (w), 844 (m). MS (EI, 70 eV): m/z (%) = 298 (M^+ , 100), 256 (10), 214 (22), 198 (80), 183 (26).

2-Acetyl-4-azido-3-methoxy-butyric acid methyl ester (11a): Starting with **2** (1.967 g, 15 mmol), 3-trimethylsilanyloxy-but-2-enoic acid methyl ester (**10a**) (1.883 g, 10 mmol), and Me_3SiOTf (1.111 g, 5 mmol) in CH_2Cl_2 (100 mL), **11a** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 100:1 $\rightarrow$ 1:1) as a yellow oil (1.081 g, 50%). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.27, 2.33 (ds, 3 H, CH_3), 3.23 – 3.40 (m, 1 H, $\text{CH}_2\text{-N}_3$), 3.44, 3.47 (ds, 3 H, OCH_3), 3.63 (dt, J = 13.2, 3.0 Hz, 1 H, $\text{CH}_2\text{-N}_3$), 3.73, 3.78 (ds, 3 H, OCH_3), 3.92 (dd, J = 9.6, 3.0 Hz, 1 H, CH), 3.99 – 4.03 (m, 1 H, CH). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 3.6, 31.7, 51.1, 51.1, 52.8, 52.9, 58.5, 58.6, 60.2, 61.7, 78.5, 79.1, 167.6, 167.9, 201.0, 201.5. IR (neat, cm^{-1}): $\tilde{\nu}$ = 2998 (w), 2956 (w), 2938 (w), 2105 (s), 1746 (s), 1720 (s), 1648 (w), 1438 (m), 1360 (m), 1260 (s), 1212 (m), 1197 (m), 1173 (m), 1150 (m), 1117 (m), 1096 (s), 1050 (w), 994 (w). MS (EI, 70 eV): m/z (%) = 139 ($\text{M}^+\text{-OMe}$, 14), 159 ($\text{M}^+\text{-CH}_2\text{N}_3$, 3), 155 (1), 146 (3), 117 (79), 101 (6), 85 (85), 55 (9), 43 (100).

2-Acetyl-4-azido-3-methoxy-butyric acid ethyl ester (11b): Starting with **2** (1.967 g, 15 mmol) 3-trimethylsilanyloxy-but-2-enoic acid ethyl ester (**10b**) (2.023 g, 10 mmol), and Me_3SiOTf (1.111 g, 5 mmol) in CH_2Cl_2 (100 mL) **11b** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 100:1 $\rightarrow$ 1:1) as a yellow oil (1.144 g, 50%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.29 (m, 3 H, CH_3), 2.27, 2.33 (ds, 3 H, CH_3), 3.25 – 3.37 (m, 1 H, $\text{CH}_2\text{-N}_3$),

3.41, 3.44 (ds, 3 H, OCH₃), 3.63 (m, 1 H, CH₂-N₃), 3.89 (dd, $J = 8.7, 2.4$ Hz, 1 H, CH), 3.99 – 4.04 (m, 1 H, CH), 4.17 – 4.26 (m, 2 H, OCH₂). ¹³C NMR (CDCl₃, 150 MHz): $\delta_c = 13.9, 14.0, 30.1, 31.2, 50.0, 50.9, 58.2, 58.3, 60.3, 61.7, 61.6, 61.7, 78.8, 166.8, 167.1, 200.8, 201.2$. IR (neat, cm⁻¹): $\tilde{\nu} = 2986$ (w), 2939 (w), 2104 (s), 1742 (s), 1718 (s), 1642 (w), 1447 (w), 1366 (m), 1295 (s), 1256 (s), 1212 (m), 1179 (s), 1151 (m), 1115 (m), 1097 (s), 1044 (w), 1025 (w). MS (EI, 70 eV): m/z (%) = 198 (M⁺-OMe, 1), 173 (M⁺-CH₂N₃, 2), 141 (1), 131 (84), 128 (3), 113 (2), 103 (26), 99 (3), 85 (100), 82 (2), 55 (6), 43 (68).

2-Acetyl-4-azido-3-methoxy-butyric acid 2-methoxy-ethyl ester (11c): Starting with **2** (0.171 g, 1.3 mmol), 3-trimethylsilanyloxy-but-2-enoic acid 2-methoxy-ethyl ester (**10c**) (0.236 g, 1.0 mmol), and Me₃SiOTf (0.111 g, 0.5 mmol) in CH₂Cl₂ (20 mL), crude **11c** was isolated as a brownish oil and used for the next step without further purification.

3-(2-Azido-1-methoxy-ethyl)-pentane-2,4-dione (11d): Starting with **2** (0.786 g, 6.0 mmol), 4-trimethylsilanyloxy-pent-3-en-2-one (**10d**) (0.862 g, 5 mmol) and Me₃SiOTf (0.556 g, 2.5 mmol) in CH₂Cl₂ (100 mL), **11d** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 50:1 → 1:1) as a yellow oil (0.540 g, 59%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 2.21$ (s, 3 H, CH₃), 2.29 (s, 3 H, CH₃), 3.17 (dd, $J = 13.2, 3.6$ Hz, 1 H, CH₂-N₃), 3.39 (s, 3 H, OCH₃), 3.63 (dd, $J = 13.2, 3.0$ Hz, 1 H, CH₂-N₃), 4.06 (dt, $J = 9.3, 3.6$ Hz, 1 H, CH), 4.15 (d, $J = 9.3$ Hz, 1 H, CH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 30.6, 30.7, 50.4, 57.8, 69.2, 79.0, 201.2, 201.7$. IR (neat, cm⁻¹): $\tilde{\nu} = 2993$ (w), 2938 (m), 2833 (w), 2104 (s), 1725 (s), 1703 (s), 1558 (m), 1423 (m), 1360 (s), 1276 (s), 1187 (m), 1153 (m), 1110 (s), 1197 (s), 1048 (w), 1024 (w), 956 (w), 930 (w), 579 (w). MS (EI, 70 eV): m/z (%) = 199 (M⁺, 2), 157 (2), 143 (9), 130 (1), 125 (3), 101 (58), 83 (15), 70 (5), 43 (100). Anal. calcd. for C₈H₁₃O₃N₃ (199.210): C 48.24, H 6.58, N 21.09; found C 48.70, H 6.17, N 21.30.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-hept-6-enoic acid ethyl ester (11e): Starting with **2** (0.197 g, 1.5 mmol), 3-trimethylsilanyloxy-hepta-2,6-dienoic acid ethyl ester (**10e**) (0.242 g, 1.0 mmol), and Me₃SiOTf (0.111 g, 0.5 mmol) in CH₂Cl₂ (20 mL), crude **11e** was isolated as a brownish oil and used for the next step without further purification.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-hexanoic acid ethyl ester (11f): Starting with **2** (0.184 g, 1.4 mmol), 3-trimethylsilanyloxy-hex-2-enoic acid ethyl ester (**10f**) (0.230 g, 1.0 mmol), and Me₃SiOTf (0.111 g, 0.5 mmol) in CH₂Cl₂ (20 mL), crude **11f** was isolated as a brownish oil and used for the next step without further purification.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-heptanoic acid ethyl ester (11g): Starting with **2** (0.184 g, 1.4 mmol), 3-trimethylsilanyloxy-hept-2-enoic acid ethyl ester (**10g**) (0.244 g, 1.0 mmol), and Me₃SiOTf (0.111 g, 0.5 mmol) in CH₂Cl₂ (20 mL), crude **11g** was isolated as a brownish oil and used for the next step without further purification.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-octanoic acid ethyl ester (11h): Starting with **2** (0.184 g, 1.4 mmol), 3-trimethylsilanyloxy-oct-2-enoic acid ethyl ester (**10h**) (0.258 g, 1.0 mmol), and Me₃SiOTf (0.111 g, 0.5 mmol) in CH₂Cl₂ (20 mL), crude **11h** was isolated as a brownish oil and used for the next step without further purification.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-decanoic acid ethyl ester (11i): Starting with **2** (0.984 g, 7.5 mmol), 3-trimethylsilanyloxy-dec-2-enoic acid ethyl ester (**10i**) (1.432 g, 5.0 mmol), and Me₃SiOTf (0.556 g, 2.5 mmol) in CH₂Cl₂ (100 mL), **11i** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 5:1) as a yellow oil (2.058 g, 92%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.88 (t, *J* = 6.9 Hz, 3 H, CH₃), 1.23 – 1.33 (m, 11 H, 4×CH₂, OCH₂CH₃), 1.54 – 1.62 (m, 2 H, CH₂), 2.53 (t, *J* = 7.2 Hz, 2 H, CH₂), 3.15 – 3.37 (m, 1 H,

CH₂-N₃), 3.38, 3.43 (ds, 3 H, OCH₃), 3.46 – 3.70 (m, 2 H, CH, CH₂-N₃), 4.20 (q, $J = 7.2$ Hz, 2 H, OCH₂), 4.15 – 4.23 (m, 1 H, CH). IR (neat, cm⁻¹): $\tilde{\nu} = 2957$ (s), 2930 (s), 2858 (s), 2120 (s), 1745 (s), 1717 (s), 1647 (w), 1463 (m), 1410 (w), 1371(m), 1300 (s), 1234 (s), 1178 (s), 1158 (s), 1158 (m), 1126 (m), 1100 (s), 1033 (m). MS (EI, 70 eV): m/z (%) = 257 (M⁺-N₃, 7), 196 (4), 185 (2), 169 (12), 143 (22), 130 (90), 127 (57), 114 (18), 102 (11), 85 (30), 70 (16), 57 (100), 43 (74). HRMS (ESI): calcd. for C₁₅H₂₇O₄N₃Na ([M+Na]⁺) = 336.18993; found = 336.18912.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-dodecanoic acid ethyl ester (11j): Starting with **2** (0.184 g, 1.4 mmol), 3-trimethylsilanyloxy-dodec-2-enoic acid ethyl ester (**10j**) (0.315 g, 1.0 mmol), and Me₃SiOTf (0.111 g, 0.5 mmol) in CH₂Cl₂ (20 mL), crude **11j** was isolated as a brownish oil and used for the next step without further purification.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-tridecanoic acid ethyl ester (11k): Starting with **2** (0.092 g, 0.7 mmol), 3-trimethylsilanyloxy-tridec-2-enoic acid ethyl ester (**10k**) (0.164 g, 0.5 mmol), and Me₃SiOTf (0.056 g, 0.25 mmol) in CH₂Cl₂ (10 mL), crude **11k** was isolated as a brownish oil and used for the next step without further purification.

2-(2-Azido-1-methoxy-ethyl)-3-oxo-tetradecanoic acid ethyl ester (11l): Starting with **2** (0.092 g, 0.7 mmol), 3-trimethylsilanyloxy-tetradec-2-enoic acid ethyl ester (**10l**) (0.171 g, 0.5 mmol), and Me₃SiOTf (0.056 g, 0.25 mmol) in CH₂Cl₂ (10 mL), crude **11l** was isolated as a brownish oil and used for the next step without further purification.

General procedure for the preparation of 2-alkylidene-4-methoxypyrrolidines (4): To a THF-solution (10 mL/mmol) of **3** (1 equiv.) was added triphenylphosphine (1.2 equiv.) at room temperature. The reaction mixture was heated and stirred for 4 h at 80 °C. After cooling

to 20 °C, the solvent was removed *in vacuo* and the residue was purified by chromatography (silica gel, *n*-hexane/EtOAc) to give **4**.

(4-Methoxy-pyrrolidin-2-ylidene)-acetic acid methyl ester (4a): Starting with **3a** (0.400g, 1.86 mmol) and PPh₃ (0.585 g, 2.23 mmol) in THF (10 mL), **4a** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a yellow solid (0.257 g, 81%). M.p. = 42.6 °C. ¹H NMR (CDCl₃, 300 MHz): δ = 2.64 (dd, *J* = 17.1, 2.7 Hz, 1 H, CH₂), 2.77 (dd, *J* = 17.1, 6.3 Hz, 1 H, CH₂), 3.32 (s, 3 H, OCH₃), 3.56 (d, *J* = 10.5 Hz, 1 H, CH₂NH), 3.62 (s, 3 H, OCH₃), 3.60 – 3.68 (m, 1 H, CH₂NH), 4.05 (quint, *J* = 1.8 Hz, 1 H, CH), 4.55 (s, 1 H, CH=C), 7.82 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 38.2, 49.9, 52.1, 56.3, 76.9, 77.1, 163.8, 170.5. IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3367 (m), 2982 (w), 2940 (m), 2881 (w), 1663 (s), 1603 (s), 1489 (m), 1458 (w), 1433 (w), 1340 (m), 1293 (m), 1225 (s), 1188 (m), 1152 (s), 1097 (s), 1045 (s), 904 (w), 782 (m). MS (EI, 70 eV): *m/z* (%) = 171 (M⁺, 87), 156 (4), 141 (15), 140 (100), 124 (17), 112 (28), 109 (22), 80 (41). Anal. calcd. for C₈H₁₃O₃N (171.196): C 56.13, H 7.65, N 8.18; found C 55.76, H 7.30, N 8.01.

(4-Methoxy-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4b): Starting with **3b** (0.850 g, 3.71 mmol) and PPh₃ (1.167 g, 4.45 mmol) in THF (30 mL), **4b** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 3:1) as a yellow solid (0.439 g, 64%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.25 (t, *J* = 7.2 Hz, 3 H, CH₃), 2.67 (dd, *J* = 17.1, 3.3 Hz, 1 H, CH₂), 2.77 (dd, *J* = 17.1, 6.3 Hz, 1 H, CH₂), 3.33 (s, 3 H, OCH₃), 3.55 (dd, *J* = 11.1, 2.4 Hz, 1 H, CH₂NH), 3.66 (dd, *J* = 11.1, 5.4 Hz, 1 H, CH₂NH), 4.04 – 4.09 (m, 1 H, CH), 4.10 (q, *J* = 7.2 Hz, 2 H, OCH₂), 4.55 (s, 1 H, CH=C), 8.81 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 14.5, 38.2, 52.1, 56.3, 58.3, 77.0, 77.7, 163.7, 170.3. IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3378 (s), 2976 (m), 2945 (w), 2925 (w), 2894 (w), 1663 (s), 1616 (s), 1493 (m), 1461 (w), 1447 (w), 1367 (m), 1340 (m), 1279 (m), 1251 (s), 1232 (s), 1190 (w), 1162 (s), 1144 (s), 1096 (s),

1079 (m), 1056 (s), 982 (w), 772 (m), 714 (w), 647 (w). MS (EI, 70 eV): m/z (%) = 185 (M^+ , 70), 154 (17), 140 (69), 124 (17), 112 (100). Anal. calcd. for $C_9H_{15}O_3N$ (185.223): C 58.36, H 8.16, N 7.56; found C 58.74, H 8.23, N 7.90.

(4-Methoxy-pyrrolidin-2-ylidene)-acetic acid isopropyl ester (4c): Starting with **3c** (0.490 g, 2.0 mmol) and PPh_3 (0.792 g, 3.0 mmol) in THF (20 mL), **4c** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a yellow solid (0.295 g, 74%). 1H NMR ($CDCl_3$, 300 MHz): δ = 1.22 (d, J = 7.8 Hz, 6 H, $2\times CH_3$), 2.63 (dd, J = 16.8, 3.0 Hz, 1 H, CH_2), 2.76 (dd, J = 16.8, 6.9 Hz, 1 H, CH_2), 3.32 (s, 3 H, OCH_3), 3.53 (dd, J = 11.1, 3.3 Hz, 1 H, CH_2NH), 3.66 (dd, J = 10.8, 5.1 Hz, 1 H, CH_2NH), 4.05 (m, 1 H, CH), 4.53 (s, 1 H, $CH=C$), 4.99 (septet, J = 6.3 Hz, 1 H, CH), 7.82 (broad s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 22.0, 38.2, 52.0, 56.3, 65.0, 77.0, 78.2, 163.4, 169.9. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3380 (m), 2977 (w), 1655 (s), 1610 (s), 1491 (m), 1370 (w), 1360 (w), 1295 (w), 1256 (m), 1230 (s), 1178 (w), 1163 (m), 1148 (m), 1102 (s), 1043 (w), 1004 (m), 778 (m). MS (EI, 70 eV): m/z (%) = 199 (M^+ , 45), 168 (11), 157 (16), 140 (54), 125 (9), 112 (100), 109 (6). Anal. calcd. for $C_{10}H_{17}O_3N$ (199.250): C 60.28, H 8.60, N 7.03; found C 60.35, H 8.16, N 7.08.

(4-Methoxy-pyrrolidin-2-ylidene)-acetic acid 2-methoxy-ethyl ester (4d): Starting with **3d** (0.300 g, 1.16 mmol) and PPh_3 (0.364 g, 1.39 mmol) in THF (30 mL), **4d** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a yellow solid (0.184 g, 74%). 1H NMR ($CDCl_3$, 300 MHz): δ = 2.64 (dd, J = 16.8, 2.7 Hz, 1 H, CH_2), 2.77 (dd, J = 17.1, 6.0 Hz, 1 H, CH_2), 3.32 (s, 3 H, OCH_3), 3.38 (s, 3 H, OCH_3), 3.53 – 3.60 (m, 2 H from OCH_2 and 1 H from CH_2NH), 3.66 (dd, J = 1.1, 5.4 Hz, 1 H, CH_2NH), 4.06 (quint, J = 3.0 Hz, 1 H, CH), 4.20 (t, J = 4.5 Hz, 2 H, OCH_2), 4.61 (s, 1 H, $CH=C$), 7.80 (broad s, 1 H, NH). ^{13}C ($CDCl_3$, 75 MHz): δ_c = 38.4, 52.3, 56.6, 59.0, 61.7, 71.1, 77.2, 77.7, 164.2, 170.2. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3384 (m), 2983 (w), 2970 (w), 2939 (w), 2921 (w), 2895 (m), 2846 (w), 2818 (w), 1658 (s),

1611 (s), 1491 (m), 1454 (w), 1371 (w), 1342 (w), 1295 (m), 1251 (s), 1235 (s), 1221 (s), 1201 (w), 1188 (w), 1164 (m), 1145 (m), 1129 (s), 1096 (s), 1078 (m), 1057 (s), 1044 (m), 774 (m), 709 (w), 655 (w). MS (EI, 70 eV): m/z (%) = 215 (M^+ , 29), 184 (21), 168 (4), 153 (20), 140 (100), 126 (6), 112 (60). The exact molecular mass $m/z = 215.1158 \pm 2$ ppm [M^+] for $C_{10}H_{17}O_4N$ was confirmed by HRMS (EI, 70 eV).

2-(4-Methoxy-pyrrolidin-2-ylidene)-1-phenyl-ethanone (4e): Starting with **3e** (2.000 g, 7.66 mmol) and PPh_3 (2.409 g, 9.19 mmol) in THF (70 mL), **4e** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a yellow solid (1.392 g, 84 %). 1H NMR ($CDCl_3$, 300 MHz): δ = 2.80 (dd, J = 17.3, 28 Hz, 1 H, CH_2), 2.92 (dd, J = 17.5, 6.3 Hz, 1 H, CH_2), 3.35 (s, 3 H, OCH_3), 3.69 (dd, J = 11.8, 2.2 Hz, 1 H, CH_2NH), 3.79 (dd, J = 11.9, 5.3 Hz, 1 H, CH_2NH), 4.13 (quint, J = 2.8 Hz, 1 H, CH), 5.82 (s, 1 H, $CH=C$), 7.36 – 7.43 (m, 3 H, 3 $\times$ CH from Ph), 7.85 – 7.88 (m, 2 H, 2 $\times$ CH from Ph), 10.17 (broad s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 39.0, 52.8, 56.4, 76.5, 87.0, 126.9, 128.0, 130.4, 140.1, 166.5, 188.2. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3270 (w), 2994 (w), 2937 (m), 2880 (w), 2832 (w), 1609 (s), 1580 (s), 1523 (s), 1485 (s), 1457 (m), 1442 (w), 1410 (m), 1362 (m), 1342 (m), 1305 (s), 1288 (m), 1265 (s), 1240 (m), 1199 (w), 1178 (m), 1162 (m), 1092 (s), 1063 (w), 1039 (m), 1026 (w), 995 (w), 943 (w), 851 (m), 749 (s), 716 (s), 679 (m), 548 (w, Ph). MS (EI, 70 eV): m/z (%) = 217 (M^+ , 100), 202 (1), 186 (68), 140 (25), 112 (1), 80 (37), 77 (59). Anal. calcd. for $C_{13}H_{15}O_2N$ (217.267): C 71.87, H 6.96, N 6.45; found C 72.01, H 7.41, N 6.44.

(4-Methoxy-3-methyl-pyrrolidin-2-ylidene)-acetic acid methyl ester (4f): Starting with **3f** (2.000 g, 8.73 mmol) and PPh_3 (3.433 g, 13.1 mmol) in THF (70 mL), **4f** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish oil (1.532 g, 95%). 1H NMR ($CDCl_3$, 300 MHz): δ = 1.19 (d, J = 7.5 Hz, 3 H, CH_3), 2.72 – 2.81 (m, 1 H, CH), 3.36 (s, 3 H, OCH_3), 3.43 (dd, J = 7.5, 3.6 Hz, 1 H, CH), 3.64 (s, 3 H, OCH_3), 3.66 – 3.76 (m,

2 H, CH₂NH), 4.53 (s, 1 H, CH=C), 7.72 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 16.6, 44.5, 50.1, 50.2, 57.1, 76.8, 84.3, 168.6, 171.1. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3373 (m), 2969 (w), 2954 (m), 2936 (m), 2903 (w), 2876 (w), 1737 (m), 1664 (s), 1604 (s), 1491 (w), 1458 (m), 1432 (m), 1314 (w), 1296 (w), 1257 (m), 1230 (s), 1203 (m), 1152 (s), 1106 (m), 1043 (m), 1018 (w), 784 (w). MS (EI, 70 eV): m/z (%) = 185 (M⁺, 100), 170 (1), 154 (92), 138 (12), 127 (17), 122(55). Anal. calcd. for C₉H₁₅O₃N (185.223): C 58.36, H 8.16, N 7.56; found C 58.87, H 7.56, N 7.80.

(3-Ethyl-4-methoxy-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4g): Starting with **3g** (1.000 g, 3.9 mmol) and PPh₃ (1.223 g, 4.7 mmol) in THF (50 mL), **4g** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish oil (0.660 g, 79%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.99 (t, *J* = 4.2 Hz, 3 H, CH₃), 1.25 (t, *J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.39 – 1.53 (m, 1 H, CH₂), 1.58 – 1.66 (m, 1 H, CH₂), 2.63 – 2.66 (m, 1 H, CH), 3.33 (s, 3 H, OCH₃), 3.49 (dt, *J* = 10.8, 1.5 Hz, 1 H, CH), 3.67 (dd, *J* = 10.8, 5.4 Hz, 1 H, CH₂NH), 3.72 – 3.75 (m, 1 H, CH₂NH), 4.10 (dq, *J* = 1.5, 7.2 Hz, 2 H, OCH₂), 4.53 (s, 1 H, CH=C), 7.79 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 11.0, 14.4, 24.3, 50.8, 51.2, 56.1, 58.1, 77.4, 81.9, 167.4, 170.5. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3368 (m), 2971 (s), 2934 (s), 2901 (m), 2877 (m), 2827 (w), 1729 (w), 1659 (s), 1604 (s), 1491 (m), 1462 (m), 1368 (m), 1345 (w), 1311 (m), 1225 (s), 1246 (s), 1199 (s), 1149 (s), 1094 (s), 1050 (s), 783 (s). UV-VIS (CH₂Cl₂, nm): λ_{max} (log ε) = 280 (4.16). MS (EI, 70 eV): m/z (%) = 213 (M⁺, 25), 185 (18), 182 (4), 154 (100), 136 (15), 107 (31). Anal. calcd. for C₁₁H₁₉O₃N (213.276): C 61.95, H 8.98, N 6.57; found C 62.30, H 8.32, N 5.98.

(3,4-Dimethoxy-pyrrolidin-2-ylidene)-acetic acid methyl ester (4h): Starting with **3h** (1.400 g, 5.71 mmol) and PPh₃ (1.797 g, 6.85 mmol) in THF (50 mL), *trans*-**4h** and *cis*-**4h**

were isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as brownish solids (0.760 g, 66%; 0.234 g, 20% = 86 %).

trans-4h: ^1H NMR (CDCl_3 , 300 MHz): δ = 3.37 (ddd, J = 10.5, 3.9, 0.9 Hz, 1 H, CH_2NH), 3.41 (s, 3 H, OCH_3), 3.49 (s, 3 H, OCH_3), 3.65 (s, 3 H, OCH_3), 3.75 (ddd, J = 10.5, 6.0, 0.9 Hz, 1 H, CH_2NH), 3.94 (dq, J = 0.6, 3.9 Hz, 1 H, CH), 4.17 (d, J = 3.9 Hz, 1 H, CH), 4.74 (s, 1 H, $\text{CH}=\text{C}$), 7.55 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 49.1, 49.8, 56.9, 78.8, 81.7, 85.0, 161.3, 170.3. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3373 (s), 2984 (w), 2945 (m), 2904 (w), 2875 (w), 2836 (w), 1736 (w), 1661 (s), 1628 (s), 1494 (m), 1461 (m), 1449 (w), 1432 (m), 1374 (m), 1358 (m), 1311 (m), 1283 (m), 1257 (m), 1224 (s), 1192 (m), 1117 (s), 1046 (s), 1007 (w), 985 (w), 784 (m). MS (EI, 70 eV): m/z (%) = 201 (M^+ , 26), 186 (4), 170 (25), 154 (9), 140 (100), 110 (9), 80 (23). Anal. calcd. for $\text{C}_9\text{H}_{15}\text{O}_4\text{N}$ (201.222): C 53.72, H 7.51, N 6.96; found C 53.45, H 7.10, N 6.57.

cis-4h: M.p. = 95 °C. ^1H NMR (CDCl_3 , 300 MHz): δ = 3.38 (s, 3 H, OCH_3), 3.44 (s, 3 H, OCH_3), 3.55 (s, 1 H, CH_2NH), 3.58 (d, J = 1.8 Hz, 1 H, CH_2NH), 4.00 (q, J = 4.7 Hz, 1 H, CH), 4.12 (d, J = 4.9 Hz, 1 H, CH), 4.76 (s, 1 H, $\text{CH}=\text{C}$), 7.49 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 48.0, 49.9, 57.7, 77.2, 78.7, 81.1, 161.2, 170.3. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3360 (s), 2981 (w), 2941 (m), 2905 (w), 2830 (w), 1668 (s), 1618 (s), 1489 (m), 1463 (w), 1429 (w), 1364 (m), 1330 (w), 1289 (m), 1251 (s), 1229 (s), 1207 (s), 1184 (m), 1152 (s), 1138 (s), 1111 (m), 1076 (w), 1043 (s), 1018 (m), 777 (m). UV-VIS (CH_2Cl_2 , nm): λ_{max} (log ϵ) = 280 (4.23). MS (EI, 70 eV): m/z (%) = 201 (M^+ , 32), 186 (4), 170 (21), 154 (10), 140 (100), 109 (17), 80 (36). Anal. calcd. for $\text{C}_9\text{H}_{15}\text{O}_4\text{N}$ (201.222): C 53.72, H 7.51, N 6.96; found C 53.94, H 7.54, N 7.01.

(3-Allyl-4-methoxy-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4i): Starting with **3i** (0.300 g, 1.11 mmol) and PPh_3 (0.351 g, 1.34 mmol) in THF (15 mL), **4i** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 100:1 → 1:1) as a brownish oil (0.236 g, 94%).

^1H NMR (CDCl_3 , 300 MHz): δ = 1.26 (t, J = 7.2 Hz, 3 H, CH_3), 2.18 – 2.42 (m, 1 H, CH), 3.63 – 3.76 (m, 2 H, CH_2NH), 4.11 (dq, J = 1.5, 7.2 Hz, 2 H, OCH_2), 4.56 (s, 1 H, $\text{CH}=\text{C}$), 5.09 – 5.15 (m, 2 H, $\text{CH}_2=\text{CH}$), 5.76 – 5.88 (m, 1 H, $\text{CH}=\text{CH}_2$), 7.80 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 150 MHz): δ_{c} = 14.8, 35.9, 49.6, 51.0, 56.8, 58.7, 77.9, 81.7, 117.7, 134.9, 170.0, 171.0. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3367 (m), 2980 (s), 2934 (m), 2905 (m), 2829 (w), 1732 (s), 1661 (s), 1603 (s), 1488 (m), 1463 (m), 1444 (m), 1418 (w), 1369 (m), 1299 (s), 1245 (s), 1221 (s), 1183 (s), 1151 (s), 1099 (s), 1051 (s), 998 (w), 974 (w), 918 (m), 854 (m), 785 (w). MS (EI, 70 eV): m/z (%) = 225 (M^+ , 22), 194 (71), 180 (17), 151 (32), 120 (100). The exact molecular mass m/z = 225.1365 ± 2 ppm [M^+] for $\text{C}_{12}\text{H}_{19}\text{O}_3\text{N}$ was confirmed by HRMS (EI, 70 eV).

(4-Methoxy-3-propyl-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4j): Starting with **3j** (0.350 g, 1.29 mmol) and PPh_3 (0.406 g, 1.55 mmol) in THF (15 mL), **4j** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 100:1 $\rightarrow$ 1:1), as a brownish oil (0.252 g, 86%). ^1H NMR (CDCl_3 , 300 MHz): δ = 0.98 (t, J = 6.6 Hz, 3 H, CH_3), 1.25 (t, J = 7.1 Hz, 3 H, OCH_2CH_3), 1.39 – 1.49 (m, 4 H, $2\times\text{CH}_2$), 2.68 – 2.72 (m, 1 H, CH), 3.32 (s, 3 H, OCH_3), 3.49 (dt, J = 10.5, 1.5 Hz, 1 H, CH), 3.64 – 3.72 (m, 2 H, CH_2NH), 4.10 (dq, J = 1.5, 7.2 Hz, 2 H, OCH_2), 4.53 (s, 1 H, $\text{CH}=\text{C}$), 7.78 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 150 MHz): δ_{c} = 14.1, 14.7, 20.3, 34.0, 49.9, 51.0, 56.5, 58.5, 77.7, 82.7, 168.0, 170.8. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3367 (w), 2960 (m), 2934 (m), 2873 (w), 1661 (s), 1605 (s), 1489 (w), 1462 (w), 1369 (w), 1313 (w), 1286 (w), 1241 (s), 1220 (s), 1192 (m), 1149 (s), 1097 (m), 1050 (m), 783 (w). UV-VIS (CH_2Cl_2 , nm): λ_{max} (log ϵ) = 280 (4.18). MS (EI, 70 eV): m/z (%) = 227 (M^+ , 12), 196 (4), 182 (17), 167 (7), 154 (100). HRMS (ESI): calcd. for $\text{C}_{12}\text{H}_{22}\text{O}_3\text{N}$ ($[\text{M}+1]^+$) = 228.15997; found = 228.15923.

(3-Butyl-4-methoxy-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4k): Starting with **3k** (0.400 g, 1.40 mmol), and PPh₃ (0.441 g, 1.68 mmol) in THF (15 mL), **4k** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish solid (0.313 g, 93%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.91 (t, *J* = 7.3 Hz, 3 H, CH₃), 1.25 (dt, *J* = 1.5, 7.1 Hz, 3 H, OCH₂CH₃), 1.30 – 1.43 (m, 4 H, 2×CH₂), 1.52 – 1.59 (m, 2 H, CH₂), 2.60 – 2.71 (m, 1 H, CH), 3.22 (s, 3 H, OCH₃), 3.49 (dt, *J* = 10.6, 1.7 Hz, 1 H, CH), 3.55 – 3.73 (m, 1 H, CH₂NH), 4.10 (dq, *J* = 2.0, 7.1 Hz, 2 H, OCH₂), 4.53 (s, 1 H, CH=C), 7.80 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.0, 14.7, 22.7, 29.2, 31.4, 50.2, 51.0, 56.5, 58.5, 78.9, 82.7, 168.1, 170.8. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3366 (m), 2956 (s), 2932 (s), 2868 (m), 1661 (s), 1604 (s), 1488 (m), 1463 (m), 1368 (m), 1343 (w), 1318 (w), 1237 (s), 1187 (m), 1150 (s), 1098 (m), 1051 (m), 783 (m). MS (EI, 70 eV): *m/z* (%) = 241 (M⁺, 16), 210 (29), 196 (17), 168 (5), 153 (100), 136 (19). The exact molecular mass *m/z* = 241.1678 ± 2 ppm [M⁺] for C₁₃H₂₃O₃N was confirmed by HRMS (EI, 70 eV).

(3-Hexyl-4-methoxy-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4l): Starting with **3l** (0.450 g, 1.43 mmol) and PPh₃ (0.452 g, 1.72 mmol) in THF (20 mL), **4l** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish solid (0.377 g, 98%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.89 (t, *J* = 6.6 Hz, 3 H, CH₃), 1.28 (t, *J* = 7.2 Hz, 3 H, OCH₂CH₃), 1.23 – 1.54 (m, 10 H, 5×CH₂), 2.66 – 2.72 (m, 1 H, CH), 3.32 (s, 3 H, OCH₃), 3.49 (dt, *J* = 10.8, 1.5 Hz, 1 H, CH), 3.64 – 3.73 (m, 2 H, CH₂NH), 4.10 (dq, *J* = 1.8, 7.2 Hz, 2 H, OCH₂), 4.53 (s, 1 H, CH=C), 7.79 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.0, 14.6, 22.5, 26.9, 29.2, 31.6, 31.8, 50.0, 50.9, 56.4, 58.3, 77.6, 82.6, 167.9, 170.6. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3366 (w), 2930 (m), 2859 (w), 1662 (s), 1606 (s), 1489 (w), 1463 (w), 1369 (w), 1230 (s), 1149 (s), 1100 (m), 1051 (m), 783 (w). UV-VIS (CH₂Cl₂, nm): λ_{max} (log ε) = 280 (4.26). MS (EI, 70 eV): *m/z* (%) = 269 (M⁺, 16), 238 (56), 224 (15), 185 (13), 164 (21),

153 (100). The exact molecular mass $m/z = 269.1991 \pm 2$ ppm [M^+] for $C_{15}H_{27}O_3N$ was confirmed by HRMS (EI, 70 eV).

(4-Methoxy-3-octyl-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4m): Starting with **3m** (0.350 g, 1.03 mmol) and PPh_3 (0.296 g, 1.13 mmol) in THF (10 mL), **4m** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish oil (0.262 g, 86%). 1H NMR ($CDCl_3$, 300 MHz): δ = 0.88 (t, J = 6.6 Hz, 3 H, CH_3), 1.221.28 (m, 13 H, $5 \times CH_2$, OCH_2CH_3), 1.37 – 1.44 (m, 3 H, $2 \times CH_2$), 1.54 – 1.58 (m, 1 H, CH_2), 2.67 – 2.72 (m, 1 H, CH), 3.32 (s, 3 H, OCH_3), 3.45 – 3.66 (m, 2 H, CH_2NH), 3.71 – 3.73 (m, 1 H, CH), 4.10 (dq, J = 1.8, 7.2 Hz, 2 H, OCH_2), 4.53 (s, 1 H, $CH=C$), 7.79 (broad s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 14.0, 14.6, 22.6, 26.9, 27.4, 29.2, 29.3, 31.6, 31.7, 50.0, 50.8, 57.0, 58.4, 78.7, 82.6, 167.9, 170.7. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3368(w) 2954 (w), 2927 (s), 2856 (w), 1734 (w), 1662 (s), 1605 (s), 1463 (m), 1230 (s), 1206 (m), 1180 (m), 1151 (s), 1100 (s), 1052 (m). MS (EI, 70 eV): m/z (%) = 297 (M^+ , 21), 266 (10), 252 (22), 220 (5), 192 (49), 180 (10). Anal. calcd. for $C_{17}H_{31}O_3N$ (297.437): C 68.65, H 10.51, N 4.71; found C 69.23, H 9.58, N 4.88.

(4-Methoxy-3-nonyl-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4n): Starting with **3n** (1.700 g, 4.78 mmol) and PPh_3 (1.505 g, 5.74 mmol) in THF (20 mL), **4n** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish solid (1.204 g, 81%). 1H NMR ($CDCl_3$, 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.21 – 1.34 (m, 17 H, $7 \times CH_2$, OCH_2CH_3), 1.36 – 1.54 (m, 2 H, CH_2), 2.49 – 2.72 (m, 1 H, CH), 3.32 (s, 3 H, OCH_3), 3.49 (dt, J = 12.3, 1.5 Hz, 1 H, CH), 3.55 – 3.72 (m, 2 H, CH_2NH), 4.10 (dq, J = 2.0, 7.2 Hz, 2 H, OCH_2), 4.53 (s, 1 H, $CH=C$), 7.80 (broad s, 1H, NH). ^{13}C NMR ($CDCl_3$, 150 MHz): δ_c = 14.68, 14.71, 22.7, 25.0, 25.8, 27.1, 27.6, 29.4, 29.7, 50.2, 51.0, 56.5, 58.5, 78.9, 82.7, 168.0, 170.8. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3352 (m), 2925 (s), 2854 (s), 1733 (w), 1661 (s), 1607 (s), 1489

(m), 1463 (m), 1369 (m), 1345 (w), 1319 (w), 1288 (w), 1234 (s), 1151 (s), 1101 (m), 1053 (m), 781 (m), 722 (w), 695 (w). UV-VIS (CH₂Cl₂, nm): λ_{max} (log ϵ) = 280 (4.26). MS (EI, 70 eV): m/z (%) = 311 (M⁺, 11), 280 (62), 266 (11), 206 (16), 185 (16), 153 (100). The exact molecular mass $m/z = 311.2460 \pm 2$ ppm [M⁺] for C₁₈H₃₃O₃N was confirmed by HRMS (EI, 70 eV).

(3-Decyl-4-methoxy-pyrrolidin-2-ylidene)-acetic acid ethyl ester (4o): Starting with **3o** (1.150 g, 3.11 mmol) and PPh₃ (0.980 g, 3.374 mmol) in THF (40 mL), **4o** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish solid (0.835 g, 83%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.91 (t, J = 6.9 Hz, 3 H, CH₃), 1.20 – 1.58 (m, 21 H, 9×CH₂, OCH₂CH₃), 2.62 – 2.69 (m, 1 H, CH), 3.32 (s, 3 H, OCH₃), 3.49 (dt, J = 12.3, 1.8 Hz, 1 H, CH), 3.55 – 3.73 (m, 2 H, CH₂NH), 4.10 (dq, J = 1.4, 7.2 Hz, 2 H, OCH₂), 4.53 (s, 1 H, CH=C), 7.79 (broad s, 1H, NH). ¹³C NMR (CDCl₃, 150 MHz): δ_c = 14.1, 14.7, 22.7, 24.9, 27.0, 27.5, 29.3, 29.5, 29.7, 31.8, 31.9, 50.1, 50.9, 56.5, 58.4, 78.8, 82.7, 168.0, 170.7. IR (KBr, cm⁻¹): $\tilde{\nu}$ = 3367 (w), 2978 (w), 2922 (s), 2905 (m), 2825 (w), 1659 (s), 1604 (s), 1490 (w), 1468 (w), 1369 (w), 1348 (w), 1310 (w), 1279 (w), 1255 (m), 1234 (s), 1191 (w), 1159 (s), 1115 (w), 1089 (w), 1064 (m), 782 (m), 723 (w). MS (EI, 70 eV): m/z (%) = 325 (M⁺, 18), 294 (100), 280 (18), 220 (36). The exact molecular mass $m/z = 325.2617 \pm 2$ ppm [M⁺] for C₁₉H₃₅O₃N was confirmed by HRMS (EI, 70 eV).

3-Methoxy-2,3,3a,4,5,6,7,8,9,10,11,12-dodecahydro-1H-cyclododeca[b]pyrrole-13-carboxylic acid ethyl ester (4p): Starting with **3p** (1.900 g, 5.38 mmol) and PPh₃ (1.692 g, 6.45 mmol) in THF (10 mL), **4p** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish oil (1.624 g, 98%). ¹H NMR (CDCl₃, 600 MHz): δ = 1.23 (t, J = 7.2 Hz, 3 H, CH₃), 1.25 – 1.45 (m, 12 H, 6×CH₂), 1.52 – 1.65 (m, 2 H, CH₂), 1.71 – 1.76 (m, 1 H, CH₂), 1.82 – 1.87 (m, 1 H, CH₂), 2.11 – 2.19 (m, 2 H, CH₂), 3.00 (dd, J = 9.2 Hz, 1

H, CH), 3.28 (s, 3 H, OCH₃), 3.53 (dd, $J = 9.2$ Hz, 1 H, CH), 3.58 (d, $J = 4.6$ Hz, 1 H, CH₂NH), 3.65 (dd, $J = 18.4, 4.6$ Hz, 1 H, CH₂NH), 4.04 – 4.14 (m, 2 H, OCH₂), 8.04 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 50 MHz): $\delta_c = 14.1, 21.1, 23.1, 24.1, 24.9, 25.6, 26.7, 27.9, 28.3, 37.3, 50.4, 50.7, 56.0, 58.6, 82.4, 90.3, 166.1, 175.3$. IR (neat, cm⁻¹): $\tilde{\nu} = 3355$ (m), 2929 (s), 2860 (s), 1727 (s), 1656 (s), 1583 (s), 1468 (m), 1445 (m), 1367 (w), 1315 (w), 1296 (w), 1337 (s), 1226 (s), 1178 (s), 1146 (m), 1095 (s), 1049 (s). MS (EI, 70 eV): m/z (%) = 309 (M^+ , 29), 278 (100), 264 (9), 236 (4), 204 (51). The exact molecular mass $m/z = 309.2304 \pm 2$ ppm [M^+] for C₁₈H₃₁O₃N was confirmed by HRMS (EI, 70 eV).

3-Methoxy-2,3,3a,4,5,6-hexahydro-1H-indole-7-carboxylic acid ethyl ester (4q): Starting with **3q** (0.800 g, 2.97 mmol) and PPh₃ (1.170 g, 4.46 mmol) in THF (50 mL), *cis*-**4q** and *trans*-**4q** were isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as yellow and brownish oils (0.490 g, 73%; 0.103 g, 15% = 88%).

***cis*-4q:** ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.26$ (t, $J = 6.9$ Hz, 3 H, CH₃), 1.36 – 1.53 (m, 1 H, CH₂), 1.90 – 1.99 (m, 1 H, CH₂), 2.03 – 2.10 (m, 1 H, CH₂), 2.11 – 2.19 (m, 2 H, CH₂), 2.35 (dd, $J = 15.9, 6.6$ Hz, 1 H, CH₂), 2.50 – 2.60 (m, 1 H, CH), 3.21 (dt, $J = 14.1, 4.5$ Hz, 1 H, CH), 3.42 (s, 3 H, OCH₃), 3.66 – 3.76 (m, 2 H, CH₂NH), 4.13 (dq, $J = 1.2, 6.9$ Hz, 2 H, OCH₂), 7.25 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 14.5, 22.1, 22.3, 26.1, 46.4, 49.2, 57.9, 58.5, 84.0, 87.0, 160.0, 169.8$. IR (neat, cm⁻¹): $\tilde{\nu} = 3391$ (m), 2977 (s), 2934 (s), 2861 (s), 1727 (m), 1674 (s), 1610 (s), 1480 (w), 1462 (m), 1446 (m), 1427 (w), 1368 (m), 1295 (s), 1243 (s), 1228 (s), 1196 (s), 1176 (m), 1140 (s), 1107 (s), 1072 (s), 1036 (m), 994 (w). MS (EI, 70 eV): m/z (%) = 225 (M^+ , 100), 196 (93), 180 (43), 165 (71), 152 (93), 120 (62). Anal. calcd. for C₁₂H₁₉O₃N (225.287): C 63.98, H 8.50, N 6.22; found C 64.43, H 8.85, N 5.75.

***trans*-4q:** ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.25$ (t, $J = 7.2$ Hz, 3 H, CH₃), 1.42 – 1.51 (m, 1 H, CH₂), 1.61 (dq, $J = 2.4, 12.0$ Hz, 1 H, CH₂), 1.82 – 1.89 (m, 1 H, CH₂), 1.94 – 2.01 (m,

1 H, CH₂), 2.31 – 2.23 (m, 1 H, CH₂), 2.33 (dd, $J = 15.9, 6.0$ Hz, 1 H, CH₂), 2.51 – 2.63 (m, 1 H, CH), 3.33 (s, 3 H, OCH₃), 3.46 (dd, $J = 11.4, 3.3$ Hz, 1 H, CH₂NH), 3.57 (dd, $J = 11.4, 2.4$ Hz, 1 H, CH₂NH), 3.87 (t, $J = 3.6$ Hz, 1 H, CH), 4.12 (q, $J = 7.2$ Hz, 2 H, OCH₂), 7.34 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 14.7, 21.5, 22.4, 22.8, 46.3, 49.9, 57.0, 58.5, 80.4, 87.2, 160.7, 169.9$. IR (neat, cm⁻¹): $\tilde{\nu} = 3391$ (s), 2977(s), 2934 (s), 2858 (m), 1729 (m), 1674 (s), 1600 (s), 1463 (m), 1445 (m), 1409 (w), 1368 (m), 1335 (w), 1295 (m), 1259 (s), 1232 (s), 1180 (s), 1164 (s), 1116 (s), 1098 (s), 1054 (m), 1028 (s), 801 (m), 723 (w). MS (EI, 70 eV): m/z (%) = 225 (M⁺, 100), 196 (90), 180 (72), 152 (95), 120 (97). Anal. calcd. for C₁₂H₁₉O₃N (225.287): C 63.98, H 8.50, N 6.22; found C 64.09, H 8.16, N 6.85.

3-Methoxy-2,3,3a,4,5,6,7,8-octahydro-1H-cycloocta[b]pyrrole-9-carboxylic acid ethyl ester (4r): Starting with **3r** (0.180 g, 0.605 mmol) and PPh₃ (0.191 g, 0.73 mmol) in THF (10 mL), *cis*-**4r** and *trans*-**4r** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as yellow solids (0.021 g, 14%; 0.116 g, 76% = 90%).

***cis*-4r:** ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.25$ (t, $J = 6.9$ Hz, 3 H, CH₃), 1.20 – 1.32 (m, 3 H, 2×CH₂), 1.39 – 1.50 (m, 1 H, CH₂), 1.53 – 1.64 (m, 2 H, CH₂), 1.81 – 1.91 (m, 1 H, CH₂), 1.84 – 2.11 (m, 2 H, CH₂), 2.62 (dt, $J = 14.4, 2.7$ Hz, 1 H, CH₂), 2.95 – 3.05 (m, 1 H, CH), 3.32 (t, $J = 9.3$ Hz, 1 H, CH₂NH), 3.37 (s, 3 H, OCH₃), 3.60 – 3.67 (m, 1 H, CH₂NH), 4.04 (dq, $J = 1.8, 7.2$ Hz, 1 H, CH), 4.11 (dq, $J = 0.6, 6.9$ Hz, 2 H, OCH₂), 7.86 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 14.9, 25.0, 25.95, 25.97, 27.5, 29.7, 44.6, 48.4, 57.8, 58.7, 79.9, 88.8, 165.7, 170.7$. IR (KBr, cm⁻¹): $\tilde{\nu} = 3369$ (w), 2940 (s), 2928(s), 2852 (w), 1654 (s), 1591 (s), 1460 (w), 1369 (w), 1304 (m), 1236 (m), 1213 (s), 1183 (m), 1146 (m), 1091 (m), 1053 (m). MS (EI, 70 eV): m/z (%) = 253 (M⁺, 58), 238 (10), 224 (10), 222 (65), 208 (30), 180 (10), 164 (11), 147 (100). The exact molecular mass $m/z = 253.1678 \pm 2$ ppm [M⁺] for C₁₄H₂₃O₃N was confirmed by HRMS (EI, 70 eV).

***trans*-4r:** ^1H NMR (CDCl_3 , 300 MHz): δ = 1.24 (t, J = 7.2 Hz, 3 H, CH_3), 1.26 – 1.46 (m, 4 H, $2\times\text{CH}_2$), 1.51 – 1.62 (m, 2 H, CH_2), 1.73 – 1.88 (m, 2 H, CH_2), 2.00 – 2.11 (m, 1 H, CH_2), 2.67 (dt, J = 13.8, 3.9 Hz, 1 H, CH_2), 2.96 (dd, J = 12.6, 3.0 Hz, 1 H, CH), 3.32 (s, 3 H, OCH_3), 3.52 – 3.58 (m, 2 H, CH_2NH), 3.62 – 3.67 (m, 1 H, CH), 4.11 (q, J = 7.2 Hz, 2 H, OCH_2), 7.97 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 14.5, 24.8, 25.3, 25.8, 29.2, 33.6, 49.6, 49.9, 55.8, 58.3, 84.9, 88.3, 164.8, 170.0. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3364 (m), 2927 (s), 2847 (w), 1657 (s), 1597 (s), 1458 (m), 1364 (w), 1304 (m), 1234 (s), 1217 (s), 1170 (m), 1147 (w), 1130 (m), 1099 (s), 1044 (m). MS (EI, 70 eV): m/z (%) = 253 (M^+ , 100), 238 (11), 224 (10), 222 (86), 208 (42), 180 (8), 149 (4). The exact molecular mass m/z = 253.1678 $\pm$ 2 ppm [M^+] for $\text{C}_{14}\text{H}_{23}\text{O}_3\text{N}$ was confirmed by HRMS (EI, 70 eV).

3-Methoxy-4-methyl-2,3,3a,4,5,6-hexahydro-1H-indole-7-carboxylic acid ethyl ester (4s):

Starting with **3s** (1.250 g, 4.41 mmol) and PPh_3 (1.389 g, 5.3 mmol) in THF (20 mL), ***cis*-4s** and ***trans*-4s** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 100:1 $\rightarrow$ 1:1) as yellow solids (0.385 g, 37%; 0.358 g, 34% = 71%).

***cis*-4s:** ^1H NMR (CDCl_3 , 300 MHz): δ = 1.09 (d, J = 6.6 Hz, 3 H, CH_3), 1.18 – 1.24 (m, 1 H, CH_2), 1.27 (t, J = 7.2 Hz, 3 H, OCH_2CH_3), 1.41 – 1.53 (m, 1 H, CH_2), 1.74 – 1.82 (m, 1 H, CH), 3.37 (s, 3 H, OCH_3), 3.70 – 3.79 (m, 2 H, CH_2NH), 4.12 (dq, J = 1.5, 7.2 Hz, 2 H, OCH_2), 7.34 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 14.3, 19.3, 22.7, 31.9, 33.4, 49.3, 51.5, 57.2, 58.2, 83.6, 86.5, 159.8, 169.6. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3371 (m), 2954 (w), 2930 (m), 2873 (w), 2854 (w), 1664 (s), 1603 (s), 1370 (m), 1294 (m), 1248 (s), 1229 (s), 1201 (m), 1126 (s), 1053 (m). MS (EI, 70 eV): m/z (%) = 239 (M^+ , 95), 224 (16), 210 (45), 194 (25), 166 (100), 134 (68). Anal. calcd. for $\text{C}_{13}\text{H}_{21}\text{O}_3\text{N}$ (239.314): C 65.25, H 8.84, N 5.85; found C 65.50, H 8.47, N 5.40.

trans-4s: ^1H NMR (CDCl_3 , 300 MHz): δ = 1.04 (d, J = 6.3 Hz, 3 H, CH_3), 1.18 – 1.29 (m, 1 H, CH_2), 1.26 (t, J = 7.2 Hz, 3 H, OCH_2CH_3), 1.81 – 1.88 (m, 2 H, CH_2 , CH), 2.15 – 2.26 (m, 2 H, CH_2), 2.35 (dd, J = 6.6 Hz, 1 H, CH), 3.33 (s, 3 H, OCH_3), 3.42 (dd, J = 11.7, 3.3 Hz, 1 H, CH), 3.59 (dd, J = 11.4, 2.6 Hz, 1 H, CH_2NH), 3.42 (dd, J = 11.7, 3.3 Hz, 1 H, CH), 3.59 (dd, J = 11.4, 2.6 Hz, 1 H, CH_2NH), 3.93 – 3.96 (m, 1 H, CH_2NH), 4.12 (q, J = 7.2 Hz, 2 H, OCH_2), 7.33 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 14.5, 19.8, 22.9, 27.2, 31.6, 49.4, 52.8, 56.7, 58.3, 78.7, 86.7, 160.0, 169.6. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3409 (m), 2980 (w), 2952 (w), 2930 (m), 2904 (w), 2865 (w), 2851 (w), 1663 (s), 1600 (s), 1438 (w), 1367 (w), 1308 (w), 1260 (s), 1233 (s), 1182 (w), 1116 (s), 1096 (m), 1075 (w), 1051 (m). MS (EI, 70 eV): m/z (%) = 239 (M^+ , 100), 224 (2), 210 (56), 194 (31), 166 (98), 134 (31). The exact molecular mass m/z = 239.1521 ± 2 ppm [M^+] for $\text{C}_{13}\text{H}_{21}\text{O}_3\text{N}$ was confirmed by HRMS (EI, 70 eV).

3-Methoxy-5-phenyl-2,3,3a,4,5,6-hexahydro-1H-indole-7-carboxylic acid methyl ester (4t): Starting with **3t** (0.300 g, 0.905 mmol) and PPh_3 (0.285 g, 1.09 mmol) in THF (15 mL), **trans-4t** and **cis-4t** were isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 100:1 $\rightarrow$ 4:1) as brownish oils (0.134 g, 52%, 0.102 g, 39% = 91%).

trans-4t: ^1H NMR (CDCl_3 , 300 MHz): δ = 1.52 – 1.62 (m, 1 H, CH_2), 2.16 – 2.32 (m, 2 H, CH_2 , CH), 2.60 – 2.69 (m, 1 H, CH), 2.75 – 2.90 (m, 2 H, CH_2), 3.11 – 3.31 (m, 1 H, CH), 3.41 (s, 3 H, OCH_3), 3.64 (s, 3 H, OCH_3), 3.77 – 3.81 (m, 2 H, $\text{CH}_2\text{-NH}$), 7.17 – 7.32 (m, 6 H, 5 $\times$ CH from Ph, NH). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 32.0, 33.2, 41.1, 47.8, 50.1, 50.6, 58.5, 84.2, 87.3, 126.5, 127.1, 128.6, 146.4, 160.2, 170.4. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3392 (w), 2975 (w), 2938 (m), 2900 (w), 2857 (w), 1737 (w), 1673 (s), 1606 (s), 1492 (w), 1441 (m), 1369 (w), 1328 (w), 1300 (w), 1227 (s), 1195 (m), 1137 (m), 1114 (m), 1079 (m), 1031 (w), 991 (w), 760 (w), 701 (m). MS (EI, 70 eV): m/z (%) = 287 (M^+ , 58), 255 (18), 212 (4), 196 (23), 183 (21), 151 (100).

cis-4t: ^1H NMR (CDCl_3 , 300 MHz): δ = 1.89 – 2.09 (m, 2 H, CH_2), 2.23 – 2.33 (m, 1 H, CH), 2.63 (dd, J = 15.6, 5.7 Hz, 1 H, CH), 2.81 – 2.92 (m, 2 H, CH_2), 3.35 (s, 3 H, OCH_3), 3.48 – 3.61 (m, 1 H, $\text{CH}_2\text{-NH}$), 3.64 (s, 3 H, OCH_3), 3.65 – 3.82 (m, 1 H, $\text{CH}_2\text{-NH}$), 3.90 – 3.94 (m, 1 H, CH), 7.22 – 7.32 (m, 5 H, $5\times\text{CH}$ from Ph), 7.41 (broad s, 1 H, N-H). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 28.4, 32.3, 41.6, 47.5, 50.6, 50.6, 57.4, 80.7, 87.3, 126.4, 127.2, 128.6, 146.7, 160.6, 170.3. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3379 (w), 2948 (m), 2929 (m), 2869 (w), 2856 (w), 1735 (s), 1666 (m), 1603 (m), 1494 (w), 1440 (s), 1379 (w), 1358 (w), 1332 (w), 1236 (s), 1203 (s), 1180 (m), 1158 (m), 1115 (s), 1091 (s), 1027 (m), 972 (w), 909 (w), 843 (w), 760 (m), 733 (w), 700 (m). MS (EI, 70 eV): m/z (%) = 287 (M^+ , 19), 212 (7), 196 (11), 157 (8), 144 (7), 131 (6), 117 (40), 104 (100), 91 (99), 77 (17).

1-(4-Methoxy-pyrrolidin-2-ylidene)-propan-2-one (4u): Starting with **3u** (0.130 g, 0.653 mmol) and PPh_3 (0.188 g, 0.718 mmol) in THF (5 mL), **4u** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 50:1 $\rightarrow$ 1:1) as a yellow solid (1:1 mixture of product and PPh_3 , calculated yield of product: 0.082 g, 81%). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.03 (s, 3 H, CH_3), 2.64 (dd, J = 17.1, 1.8 Hz, 1 H, CH_2), 2.78 (ddd, J = 17.1, 6.3, 0.6 Hz, 1 H, CH_2), 3.32 (s, 3 H, OCH_3), 3.60 (dd, J = 11.7, 2.4 Hz, 1 H, CH_2NH), 3.69 (dd, J = 11.7, 5.1 Hz, 1 H, CH_2NH), 4.06 (sept, J = 3.0 Hz, 1 H, CH), 5.12 (s, 1 H, $\text{CH}=\text{C}$), 9.68 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 28.7, 38.6, 52.7, 56.5, 76.7, 90.5, 164.9, 195.4. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3297 (w), 2929 (m), 1624 (s), 1550 (s), 1504 (m), 1463 (w), 1438 (m), 1417 (w), 1361 (m), 1294 (m), 1252 (m), 1188 (s), 1097 (s), 21038 (w). MS (EI, 70 eV): m/z (%) = 155 (M^+ , 82), 140 (100), 124 (48), 80 (26), 68 (9). The exact molecular mass m/z = 155.0946 ± 2 ppm [M^+] for $\text{C}_8\text{H}_{13}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

General procedure for the synthesis of pyrroles (5). A solution of **4** in CH_2Cl_2 (10 mL/mmol) or in 1,4-dioxane (5 mL/mmol) was stirred at reflux for 24 h. After cooling to 20

°C, the solvent was removed in *vacuo* and the residue was purified by chromatography (silica gel, *n*-Hex/EtOAc) to give **5**.

(1H-Pyrrol-2-yl)-acetic acid methyl ester (5a): Starting with **4a** (0.100 g, 0.32 mmol) in 1,4-dioxane (10 mL), **5a** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 10:1) as a black oil (0.068 g, 76%). ¹H NMR (CDCl₃, 600 MHz): δ = 3.69 (s, 2 H, CH₂), 3.72 (s, 3 H, OCH₃), 6.01 (m, 1 H, CH), 6.15 (m, 1 H, CH), 6.75 (m, 1 H, CH), 8.70 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 33.0, 52.2, 107.4, 108.3, 115.0, 117.8, 171.6. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3382 (s), 2955 (m), 2926 (m), 1734 (s), 1646 (m), 1599 (w), 1574 (w), 1436 (s), 1343 (w), 1330 (w), 1260 (s), 1201 (m), 1150 (m), 1128 (m), 1095 (m), 1045 (w), 1024 (m), 796 (w), 723 (m). MS (EI, 70 eV): *m/z* (%) = 139 (M⁺, 33), 124 (2), 107 (5), 80 (100). The exact molecular mass *m/z* = 139.0633 ± 2 ppm [M⁺] for C₇H₉O₂N was confirmed by HRMS (EI, 70 eV).

(1H-Pyrrol-2-yl)-acetic acid ethyl ester (5b): Starting with **4b** (0.020 g, 0.108 mmol) in 1,4-dioxane (5 mL), **5b** was without further purification as a black oil (0.017 g, 100%). ¹H NMR (CDCl₃, 300 MHz): δ = 1.28 (t, *J* = 7.2 Hz, 3 H, CH₃), 3.67 (s, 2 H, CH₂), 4.18 (q, *J* = 7.2 Hz, 2 H, OCH₂), 6.02 (m, 1 H, CH), 6.12 – 6.15 (m, 1 H, CH), 6.75 (m, 1 H, CH), 8.70 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 14.2, 33.2, 61.1, 107.3, 108.3, 117.7, 123.6, 171.3. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3394 (m), 2985 (m), 2938 (w), 2906 (w), 1735 (s), 1578 (m), 1499 (m), 1447 (m), 1411 (m), 1369 (m), 1325 (s), 1267 (s), 1227 (w), 1201 (s), 1127 (s), 1095 (s), 1053 (s), 781 (m), 754 (m), 722 (m). MS (EI, 70 eV): *m/z* (%) = 135 (M⁺, 75), 124 (56), 108 (100). HRMS (ESI): calcd. for C₈H₁₂O₂N ([M+1]⁺) = 154.08680; found = 154.08601.

(1H-Pyrrol-2-yl)-acetic acid isopropyl ester (5c): Starting with **4c** (0.015 g, 0.075 mmol) in 1,4-dioxane (5 mL), **5c** was without further purification as a black oil (0.013 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.26 (d, J = 6.3 Hz, 6 H, $2\times\text{CH}_3$), 3.64 (s, 2 H, CH_2), 5.04 (sept, J = 6.3 Hz, 1 H, OCH), 6.01 (m, 1 H, CH), 6.12 – 6.15 (m, 1 H, CH), 6.75 (m, 1 H, CH), 8.75 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 21.8, 33.4, 68.6, 107.1, 108.2, 117.6, 123.5, 170.8. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3373 (m), 2981 (s), 2935 (m), 2877 (w), 1731 (s), 1609 (w), 1553 (w), 1464 (w), 1435 (m), 1374 (m), 1343 (m), 1262 (s), 1175 (s), 1224 (s), 1150 (s), 1106 (s), 1043 (w), 964 (w), 766 (m). MS (EI, 70 eV): m/z (%) = 167 (M^+ , 27), 125 (4), 80 (100). HRMS (ESI): calcd. for $\text{C}_9\text{H}_{14}\text{O}_2\text{N}$ ($[\text{M}+1]^+$) = 168.10245; found = 168.10161.

(1H-Pyrrol-2-yl)-acetic acid 2-methoxy-ethyl ester (5d): Starting with **4d** (0.030 g, 0.14 mmol) in 1,4-dioxane (3 mL), **5d** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 10:1) as a black oil (0.025 g, 98%). ^1H NMR (CDCl_3 , 300 MHz): δ = 3.39 (s, 3 H, OCH_3), 3.60 – 3.63 (m, 2 H, OCH_2), 3.72 (s, 2 H, CH_2), 4.27 – 4.32 (m, 2 H, OCH_2), 6.02 (m, 1 H, CH), 6.14 (q, J = 3.0 Hz, 1 H, CH), 6.75 (m, 1 H, CH), 8.82 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 33.1, 58.8, 64.1, 70.1, 107.2, 108.1, 117.6, 122.9, 170.9. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3359 (m), 2932 (m), 2895 (m), 2825 (w), 1741 (s), 1643 (m), 1577 (w), 1452 (m), 1409 (m), 1369 (m), 1320 (s), 1264 (s), 1197 (s), 1178 (s), 1152 (s), 1227 (s), 1097 (s), 1037 (s), 861 (w), 790 (w), 728 (m). MS (EI, 70 eV): m/z (%) = 183 (M^+ , 25), 125 (11), 107 (46), 80 (100). The exact molecular mass m/z = 183.0895 ± 2 ppm [M^+] for $\text{C}_9\text{H}_{13}\text{O}_3\text{N}$ was confirmed by HRMS (EI, 70 eV).

1-Phenyl-2-(1H-pyrrol-2-yl)-ethanone (5e): Starting with **4e** (0.026 g, 0.12 mmol) in 1,4-dioxane (5 mL), **5e** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish solid (0.018 g, 82%). ^1H NMR (CDCl_3 , 300 MHz): δ = 4.32 (s, 2 H, CH_2),

6.07 (m, 1 H, CH),), 6.15 (dd, $J = 6.0, 1.8$ Hz, 1 H, CH), 6.76 (m, 1 H, CH), 7.44 – 7.50 (m, 2 H, 2×CH of Ph), 7.55 – 7.60 (m, 1 H, CH of Ph), 7.99 – 8.03 (m, 2 H, 2×CH from Ph), 8.85 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta_{\text{c}} = 36.9, 107.4, 108.2, 117.8, 123.9, 128.4, 128.7, 133.4, 136.3, 197.7$. IR (KBr, cm^{-1}): $\tilde{\nu} = 3351$ (s), 2902 (w), 1676 (s), 1587 (w), 1341 (m), 1285 (w), 1239 (m), 1215 (m), 999 (m), 796 (m), 742 (s), 731 (s), 690 (m), 600 (w). MS (EI, 70 eV): m/z (%) = 185 (M^+ , 33), 105 (79), 80 (100). The exact molecular mass $m/z = 185.0841 \pm 2$ ppm [M^+] for $\text{C}_{12}\text{H}_{11}\text{ON}$ was confirmed by HRMS (EI, 70 eV).

(3-Methyl-1H-pyrrol-2-yl)-acetic acid methyl ester (5f): Starting with **4f** (0.300g, 1.62 mmol) in CH_2Cl_2 (30 mL), **5f** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 10:1) as a brownish solid (0.248 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 2.04$ (s, 3 H, CH_3), 3.60 (s, 2 H, CH_2), 3.71 (s, 3 H, OCH_3), 6.00 (t, $J = 1.8$ Hz, 1 H, CH), 6.67 (t, $J = 1.8$ Hz, 1 H, CH), 8.49 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta_{\text{c}} = 10.6, 30.9, 51.9, 109.7, 115.7, 116.4, 119.2, 171.7$. IR (KBr, cm^{-1}): $\tilde{\nu} = 3353$ (s), 2953 (w), 2922 (w), 1717 (s), 1448 (w), 1436 (m), 1401 (m), 1344 (m), 1290 (w), 1256 (m), 1224 (s), 1178 (m), 1099 (m), 986 (m), 848 (w), 787 (w), 721 (s), 592 (m), 568 (w). UV-VIS (CH_2Cl_2 , nm): λ_{max} (log ϵ) = 227 (3.65), 460 (2.39). MS (EI, 70 eV): m/z (%) = 153 (M^+ , 49), 138 (1), 94 (100), 80 (2). Anal. calcd. for $\text{C}_8\text{H}_{11}\text{O}_2\text{N}$ (153.181): C 62.73, H 7.24, N 9.14; found C 63.04, H 7.82, N 8.73.

(3-Ethyl-1H-pyrrol-2-yl)-acetic acid ethyl ester (5g): Starting with **4g** (0.120 g, 0.56 mmol) in CH_2Cl_2 (12 mL), **5g** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish oil (0.102 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.16$ (t, $J = 7.5$ Hz, 3 H, CH_3), 1.27 (t, $J = 7.2$ Hz, 3 H, OCH_2CH_3), 2.43 (q, $J = 7.5$ Hz, 2 H, CH_2), 3.59 (s, 2

H, CH₂), 4.17 (q, $J = 7.2$ Hz, 2 H, OCH₂), 6.05 (t, $J = 1.8$ Hz, 1 H, CH), 6.68 (t, $J = 1.8$ Hz, 1 H, CH), 8.53 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 13.9, 15.4, 18.6, 31.1, 60.8, 107.8, 116.4, 118.5, 122.6, 171.2$. IR (neat, cm⁻¹): $\tilde{\nu} = 3392$ (m), 2965 (m), 2933 (w), 2873 (w), 1730 (s), 1478 (w), 1460 (m), 1425 (w), 1401 (w), 1370 (m), 1331 (w), 1297 (m), 1251 (m), 1182 (m), 1139 (m), 1096 (w), 1030 (m), 724 (m). MS (EI, 70 eV): m/z (%) = 181 (M⁺, 37), 166 (5), 120 (3), 107 (100), 93 (23). The exact molecular mass $m/z = 181.1103 \pm 2$ ppm [M⁺] for C₁₀H₁₅O₂N was confirmed by HRMS (EI, 70 eV).

(3-Methoxy-1H-pyrrol-2-yl)-acetic acid methyl ester (5h): Starting with *cis*-**4h** (0.150 g, 0.75 mmol) in CH₂Cl₂ (15 mL), **5h** was isolated without further purification as a black oil (0.127g, 100%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 3.63$ (s, 2 H, CH₂), 3.72 (s, 3 H, OCH₃), 3.75 (s, 3 H, OCH₃), 5.95 (t, $J = 3.0$ Hz, 1 H, CH), 6.54 (t, $J = 3.0$ Hz, 1 H, CH), 8.25 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 29.3, 51.8, 58.8, 96.3, 106.9, 114.5, 144.6, 171.7$. IR (neat, cm⁻¹): $\tilde{\nu} = 3383$ (s), 2952 (w), 1735 (s), 1669 (w), 1604 (m), 1490 (w), 1463 (m), 1344 (w), 1308 (m), 1263 (s), 1206 (s), 1179 (m), 1152 (s), 1095 (m), 1077 (m), 1050 (w), 1015 (m). MS (EI, 70 eV): m/z (%) = 169 (M⁺, 40), 154 (s), 140 (12), 122 (2), 110 (100), 94 (14), 80 (11). Anal. calcd. for C₈H₁₁O₃N (169.180): C 56.80, H 6.55, N 8.28; found C 56.70, H 6.35, N 7.74.

(3-Propyl-1H-pyrrol-2-yl)-acetic acid ethyl ester (5j): Starting with **4j** (0.200 g, 0.88 mmol) in CH₂Cl₂ (20 mL), **5j** was isolated without further purification as a brownish oil (0.172 g, 100%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 0.93$ (t, $J = 7.2$ Hz, 3 H, CH₃), 1.27 (t, $J = 7.2$ Hz, 3 H, OCH₂CH₃), 1.55 (sextet, $J = 7.5$ Hz, 2 H, CH₂), 2.37 (t, $J = 7.2$ Hz, 2 H, CH₂), 3.59 (s, 2 H, CH₂), 4.17 (q, $J = 7.2$ Hz, 2 H, OCH₂), 6.03 (t, $J = 2.7$ Hz, 1 H, CH), 6.68 (t, $J = 2.7$ Hz, 1 H, CH), 8.56 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 150 MHz): $\delta_c = 14.15, 14.20, 24.5, 27.9,$

31.4, 61.0, 108.7, 116.6, 119.2, 121.2, 171.5. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3393 (s), 2960 (s), 2931 (s), 2871 (m), 1731 (s), 1639 (w), 1461 (m), 1399 (w), 1371 (m), 1327 (m), 1303 (m), 1248 (s), 1231 (s), 1181 (s), 1140 (s), 1098 (m), 1067 (w), 1031 (s), 721 (m). MS (EI, 70 eV): m/z (%) = 195 (M^+ , 48), 180 (1), 166 (49), 122 (100). The exact molecular mass m/z = 195.1259 $\pm$ 2 ppm [M^+] for $\text{C}_{11}\text{H}_{17}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

(3-Butyl-1H-pyrrol-2-yl)-acetic acid ethyl ester (5k): Starting with **4k** (0.020 g, 0.083 mmol) in CH_2Cl_2 (3 mL), **5k** was isolated without further purification as a brownish oil (0.017 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): δ = 0.91 (t, J = 7.2 Hz, 3 H, CH_3), 1.27 (t, J = 7.2 Hz, 3 H, OCH_2CH_3), 1.25 – 1.38 (m, 2 H, CH_2), 1.46 – 1.54 (m, 2 H, CH_2), 2.42 (t, J = 7.4 Hz, 2 H, CH_2), 3.58 (s, 2 H, CH_2), 4.17 (q, J = 7.2 Hz, 2 H, OCH_2), 6.03 (t, J = 2.7 Hz, 1 H, CH), 6.68 (t, J = 2.7 Hz, 1 H, CH), 8.55 (broad s, 1H, NH). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3390 (m), 2958 (m), 2928 (m), 2861 (w), 1731 (s), 1677 (m), 1645 (m), 1575 (w), 1549 (w), 1459 (m), 1402 (m), 1372 (m), 1328 (m), 1295 (m), 1257 (s), 1178 (s), 1139 (s), 1102 (m), 1072 (w), 1031 (m), 842 (w), 758 (w), 722 (m). MS (EI, 70 eV): m/z (%) = 209 (M^+ , 51), 193 (3), 180 (5), 166 (65), 136 (63), 120 (17), 106 (12), 94 (100). The exact molecular mass m/z = 209.1416 $\pm$ 2 ppm [M^+] for $\text{C}_{12}\text{H}_{19}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

(3-Hexyl-1H-pyrrol-2-yl)-acetic acid ethyl ester (5l): Starting with **4l** (0.250 g, 0.93 mmol) in CH_2Cl_2 (25 mL), **5l** was isolated without further purification as a brownish oil (0.221 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.27 (t, J = 7.2 Hz, 3 H, OCH_2CH_3), 1.21 – 1.36 (m, 6 H, $3\times\text{CH}_2$), 1.52 (quint, J = 7.6 Hz, 2 H, CH_2), 2.39 (t, J = 7.6 Hz, 2 H, CH_2), 3.58 (s, 2 H, CH_2), 4.17 (q, J = 7.2 Hz, 2 H, OCH_2), 6.02 (t, J = 2.7 Hz, 1 H, CH), 6.68 (t, J = 2.7 Hz, 1 H, CH), 8.50 (broad s, 1H, NH). ^{13}C NMR (CDCl_3 , 150 MHz): δ_c = 14.2, 14.3, 22.8, 25.9, 29.3, 29.8, 31.4, 31.9, 61.1, 108.7, 116.6, 119.2, 121.5, 171.5. IR

(neat, cm^{-1}): $\tilde{\nu}$ = 3390 (s), 2957 (s), 2927 (s), 2855 (s), 1730 (s), 1597 (w), 1463 (m), 1399 (w), 1371 (s), 21331 (m), 1300 (m), 1249 (s), 1197 (s), 1183 (s), 1165 (s), 1139 (s), 1102 (m), 1075 (w), 1030 (m), 720 (m). MS (EI, 70 eV): m/z (%) = 237 (M^+ , 100), 208 (2), 164 (94). The exact molecular mass m/z = 237.1729 ± 2 ppm [M^+] for $\text{C}_{14}\text{H}_{23}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

(3-Nonyl-1H-pyrrol-2-yl)-acetic acid ethyl ester (5n): Starting with **4n** (0.188 g, 0.604 mmol) in CH_2Cl_2 (20 mL), **5n** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish oil (0.157 g, 93%). ^1H NMR (CDCl_3 , 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.10 – 1.36 (m, 15 H, $6 \times \text{CH}_2$, OCH_2CH_3), 1.53 (quint, J = 7.2 Hz, 2 H, CH_2), 2.38 (t, J = 7.8 Hz, 2 H, CH_2), 3.58 (s, 2 H, CH_2), 4.17 (q, J = 7.2 Hz, 2 H, OCH_2), 6.02 (t, J = 2.7 Hz, 1 H, CH), 6.68 (t, J = 2.7 Hz, 1 H, CH), 8.54 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_{c} = 14.01, 14.04, 22.6, 25.7, 29.3, 29.45, 29.51, 29.54, 31.2, 31.3, 31.8, 60.9, 108.5, 116.4, 118.9, 121.3, 171.3. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3393 (m), 2957 (m), 2926 (s), 2854 (m), 1730 (s), 1463 (m), 1422 (w), 1401 (w), 1371 (w), 1332 (w), 1300 (w), 1249 (m), 1221 (m), 1188 (m), 1138 (m), 1102 (w), 1030 (m), 719 (m). UV-VIS (CH_2Cl_2 , nm): λ_{max} (log ϵ) = 226 (4.18), 317 (2.41). MS (EI, 70 eV): m/z (%) = 279 (M^+ , 50), 250 (3), 234 (1), 206 (41), 192 (5), 166 (98), 138 (5), 120 (14), 94 (100). The exact molecular mass m/z = 279.2198 ± 2 ppm [M^+] for $\text{C}_{17}\text{H}_{29}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

(3-Decyl-1H-pyrrol-2-yl)-acetic acid ethyl ester (5o): Starting with **4o** (0.085 g, 0.261 mmol) in CH_2Cl_2 (10 mL), **5o** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a brownish oil (0.070 g, 91%). ^1H NMR (CDCl_3 , 300 MHz): δ = 0.88 (t, J = 6.9 Hz, 3 H, CH_3), 1.14 – 1.36 (m, 17 H, $7 \times \text{CH}_2$, OCH_2CH_3), 1.49 (quint, J = 6.9 Hz, 2 H, CH_2), 2.38 (t, J = 7.8 Hz, 2 H, CH_2), 3.58 (s, 2 H, CH_2), 4.17 (q, J = 7.2 Hz, 2 H, OCH_2), 6.02

(t, $J = 2.7$ Hz, 1 H, CH), 6.68 (t, $J = 2.7$ Hz, 1 H, CH), 8.54 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta_{\text{c}} = 14.1, 14.14, 22.7, 25.7, 29.3, 29.5, 29.63, 29.64, 31.2, 31.3, 31.9, 61.0, 108.6, 116.5, 119.0, 121.3, 171.4$. IR (neat, cm^{-1}): $\tilde{\nu} = 3394$ (w), 2957 (w), 2926 (s), 2859 (s), 1730 (s), 1463 (m), 1371 (w), 1332 (w), 1301 (w), 1248 (m), 1182 (m), 1140 (m), 1030 (m), 719 (w). UV-VIS (CH_2Cl_2 , nm): λ_{max} ($\log \epsilon$) = 225 (3.67), 227 (3.67), 460 (2.21). MS (EI, 70 eV): m/z (%) = 293 (M^+ , 71), 264 (2), 220 (44), 166 (100), 138 (4), 120 (15), 94 (97). The exact molecular mass $m/z = 209.1416 \pm 2$ ppm [M^+] for $\text{C}_{12}\text{H}_{19}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

4,5,6,7,8,9,10,11,12,13-Decahydro-1H-cyclododeca[b]pyrrole-13-carboxylic acid ethyl ester (5p): Starting with **4p** (0.800 g, 2.59 mmol) in CH_2Cl_2 (40 mL), **5p** was isolated without further purification as a black solid (0.719 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.25$ (t, $J = 7.11$ Hz, 3 H, CH_3), 1.18 – 1.43 (m, 12 H, $6 \times \text{CH}_2$), 1.54 – 1.68 (m, 2 H, CH_2), 1.88 – 1.95 (m, 2 H, CH_2), 2.40 – 2.51 (m, 2 H, OCH_2), 3.84 (t, $J = 7.5$ Hz, 1 H, CH), 4.06 – 4.20 (m, 2 H, OCH_2), 6.00 (t, $J = 2.7$ Hz, 1 H, CH), 6.71 (t, $J = 2.7$ Hz, 1 H, CH), 8.39 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 50 MHz): $\delta_{\text{c}} = 14.2, 22.1, 22.3, 22.5, 24.1, 24.16, 24.2, 24.7, 29.0, 32.7, 39.3, 60.8, 108.0, 117.2, 121.2, 124.6, 174.9$. IR (KBr, cm^{-1}): $\tilde{\nu} = 3345$ (s), 2928 (s), 2860 (m), 1721 (s), 1458 (w), 1232 (m), 1201 (m), 1153 (m). MS (EI, 70 eV): m/z (%) = 277 (M^+ , 100), 248 (1), 204 (100). UV-VIS (CH_2Cl_2 , nm): λ_{max} ($\log \epsilon$) = 231 (3.71). The exact molecular mass $m/z = 277.2042 \pm 2$ ppm [M^+] for $\text{C}_{17}\text{H}_{27}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV). The structure was confirmed by X-Ray crystallography.

4,5,6,7-Tetrahydro-1H-indole-7-carboxylic acid ethyl ester (5q): Starting with *trans*-**4q** (0.103 g, 0.46 mmol) in 1,4-dioxane (10 mL), **5q** was isolated without further purification as a red oil (0.089 g, 100%). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 1.30$ (t, $J = 7.2$ Hz, 3 H, CH_3), 1.64

– 1.77 (m, 1 H, CH₂), 1.88 – 2.00 (m, 1 H, CH₂), 2.01 – 2.11 (m, 2 H, CH₂), 2.52 (dt, $J = 1.5$, 6.2 Hz, 2 H, CH₂), 3.68 (t, $J = 6.9$ Hz, 1 H, CH), 8.41 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 14.1, 22.3, 22.6, 26.0, 39.5, 60.8, 107.1, 116.8, 118.3, 122.3, 173.0$. IR (neat, cm⁻¹): $\tilde{\nu} = 3393$ (s), 2977 (s), 2935 (s), 2856 (m), 1724 (s), 1591 (w), 1448 (s), 1400 (w), 1369 (m), 1292 (s), 1248 (s), 1216 (s), 1180 (s), 1095 (s), 12054 (w), 1027 (s), 852 (w), 722 (m). MS (EI, 70 eV): m/z (%) = 193 (M⁺, 27), 120 (100). The exact molecular mass $m/z = 193.1103 \pm 2$ ppm (M⁺) for C₁₁H₁₅O₂N was confirmed by HRMS (EI, 70 eV).

4,5,6,7,8,9-Hexahydro-1H-cycloocta[b]pyrrole-9-carboxylic acid ethyl ester (5r): Starting with *cis*-**4r** (0.040 g, 0.158 mmol) in 1,4-dioxane (5 mL), **5r** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 100:1 → 30:1) as a brownish oil (0.029 g, 83%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.10 - 1.29$ (m, 2 H, CH₂), 1.33 (t, $J = 7.2$ Hz, 3 H, CH₃), 1.34 – 1.44 (m, 2 H, CH₂), 1.65 – 1.72 (m, 2 H, CH₂), 1.80 – 1.88 (m, 1 H, CH₂), 2.04 – 2.09 (m, 1 H, CH₂), 2.40 – 2.44 (m, 1 H, CH₂), 2.69 – 2.75 (m, 1 H, CH₂), 3.88 (dd, $J = 12.3, 1.5$ Hz, 1 H, CH), 4.21 (q, $J = 7.2$ Hz, 2 H, OCH₂), 5.96 (t, $J = 1.8$ Hz, 1 H, CH), 6.65 (t, $J = 1.8$ Hz, 1 H, CH), 8.95 (broad s, 1 H, NH). IR (neat, cm⁻¹): $\tilde{\nu} = 3368$ (w), 2960 (w), 2925 (m), 2853 (w), 1721 (m), 1477 (w), 1434 (s), 1261 (w), 1197 (s), 1180 (s), 1118 (s), 1092 (m), 1070 (m), 1025 (m). MS (EI, 70 eV): m/z (%) = 221 (M⁺, 30), 192 (4), 148 (100).

5-Phenyl-4,5,6,7-tetrahydro-1H-indole-7-carboxylic acid methyl ester (5t): Starting with **4t** (0.050 g, 0.174 mmol) in 1,4-dioxane (5 mL), **5t** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a brownish oil (0.035 g, 79%). ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.85 - 2.04$ (m, 2 H, CH₂), 2.06 – 2.16 (m, 1 H, CH₂), 2.46 – 2.52 (m, 1 H, CH), 2.64 – 2.73 (m, 1 H, CH₂), 2.82 – 2.89 (dd, $J = 15.6, 5.1$ Hz, 1 H, CH₂), 3.17 – 3.27 (m, 1 H, CH), 3.73 (s, 1 H, CH), 6.03 (t, $J = 1.8$ Hz, 1 H, CH), 6.74 (t, $J = 1.8$ Hz, 1 H, CH), 7.17 – 7.36 (m, 5 H, 5×CH of Ph), 8.29 (broad s, 1 H, NH). IR (neat, cm⁻¹): $\tilde{\nu} = 3404$ (w), 2950 (w),

2926 (w), 1731 (s), 1646 (w), 1444 (m), 1232 (s), 1201 (s), 1174 (m), 1162 (m), 1108 (m), 702 (m). MS (EI, 70 eV): m/z (%) = 255 (M^+ , 84), 196 (82), 118 (100), 104 (7), 91 (39), 77 (42). HRMS (ESI): calcd. for $C_{16}H_{18}O_2N$ ($[M+1]^+$) = 256.13375; found = 256.13313.

1-(1H-Pyrrol-2-yl)-propan-2-one (5u): Starting with **4u** (0.050 g, 0.32 mmol) in 1,4-dioxane (5 mL), **5u** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 10:1 $\rightarrow$ 1:1) as a brownish solid (0.034 g, 87%). 1H NMR ($CDCl_3$, 300 MHz): δ = 2.20 (s, 3 H, CH_3), 3.74 (s, 2 H, CH_2), 6.00 (m, 1 H, CH), 6.15 (dd, J = 6.0, 3.0 Hz, 1 H, CH), 6.75 (m, 1 H, CH), 8.62 (broad s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 29.5, 42.1, 107.4, 108.3, 117.8, 123.8, 206.8. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3328 (s), 2912 (w), 1706 (s), 1427 (w), 1390 (w), 1358 (w), 1331 (w), 1286 (w), 1234 (w), 1168 (w), 1127 (w), 1029 (w), 799 (m), 758 (m), 728 (m), 607 (w), 561 (w). MS (EI, 70 eV): m/z (%) = 123 (M^+ , 22), 82 (100). The exact molecular mass m/z = 123.0684 $\pm$ 2 ppm [M^+] for C_7H_9ON was confirmed by HRMS (EI, 70 eV).

General procedure for the TFA reactions of pyrrolidines (4): To a CH_2Cl_2 -solution (20 mL/mmol) of **4** (1 equiv.) was added TFA (13 equiv.) and the solution was stirred for 24 h at 20 °C. The solvent was removed *in vacuo* and the residue was purified by chromatography (silica gel, *n*-hexane/EtOAc) to give **5**, **6**, and **7**.

5a, [5-(2,2,2-Trifluoro-acetyl)-1H-pyrrol-2-yl]-acetic acid methyl ester (6a), (7-Methoxycarbonylmethyl-1H-indol-2-yl)-acetic acid methyl ester (7a): Starting with **4a** (0.150 g, 0.88 mmol) and TFA (0.88 mL, 11.4 mmol) in CH_2Cl_2 (15 mL), **5-6a** and **7a** were isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as black solid and brownish oil (0.047 g {0.039 g, 32%; 0.008 g, 4%}; 0.056 g, 49% = 85%). [Inseparable mixture of **5a/6a** = 4.5:1].

5a, 6a: [*Signals of **6a**] ^1H NMR (CDCl_3 , 300 MHz): δ = 3.69 (s, 2 H, CH_2), 3.72 (s, 3 H, OCH_3), *3.77 (s, 2 H, CH_2), *3.78 (s, 3 H, OCH_3), 6.01 (m, 1 H, CH), 6.13 – 6.16 (m, 1 H, CH), *6.23 (m, 1 H, CH), 6.76 (m, 1 H, CH), 7.13 (m, 1 H, CH), 8.71 (broad s, 1 H, NH), *9.98 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = *32.9, 33.0, 52.1, *52.6, 107.4, 108.3, *112.3, 115.1, 117.8, *118.9, *122.0, *123.1, *125.8, *135.6, *169.7, 171.7. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3413 (w), 3298 (m), 2925 (w), 1746 (s), 1650 (s), 1437 (m), 1353 (m), 1261 (m), 1201 (s), 1145 (s), 1103 (w), 1062 (m), 1027 (w), 886 (w), 804 (m), 766 (m). MS (EI, 70 eV): m/z (%) = *235 (M^+ , 60), *176 (100), *166 (47), 139 (M^+ , 1), 124 (1), 106 (52), 79 (36). The exact molecular mass m/z = 139.0633 ± 2 ppm [M^+] for $\text{C}_7\text{H}_9\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

7a: ^1H NMR (CDCl_3 , 300 MHz): δ = 3.67 (s, 3 H, OCH_3), 3.74 (s, 3 H, OCH_3), 3.84 (s, 2 H, CH_2), 3.86 (s, 2 H, CH_2), 6.40 (m, 1 H, CH), 6.98 (d, J = 7.2 Hz, 1 H, CH), 7.11 (t, J = 7.2 Hz, 1 H, CH), 7.26 (d, J = 7.2 Hz, 1 H, CH), 8.76 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 33.7, 39.2, 51.9, 52.3, 100.3, 110.1, 120.7, 121.9, 125.4, 127.8, 130.5, 136.3, 171.0, 172.2. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3374 (w), 2954 (w), 1735 (s), 1641 (m), 1551 (w), 1437 (m), 1340 (w), 1262 (m), 1203 (m), 1161 (s), 1103 (w), 1045 (w), 1015 (w), 769 (w). MS (EI, 70 eV): m/z (%) = 261 (M^+ , 74), 215 (1), 202 (100), 166 (8), 143 (32), 115 (10). HRMS (ESI): calcd. for $\text{C}_{14}\text{H}_{16}\text{O}_4\text{N}$ ($[\text{M}+1]^+$) = 262.10793; found = 262.10718.

[5-(2,2,2-Trifluoro-acetyl)-1H-pyrrol-2-yl]-acetic acid ethyl ester (6b), (7-Ethoxycarbonylmethyl-1H-indol-2-yl)-acetic acid ethyl ester (7b): Starting with **4b** (0.100 g, 0.54 mmol) and TFA (0.54 mL, 7.0 mmol) in CH_2Cl_2 (10 mL), **6b** and **7b** were isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 100:1 $\rightarrow$ 1:1) as black solid and brownish oil (0.051 g, 38%; 0.042 g, 54%; combined yield: 92%).

6b: ^1H NMR (CDCl_3 , 300 MHz): δ = 1.29 (t, J = 7.2 Hz, 3 H, CH_3), 3.76 (s, 2 H, CH_2), 4.24 (q, J = 7.2 Hz, 2 H, OCH_2), 6.24 (m, 1 H, CH), 7.15 (m, 1 H, CH), 10.07 (broad s, 1 H, NH). MS (EI, 70 eV): m/z (%) = 249 (M^+ , 35), 180 (21), 176 (100), 105 (32), 79 (41).

7b: ^1H NMR (CDCl_3 , 300 MHz): δ = 1.24 (t, J = 7.2 Hz, 3 H, CH_3), 1.29 (t, J = 7.2 Hz, 3 H, CH_3), 3.82 (s, 2 H, CH_2), 3.85 (s, 2 H, CH_2), 4.14 (q, J = 7.2 Hz, 2 H, OCH_2), 4.19 (q, J = 7.2 Hz, 2 H, OCH_2), 6.40 (m, 1 H, CH), 6.98 (d, J = 7.2 Hz, 1 H, CH), 7.10 (t, J = 7.2 Hz, 1 H, CH), 7.26 (d, J = 7.2 Hz, 1 H, CH), 8.80 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 14.1, 14.2, 34.0, 39.4, 61.0, 61.5, 100.3, 110.1, 120.7, 121.9, 125.5, 127.9, 130.6, 136.3, 170.8, 172.3. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3373 (w), 2982 (m), 2936 (w), 1729 (s), 1616 (w), 1464 (w), 1437 (m), 1396 (w), 1370 (m), 1342 (m), 1298 (m), 1259 (s), 1224 (s), 1178 (s), 1159 (s), 1097 (w), 1030 (m), 768 (m). MS (EI, 70 eV): m/z (%) = 289 (M^+ , 54), 216 (100), 188 (15), 158 (2), 143 (65), 130 (3), 114 (19). HRMS (ESI): calcd. for $\text{C}_{16}\text{H}_{20}\text{O}_4\text{N}$ ($[\text{M}+1]^+$) = 290.13923; found = 290.13829.

(7-Isopropoxycarbonylmethyl-1H-indol-2-yl)-acetic acid isopropyl ester (7c): Starting with **4c** (0.100 g, 0.5 mmol) and TFA (0.50 mL, 6.5 mmol) in CH_2Cl_2 (10 mL), **7c** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc}$ = 50:1 $\rightarrow$ 1:1) as a brownish oil (0.072 g, 91%). ^1H NMR (CDCl_3 , 300 MHz): δ = 1.22 (d, J = 6.3 Hz, 6 H, $2\times\text{CH}_3$), 1.27 (d, J = 6.3 Hz, 6 H, $2\times\text{CH}_3$), 3.80 (s, 2 H, CH_2), 3.81 (s, 2 H, CH_2), 5.02 (quint, J = 6.3 Hz, 1 H, OCH), 5.07 (quint, J = 6.3 Hz, 1 H, OCH), 6.40 (m, 1 H, CH), 6.98 (d, J = 7.2 Hz, 1 H, CH), 7.11 (t, J = 7.2 Hz, 1 H, CH), 7.26 (d, J = 7.2 Hz, 1 H, CH), 8.78 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 21.75, 21.77, 34.2, 39.7, 68.0, 69.0, 100.2, 109.9, 120.5, 121.7, 125.7, 127.9, 130.7, 136.3, 170.1, 171.3. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3373 (m), 2981 (s), 2935 (m), 2877 (w), 1729 (s), 1609 (w), 1553 (w), 1464 (w), 1435 (m), 1374 (m), 1343 (m), 1262 (s), 1175 (s), 1224 (s), 1150 (s), 1106 (s), 1043 (w), 964 (w), 766 (m). MS (EI, 70 eV): m/z (%) =

317 (M^+ , 72), 275 (19), 230 (73), 188 (100), 143 (44), 115 (12). HRMS (ESI): calcd. for $C_{18}H_{24}O_4N$ ($[M+1]^+$) = 318.17053; found = 318.16991.

5d, [5-(2,2,2-Trifluoro-acetyl)-1H-pyrrol-2-yl]-acetic acid 2-methoxy-ethyl ester (**6d**), [7-(2-Methoxy-ethoxycarbonylmethyl)-1H-indol-2-yl]-acetic acid 2-methoxy-ethyl ester (**7d**): Starting with **4d** (0.050 g, 0.23 mmol) and TFA (0.23 mL, 3.02 mmol) in CH_2Cl_2 (5 mL), **5-6d** and **7d** were isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as black and brownish oils (0.019 g {0.008 g, 19%; 0.011 g, 17% }; 0.018, 45% = 81%). [Inseparable mixture of **5d/6d** = 1:1.4].

5d, **6d**: [*Signals of **6d**] 1H NMR ($CDCl_3$, 300 MHz): δ = 3.40 (s, 3 H, OCH_3), 3.42 (s, 3 H, OCH_3), 3.60 – 3.67 (m, 4 H, $2 \times OCH_2$), *3.72 (s, 2 H, CH_2), 3.80 (s, 2 H, CH_2), 4.27 – 4.30 (m, 2 H, OCH_2), 4.32 – 4.36 (m, 2 H, OCH_2), 6.02 (m, 1 H, CH), 6.12 – 6.15 (m, 1 H, CH), *6.23 (m, 1 H, CH), 6.75 (m, 1 H, CH), *7.13 (m, 1 H, CH), 8.78 (broad s, 1 H, NH), *10.18 (broad s, 1 H, NH). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3375 (w), 3300 (m), 2978 (w), 2954 (w), 2926 (w), 2893 (w), 2856 (w), 1741 (m), 1653 (m), 1503 (w), 1452 (w), 1435 (w), 1407 (w), 1373 (w), 1342 (w), 1259 (m), 1192 (s), 1145 (s), 1102 (m), 1059 (m), 1034 (m), 883 (w), 804 (w), 765 (m), 731 (w). MS (EI, 70 eV): m/z (%) = *279 (M^+ , 1), *203 (11), 183 (M^+ , 18), *176 (6), 107 (34), 80 (100). The exact molecular mass m/z = 183.0895 ± 2 ppm [M^+] for $C_9H_{13}O_3N$ was confirmed by HRMS (EI, 70 eV).

7d: 1H NMR ($CDCl_3$, 300 MHz): δ = 3.35 (s, 3 H, OCH_3), 3.42 (s, 3 H, CH_3), 3.56 – 3.67 (m, 4 H, $2 \times OCH_2$), 3.88 (s, 2 H, CH_2), 3.89 (s, 2 H, CH_2), 4.23 – 4.26 (m, 2 H, OCH_2), 4.29 – 4.34 (m, 2 H, OCH_2), 6.42 (m, 1 H, CH), 6.99 (d, J = 7.2 Hz, 1 H, CH), 7.11 (t, J = 7.2 Hz, 1 H, CH), 7.26 (d, J = 7.2 Hz, 1 H, CH), 8.89 (broad s, 1 H, NH). IR (neat, cm^{-1}): $\tilde{\nu}$ = 3364 (w), 2928 (w), 1736 (s), 1643 (m), 1552 (w), 1441 (m), 1407 (m), 1376 (w), 1325 (w), 1254 (m), 1159 (s), 1129 (s), 1037 (m), 769 (w). MS (EI, 70 eV): m/z (%) = 349 (M^+ , 23), 199 (14),

167 (27), 143 (30), 141 (100), 114 (12), 104 (58). HRMS (ESI): calcd. for $C_{18}H_{24}O_6N$ ($[M+1]^+$) = 350.16036; found = 350.15946.

2,2,2-Trifluoro-1-[5-(2-oxo-2-phenyl-ethyl)-1H-pyrrol-2-yl]-ethanone (6e): Starting with **4e** (0.200 g, 0.92 mmol) and TFA (0.9 mL, 12.0 mmol) in CH_2Cl_2 (20 mL), **6e** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a black solid (0.139 g, 54%). M.p. = 121.7 °C. 1H NMR ($CDCl_3$, 300 MHz): δ = 4.44 (s, 2 H, CH_2), 6.28 (dd, J = 3.9, 2.7 Hz, 1 H, CH), 7.16 (m, J = 2.0 Hz, 1 H, CH), 7.54 (m, 2 H, 2 $\times$ CH from Ph), 7.61 – 7.66 (m, 1 H, CH from Ph), 8.00 – 8.07 (m, 2 H, 2 $\times$ CH from Ph), 10.34 (broad s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 36.5, 112.6, 115.2, 119.0, 122.0, 125.9, 128.4, 129.0, 134.1, 135.9, 136.7, 195.2. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3295 (m), 2962 (w), 2925 (w), 1692 (m), 1641 (s), 1559 (w), 1497 (w), 1448 (w), 1430 (w), 1406 (w), 1355 (w), 1332 (w), 1261 (m), 1187 (m), 1142 (s), 1098 (m), 1063 (m), 1024 (m), 883 (w), 794 (m), 758 (m), 687 (w). MS (EI, 70 eV): m/z (%) = 281 (M^+ , 4), 212 (5), 176 (4), 105 (100), 77 (45). HRMS (ESI): calcd. for $C_{14}H_{11}O_2NF_3$ ($[M+1]^+$) = 282.07419; found = 282.07346.

2-(2,2,2-Trifluoro-acetyl)-4,5,6,7,8,9-hexahydro-1H-cycloocta[b]pyrrole-9-carboxylic acid ethyl ester (6f): Starting with *trans*-**4r** (0.040 g, 0.158 mmol) and TFA (0.16 mL, 2.05 mmol) in CH_2Cl_2 (5 mL), **6f** was isolated after chromatography (silica gel, *n*-hex/EtOAc = 100:1 $\rightarrow$ 10:1) as a reddish oil (0.029 g, 58%). 1H NMR ($CDCl_3$, 300 MHz): δ = 1.10 – 1.29 (m, 2 H, CH_2), 1.33 (t, J = 7.2 Hz, 3 H, CH_3), 1.34 – 1.44 (m, 2 H, CH_2), 1.61 – 1.75 (m, 2 H, CH_2), 1.80 – 1.88 (m, 1 H, CH_2), 2.04 – 2.13 (m, 1 H, CH_2), 2.36 – 2.45 (m, 1 H, CH_2), 2.69 – 2.77 (m, 1 H, CH_2), 3.91 (dd, J = 12.2, 4.8 Hz, 1 H, CH), 4.26 (dq, J = 7.2, 1.5 Hz, 2 H, OCH_2), 6.93 (t, J = 2.1 Hz, 1 H, CH), 10.23 (broad s, 1 H, NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 14.2, 25.0, 25.7, 26.0, 31.8, 35.3, 40.8, 61.7, 115.3, 119.1, 121.0, 124.2, 126.6, 137.9, 173.7. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3411 (w), 2931 (m), 2858 (w), 1725 (m), 1660 (s), 1471 (m),

1446 (w), 1408 (w), 1378 (w), 1333 (w), 1299 (w), 1257 (m), 1236 (m), 1158 (s), 1144 (s), 1096 (w), 1078 (w), 1027 (w), 898 (m), 765 (w). MS (EI, 70 eV): m/z (%) = 317 (M^+ , 42), 289 (3), 248 (9), 244 (100), 202 (21), 190 (41), 174 (13), 145 (20). HRMS (ESI): calcd. for $C_{15}H_{19}O_3NF_3$ ($[M+1]^+$) = 318.13171; found = 318.13083.

General procedure for the preparation of pyrroles (9) and (12): To a THF-solution (10 mL/mmol) of **11** (1 equiv.) was added triphenylphosphine (1.2 equiv.) at room temperature. The reaction mixture was heated and stirred for 4 h at 80 °C. After cooling to 20 °C, the solvent was removed *in vacuo* and the residue was purified by chromatography (silica gel, *n*-hexane/EtOAc) to give **12**.

2-1H-Pyrrole-2-ylmethyl-1H-pyrrole (9): Starting with **8** (0.100 g, 0.34 mmol), and PPh_3 (0.198 g, 0.75 mmol) in THF (10 mL), **9** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 75:1 → 10:1) as a brownish solid (0.041 g, 83%). 1H NMR ($CDCl_3$, 300 MHz): δ = 3.97 (s, 2 H, CH_2), 6.02 – 6.042 (m, 2 H, 2×CH), 6.14 (dd, J = 5.7, 2.7 Hz, 2 H, 2×CH), 6.64 (dd, J = 4.2, 2.7 Hz, 2 H, 2×CH), 6.64 (dd, J = 4.2, 2.7 Hz, 2 H, 2×CH), 7.83 (broad s, 2 H, 2×NH). ^{13}C NMR ($CDCl_3$, 75 MHz): δ_c = 26.4, 106.4, 108.4, 117.2, 129.0. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3333 (s), 2923 (w), 1631 (w), 1564 (w), 1468 (w), 1439 (w), 1425 (w), 1332 (w), 1261 (w), 1250 (w), 1183 (w), 1114 (m), 1094 (m), 1026 (m), 804 (m), 729 (s), 682 (w), 689 (w), 625 (w), 586 (m), 549 (w). MS (EI, 70 eV): m/z (%) = 146 (M^+ , 100), 80 (39), 67 (26). The exact molecular mass m/z = 146.0844 ± 2 ppm [M^+] for $C_9H_{10}N_2$ was confirmed by HRMS (EI, 70 eV).

2-Methyl-1H-pyrrole-3-carboxylic acid methyl ester (12a): Starting with **11a** (0.030 g, 0.14 mmol) and PPh_3 (0.044 g, 0.17 mmol) in THF (5 mL), **12a** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 5:1) as a brownish solid (0.017 g, 85%).

^1H NMR (CDCl_3 , 300 MHz): δ = 2.53 (s, 3 H, CH_3), 3.80 (s, 3 H, OCH_3), 6.55 (d, J = 2.7 Hz, 1 H, CH), 6.56 (d, J = 2.7 Hz, 1 H, CH), 8.23 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 13.2, 50.8, 110.4, 110.5, 115.8, 135.3, 166.1. IR (neat, cm^{-1}): $\tilde{\nu}$ = 3312 (m), 2978 (s), 2952 (w), 2933 (w), 2867 (m), 1707 (s), 1680 (s), 1607 (w), 1580 (m), 1493 (w), 1452 (s), 1408 (w), 1381 (m), 1348 (w), 1329 (m), 1268 (m), 1226 (w), 1202 (m), 1177 (w), 1125 (s), 1076 (w), 1053 (w), 1026 (w), 900 (w), 844 (w), 790 (w), 726 (w). MS (EI, 70 eV): m/z (%) = 139 (M^+ , 46), 124 (19), 107 (100), 80 (33). The exact molecular mass m/z = 139.0633 $\pm$ 2 ppm [M^+] for $\text{C}_7\text{H}_9\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

2-Methyl-1H-pyrrole-3-carboxylic acid ethyl ester (12b): Starting with **11b** (0.100 g, 0.44 mmol) and PPh_3 (0.137 g, 0.52 mmol) in THF (10 mL), **12b** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 5:1) as a brownish solid (0.061 g, 91%). M.p. = 64 $^\circ\text{C}$. ^1H NMR (CDCl_3 , 300 MHz): δ = 1.34 (t, J = 7.2 Hz, 3 H, OCH_2CH_3), 2.52 (s, 3 H, CH_3), 4.27 (q, J = 7.2 Hz, 2 H, OCH_2), 6.56 (d, J = 2.4 Hz, 2 H, 2 $\times$ CH), 8.51 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): δ_c = 13.2, 14.5, 59.4, 110.4, 111.7, 15.7, 135.2, 165.8. IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3294 (s), 2985 (m), 2938 (w), 2906 (w), 1697 (s), 1675 (s), 1578 (m), 1499 (m), 1447 (m), 1411 (m), 1369 (m), 1325 (s), 1267 (s), 1227 (w), 1201 (s), 1127 (s), 1095 (s), 1053 (s), 901 (m), 781 (m), 754 (m), 722 (m). MS (EI, 70 eV): m/z (%) = 153 (M^+ , 95), 124 (61), 108 (100). The exact molecular mass m/z = 153.0790 $\pm$ 2 ppm [M^+] for $\text{C}_8\text{H}_{11}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

2-Methyl-1H-pyrrole-3-carboxylic acid 2-methoxy-ethyl ester (12c): Starting with crude **11c** (0.259 g, 1.0 mmol) and PPh_3 (0.315 g, 1.2 mmol) in THF (10 mL), **12c** was isolated after chromatography (silica gel, n -Hex/EtOAc = 100:1 $\rightarrow$ 1:1) as a black oil (0.129 g, 71% over two steps). ^1H NMR (CDCl_3 , 300 MHz): δ = 2.53 (s, 3 H, CH_3), 3.42 (s, 3 H, OCH_3), 3.67 –

3.71 (m, 2 H, OCH₂), 4.35 – 4.39 (m, 2 H, OCH₂), 6.55 (t, $J = 3.0$ Hz, 1 H, CH), 6.58 (t, $J = 3.0$ Hz, 1 H, CH), 8.35 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 13.0, 58.8, 63.7, 70.8, 110.4, 111.2, 115.8, 135.5, 165.5$. IR (neat, cm⁻¹): $\tilde{\nu} = 3359$ (m), 2932 (m), 2895 (m), 2825 (w), 1717 (s), 1643 (m), 1577 (w), 1452 (m), 1409 (m), 1369 (m), 1320 (s), 1264 (s), 1197 (s), 1178 (s), 1152 (s), 1227 (s), 1097 (s), 1037 (s), 861 (w), 790 (w), 728 (m). MS (EI, 70 eV): m/z (%) = 183 (M⁺, 25), 125 (11), 107 (46), 80 (100). The exact molecular mass $m/z = 183.0895 \pm 2$ ppm [M⁺] for C₉H₁₃O₃N was confirmed by HRMS (EI, 70 eV).

1-(2-Methyl-1H-pyrrol-3-yl)-ethanone (12d): Starting with **11d** (0.100 g, 0.55 mmol) and PPh₃ (0.172 g, 0.66 mmol) in THF (10 mL), **12d** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 30:1 → 2:1) as a brownish solid (0.066 g, 97%). M.p. = 75 °C. ¹H NMR (CDCl₃, 300 MHz): $\delta = 2.43$ (s, 3 H, CH₃), 2.55 (s, 3 H, CH₃), 6.52 – 6.54 (m, 1 H, CH), 6.57 – 6.59 (m, 1 H, CH), 8.52 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 13.8, 28.3, 110.5, 115.9, 120.5, 135.4, 195.8$. IR (KBr, cm⁻¹): $\tilde{\nu} = 3211$ (s), 3110 (s), 3021 (m), 2981 (w), 2962 (m), 2926 (m), 2843 (w), 1636 (s), 1569 (s), 1491 (s), 1465 (s), 1402 (s), 1364 (s), 1333 (w), 1279 (m), 944 (m), 892 (m), 858 (w), 799 (w), 775 (w), 733 (m), 677 (w), 636 (w). MS (EI, 70 eV): m/z (%) = 123 (M⁺, 44), 107 (100), 94 (3), 80 (25), 66 (2), 43 (11). Anal. calcd. for C₇H₉ON (123.155): C 68.27, H 7.37, N 11.37; found C 68.12, H 7.15, N 11.53.

2-But-3-enyl-1H-pyrrole-3-carboxylic acid ethyl ester (12e): Starting with crude **11e** (0.269 g, 1.0 mmol) and PPh₃ (0.315 g, 1.2 mmol) in THF (10 mL), **12e** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 1:1) as a black oil (0.135 g, 70% over two steps). ¹H NMR (CDCl₃, 300 MHz): $\delta = 1.34$ (t, $J = 7.2$ Hz, 3 H, CH₃), 2.42 (dq, $J = 7.5, 1.2$ Hz, 2 H, CH₂), 3.07 (t, $J = 7.5$ Hz, 2 H, CH₂), 4.27 (q, $J = 7.2$ Hz, 2 H, OCH₂), 5.01 – 5.11 (m, 2 H, CH=CH₂), 5.80 – 5.93 (m, 1 H, CH=CH₂), 6.57 (m, 2 H, 2×CH), 8.26 (broad s, 1 H,

NH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta_{\text{c}} = 13.0, 58.8, 63.7, 70.8, 110.4, 111.2, 115.8, 135.5, 165.5$. IR (neat, cm^{-1}): $\tilde{\nu} = 3389$ (m), 2981 (m), 2957 (m), 2906 (w), 1732 (s), 1640 (w), 1462 (w), 1446 (w), 1370 (m), 1326 (m), 1306 (m), 1250 (s), 1181 (s), 1158 (s), 1098 (w), 1080 (w), 1030 (s), 998 (w), 914 (w), 857 (s), 723 (m). MS (EI, 70 eV): m/z (%) = 193 (M^+ , 56), 178 (2), 164 (3), 146 (3), 120 (100), 106 (19), 93 (14), 80 (9), 65 (5). The exact molecular mass $m/z = 193.1103 \pm 2$ ppm [M^+] for $\text{C}_{11}\text{H}_{15}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

2-Propyl-1H-pyrrole-3-carboxylic acid ethyl ester (12f): Starting with crude **11f** (0.257 g, 1.0 mmol) and PPh_3 (0.315 g, 1.2 mmol) in THF (10 mL), **12f** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc} = 50:1 \rightarrow 3:1$) as a brownish oil (0.099 g, 55% over two steps). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 0.95$ (t, $J = 7.2$ Hz, 3 H, CH_3), 1.34 (t, $J = 7.2$ Hz, 3 H, CH_3), 1.67 (sext, $J = 7.5$ Hz, 2 H, CH_2), 2.92 (t, $J = 7.5$ Hz, 2 H, CH_2), 4.27 (q, $J = 7.2$ Hz, 2 H, OCH_2), 6.54 – 6.58 (m, $J = 2.7$ Hz, 2 H, $2\times\text{CH}$), 8.31 (broad s, 1 H, NH). ^{13}C NMR (CDCl_3 , 75 MHz): $\delta_{\text{c}} = 13.7, 14.4, 22.7, 29.2, 59.3, 110.3, 111.1, 115.8, 139.9, 165.8$. IR (neat, cm^{-1}): $\tilde{\nu} = 3345$ (m), 2959 (m), 2873 (w), 1645 (s), 1582 (m), 1551 (m), 1491 (w), 1459 (m), 1448 (m), 1375 (m), 1305 (m), 1251 (s), 1174 (s), 1131 (s), 1103 (m), 1040 (m), 1019 (m), 843 (w), 759 (w). MS (EI, 70 eV): m/z (%) = 181 (M^+ , 62), 166 (2), 152 (100), 136 (45), 124 (87), 120 (11), 108 (49). The exact molecular mass $m/z = 181.1103 \pm 2$ ppm [M^+] for $\text{C}_{10}\text{H}_{15}\text{O}_2\text{N}$ was confirmed by HRMS (EI, 70 eV).

2-Butyl-1H-pyrrole-3-carboxylic acid ethyl ester (12g): Starting with crude **11g** (0.271 g, 1.0 mmol) and PPh_3 (0.315 g, 1.2 mmol) in THF (10 mL), **12g** was isolated after chromatography (silica gel, $n\text{-Hex/EtOAc} = 100:1 \rightarrow 5:1$) as a brownish oil (0.123 g, 63% over two steps). ^1H NMR (CDCl_3 , 300 MHz): $\delta = 0.93$ (t, $J = 7.2$ Hz, 3 H, CH_3), 1.34 (t, $J =$

7.2 Hz, 3 H, CH₃), 1.25 – 1.41 (m, 2 H, CH₂), 1.57 – 1.65 (m, 2 H, CH₂), 2.95 (t, J = 7.8 Hz, 2 H, CH₂), 4.27 (q, J = 7.2 Hz, 2 H, OCH₂), 6.57 (d, J = 2.7 Hz, 2 H, 2×CH), 8.28 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): δ_c = 13.8, 14.5, 22.5, 27.0, 31.6, 59.3, 110.5, 111.3, 115.7, 139.9, 165.5. IR (neat, cm⁻¹): $\tilde{\nu}$ = 3349 (w), 2960 (s), 2933 (m), 2871 (w), 1676 (s), 1464 (s), 1395 (w), 1371 (w), 1327 (m), 1301 (w), 1262 (m), 1182 (s), 1128 (s), 1039 (m). MS (EI, 70 eV): m/z (%) = 195 (M⁺, 48), 180 (1), 166 (49), 122 (100). The exact molecular mass m/z = 195.1259 ± 2 ppm [M⁺] for C₁₁H₁₇O₂N was confirmed by HRMS (EI, 70 eV).

2-Pentyl-1H-pyrrole-3-carboxylic acid ethyl ester (12h): Starting with crude **11h** (0.285 g, 1.0 mmol) and PPh₃ (0.315 g, 1.2 mmol) in THF (10 mL), **12h** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 50:1 → 5:1) as a brownish oil (0.129 g, 62% over two steps). ¹H NMR (CDCl₃, 300 MHz): δ = 0.88 – 1.01 (m, 3 H, CH₃), 1.26 – 1.51 (m, 7 H, 2×CH₂, CH₃), 1.67 – 1.81 (m, 2 H, CH₂), 3.01 (t, J = 7.7 Hz, 2 H, CH₂), 4.34 (q, J = 7.2 Hz, 2 H, OCH₂), 6.63 – 6.66 (m, 2 H, 2×CH), 8.29 (broad s, 1 H, NH). IR (neat, cm⁻¹): $\tilde{\nu}$ = 3343 (m), 2958 (m), 2929 (m), 2863 (w), 1647 (s), 1583 (m), 1549 (m), 1490 (w), 1460 (m), 1450 (m), 1399 (m), 1374 (m), 1303 (m), 1254 (s), 1176 (s), 1132 (s), 1102 (m), 1044 (m), 1021 (m), 841 (m), 795 (w), 761 (w), 728 (w). MS (EI, 70 eV): m/z (%) = 209 (M⁺, 51), 193 (3), 180 (5), 166 (65), 136 (63), 120 (17), 106 (12), 94 (100). The exact molecular mass m/z = 209.1416 ± 2 ppm [M⁺] for C₁₂H₁₉O₂N was confirmed by HRMS (EI, 70 eV).

2-Heptyl-1H-pyrrole-3-carboxylic acid ethyl ester (12i): Starting with **11i** (0.100 g, 0.33 mmol) and PPh₃ (0.105 g, 0.40 mmol) in THF (10 mL), **12i** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 5:1) as a brownish solid (0.069 g, 89%). ¹H NMR (CDCl₃, 300 MHz): δ = 0.86 (t, J = 6.9 Hz, 3 H, CH₃), 1.15 – 1.40 (m, 11 H, 4×CH₂, CH₃), 1.50 – 1.64 (m, 2 H, CH₂), 2.92 (t, J = 7.5 Hz, 2 H, CH₂), 4.27 (q, J = 7.2 Hz, 2 H,

OCH₂), 6.56 (d, $J = 3.0$ Hz, 1 H, CH), 6.58 (d, $J = 3.3$ Hz, 1 H, CH), 8.21 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 13.9, 14.3, 22.5, 27.3, 29.0, 29.3, 29.5, 31.7, 59.2, 110.9, 110.9, 115.8, 140.1, 165.8$. IR (neat, cm⁻¹): $\tilde{\nu} = 3324$ (s), 2956 (s), 2928 (s), 2871 (m), 2857 (m), 1726 (m), 1702 (s), 1673 (s), 1574 (m), 1496 (m), 1466 (s), 1394 (m), 1370 (m), 1331 (m), 1297 (m), 1264 (m), 1197 (m), 1181 (m), 1128 (s), 1096 (w), 1077 (w), 1049 (m), 1028 (w), 902 (w), 790 (w), 724 (m). MS (EI, 70 eV): m/z (%) = 237 (M⁺, 40), 208 (18), 192 (12), 164 (39), 151 (38), 138 (6), 124 (100). Anal. calcd. for C₁₄H₂₃O₂N (237.342): C 70.85, H 9.77, N 5.90; found C 70.47, H 10.10, N 5.91.

2-Nonyl-1H-pyrrole-3-carboxylic acid ethyl ester (12j): Starting with crude **11j** (0.341 g, 1.0 mmol) and PPh₃ (0.315 g, 1.2 mmol) in THF (10 mL), **12j** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 50:1 → 5:1) as a brownish oil (0.171 g, 65% over two steps). ¹H NMR (CDCl₃, 300 MHz): $\delta = 0.88$ (t, $J = 6.9$ Hz, 3 H, CH₃), 1.19 – 1.39 (m, 12 H, 6×CH₂), 1.34 (t, $J = 7.2$ Hz, 3 H, CH₃), 1.58 – 1.68 (m, 2 H, CH₂), 2.94 (t, $J = 7.6$ Hz, 2 H, CH₂), 4.27 (q, $J = 7.2$ Hz, 2 H, OCH₂), 6.57 (m, 2 H, 2×CH), 8.20 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 14.1, 14.5, 22.6, 27.3, 29.3, 29.4, 29.47, 29.51, 29.7, 31.8, 59.3, 110.5, 111.1, 115.7, 140.0, 165.5$. IR (neat, cm⁻¹): $\tilde{\nu} = 3332$ (m), 2957 (m), 2926 (s), 2855 (m), 1675 (s), 1464 (m), 1371 (w), 1329 (w), 1263 (w), 1196 (w), 1182 (w), 1128 (s), 1043 (w), 723 (w). MS (EI, 70 eV): m/z (%) = 265 (M⁺, 64), 236 (8), 220 (5), 192 (52), 166 (98), 154 (22), 138 (6), 124 (37), 120 (17), 94 (100). The exact molecular mass $m/z = 265.2042 \pm 2$ ppm [M⁺] for C₁₆H₂₇O₂N was confirmed by HRMS (EI, 70 eV).

2-Decyl-1H-pyrrole-3-carboxylic acid ethyl ester (12k): Starting with crude **11k** (0.178 g, 0.5 mmol) and PPh₃ (0.158 g, 0.6 mmol) in THF (10 mL), **12k** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 5:1) as a brownish oil (0.095 g, 68% over two steps). ¹H NMR (CDCl₃, 300 MHz): $\delta = 0.88$ (t, $J = 7.2$ Hz, 3 H, CH₃), 1.19 – 1.36

(m, 17 H, CH₃, 7×CH₂), 1.49 – 1.65 (m, 2 H, CH₂), 2.94 (t, $J = 7.8$ Hz, 2 H, CH₂), 4.27 (q, $J = 7.2$ Hz, 2 H, OCH₂), 6.57 (d, $J = 2.7$ Hz, 2 H, 2×CH), 8.15 (broad s, 1 H, NH). IR (neat, cm⁻¹): $\tilde{\nu} = 3366$ (s), 2956 (s), 2926 (s), 2854 (s), 1676 (s), 1598 (w), 1574 (w), 1492 (w), 1463 (s), 1396 (w), 1371 (m), 1330 (m), 1299 (m), 1259 (m), 1195 (s), 1184 (s), 1129 (s), 1033 (m), 722 (m). MS (EI, 70 eV): m/z (%) = 279 (M⁺, 50), 250 (3), 234 (1), 206 (41), 192 (5), 166 (98), 138 (5), 120 (14), 94 (100). The exact molecular mass $m/z = 279.2198 \pm 2$ ppm [M⁺] for C₁₇H₂₉O₂N was confirmed by HRMS (EI, 70 eV).

2-Undecyl-1H-pyrrole-3-carboxylic acid ethyl ester (12l): Starting with crude **11l** (0.185 g, 1.0 mmol) and PPh₃ (0.158 g, 0.6 mmol) in THF (10 mL), **12l** was isolated after chromatography (silica gel, *n*-Hex/EtOAc = 100:1 → 5:1) as a brownish oil (0.098 g, 67% over two steps). ¹H NMR (CDCl₃, 300 MHz): $\delta = 0.88$ (t, $J = 7.2$ Hz, 3 H, CH₃), 1.21 – 1.36 (m, 19 H, CH₃, 8×CH₂), 1.48 – 1.58 (m, 2 H, CH₂), 2.94 (t, $J = 7.8$ Hz, 2 H, CH₂), 4.27 (q, $J = 7.2$ Hz, 2 H, OCH₂), 6.57 (d, $J = 2.7$ Hz, 2 H, 2×CH), 8.14 (broad s, 1 H, NH). ¹³C NMR (CDCl₃, 75 MHz): $\delta_c = 14.1, 14.5, 22.7, 27.3, 29.3, 29.43, 29.46, 29.57, 29.60$ (2C), 31.2, 31.9, 59.3, 110.5, 111.3, 115.6, 139.9, 165.5. IR (neat, cm⁻¹): $\tilde{\nu} = 3377$ (m), 2956 (s), 2926 (s), 2854 (s), 1676 (s), 1463 (s), 1371 (w), 1330 (w), 1300 (w), 1257 (m), 1185 (m), 1183 (m), 1131 (s), 1032 (m), 721 (w). MS (EI, 70 eV): m/z (%) = 293 (M⁺, 71), 264 (2), 220 (4), 166 (100), 138 (4), 120 (15), 94 (97). The exact molecular mass $m/z = 293.2355 \pm 2$ ppm [M⁺] for C₁₈H₃₁O₂N was confirmed by HRMS (EI, 70 eV).

Crystal Structure Determination. The intensity data for the compounds were collected on a Nonius Kappa CCD diffractometer, using graphite-monochromated Mo-K α radiation. Data were corrected for Lorentz and polarization effects, but not for absorption effects.^{1, 2} The

structures were solved by direct methods (SHELXS)³ and refined by full-matrix least squares techniques against F_o^2 (SHELXL-97).⁴ For the C3-group of both symmetry independent molecules of **4**, for the amine-groups (not for **12b**), for the whole compound **6e** the hydrogen atoms were located by difference Fourier synthesis and refined isotropically. All other hydrogen atoms were included at calculated positions with fixed thermal parameters. All nonhydrogen atoms were refined anisotropically. XP (SIEMENS Analytical X-ray Instruments, Inc.) was used for structure representations.

- (1) COLLECT, Data Collection Software; Nonius, B.V., Netherlands, 1998.
- (2) Otwinowski, Z.; Minor, W. „Processing of X-Ray Diffraction Data Collected in Oscillation Mode“, in *Methods in Enzymology*, Vol. 276, Macromolecular Crystallography, Part A, edited by Carter, C. W.; Sweet, R. M. pp. 307-326, Academic Press 1997.
- (3) Sheldrick, G. M. *Acta Crystallogr. Sect. A* **1990**, 46, 467.
- (4) Sheldrick, G. M. SHELXL-97 (Release 97-2), University of Göttingen, Germany, 1997.

Table 1. Crystal data and structure refinement for **4e**.

Identification code	FO1921	
Empirical formula	C13 H15 N O2	
Formula weight	217.26	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 7.4218(5) Å	$\alpha = 90^\circ$.
	b = 8.7401(6) Å	$\beta = 90^\circ$.

	$c = 17.2870(9) \text{ \AA}$	$\gamma = 90^\circ$.
Volume	$1121.4(1) \text{ \AA}^3$	
Z	4	
Density (calculated)	1.287 Mg/m^3	
Absorption coefficient	0.087 mm^{-1}	
F(000)	464	
Crystal size	$0.20 \times 0.03 \times 0.03 \text{ mm}^3$	
Theta range for data collection	2.36 to 27.38° .	
Index ranges	$-9 \leq h \leq 9$, $-11 \leq k \leq 10$, $-22 \leq l \leq 22$	
Reflections collected	4080	
Independent reflections	2446 [$R(\text{int}) = 0.0389$]	
Completeness to $\theta = 27.38^\circ$	97.7 %	
Absorption correction	NONE	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2446 / 0 / 149	
Goodness-of-fit on F^2	1.055	
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0530$, $wR2 = 0.1171$	
R indices (all data)	$R1 = 0.0774$, $wR2 = 0.1297$	
Absolute structure parameter	$0.1(19)$	
Largest diff. peak and hole	0.181 and $-0.203 \text{ e.\AA}^{-3}$	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

for **4e**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
N	52(3)	7482(2)	5480(1)	44(1)
O(1)	784(2)	7619(2)	7248(1)	45(1)
C(1)	186(4)	8751(3)	6020(2)	50(1)
O(2)	559(2)	5653(2)	4256(1)	49(1)
C(2)	-660(3)	8109(2)	6763(2)	40(1)
C(3)	-1808(3)	6766(3)	6480(1)	40(1)
C(4)	-1015(3)	6346(2)	5701(1)	37(1)
C(5)	-1339(3)	5014(3)	5294(1)	38(1)
C(6)	-459(3)	4700(3)	4584(1)	39(1)
C(7)	-703(3)	3170(3)	4197(1)	37(1)
C(8)	-1487(3)	1913(3)	4564(1)	41(1)
C(9)	-1627(3)	514(3)	4193(2)	43(1)
C(10)	-985(3)	350(3)	3438(1)	44(1)
C(11)	-204(3)	1575(3)	3069(1)	46(1)
C(12)	-56(3)	2973(3)	3442(1)	42(1)
C(13)	185(3)	7337(3)	8020(1)	50(1)

Table 3. Bond lengths [Å] and angles [°] for 4e.

N-C(4)	1.326(3)
N-C(1)	1.453(3)
O(1)-C(2)	1.427(3)
O(1)-C(13)	1.428(3)
C(1)-C(2)	1.535(3)
O(2)-C(6)	1.260(3)
C(2)-C(3)	1.530(3)
C(3)-C(4)	1.514(3)
C(4)-C(5)	1.382(3)
C(5)-C(6)	1.416(3)
C(6)-C(7)	1.506(3)
C(7)-C(8)	1.396(3)
C(7)-C(12)	1.401(3)
C(8)-C(9)	1.384(3)
C(9)-C(10)	1.397(3)
C(10)-C(11)	1.374(3)
C(11)-C(12)	1.386(3)
C(4)-N-C(1)	115.3(2)
C(2)-O(1)-C(13)	111.55(18)
N-C(1)-C(2)	103.32(19)
O(1)-C(2)-C(3)	112.06(18)
O(1)-C(2)-C(1)	107.10(19)
C(3)-C(2)-C(1)	103.9(2)
C(4)-C(3)-C(2)	104.68(18)
N-C(4)-C(5)	126.0(2)
N-C(4)-C(3)	107.87(19)
C(5)-C(4)-C(3)	126.2(2)
C(4)-C(5)-C(6)	121.7(2)
O(2)-C(6)-C(5)	122.6(2)
O(2)-C(6)-C(7)	117.3(2)
C(5)-C(6)-C(7)	120.1(2)
C(8)-C(7)-C(12)	118.0(2)
C(8)-C(7)-C(6)	123.2(2)
C(12)-C(7)-C(6)	118.8(2)
C(9)-C(8)-C(7)	121.0(2)
C(8)-C(9)-C(10)	119.9(2)

C(11)-C(10)-C(9)	119.8(2)
C(10)-C(11)-C(12)	120.3(2)
C(11)-C(12)-C(7)	120.9(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 4e. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N	52(1)	38(1)	43(1)	2(1)	9(1)	-3(1)
O(1)	34(1)	58(1)	44(1)	2(1)	1(1)	0(1)
C(1)	64(2)	38(1)	48(1)	0(1)	9(1)	-8(1)
O(2)	58(1)	45(1)	44(1)	2(1)	9(1)	-10(1)
C(2)	38(1)	39(1)	44(1)	3(1)	1(1)	1(1)
C(3)	36(1)	43(1)	40(1)	1(1)	2(1)	2(1)
C(4)	34(1)	37(1)	41(1)	5(1)	0(1)	5(1)
C(5)	34(1)	39(1)	41(1)	3(1)	0(1)	-1(1)
C(6)	35(1)	40(1)	41(1)	5(1)	-2(1)	1(1)
C(7)	29(1)	42(1)	38(1)	2(1)	-4(1)	-1(1)
C(8)	40(1)	44(1)	38(1)	1(1)	2(1)	-1(1)
C(9)	38(1)	42(1)	50(2)	2(1)	0(1)	-1(1)
C(10)	39(1)	45(1)	49(2)	-9(1)	-2(1)	2(1)
C(11)	43(1)	55(1)	40(1)	-5(1)	3(1)	1(1)
C(12)	38(1)	48(1)	39(1)	3(1)	1(1)	1(1)
C(13)	46(1)	61(2)	41(1)	4(1)	-1(1)	-1(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 4e.

	x	y	z	U(eq)
H(1A)	-491	9652	5831	59
H(1B)	1458	9046	6106	59
H(2A)	-1419	8897	7027	48
H(3A)	-1731	5894	6844	47
H(3B)	-3086	7073	6427	47
H(5A)	-2173	4291	5494	46
H(8A)	-1931	2018	5077	49
H(9A)	-2159	-332	4451	52
H(10A)	-1088	-605	3180	53
H(11A)	237	1463	2557	55
H(12A)	491	3810	3182	50
H(13A)	1207	7003	8337	74
H(13B)	-740	6537	8016	74
H(13C)	-322	8279	8238	74
H(1N)	520(40)	7430(30)	5070(18)	54(9)

Table 1. Crystal data and structure refinement for 4f.

Identification code	FO1964	
Empirical formula	C ₉ H ₁₅ N O ₃	
Formula weight	185.22	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 9.1700(3) Å	$\alpha = 82.609(1)^\circ$.
	b = 10.4696(4) Å	$\beta = 87.188(1)^\circ$.
	c = 10.6924(5) Å	$\gamma = 75.131(1)^\circ$.
Volume	983.78(7) Å ³	
Z	4	
Density (calculated)	1.251 Mg/m ³	
Absorption coefficient	0.094 mm ⁻¹	
F(000)	400	
Crystal size	0.03 x 0.03 x 0.02 mm ³	
Theta range for data collection	2.03 to 27.50°.	
Index ranges	-11 ≤ h ≤ 11, -13 ≤ k ≤ 12, -13 ≤ l ≤ 13	
Reflections collected	7039	
Independent reflections	4481 [R(int) = 0.0285]	
Completeness to theta = 27.50°	99.1 %	
Absorption correction	NONE	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4481 / 0 / 243	
Goodness-of-fit on F ²	1.134	
Final R indices [I > 2sigma(I)]	R1 = 0.0709, wR2 = 0.1774	
R indices (all data)	R1 = 0.0931, wR2 = 0.1869	
Largest diff. peak and hole	0.375 and -0.303 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

for 4f. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1A)	4110(2)	9174(2)	4187(2)	25(1)
O(2A)	3490(3)	7686(2)	3100(2)	30(1)
O(3A)	5431(2)	6298(2)	8686(2)	30(1)
N(1A)	5920(3)	8016(2)	6330(2)	23(1)
C(1A)	6844(3)	7722(3)	7465(3)	26(1)
C(2A)	6771(3)	6301(3)	7942(3)	24(1)
C(3A)	6598(3)	5716(3)	6735(3)	24(1)
C(4A)	5760(3)	6926(3)	5886(3)	20(1)
C(5A)	5005(3)	6864(3)	4832(3)	22(1)
C(6A)	4201(3)	8013(3)	4058(3)	21(1)
C(7A)	2580(4)	8779(3)	2304(3)	31(1)
C(8A)	8110(4)	5067(3)	6135(3)	35(1)
C(9A)	5567(5)	6497(4)	9958(3)	40(1)
O(1B)	579(2)	6134(2)	-882(2)	28(1)
O(2B)	1066(2)	7939(2)	-2060(2)	26(1)
O(3B)	-745(2)	7764(2)	3801(2)	26(1)
N(1B)	-1115(3)	6705(2)	1363(2)	23(1)
C(1B)	-1994(3)	6682(3)	2540(3)	24(1)
C(2B)	-2005(3)	8005(3)	2995(3)	21(1)
C(3B)	-1818(3)	8913(3)	1777(3)	21(1)
C(4B)	-1023(3)	7931(3)	881(3)	20(1)
C(5B)	-346(3)	8289(3)	-225(3)	22(1)
C(6B)	443(3)	7339(3)	-1036(3)	21(1)
C(7B)	1990(4)	7053(3)	-2878(3)	31(1)
C(8B)	-3327(4)	9778(3)	1275(3)	35(1)
C(9B)	-774(4)	8859(3)	4468(3)	31(1)

Table 3. Bond lengths [Å] and angles [°] for 4f.

O(1A)-C(6A)	1.222(3)
O(2A)-C(6A)	1.365(3)
O(2A)-C(7A)	1.435(4)
O(3A)-C(9A)	1.419(4)
O(3A)-C(2A)	1.431(4)
N(1A)-C(4A)	1.335(4)
N(1A)-C(1A)	1.468(4)
C(1A)-C(2A)	1.527(4)
C(2A)-C(3A)	1.530(4)
C(3A)-C(4A)	1.514(4)
C(3A)-C(8A)	1.528(4)
C(4A)-C(5A)	1.369(4)
C(5A)-C(6A)	1.423(4)
O(1B)-C(6B)	1.226(3)
O(2B)-C(6B)	1.365(3)
O(2B)-C(7B)	1.441(3)
O(3B)-C(9B)	1.420(4)
O(3B)-C(2B)	1.426(3)
N(1B)-C(4B)	1.343(4)
N(1B)-C(1B)	1.462(4)
C(1B)-C(2B)	1.523(4)
C(2B)-C(3B)	1.540(4)
C(3B)-C(4B)	1.518(4)
C(3B)-C(8B)	1.526(4)
C(4B)-C(5B)	1.363(4)
C(5B)-C(6B)	1.434(4)
C(6A)-O(2A)-C(7A)	116.1(2)
C(9A)-O(3A)-C(2A)	113.4(3)
C(4A)-N(1A)-C(1A)	113.2(2)
N(1A)-C(1A)-C(2A)	102.0(2)
O(3A)-C(2A)-C(1A)	110.8(2)
O(3A)-C(2A)-C(3A)	106.6(2)
C(1A)-C(2A)-C(3A)	103.4(2)
C(4A)-C(3A)-C(8A)	110.8(2)
C(4A)-C(3A)-C(2A)	102.5(2)
C(8A)-C(3A)-C(2A)	113.0(3)

N(1A)-C(4A)-C(5A)	127.4(3)
N(1A)-C(4A)-C(3A)	108.6(2)
C(5A)-C(4A)-C(3A)	124.0(2)
C(4A)-C(5A)-C(6A)	123.2(3)
O(1A)-C(6A)-O(2A)	121.3(3)
O(1A)-C(6A)-C(5A)	126.9(3)
O(2A)-C(6A)-C(5A)	111.8(2)
C(6B)-O(2B)-C(7B)	115.6(2)
C(9B)-O(3B)-C(2B)	112.7(2)
C(4B)-N(1B)-C(1B)	113.3(2)
N(1B)-C(1B)-C(2B)	103.1(2)
O(3B)-C(2B)-C(1B)	107.0(2)
O(3B)-C(2B)-C(3B)	111.7(2)
C(1B)-C(2B)-C(3B)	104.1(2)
C(4B)-C(3B)-C(8B)	112.5(2)
C(4B)-C(3B)-C(2B)	103.1(2)
C(8B)-C(3B)-C(2B)	112.3(2)
N(1B)-C(4B)-C(5B)	127.5(3)
N(1B)-C(4B)-C(3B)	108.9(2)
C(5B)-C(4B)-C(3B)	123.6(3)
C(4B)-C(5B)-C(6B)	122.7(3)
O(1B)-C(6B)-O(2B)	121.6(3)
O(1B)-C(6B)-C(5B)	126.9(3)
O(2B)-C(6B)-C(5B)	111.5(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 4f. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2}U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1A)	29(1)	16(1)	30(1)	-3(1)	-5(1)	-4(1)
O(2A)	40(1)	26(1)	26(1)	1(1)	-12(1)	-12(1)
O(3A)	37(1)	36(1)	19(1)	-3(1)	0(1)	-15(1)
N(1A)	27(1)	16(1)	26(1)	-2(1)	-4(1)	-5(1)
C(1A)	26(2)	23(2)	28(2)	-2(1)	-7(1)	-7(1)
C(2A)	25(1)	21(1)	26(2)	0(1)	-6(1)	-6(1)
C(3A)	28(2)	18(1)	25(2)	-2(1)	-2(1)	-5(1)
C(4A)	22(1)	16(1)	21(1)	-3(1)	3(1)	-6(1)
C(5A)	28(1)	16(1)	22(1)	-2(1)	-1(1)	-7(1)
C(6A)	21(1)	23(1)	19(1)	-2(1)	3(1)	-8(1)
C(7A)	30(2)	34(2)	28(2)	4(1)	-11(1)	-7(1)
C(8A)	35(2)	24(2)	38(2)	-2(1)	-2(1)	6(1)
C(9A)	60(2)	41(2)	24(2)	-5(1)	-2(2)	-20(2)
O(1B)	37(1)	19(1)	28(1)	-6(1)	7(1)	-9(1)
O(2B)	31(1)	24(1)	23(1)	-5(1)	8(1)	-7(1)
O(3B)	29(1)	21(1)	26(1)	-5(1)	-5(1)	-3(1)
N(1B)	29(1)	19(1)	23(1)	-7(1)	6(1)	-9(1)
C(1B)	29(2)	25(2)	23(2)	-5(1)	6(1)	-13(1)
C(2B)	21(1)	20(1)	21(1)	-5(1)	1(1)	-5(1)
C(3B)	23(1)	20(1)	21(1)	-4(1)	3(1)	-5(1)
C(4B)	19(1)	19(1)	20(1)	-4(1)	-1(1)	-4(1)
C(5B)	26(1)	18(1)	22(1)	-4(1)	2(1)	-6(1)
C(6B)	22(1)	22(1)	20(1)	-4(1)	2(1)	-7(1)
C(7B)	34(2)	32(2)	25(2)	-8(1)	10(1)	-2(1)
C(8B)	36(2)	33(2)	25(2)	-5(1)	0(1)	8(1)
C(9B)	38(2)	29(2)	30(2)	-10(1)	-4(1)	-10(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 4f.

	x	y	z	U(eq)
H(1AA)	6411	8340	8093	31
H(1AB)	7894	7770	7256	31
H(2AA)	7700	5789	8408	29
H(3AA)	5969	5059	6910	29
H(5AA)	5018	6013	4606	26
H(7AA)	2123	8437	1652	47
H(7AB)	3213	9347	1904	47
H(7AC)	1783	9301	2812	47
H(8AA)	7931	4713	5368	53
H(8AB)	8674	4339	6732	53
H(8AC)	8693	5731	5922	53
H(9AA)	4618	6483	10416	60
H(9AB)	5791	7359	9984	60
H(9AC)	6386	5785	10353	60
H(1BA)	-1505	5923	3159	29
H(1BB)	-3031	6625	2390	29
H(2BA)	-2972	8374	3447	25
H(3BA)	-1151	9492	1937	25
H(5BA)	-399	9205	-468	26
H(7BA)	2386	7574	-3578	47
H(7BB)	1381	6533	-3211	47
H(7BC)	2831	6449	-2401	47
H(8BA)	-3153	10340	504	52
H(8BB)	-3833	10342	1912	52
H(8BC)	-3964	9207	1085	52
H(9BA)	115	8647	5001	47
H(9BB)	-1690	9039	4997	47
H(9BC)	-768	9647	3866	47
H(1NB)	-740(40)	5990(40)	920(40)	41(10)
H(1NA)	5570(40)	8790(40)	5960(40)	39(11)

Table 1. Crystal data and structure refinement for cis-4h.

Identification code	FO1921	
Empirical formula	C ₉ H ₁₅ N O ₄	
Formula weight	201.22	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 13.3623(5) Å	$\alpha = 90^\circ$.
	b = 4.4324(2) Å	$\beta = 93.147(2)^\circ$.
	c = 17.5285(6) Å	$\gamma = 90^\circ$.
Volume	1036.60(7) Å ³	
Z	4	
Density (calculated)	1.289 Mg/m ³	
Absorption coefficient	0.101 mm ⁻¹	
F(000)	432	
Crystal size	0.03 x 0.03 x 0.02 mm ³	
Theta range for data collection	2.85 to 27.48°.	
Index ranges	-15 ≤ h ≤ 17, -5 ≤ k ≤ 5, -22 ≤ l ≤ 22	
Reflections collected	6863	
Independent reflections	2366 [R(int) = 0.0691]	
Completeness to theta = 27.48°	99.2 %	
Absorption correction	NONE	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2366 / 0 / 131	
Goodness-of-fit on F ²	1.048	
Final R indices [I > 2sigma(I)]	R1 = 0.0543, wR2 = 0.1396	
R indices (all data)	R1 = 0.0771, wR2 = 0.1539	
Largest diff. peak and hole	0.475 and -0.259 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

for cis-4h. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	9427(1)	1(3)	3070(1)	40(1)
N(1)	8434(1)	3464(4)	1914(1)	33(1)
C(1)	7658(1)	2281(4)	2258(1)	29(1)
O(2)	8414(1)	-2582(3)	3822(1)	43(1)
C(2)	6714(1)	3429(4)	1842(1)	32(1)
O(3)	7011(1)	1816(3)	573(1)	42(1)
C(3)	7064(1)	4382(4)	1059(1)	35(1)
O(4)	5949(1)	1219(3)	1847(1)	40(1)
C(4)	8141(1)	5347(4)	1256(1)	38(1)
C(5)	7665(1)	389(4)	2869(1)	31(1)
C(6)	8581(1)	-676(4)	3235(1)	31(1)
C(7)	9291(2)	-3757(5)	4224(1)	52(1)
C(8)	6922(2)	2522(6)	-203(1)	59(1)
C(9)	5022(1)	2317(6)	1533(1)	50(1)

Table 3. Bond lengths [Å] and angles [°] for *cis*-4h.

O(1)-C(6)	1.219(2)
N(1)-C(1)	1.335(2)
N(1)-C(4)	1.459(2)
C(1)-C(5)	1.361(2)
C(1)-C(2)	1.510(2)
O(2)-C(6)	1.360(2)
O(2)-C(7)	1.432(2)
C(2)-O(4)	1.416(2)
C(2)-C(3)	1.533(2)
O(3)-C(8)	1.394(2)
O(3)-C(3)	1.421(2)
C(3)-C(4)	1.522(3)
O(4)-C(9)	1.414(2)
C(5)-C(6)	1.431(2)
C(1)-N(1)-C(4)	113.58(15)
N(1)-C(1)-C(5)	128.74(16)
N(1)-C(1)-C(2)	107.38(15)
C(5)-C(1)-C(2)	123.88(15)
C(6)-O(2)-C(7)	115.75(16)
O(4)-C(2)-C(1)	110.34(14)
O(4)-C(2)-C(3)	116.91(14)
C(1)-C(2)-C(3)	103.67(14)
C(8)-O(3)-C(3)	113.83(15)
O(3)-C(3)-C(4)	112.13(15)
O(3)-C(3)-C(2)	107.95(14)
C(4)-C(3)-C(2)	101.90(13)
C(9)-O(4)-C(2)	112.11(15)
N(1)-C(4)-C(3)	103.14(14)
C(1)-C(5)-C(6)	121.67(16)
O(1)-C(6)-O(2)	121.68(16)
O(1)-C(6)-C(5)	126.48(16)
O(2)-C(6)-C(5)	111.83(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cis-4h. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	26(1)	42(1)	51(1)	3(1)	0(1)	2(1)
N(1)	28(1)	33(1)	40(1)	3(1)	3(1)	0(1)
C(1)	26(1)	26(1)	34(1)	-6(1)	1(1)	1(1)
O(2)	41(1)	47(1)	40(1)	12(1)	-5(1)	0(1)
C(2)	28(1)	29(1)	39(1)	-3(1)	-1(1)	2(1)
O(3)	60(1)	31(1)	34(1)	-1(1)	-5(1)	5(1)
C(3)	40(1)	25(1)	40(1)	2(1)	-2(1)	7(1)
O(4)	25(1)	43(1)	50(1)	2(1)	-5(1)	-2(1)
C(4)	44(1)	29(1)	42(1)	5(1)	7(1)	0(1)
C(5)	25(1)	35(1)	33(1)	-2(1)	2(1)	-1(1)
C(6)	32(1)	31(1)	31(1)	-3(1)	-1(1)	0(1)
C(7)	52(1)	51(1)	53(1)	12(1)	-17(1)	3(1)
C(8)	77(2)	63(2)	37(1)	0(1)	-2(1)	12(1)
C(9)	25(1)	67(2)	57(1)	-1(1)	-5(1)	5(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for cis-4h.

	x	y	z	U(eq)
H(2A)	6482	5266	2112	38
H(3A)	6654	6090	839	42
H(4A)	8572	4957	825	46
H(4B)	8178	7516	1390	46
H(5A)	7046	-242	3058	37
H(7A)	9092	-5097	4634	79
H(7B)	9692	-2087	4444	79
H(7C)	9690	-4892	3869	79
H(8A)	6893	656	-503	89
H(8B)	7502	3718	-339	89
H(8C)	6307	3688	-311	89
H(9A)	4515	725	1549	75
H(9B)	5096	2930	1002	75
H(9C)	4813	4057	1830	75
H(1N1)	9070(20)	3270(60)	2087(13)	59(7)

Table 1. Crystal data and structure refinement for 5p.

Identification code	FO1963	
Empirical formula	C ₁₇ H ₂₇ N O ₂	
Formula weight	277.40	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 8.8774(1) Å	$\alpha = 90^\circ$.
	b = 13.3104(2) Å	$\beta = 90^\circ$.
	c = 27.0971(6) Å	$\gamma = 90^\circ$.
Volume	3201.84(9) Å ³	
Z	8	
Density (calculated)	1.151 Mg/m ³	
Absorption coefficient	0.074 mm ⁻¹	
F(000)	1216	
Crystal size	0.03 x 0.03 x 0.03 mm ³	
Theta range for data collection	3.06 to 27.48°.	
Index ranges	-11 ≤ h ≤ 11, -17 ≤ k ≤ 17, -34 ≤ l ≤ 35	
Reflections collected	6800	
Independent reflections	3652 [R(int) = 0.0341]	
Completeness to theta = 27.48°	99.5 %	
Absorption correction	NONE	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3652 / 0 / 185	
Goodness-of-fit on F ²	1.029	
Final R indices [I > 2sigma(I)]	R1 = 0.0466, wR2 = 0.1086	
R indices (all data)	R1 = 0.0750, wR2 = 0.1200	
Largest diff. peak and hole	0.194 and -0.214 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

for 5p. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	2264(1)	206(1)	281(1)	31(1)
O(2)	3181(1)	998(1)	944(1)	31(1)
N	-1226(1)	1448(1)	295(1)	25(1)
C(1)	-1928(2)	2335(1)	189(1)	29(1)
C(2)	-1447(2)	3039(1)	520(1)	28(1)
C(3)	-406(1)	2569(1)	844(1)	23(1)
C(4)	392(1)	3053(1)	1272(1)	28(1)
C(5)	-592(2)	3180(1)	1733(1)	34(1)
C(6)	-1062(2)	2200(1)	1979(1)	34(1)
C(7)	190(2)	1661(1)	2259(1)	37(1)
C(8)	-261(2)	642(1)	2477(1)	44(1)
C(9)	-649(2)	-164(1)	2096(1)	39(1)
C(10)	714(2)	-610(1)	1827(1)	39(1)
C(11)	375(2)	-1027(1)	1313(1)	35(1)
C(12)	-304(1)	-262(1)	953(1)	25(1)
C(13)	562(1)	735(1)	931(1)	22(1)
C(14)	-303(1)	1579(1)	698(1)	21(1)
C(15)	2075(1)	605(1)	677(1)	22(1)
C(16)	4702(2)	987(1)	735(1)	37(1)
C(17)	5769(2)	1089(1)	1158(1)	30(1)

Table 3. Bond lengths [Å] and angles [°] for 5p.

O(1)-C(15)	1.2088(16)
O(2)-C(15)	1.3277(15)
O(2)-C(16)	1.4647(16)
N-C(1)	1.3654(18)
N-C(14)	1.3769(17)
C(1)-C(2)	1.365(2)
C(2)-C(3)	1.4211(19)
C(3)-C(14)	1.3776(18)
C(3)-C(4)	1.5050(19)
C(4)-C(5)	1.532(2)
C(5)-C(6)	1.523(2)
C(6)-C(7)	1.526(2)
C(7)-C(8)	1.532(2)
C(8)-C(9)	1.528(2)
C(9)-C(10)	1.532(2)
C(10)-C(11)	1.528(2)
C(11)-C(12)	1.5337(19)
C(12)-C(13)	1.5345(18)
C(13)-C(14)	1.5007(18)
C(13)-C(15)	1.5198(17)
C(16)-C(17)	1.493(2)
C(15)-O(2)-C(16)	117.76(11)
C(1)-N-C(14)	109.16(12)
C(2)-C(1)-N	108.25(12)
C(1)-C(2)-C(3)	107.86(12)
C(14)-C(3)-C(2)	106.66(12)
C(14)-C(3)-C(4)	126.84(12)
C(2)-C(3)-C(4)	126.48(12)
C(3)-C(4)-C(5)	114.00(11)
C(6)-C(5)-C(4)	114.74(12)
C(5)-C(6)-C(7)	114.89(12)
C(6)-C(7)-C(8)	114.70(12)
C(9)-C(8)-C(7)	114.89(13)
C(8)-C(9)-C(10)	114.51(13)
C(11)-C(10)-C(9)	114.70(12)
C(10)-C(11)-C(12)	114.62(12)

C(11)-C(12)-C(13)	113.66(11)
C(14)-C(13)-C(15)	110.32(10)
C(14)-C(13)-C(12)	114.03(10)
C(15)-C(13)-C(12)	111.22(10)
N-C(14)-C(3)	108.06(11)
N-C(14)-C(13)	122.88(11)
C(3)-C(14)-C(13)	128.98(12)
O(1)-C(15)-O(2)	123.76(12)
O(1)-C(15)-C(13)	125.10(11)
O(2)-C(15)-C(13)	111.14(11)
O(2)-C(16)-C(17)	106.69(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 5p. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	29(1)	36(1)	28(1)	-7(1)	4(1)	-4(1)
O(2)	17(1)	47(1)	28(1)	-7(1)	1(1)	-3(1)
N	27(1)	28(1)	21(1)	-1(1)	-2(1)	1(1)
C(1)	29(1)	35(1)	24(1)	9(1)	-1(1)	3(1)
C(2)	28(1)	26(1)	31(1)	6(1)	3(1)	5(1)
C(3)	21(1)	24(1)	24(1)	2(1)	3(1)	-1(1)
C(4)	25(1)	22(1)	35(1)	-1(1)	-2(1)	-3(1)
C(5)	32(1)	36(1)	34(1)	-13(1)	-2(1)	2(1)
C(6)	26(1)	51(1)	24(1)	-3(1)	2(1)	-2(1)
C(7)	32(1)	54(1)	24(1)	-1(1)	-6(1)	-8(1)
C(8)	39(1)	66(1)	27(1)	11(1)	-5(1)	-10(1)
C(9)	36(1)	49(1)	34(1)	14(1)	-4(1)	-11(1)
C(10)	33(1)	40(1)	44(1)	20(1)	-8(1)	-1(1)
C(11)	28(1)	25(1)	53(1)	9(1)	-1(1)	1(1)
C(12)	21(1)	24(1)	30(1)	0(1)	1(1)	-1(1)
C(13)	21(1)	24(1)	21(1)	-1(1)	0(1)	0(1)
C(14)	18(1)	26(1)	20(1)	1(1)	2(1)	-2(1)
C(15)	23(1)	20(1)	24(1)	3(1)	-2(1)	0(1)
C(16)	17(1)	62(1)	33(1)	2(1)	3(1)	0(1)
C(17)	22(1)	34(1)	36(1)	0(1)	-3(1)	0(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 5p.

	x	y	z	U(eq)
H(1A)	-2630	2443	-70	35
H(2A)	-1753	3722	531	34
H(4A)	761	3722	1168	33
H(4B)	1281	2640	1360	33
H(5A)	-34	3591	1976	41
H(5B)	-1511	3556	1640	41
H(6A)	-1457	1741	1722	41
H(6B)	-1895	2344	2211	41
H(7A)	536	2101	2531	44
H(7B)	1052	1560	2033	44
H(8A)	578	393	2684	52
H(8B)	-1143	740	2695	52
H(9A)	-1339	131	1848	47
H(9B)	-1197	-715	2264	47
H(10A)	1495	-84	1797	47
H(10B)	1137	-1158	2032	47
H(11A)	-332	-1598	1347	42
H(11B)	1321	-1290	1170	42
H(12A)	-1357	-123	1052	30
H(12B)	-326	-561	619	30
H(13A)	775	938	1280	26
H(16A)	4832	1551	501	44
H(16B)	4884	349	557	44
H(17A)	6807	1082	1034	46
H(17B)	5624	528	1387	46
H(17C)	5578	1724	1329	46
H(1N)	-1421(17)	827(13)	139(6)	43(5)

Table 1. Crystal data and structure refinement for 6e.

Identification code	FO2265	
Empirical formula	C ₁₄ H ₁₀ F ₃ N O ₂	
Formula weight	281.23	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 14.6936(11) Å	$\alpha = 90^\circ$.
	b = 15.9165(17) Å	$\beta = 100.235(6)^\circ$.
	c = 5.3874(6) Å	$\gamma = 90^\circ$.
Volume	1239.9(2) Å ³	
Z	4	
Density (calculated)	1.507 Mg/m ³	
Absorption coefficient	0.131 mm ⁻¹	
F(000)	576	
Crystal size	0.03 x 0.03 x 0.02 mm ³	
Theta range for data collection	2.92 to 27.48°.	
Index ranges	-15 ≤ h ≤ 19, -17 ≤ k ≤ 20, -6 ≤ l ≤ 6	
Reflections collected	7987	
Independent reflections	2776 [R(int) = 0.0886]	
Completeness to theta = 27.48°	98.1 %	
Absorption correction	NONE	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2776 / 0 / 221	
Goodness-of-fit on F ²	1.013	
Final R indices [I > 2sigma(I)]	R1 = 0.0614, wR2 = 0.1318	
R indices (all data)	R1 = 0.1269, wR2 = 0.1616	
Largest diff. peak and hole	0.199 and -0.264 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

for 6e. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	3105(1)	6795(1)	-1806(4)	53(1)
O(2)	6294(1)	5263(1)	633(4)	52(1)
F(1)	8008(1)	5594(1)	470(4)	69(1)
F(2)	7649(1)	6182(2)	-3066(4)	82(1)
F(3)	7492(1)	6847(1)	269(5)	93(1)
N(1)	4801(2)	5832(2)	-3033(4)	42(1)
C(1)	3237(2)	6046(2)	-5576(6)	43(1)
C(2)	2711(2)	6432(2)	-3674(5)	40(1)
C(3)	1682(2)	6342(2)	-4179(5)	38(1)
C(4)	1202(2)	5932(2)	-6293(5)	44(1)
C(5)	246(2)	5888(2)	-6707(6)	51(1)
C(6)	-241(2)	6245(2)	-5023(6)	54(1)
C(7)	225(2)	6648(2)	-2915(6)	52(1)
C(8)	1183(2)	6694(2)	-2471(6)	44(1)
C(9)	4257(2)	6192(2)	-5009(5)	41(1)
C(10)	4806(2)	6680(2)	-6282(6)	46(1)
C(11)	5708(2)	6614(2)	-4999(6)	46(1)
C(12)	5710(2)	6077(2)	-2939(5)	40(1)
C(13)	6410(2)	5769(2)	-1006(5)	41(1)
C(14)	7392(2)	6104(2)	-844(6)	49(1)

Table 3. Bond lengths [Å] and angles [°] for 6e.

O(1)-C(2)	1.214(3)
O(2)-C(13)	1.230(3)
F(1)-C(14)	1.324(3)
F(2)-C(14)	1.324(3)
F(3)-C(14)	1.322(4)
N(1)-C(9)	1.340(4)
N(1)-C(12)	1.384(3)
C(1)-C(9)	1.495(4)
C(1)-C(2)	1.518(4)
C(2)-C(3)	1.495(4)
C(3)-C(4)	1.391(4)
C(3)-C(8)	1.392(4)
C(4)-C(5)	1.384(4)
C(5)-C(6)	1.375(4)
C(6)-C(7)	1.376(5)
C(7)-C(8)	1.387(4)
C(9)-C(10)	1.385(4)
C(10)-C(11)	1.385(4)
C(11)-C(12)	1.400(4)
C(12)-C(13)	1.415(4)
C(13)-C(14)	1.526(4)
C(9)-N(1)-C(12)	110.1(2)
C(9)-C(1)-C(2)	114.3(2)
O(1)-C(2)-C(3)	121.0(2)
O(1)-C(2)-C(1)	121.8(2)
C(3)-C(2)-C(1)	117.3(2)
C(4)-C(3)-C(8)	118.7(2)
C(4)-C(3)-C(2)	122.9(2)
C(8)-C(3)-C(2)	118.4(2)
C(5)-C(4)-C(3)	120.5(3)
C(6)-C(5)-C(4)	120.3(3)
C(7)-C(6)-C(5)	119.8(3)
C(6)-C(7)-C(8)	120.5(3)
C(7)-C(8)-C(3)	120.2(3)
N(1)-C(9)-C(10)	108.4(2)
N(1)-C(9)-C(1)	122.0(3)

C(10)-C(9)-C(1)	129.6(3)
C(11)-C(10)-C(9)	107.4(3)
C(10)-C(11)-C(12)	108.0(3)
N(1)-C(12)-C(11)	106.1(2)
N(1)-C(12)-C(13)	120.0(3)
C(11)-C(12)-C(13)	133.9(3)
O(2)-C(13)-C(12)	125.4(3)
O(2)-C(13)-C(14)	116.0(2)
C(12)-C(13)-C(14)	118.5(3)
F(3)-C(14)-F(2)	107.4(3)
F(3)-C(14)-F(1)	107.2(3)
F(2)-C(14)-F(1)	105.2(2)
F(3)-C(14)-C(13)	111.5(2)
F(2)-C(14)-C(13)	113.6(2)
F(1)-C(14)-C(13)	111.5(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 6e. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	51(1)	67(1)	38(1)	-10(1)	4(1)	-5(1)
O(2)	44(1)	64(1)	48(1)	10(1)	5(1)	-3(1)
F(1)	40(1)	73(1)	91(2)	9(1)	0(1)	3(1)
F(2)	48(1)	130(2)	72(1)	18(1)	21(1)	-8(1)
F(3)	56(1)	65(1)	157(2)	-42(2)	15(1)	-11(1)
N(1)	39(1)	51(2)	37(1)	3(1)	7(1)	-3(1)
C(1)	42(2)	53(2)	33(2)	-3(1)	3(1)	3(1)
C(2)	47(2)	41(2)	30(1)	2(1)	5(1)	-2(1)
C(3)	44(1)	38(2)	30(1)	4(1)	5(1)	-1(1)
C(4)	44(2)	50(2)	39(2)	-4(1)	9(1)	1(1)
C(5)	46(2)	63(2)	42(2)	-8(2)	4(1)	-7(1)
C(6)	44(2)	66(2)	52(2)	6(2)	11(1)	0(1)
C(7)	55(2)	58(2)	48(2)	-3(2)	21(2)	6(2)
C(8)	51(2)	47(2)	36(2)	-2(1)	10(1)	-1(1)
C(9)	44(2)	48(2)	32(1)	0(1)	6(1)	2(1)
C(10)	50(2)	50(2)	37(2)	7(1)	9(1)	4(1)
C(11)	48(2)	47(2)	45(2)	2(1)	15(1)	-3(1)
C(12)	40(2)	45(2)	36(2)	-3(1)	9(1)	-1(1)
C(13)	45(2)	43(2)	36(2)	-4(1)	9(1)	0(1)
C(14)	41(2)	53(2)	52(2)	-4(2)	6(1)	1(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 6e.

	x	y	z	U(eq)
H(1N1)	4600(20)	5478(19)	-1880(60)	51(8)
H(1A)	2988(19)	6268(18)	-7270(60)	47(8)
H(1B)	3150(20)	5450(20)	-5510(60)	57(9)
H(4)	1519(19)	5693(18)	-7450(60)	46(8)
H(5)	-60(19)	5619(19)	-8200(60)	48(8)
H(6)	-900(20)	6210(20)	-5150(60)	59(9)
H(7)	-110(20)	6913(19)	-1820(60)	50(8)
H(8)	1511(19)	6962(19)	-1060(60)	44(8)
H(10)	4580(20)	7020(20)	-7710(60)	53(8)
H(11)	6220(20)	6869(19)	-5370(60)	48(8)

Table 1. Crystal data and structure refinement for 12b.

Identification code	FO2063	
Empirical formula	C ₈ H ₁₁ N O ₂	
Formula weight	153.18	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 12.3895(4) Å	α = 90°.
	b = 13.7959(6) Å	β = 90°.
	c = 19.3409(8) Å	γ = 90°.
Volume	3305.8(2) Å ³	
Z	16	
Density (calculated)	1.231 Mg/m ³	
Absorption coefficient	0.089 mm ⁻¹	
F(000)	1312	
Crystal size	0.03 x 0.03 x 0.02 mm ³	
Theta range for data collection	2.45 to 27.47°.	
Index ranges	-16 ≤ h ≤ 15, -17 ≤ k ≤ 15, -25 ≤ l ≤ 25	
Reflections collected	18395	
Independent reflections	3778 [R(int) = 0.0734]	
Completeness to theta = 27.47°	99.8 %	
Absorption correction	NONE	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3778 / 0 / 199	
Goodness-of-fit on F ²	0.758	
Final R indices [I > 2σ(I)]	R1 = 0.0600, wR2 = 0.1727	
R indices (all data)	R1 = 0.1200, wR2 = 0.2274	
Largest diff. peak and hole	0.195 and -0.201 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

for 12b. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1B)	-528(2)	1642(2)	6808(1)	54(1)
C(1B)	1775(2)	1557(2)	7221(1)	41(1)
N(1B)	2859(2)	1480(2)	7154(1)	51(1)
O(2B)	21(1)	1447(1)	5713(1)	44(1)
C(2B)	3140(2)	1398(2)	6471(2)	53(1)
C(3B)	2226(2)	1418(2)	6091(1)	47(1)
C(4B)	1346(2)	1518(2)	6559(1)	39(1)
C(5B)	1254(3)	1638(2)	7911(1)	59(1)
C(6B)	204(2)	1544(2)	6397(1)	38(1)
C(7B)	-1098(2)	1479(2)	5493(1)	47(1)
C(8B)	-1121(2)	1152(2)	4751(2)	61(1)
O(1A)	5156(1)	912(2)	3171(1)	59(1)
C(1A)	2830(2)	953(2)	2795(1)	42(1)
N(1A)	1748(2)	883(2)	2888(1)	48(1)
O(2A)	4644(1)	1117(1)	4267(1)	44(1)
C(2A)	1501(2)	850(2)	3576(2)	52(1)
C(3A)	2434(2)	899(2)	3940(1)	46(1)
C(4A)	3286(2)	961(2)	3448(1)	38(1)
C(5A)	3305(3)	1015(2)	2094(1)	57(1)
C(6A)	4439(2)	994(2)	3594(1)	40(1)
C(7A)	5773(2)	1136(2)	4469(1)	47(1)
C(8A)	5810(2)	1364(2)	5224(2)	56(1)

Table 3. Bond lengths [Å] and angles [°] for 12b.

O(1B)-C(6B)	1.213(3)
C(1B)-N(1B)	1.354(3)
C(1B)-C(4B)	1.387(3)
C(1B)-C(5B)	1.486(3)
N(1B)-C(2B)	1.371(4)
O(2B)-C(6B)	1.349(3)
O(2B)-C(7B)	1.451(3)
C(2B)-C(3B)	1.351(4)
C(3B)-C(4B)	1.424(3)
C(4B)-C(6B)	1.450(3)
C(7B)-C(8B)	1.505(4)
O(1A)-C(6A)	1.214(3)
C(1A)-N(1A)	1.355(3)
C(1A)-C(4A)	1.383(3)
C(1A)-C(5A)	1.480(4)
N(1A)-C(2A)	1.366(4)
O(2A)-C(6A)	1.337(3)
O(2A)-C(7A)	1.452(3)
C(2A)-C(3A)	1.356(4)
C(3A)-C(4A)	1.423(3)
C(4A)-C(6A)	1.458(3)
C(7A)-C(8A)	1.495(4)
N(1B)-C(1B)-C(4B)	106.7(2)
N(1B)-C(1B)-C(5B)	121.5(2)
C(4B)-C(1B)-C(5B)	131.7(2)
C(1B)-N(1B)-C(2B)	110.5(2)
C(6B)-O(2B)-C(7B)	116.42(19)
C(3B)-C(2B)-N(1B)	108.1(2)
C(2B)-C(3B)-C(4B)	107.3(2)
C(1B)-C(4B)-C(3B)	107.4(2)
C(1B)-C(4B)-C(6B)	124.9(2)
C(3B)-C(4B)-C(6B)	127.7(2)
O(1B)-C(6B)-O(2B)	121.9(2)
O(1B)-C(6B)-C(4B)	126.2(2)
O(2B)-C(6B)-C(4B)	111.9(2)
O(2B)-C(7B)-C(8B)	106.7(2)

N(1A)-C(1A)-C(4A)	106.5(2)
N(1A)-C(1A)-C(5A)	121.2(2)
C(4A)-C(1A)-C(5A)	132.3(2)
C(2A)-N(1A)-C(1A)	110.6(2)
C(6A)-O(2A)-C(7A)	116.53(19)
C(3A)-C(2A)-N(1A)	108.3(2)
C(2A)-C(3A)-C(4A)	106.7(2)
C(1A)-C(4A)-C(3A)	107.9(2)
C(1A)-C(4A)-C(6A)	125.3(2)
C(3A)-C(4A)-C(6A)	126.8(2)
O(1A)-C(6A)-O(2A)	122.0(2)
O(1A)-C(6A)-C(4A)	125.7(2)
O(2A)-C(6A)-C(4A)	112.3(2)
O(2A)-C(7A)-C(8A)	107.2(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 12b. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1B)	41(1)	75(1)	46(1)	-1(1)	9(1)	0(1)
C(1B)	46(1)	37(1)	40(1)	2(1)	-6(1)	-2(1)
N(1B)	46(1)	53(1)	52(1)	1(1)	-18(1)	1(1)
O(2B)	31(1)	61(1)	39(1)	0(1)	-2(1)	0(1)
C(2B)	34(1)	67(2)	58(2)	-9(1)	-2(1)	4(1)
C(3B)	38(1)	61(2)	42(1)	-9(1)	1(1)	3(1)
C(4B)	37(1)	44(1)	37(1)	0(1)	-3(1)	1(1)
C(5B)	80(2)	62(2)	35(1)	1(1)	1(1)	-6(2)
C(6B)	35(1)	41(1)	38(1)	1(1)	1(1)	-2(1)
C(7B)	30(1)	61(2)	51(2)	1(1)	-7(1)	-1(1)
C(8B)	43(2)	87(2)	52(2)	-6(2)	-12(1)	-2(1)
O(1A)	40(1)	85(2)	50(1)	-11(1)	10(1)	1(1)
C(1A)	47(1)	38(1)	41(1)	-2(1)	-3(1)	3(1)
N(1A)	43(1)	53(1)	49(1)	-3(1)	-13(1)	2(1)
O(2A)	31(1)	58(1)	41(1)	-4(1)	-1(1)	0(1)
C(2A)	35(1)	65(2)	56(2)	2(1)	0(1)	-1(1)
C(3A)	38(1)	61(2)	40(1)	1(1)	3(1)	0(1)
C(4A)	36(1)	42(1)	36(1)	-1(1)	1(1)	-2(1)
C(5A)	75(2)	57(2)	39(1)	-2(1)	1(1)	4(1)
C(6A)	38(1)	43(1)	39(1)	-3(1)	4(1)	2(1)
C(7A)	31(1)	55(2)	55(2)	-2(1)	-4(1)	0(1)
C(8A)	43(2)	73(2)	52(2)	-1(1)	-12(1)	1(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for 12b.

	x	y	z	U(eq)
H(1BA)	3319	1483	7500	61
H(2BA)	3853	1337	6296	63
H(3BA)	2178	1373	5601	57
H(5BA)	1811	1650	8271	89
H(5BB)	778	1081	7985	89
H(5BC)	831	2237	7932	89
H(7BA)	-1385	2147	5534	57
H(7BB)	-1544	1044	5783	57
H(8BA)	-1865	1166	4580	91
H(8BB)	-839	489	4719	91
H(8BC)	-672	1586	4471	91
H(1AA)	1271	862	2551	58
H(2AA)	796	801	3766	62
H(3AA)	2506	892	4429	55
H(5AA)	2728	982	1748	85
H(5AB)	3806	474	2025	85
H(5AC)	3694	1629	2046	85
H(7AA)	6166	1636	4202	57
H(7AB)	6113	499	4378	57
H(8AA)	6563	1377	5379	84
H(8AB)	5415	865	5481	84
H(8AC)	5478	1998	5306	84

Figure 1. ORTEP plot of **4e**. The thermal ellipsoids of 50% probability are shown for the non-hydrogen atoms. Selected bond lengths [$\text{\AA}$] and angles [$^\circ$]: N-C(4) 1.326(3), N-C(1) 1.453(3), O(2)-C(6) 1.260(3), C(1)-C(2) 1.535(3), C(2)-C(3) 1.530(3), C(3)-C(4) 1.514(3), C(4)-C(5) 1.382(3), C(5)-C(6) 1.416(3); C(4)-N-C(1) 115.3(2), N-C(1)-C(2) 103.32(19), C(1)-C(2)-C(3) 103.9(2), N-C(4)-C(3) 107.87(19), N-C(4)-C(5) 126.0(2).

Figure 2. ORTEP plot of **4f**. The thermal ellipsoids of 50% probability are shown for the non-hydrogen atoms. Selected bond lengths [$\text{\AA}$] and angles [$^\circ$]: N(1A)-C(4A) 1.335(4), N(1A)-C(1A) 1.468(4), O(1A)-C(6A) 1.222(3), C(1A)-C(2A) 1.527(4), C(2A)-C(3A) 1.530(4), C(3A)-C(4A) 1.514(4), C(4A)-C(5A) 1.369(4), C(5A)-C(6A) 1.423(4); C(4A)-N(1A)-C(1A) 113.2(2), N(1A)-C(1A)-C(2A) 102.0(2), C(1A)-C(2A)-C(3A) 103.4(2), C(4A)-C(3A)-C(8A) 110.8(2), N(1A)-C(4A)-C(5A) 127.4(3).

Figure 3. Crystal lattice of **4f**.

Figure 4. ORTEP plot of *cis*-**4h**. The thermal ellipsoids of 50% probability are shown for the non-hydrogen atoms. Selected bond lengths [Å] and angles [°]: N(1)-C(1) 1.335(2), N(1)-C(4) 1.459(2), O(1)-C(6) 1.219(2), C(1)-C(2) 1.510(2), C(2)-C(3) 1.533(2), C(3)-C(4) 1.522(3), C(1)-C(5) 1.361(2), C(5)-C(6) 1.431(2); C(1)-N(1)-C(4) 113.58(15), N(1)-C(4)-C(3) 103.14(14), C(4)-C(3)-C(2) 101.90(13), C(1)-C(2)-C(3) 103.67(14), O(4)-C(2)-C(1) 110.34(14), N(1)-C(1)-C(5) 128.74(16).

Figure 5. ORTEP plot of **6e**. The thermal ellipsoids of 50% probability are shown for the non-hydrogen atoms. Selected bond lengths [$\text{\AA}$] and angles [$^\circ$]: O(1)-C(2) 1.214(3), O(2)-C(13) 1.230(3), F(1)-C(14) 1.324(3), N(1)-C(9) 1.340(4), N(1)-C(12) 1.384(3), C(1)-C(9) 1.495(4), C(1)-C(2) 1.518(4), C(9)-C(10) 1.385(4), C(10)-C(11) 1.385(4), C(11)-C(12) 1.400(4); C(9)-N(1)-C(12) 110.1(2), C(9)-C(1)-C(2) 114.3(2), O(1)-C(2)-C(1) 121.8(2), N(1)-C(9)-C(1) 122.0(3), C(10)-C(9)-C(1) 129.6(3), C(11)-C(10)-C(9) 107.4(3)

Figure 6. ORTEP plot of **5p**. The thermal ellipsoids of 50% probability are shown for the non-hydrogen atoms. Selected bond lengths [Å] and angles [°]: N-C(1) 1.365(18), N-C(14) 1.377(17), C(1)-C(2) 1.365(2), C(2)-C(3) 1.421(19), C(3)-C(14) 1.378(18), C(3)-C(4) 1.505(19), C(13)-C(14) 1.501(18), C(13)-C(15) 1.520(17); C(1)-N-C(14) 109.2(12), C(14)-C(3)-C(2) 106.7(12), C(3)-C(4)-C(5) 114.0(11), C(5)-C(6)-C(7) 114.9(12), C(14)-C(13)-C(15) 110.3(10), C(15)-C(13)-C(12) 111.2(10), C(3)-C(14)-C(13) 129.0(12), N-C(14)-C(13) 122.9(11).

Figure 7. Crystal lattice of **5p**.

Figure 8. ORTEP plot of **12b**. The thermal ellipsoids of 50% probability are shown for the non-hydrogen atoms. Selected bond lengths [$\text{\AA}$] and angles [$^\circ$]: N(1A)-C(2A) 1.366(4), C(1A)-N(1A) 1.355(3), O(1A)-C(6A) 1.214(3), C(1A)-C(4A) 1.383(3), C(2A)-C(3A) 1.356(4), C(3A)-C(4A) 1.423(3), C(4A)-C(6A) 1.458(3), C(1A)-C(5A) 1.480(4); C(2A)-N(1A)-C(1A) 110.6(2), C(3A)-C(2A)-N(1A) 108.3(2), C(2A)-C(3A)-C(4A) 106.7(2), C(1A)-C(4A)-C(3A) 107.9(2), N(1A)-C(1A)-C(5A) 121.2(2), C(1A)-C(4A)-C(6A) 125.3(2).

Figure 9. Crystal lattice of **12b**.

Ernst-Moritz-Amdt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrom (Protonenbreitbandentkoppelt)
E. Behr: EB 0110-1
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77,0 ppm
Messstemperatur: 298 K (25 °C)

```

Current Data Parameters
NAME      BB_EB010-1
EXPNO    1
PROCNO   13

I2 - Acquisition Parameters
Date_     20030225
Time      11:37
INSTRUM   spect
PROBHD    5 mm Mxline
PULPROG   zgpg30
TD         32768
SOLVENT   DMSO
DS         256
NS         8
SWH        21799.311 Hz
FIDRES     0.066265 Hz
AQ         0.7573140 sec
RG          32.000
WDW         32.000 usec
DE          32.86 usec
TE         298.0 K
0.00000200 sec
DLS         30.00 dB
P1         2.00000000 sec
CPDPRG2    waltz16
P2         1.00000000 sec
DLS         22.00 dB
TE          2.50 usec
SF01       75.4761717 MHz
NUC1        13C

I2 - Processing parameters
SI         16384
SF         75.467499 MHz
WDW         HM
SSB         0
GB          0
LB          0.70 Hz
GB          0
LB          1.00

```

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektrum (Protonenbreitbandenkoppelung)
E.Bellur: EB0172-2
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77.0 ppm
Messstemperatur: 298 K (25 °C)

```

Current Data Parameters
NAME: BH_1800172-2
EXPNO: 13
PROCNO: 1

12 - Acquisition Parameters
Date_   20090415
Time    16:14
INSTRUM spect
PROBHD  5 mm Mxline
PULPROG zgpg
TD       32768
SOLVENT CDCl3
NS       1238
DS       0
SWH      2179.133 Hz
FIDRES   0.662640 Hz
AQ        0.5753740 sec
RG        32
ZWN       23.000 usec
DE        32.86 usec
TE        298.0 K
D1        0.00000000 sec
D12       30.00 usec
DL        30.00 usec
D13       2.00000000 sec
CDEPRG   waltz16
P1        11.00 usec
P2        0.10000000 sec
P3        0.20000000 sec
DE15      22.00 usec
DE16      22.50 usec
SF01      75.4761717 MHz
NUC1      13C

13 - Processing parameters
SI        16384
SF         75.607418 MHz
WDW        RM
SSB         0
GB         1.20 Hz
PC          0.8
  
```

Ernst-Moritz-Arndt-Universität Greifswald
ARX300 – Kernresonanz-Spektrometer
13C-NMR-Spektren (Protonenbreitbandenkoppelt)
E. Beller: EB0173-2a
Lösungsmittel: Deuteriochloroform, CDCl₃
Referenz: Lösungsmittel = 77.0 ppm
Messstemperatur: 298 K (25 °C)

```
Current Data Parameters
NAME      BH_J00173-24
EXPNO     13
PROCNO    1
```

F2 - Acquisition Parameters	
Date_	20030509
Time	14.00
INSTRUM	spect
PROBHD	5 mm Maltix
PULPROG	zgpg
TD	32768
SOLVENT	CDCl ₃
NS	256
DS	0
SWH	21739.131 Hz
FIDRES	0.0634626 Hz
AQ	0.7537140 sec
RG	32768
DW	23.000 nsec
DE	32.85 nsec
TI	298.0 K
D12	0.0003000 sec
D15	30.00 nsec
TI	2.00000000 sec
CPDPRG	waltz16
P21	110.00 nsec
D11	0.00000000 sec
D15	22.00 nsec
PI	2.50 nsec
SFO1	75.4761177 MHz
NUC1	¹³ C

F2 - Processing parameters	
SI	16384
SF	75.4673565 MHz
WDW	EM
SSB	0
LB	0.70 Hz
GB	0
PC	1.00

**AK
WEISZ**

```
Current Data Parameters
NAME          ED0493-3
EEDNO         2
PROONO        1
```

```

F2 - Acquisition Parameters
Date_ 20040421
Time 15.17
INSTRUM spect
PROCED 5 sec TBI 18-1
PULPROG zgpg
TD 32768
SOLVENT CACl3
NU 256
DS 0
SFO 37591.984 Hz
FIDRES 1.147277 Hz
AQ 0.4356777 sec
RG 1149.4
DE 13.50 use
WE 6.00 use
TE 277.1 K
D1 2.00000000 sec
d11 0.00000000 sec
DECAST 0.00000000 sec
MORPH 0.01000000 sec

```

```

----- CHANNEL f1 -----
MUC1          15C
P1            4.00 use
PCL           0.00 dB
SP01         150.9291628 MHz

----- CHANNEL f2 -----
CP0P902       waits14
MUC2          1M
PCP02         100.00 use
PCL           120.00 dB
PCL2          20.30 dB
SP02         600.1128051 MHz

```

```
F2 - Processing parameters
SI                32768
SF                *150.9024889 MHz
WDW               RM
SSB               0
LB                2.00 Hz
GB               0
PC               1.00
```