# Supplementary data

## $Rh_2(OAc)_4$-catalyzed reactions of α-diazoimides: a simple and novel synthesis of mono- and bis(2,3-fused perhydrooxazol-4-one) systems

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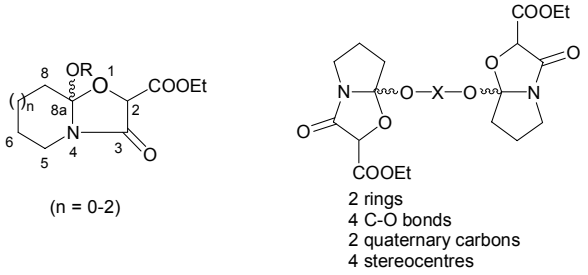


**Experimental Section**

**General Methods**. The melting points are uncorrected. The FT-IR spectra were recorded using KBr or neat method unless otherwise stated. $^1$H NMR and $^{13}$C NMR spectra were (200 MHz and 50.3 MHz, respectively) referenced to TMS. Carbon types were determined from DEPT $^{13}$C NMR experiments. Mass analyses were performed with an ionizing voltage of 70 eV or $FD^+$ method in absolute dichloromethane. All reactions were carried out under an argon atmosphere and solutions dried with anhydrous magnesium sulfate. Analytical thin layer chromatography (TLC) was performed on silica plates and components were visualized by observation under iodine. Column chromatography was performed on silica gel (100-200 mesh). Benzene was dried over sodium.

**Typical Procedures for the Rhodium(II) Acetate Catalyzed Reactions of α-Diazoimides 1. Method A:** To an oven-dried flask containing a dry benzene solution (25 mL) of the appropriate α-diazoimide (1.0 mmol) and appropriate nucleophile (water or absolute alcohol, 1.5 mmol) was added 0.3 mol% of rhodium(II) acetate catalyst under an argon atmosphere. The reaction mixture was heated at reflux for 3 h under an argon atmosphere by following TLC until the disappearance of diazo carbonyl compound. The solvent was removed under reduced pressure and the residue purified using silica column

chromatography (hexane/EtOAc) to afford the respective products **2a-p** and **3**

**Method B:** To an oven-dried flask containing a dry benzene solution (25 mL) of the appropriate α-diazoimide (2.5 mmol) and appropriate dihydroxy compound (1.0 mmol) was added 0.3 mol% of rhodium(II) acetate catalyst and followed as described in method A.

**Structural data for compounds 2a-p, 3 and 5a-f.**

**Ethyl 8a-hydroxy-3-oxo-2,3,6,7,8,8a-hexahydro-5*H*-[1,3]oxazolo[3,2-*a*]pyridine-2-carboxylate (2a).** Title compound was synthesized by following the method A. Yield: 186 mg (76%); Colorless solid; mp 104–106 $^0$C (hexane/EtOAc). Obtained as 43:57 mixture of diastereomers. IR (KBr): 3324 (OH), 1748, 1689, 1476, 1273 cm$^{-1}$. MS (EI, 70 eV), *m/z* 229 ($M^+$, 9), 212 (56), 156 (41), 126 (51), 100 (100%). Anal. Calcd for $C_{10}H_{15}NO_5$: C, 52.40; H, 6.60; N, 6.11. Found: C, 52.59; H, 6.57; N, 6.10. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 3H, $J$ = 7.1 Hz, $CH_3$), 1.39–1.46 (m, 1H), 1.64–1.84 (m, 4H), 2.26–2.33 (m, 1H), 3.05 (dt, 1H, $J_1$ = 12.9 Hz, $J_2$ = 2.4 Hz), 4.00 (dd, 1H, $J_1$ = 13.1 Hz, $J_2$ = 5.1 Hz), 4.22-4.33 (m, 2H, $OCH_2$), 4.97 (s, 1H, CH), 5.60 (br s, 1H, OH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.6 ($CH_3$), 21.7 ($CH_2$), 24.7 ($CH_2$), 36.6 ($CH_2$), 39.6 ($CH_2$), 62.8 ($OCH_2$), 77.1 (CH), 109.9 (*quat-C*), 163.8 (*quat-C*), 167.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 4.80 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.5 ($CH_3$), 21.9 ($CH_2$), 24.8 ($CH_2$), 36.9 ($CH_2$), 39.4 ($CH_2$), 63.9 ($OCH_2$), 77.4 (CH), 109.6 (*quat-C*), 163.2 (*quat-C*), 170.3 (*quat-C*).

**Ethyl 9a-hydroxy-3-oxo-2,3,5,6,7,8,9,9a-octahydro[1,3]oxazolo[3,2-*a*]azepine-2-carboxylate (2b).** Title compound was synthesized by following the method A. Yield: 165 mg (65%); Colorless solid; mp 99–101 $^0$C (hexane/EtOAc). Obtained as 30:70 mixture of diastereomers. IR (KBr): 3426 (OH), 2986, 1749, 1692, 1245 cm$^{-1}$. MS (EI, 70 eV), *m/z* 243 ($M^+$, 58), 200 (22), 170 (63), 140 (90), 114 (65), 96 (45), 85 (30), 69 (94), 55 (66), 41 (95), 29 (100). Anal. Calcd for $C_{11}H_{17}NO_5$: C, 54.31; H, 7.04; N,

5.76. Found: C, 54.38; H, 7.03; N, 5.75. Major diastereomer: $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 3H, *J* = 7.1 Hz, $CH_3$), 1.37–2.07 (m, 7H), 2.33–2.44 (m, 1H), 2.96–3.09 (m, 1H), 3.81–3.92 (m, 1H), 4.26–4.41 (m, 2H, $OCH_2$), 4.86 (s, 1H, CH), 5.09 (br s, 1H, OH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 13.9 ($CH_3$), 22.4 ($CH_2$), 27.9 ($CH_2$), 29.5 ($CH_2$), 39.1 ($CH_2$), 40.2 ($CH_2$), 63.5 ($OCH_2$), 76.9 (CH), 113.9 (*quat-C*), 164.6 (*quat-C*), 170.3 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 4.94 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.1 ($CH_3$), 27.6 ($CH_2$), 29.3 ($CH_2$), 40.4 ($CH_2$), 62.1 ($OCH_2$), 77.0 (CH).

**Ethyl 7a-methoxy-3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate (2c).** Title compound was synthesized by following the method A. Yield: 82 mg (53%); Colorless oil. Obtained as 30:70 mixture of diastereomers. IR (neat, NaCl): 2983, 1731, 1456, 1385 $cm^{-1}$. MS (EI, 70 eV), *m/z* 229 ($M^+$, 3), 198 (51), 100 (74), 60 (48), 41 (100). Anal. Calcd for $C_{10}H_{15}NO_5$: C, 52.40; H, 6.60; N, 6.11. Found: C, 52.63; H, 6.63; N, 6.12. Major diastereomer: $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 3H, *J* = 7.1 Hz, $CH_3$), 2.14–2.25 (m, 4H), 3.12–3.18 (m, 1H), 3.29 (s, 3H, $OCH_3$), 3.69–3.78 (m, 1H), 4.23–4.37 (m, 2H, $OCH_2$), 5.19 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 13.7 ($CH_3$), 23.8 ($CH_2$), 34.6 ($CH_2$), 41.5 ($CH_2$), 49.8 ($OCH_3$), 62.3 ($CH_2$), 81.3 (CH), 121.7 (*quat-C*), 166.5 (*quat-C*), 167.8 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 3.44 (s, 3H, $OCH_3$), 4.87 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 23.6 ($CH_2$), 34.5 ($CH_2$), 41.4 ($CH_2$), 50.2 ($OCH_3$), 62.2 ($CH_2$), 79.5 (CH), 120.6 (*quat-C*).

**Ethyl 8a-methoxy-3-oxo-2,3,6,7,8,8a-hexahydro-5*H*-[1,3]oxazolo[3,2-*a*]pyridine-2-carboxylate (2d).** Title compound was synthesized by following the method A. Yield: 129 mg (84%); Colorless solid; mp 104–106 $^{0}$C (hexane/EtOAc). Obtained as 34:66 mixture of diastereomers. IR (KBr): 2948, 1748, 1714, 1453 $cm^{-1}$. MS (EI, 70 eV), *m/z* 243 ($M^+$, 4), 212 (23), 100 (23), 82 (50), 55 (100), 41 (66). Anal. Calcd for $C_{11}H_{17}NO_5$: C, 54.31; H, 7.04; N, 5.76. Found: C, 54.42; H, 7.01; N, 5.78. Major diastereomer: $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.34 (t, 3H, *J* = 7.1 Hz, $CH_3$), 1.40–1.51 (m, 1H), 1.68–

1.93 (m, 4H), 2.21–2.29 (m, 1H), 2.74–2.87 (m, 1H), 3.16 (s, 3H, $OCH_3$), 4.02–4.11 (m, 1H), 4.23 (m, 2H, $OCH_2$), 4.97 (s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.0 ($CH_3$), 21.2 ($CH_2$), 24.0 ($CH_2$), 36.0 ($CH_2$), 39.0 ($CH_2$), 47.8 ($OCH_3$), 62.2 ($OCH_2$), 77.6 (CH), 111.6 (*quat-C*), 163.2 (*quat-C*), 165.9 (*quat-C*), 166.6 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1H$ NMR (200 MHz, $CDCl_3$): δ 3.34 (s, 3H, $OCH_3$), 4.77 (s, 1H, CH), $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 39.2 ($CH_2$), 48.7 ($OCH_3$), 62.1 ($OCH_2$), 76.4 (CH), 111.5 (*quat-C*).

**Ethyl 9a-methoxy-3-oxo-2,3,5,6,7,8,9,9a-octahydro[1,3]oxazolo[3,2-*a*]azepine-2-carboxylate (2e).** Title compound was synthesized by following the method A. Yield: 123 mg (77%); Colorless solid; mp 95–97 $^0C$ (hexane/EtOAc). Obtained as 24:76 mixture of diastereomers. IR (KBr): 2927,1732, 1698, 1456 $cm^{-1}$. MS (EI, 70 eV), *m/z* 257 ($M^+$, 2), 226 (33), 140 (23), 90 (34), 83 (26), 69 (57), 51 (82), 44 (100). Anal. Calcd for $C_{12}H_{19}NO_5$: C, 56.02; H, 7.44; N, 5.44. Found: C, 56.15; H, 7.40; N, 5.48. Major diastereomer: $^1H$ NMR (200 MHz, $CDCl_3$): δ 1.34 (t, 3H, $J$ = 7.1 Hz, $CH_3$), 1.56–1.82 (m, 6H), 2.0–2.1 (m, 1H), 2.31–2.47 (m, 1H), 2.78–2.96 (m, 1H), 3.15 (s, 3H, $OCH_3$), 3.76–3.89 (m, 1H), 4.25–4.38 (m, 2H, $OCH_2$), 4.91 (s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 13.9 ($CH_3$), 22.2 ($CH_2$), 27.8 ($CH_2$), 29.6 ($CH_2$), 39.1 ($CH_2$), 40.2 ($CH_2$), 47.7 ($OCH_3$), 63.2 ($OCH_2$), 76.7 (CH), 113.7 (*quat-C*), 164.7 (*quat-C*), 170.3 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1H$ NMR (200 MHz, $CDCl_3$): δ 3.32 (s, 3H, $OCH_3$), 4.82 (s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.0 ($CH_3$) 27.6 ($CH_2$), 29.5 ($CH_2$), 40.4 ($CH_2$), 48.5 ($OCH_3$), 62.1 ($OCH_2$).

**Ethyl 7a-benzyloxy-3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate (2f).** Title compound was synthesized by following the method A. Yield: 232 mg (85%); Colorless oil. Obtained as 40:60 mixture of diastereomers. IR ($CHCl_3$): 2983, 1753, 1733, 1373 $cm^{-1}$. MS (EI, 70 eV), *m/z* 305 ($M^+$, 2), 198 (100), 91 (70). Anal. Calcd for $C_{16}H_{19}NO_5$: C, 62.94; H, 6.27; N, 4.59. Found: C, 62.71; H, 6.25; N, 4.60.Major diastereomer: $^1H$ NMR (200 MHz, $CDCl_3$): δ 1.30 (t, 3H, $J$ = 7.2 Hz, $CH_3$), 2.04–2.34 (m, 4H), 3.00–3.18 (m, 1H), 3.66–3.79 (m, 1H), 4.24–4.37 (m, 2H, $OCH_2$), 4.49–4.67

(m, 2H), 5.25 (s, 1H, CH), 7.27–7.34 (m, 5H). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 13.7 ($CH_3$), 23.4 ($CH_2$), 34.6 ($CH_2$), 41.2 ($CH_2$), 62.0 ($CH_2$), 63.7 ($CH_2$), 80.8 (CH), 121.3 (*quat-C*), 127.3 (CH), 127.4 (CH), 127.5 (CH), 127.9 (CH), 128.0 (CH), 137.1 (*quat-C*), 166.1 (*quat-C*), 167.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 4.94 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 23.3 ($CH_2$), 34.5 ($CH_2$), 41.1 ($CH_2$), 61.9 ($CH_2$), 64.3 ($CH_2$), 79.2 (CH), 120.2 (*quat-C*), 137.2 (*quat-C*), 165.0 (*quat-C*), 166.3 (*quat-C*).

**Ethyl 8a-benzyloxy-3-oxo-2,3,6,7,8,8a-hexahydro-5*H*-[1,3]oxazolo[3,2-*a*]pyridine-2-carboxylate (2g).** Title compound was synthesized by following the method A. Yield: 121 mg (90%); Colorless solid; mp 63–65 $^0$C (hexane/EtOAc). Obtained as 44:56 mixture of diastereomers. IR (KBr): 2936, 1732, 1455, 1422 cm$^{-1}$. MS (EI, 70 eV), *m/z* 319 ($M^+$, 5), 212 (100), 91 (76), 55 (16). Anal. Calcd for $C_{17}H_{21}NO_5$: C, 63.94; H, 6.63; N, 4.39. Found: C, 64.02; H, 6.64; N, 4.37. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 3H, *J* = 7.1 Hz, $CH_3$), 1.42–1.54 (m, 1H), 1.61–1.89 (m, 4H), 2.26–2.39 (m, 1H), 2.71–2.89 (m, 1H), 4.02–4.11 (m, 1H), 4.23–4.30 (m, 2H, $OCH_2$), 4.39–4.67 (m, 2H), 5.00 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.2 ($CH_3$), 21.4 ($CH_2$), 24.1 ($CH_2$), 24.2 ($CH_2$), 36.4 ($CH_2$), 39.4 ($CH_2$), 62.5 ($CH_2$), 63.2 ($CH_2$), 77.7 (CH), 111.7 (*quat-C*), 127.7 (CH), 127.9 (CH), 128.4 (CH), 128.5 (CH), 136.9 (*quat-C*), 163.2 (*quat-C*), 166.6 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 4.82 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 36.5 ($CH_2$), 39.6 ($CH_2$), 62.4 ($CH_2$), 63.7 ($CH_2$), 76.6 (CH), 110.8 (*quat-C*), 137.4 (*quat-C*), 163.3 (*quat-C*), 166.1 (*quat-C*).

**Ethyl 9a-benzyloxy-3-oxo-2,3,5,6,7,8,9,9a-octahydro[1,3]oxazolo[3,2-*a*]azepine-2-carboxylate (2h).** Title compound was synthesized by following the method A. Yield: 171 mg (86%); Colorless oil. Obtained as 30:70 mixture of diastereomers. IR (neat, NaCl): 2936, 1752, 1718, 1454 cm$^{-1}$. MS (EI, 70 eV), *m/z* 333 ($M^+$, 3), 226 (100), 91 (65). Anal. Calcd for $C_{18}H_{23}NO_5$: C, 64.85; H, 6.95; N, 4.20. Found: C, 64.62; H, 6.97; N, 4.18. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.34 (t, 3H, *J* = 7.1 Hz,

$CH_3$), 1.56–1.83 (m, 6H), 2.02–2.15 (m, 1H), 2.48–2.58 (m, 1H), 2.84–2.96 (m, 1H), 3.74–3.81 (m, 1H), 4.23–4.33 (m, 2H, $OCH_2$), 4.34–4.64 (m, 2H), 4.94 (s, 1H, CH), 7.27–7.32 (m, 5H). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.7 ($CH_3$), 22.8 ($CH_2$), 27.7 ($CH_2$), 30.0 ($CH_2$), 41.2 ($CH_2$), 41.5 ($CH_2$), 63.0 ($CH_2$), 64.2 ($CH_2$), 64.6 ($CH_2$), 77.6 (CH), 116.7 (*quat-C*), 137.5 (*quat-C*), 165.8 (*quat-C*), 166.0 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): 4.87 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 23.1 ($CH_2$), 26.6 ($CH_2$), 29.6 ($CH_2$), 62.9 ($CH_2$), 65.6 ($CH_2$), 77.1 (CH), 116.5 (*quat-C*), 138.0 (*quat-C*).

**Ethyl 7a-isopropoxy-3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate (2i).** Title compound was synthesized by following the method A. Yield: 196 mg (85%); Yellow oil. Obtained as 47:53 mixture of diastereomers. IR (neat, NaCl): 2979, 1754, 1733, 1459 $cm^{-1}$. MS (EI, 70 eV), *m/z* 257 ($M^+$, 3), 198 (100), 142 (20), 126 (22), 86 (25) 41 (39). Anal. Calcd for $C_{12}H_{19}NO_5$: C, 56.02; H, 7.44; N, 5.44. Found: C, 56.09; H, 7.42; N, 5.42. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.12 (t, 3H, $J$ = 7.1 Hz, $CH_3$), 1.22–1.37 (m, 6H), 2.04–2.20 (m, 4H), 3.10–3.22 (m, 1H), 3.65–3.77 (m, 1H), 3.86–3.96 (m, 1H), 4.23–4.37 (m, 2H, $OCH_2$), 5.19 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.5 ($CH_3$), 23.9 ($CH_3$), 24.2 ($CH_2$), 35.9 ($CH_2$), 42.1 ($CH_2$), 62.8 ($CH_2$), 66.2 (OCH), 81.4 (CH), 122.1 (*quat-C*), 167.2 (*quat-C*), 168.4 (*quat-C*), Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): 4.85 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): 23.6 ($CH_3$), 24.1 ($CH_2$), 35.7 ($CH_2$), 42.2 ($CH_2$), 62.6 ($CH_2$), 79.8 (CH), 120.9 (*quat-C*), 165.9 (*quat-C*), 168.5 (*quat-C*).

**Ethyl 7a-cyclohexyloxy-3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate (2j).** Title compound was synthesized by following the method A. Yield: 160 mg (81%); Colorless thick oil. Obtained as 32:68 mixture of diastereomers. IR (neat, NaCl): 2936, 1754, 1730, 1372 $cm^{-1}$. MS (EI, 70 eV), *m/z* 297 ($M^+$, 4), 198 (100), 142 (33), 112 (16), 86 (60) 41 (48). Anal. Calcd for $C_{15}H_{23}NO_5$: C, 60.59; H, 7.80; N, 4.71. Found: C, 60.83; H, 7.78; N, 4.72. Major diastereomer: $^1$H NMR (200 MHz,

$CDCl_3$): δ 1.24–1.49 (m, 8H), 1.61–1.89 (m, 5H), 1.89–2.17 (m, 4H), 3.10–3.22 (m, 1H), 3.56–3.76 (m, 2H), 4.36 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 5.19 (s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.5 ($CH_3$), 24.3 ($CH_2$), 24.6 ($CH_2$), 24.8 ($CH_2$), 25.8 ($CH_2$), 33.9 ($CH_2$), 34.5 ($CH_2$), 35.8 ($CH_2$), 42.0 ($CH_2$), 62.8 ($CH_2$), 71.9 (CH), 81.4 (CH), 122.0 (*quat-C*), 167.1 (*quat-C*), 168.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1H$ NMR (200 MHz, $CDCl_3$): δ 4.85 (s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.6 ($CH_3$), 24.1 ($CH_2$), 24.7 ($CH_2$), 25.9 ($CH_2$), 33.7 ($CH_2$), 35.7 ($CH_2$), 42.1 ($CH_2$), 62.6 ($CH_2$), 71.6 (CH), 79.9 (CH), 120.8 (*quat-C*), 165.8 (*quat-C*), 168.5 (*quat-C*).

**Ethyl 7a-hexadecyloxy-3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate (2k).** Title compound was synthesized by following the method A. Yield: 162 mg (64%); Colorless oil. Obtained as 38:62 mixture of diastereomers. IR (neat, NaCl): 2926, 1756, 1737, 1463 $cm^{-1}$. MS (EI, 70 eV), *m/z* 439 ($M^+$, 12), 366 (6), 310 (5), 198 (100), 142 (13), 126 (22), 86 (19). Anal. Calcd for $C_{25}H_{45}NO_5$: C, 68.30; H, 10.32; N, 3.19. Found: C, 68.48; H, 10.35; N, 3.19. Major diastereomer: $^1H$ NMR (200 MHz, $CDCl_3$): δ 0.88 (t, 3H, *J* = 7.2 Hz), 1.18–1.47 (m, 25H), 1.52–1.64 (m, 3H), 2.12–2.26 (m, 4H), 3.04–3.22 (m, 1H), 3.36–3.55 (m, 2H), 3.62–3.87 (m, 2H), 4.28 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 5.16 (s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.7 ($CH_3$), 23.3 ($CH_2$), 24.5 ($CH_2$), 26.8 ($CH_2$), 30.0 ($CH_2$), 30.3 ($CH_2$), 32.5 ($CH_2$), 35.6 ($CH_2$), 42.1 ($CH_2$), 62.4 ($CH_2$), 62.9 ($CH_2$), 63.2 ($CH_2$), 81.8 (CH), 122.0 (*quat-C*), 167.2 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1H$ NMR (200 MHz, $CDCl_3$): δ 4.86(s, 1H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 24.3 ($CH_2$), 35.5 ($CH_2$), 42.0 ($CH_2$), 62.8 ($CH_2$), 80.1 (CH), 121.0 (*quat-C*), 165.9 (*quat-C*), 168.3 (*quat-C*), 168.4 (*quat-C*).

**Ethyl 8a-hexadecyloxy-3-oxo-2,3,6,7,8,8a-hexahydro-5*H*-[1,3]oxazolo[3,2-*a*]pyridine-2-carboxylate (2l).** Title compound was synthesized by following the method A. Yield: 63 mg (57%); Colorless oil. Obtained as 43:57 mixture of diastereomers. IR (neat, NaCl): 2926, 1756, 1728, 1425 $cm^{-1}$. MS (EI, 70 eV), *m/z* 453 ($M^+$, 13), 212 (100), 140 (15), 126 (44), 100 (25), 43 (46). Anal. Calcd for

$C_{26}H_{47}NO_5$: C, 68.84; H, 10.44; N, 3.09. Found: C, 68.69; H, 10.41; N, 3.08. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 0.88 (t, 3H, *J* = 7.2 Hz), 1.26–1.36 (m, 26H), 1.50–1.95 (m, 8H), 2.24–2.30 (m, 1H), 2.74–2.87 (m, 1H), 3.16–3.23 (m, 1H), 3.38–3.43 (m, 1H), 4.25 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 4.94 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.7 ($CH_3$), 22.0 ($CH_2$), 23.3 ($CH_2$), 24.8 ($CH_2$), 26.9 ($CH_2$), 29.9 ($CH_2$), 30.0 ($CH_2$), 30.3 ($CH_2$), 32.5 ($CH_2$), 37.0 ($CH_2$), 39.8 ($CH_2$), 40.0 ($CH_2$), 61.4 ($CH_2$), 62.9 ($CH_2$), 78.2 (CH), 111.9 (*quat-C*), 163.8 (*quat-C*), 167.3 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 4.76 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 24.9 ($CH_2$), 62.1 ($CH_2$), 62.7 ($CH_2$).

**Ethyl 9a-hexadecyloxy-3-oxo-2,3,5,6,7,8,9,9a-octahydro[1,3]oxazolo[3,2-*a*]azepine-2-carboxylate (2m).** Title compound was synthesized by following the method A. Yield: 119 mg (61%); Colorless oil. Obtained as 44:56 mixture of diastereomers. IR (neat, NaCl): 2925, 1756, 1726, 1454 cm$^{-1}$. MS (FD$^+$), *m/z* 467 ($M^+$). Anal. Calcd for $C_{27}H_{49}NO_5$: C, 69.34.; H, 10.56; N, 2.99. Found: C, 69.09; H, 10.59; N, 2.98. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 0.88 (t, 3H, *J* = 7.2 Hz), 1.20–1.33 (m, 27H), 1.48–1.75 (m, 6H), 1.94–2.21 (m, 3H), 2.83–3.01 (m, 1H), 3.21–3.37 (m, 2H), 3.78–3.87 (m, 2H), 4.30 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 4.80 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.7 ($CH_3$), 22.8 ($CH_2$), 23.3 ($CH_2$), 26.9 ($CH_2$), 27.8 ($CH_2$), 29.6 ($CH_2$), 30.0 ($CH_2$), 30.1 ($CH_2$), 30.3 ($CH_2$), 32.5 ($CH_2$), 41.2 ($CH_2$), 41.4 ($CH_2$), 41.5 ($CH_2$), 62.5 ($CH_2$), 62.7 ($CH_2$), 76.9 (CH), 115.6 (*quat-C*), 165.9 (*quat-C*), 168.6 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 4.87 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 23.2 ($CH_2$), 26.7 ($CH_2$), 41.1 ($CH_2$), 61.8 ($CH_2$), 62.9 ($CH_2$), 77.6 (CH), 116.4 (*quat-C*), 165.7 (*quat-C*), 168.9 (*quat-C*).

**Ethyl 3-oxo-7a-(pyren-1-ylmethoxy)-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate (2n).** Title compound was synthesized by following the method A. Yield: 178 mg (89%); Yellow oil. Obtained as 46:54 mixture of diastereomers. IR ($CHCl_3$): 3053, 2986, 1750, 1731, cm$^{-1}$. MS (EI, 70 eV), *m/z* 429 ($M^+$, 16), 232 (33), 215 (77), 198 (100), 126 (22). Anal. Calcd for $C_{26}H_{23}NO_5$: C,

72.71; H, 5.40; N, 3.26. Found: C, 72.81; H, 5.41; N, 3.26. Major diastereomer: $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.31 (t, 3H, *J* = 7.1 Hz, $CH_3$), 1.98–2.22 (m, 4H), 2.87–3.09 (m, 1H), 3.59–3.76 (m, 1H), 3.66–3.75 (m, 1H), 4.32 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 4.98 (s, 1H, CH), 5.35 (s, 2H, $OCH_2$Py), 7.78–8.22 (m, 8H), 8.26–8.31 (m, 1H). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.6 ($CH_3$), 24.3 ($CH_2$), 35.6 ($CH_2$), 42.3 ($CH_2$), 59.9 ($CH_2$), 63.6 ($CH_2$), 81.9 (CH), 122.3 (*quat-C*), 123.3 (CH), 124.9 (CH), 125.7 (CH) 126.3 (CH), 127.7 (CH), 128.2 (CH), 129.8 (*quat-C*), 130.2 (*quat-C*), 131.0 (*quat-C*), 131.5 (*quat-C*), 131.6 (*quat-C*), 167.0 (*quat-C*), 168.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 5.09 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.5 ($CH_3$), 24.1 ($CH_2$), 35.5 ($CH_2$), 42.1 ($CH_2$), 63.0 ($CH_2$), 80.1 (CH), 121.2 (*quat-C*), 123.7 (CH), 125.6 (CH), 127.5 (CH), 127.9 (CH), 128.1 (CH), 129.7 (*quat-C*), 131.1 (*quat-C*), 131.7 (*quat-C*), 166.0 (*quat-C*), 168.2 (*quat-C*).

**Ethyl 3-oxo-8a-(pyren-1-ylmethoxy)-2,3,6,7,8,8a-hexahydro-5*H*-[1,3]oxazolo[3,2-*a*]pyridine-2-carboxylate (2o).** Title compound was synthesized by following the method A. Yield: 79 mg (94%); Yellow solid. mp 96–98 $^{0}$C (hexane/EtOAc). Obtained as 34:66 mixture of diastereomers. IR (KBr): 2960, 1755, 1729, 1421 cm$^{-1}$. MS (EI, 70 eV), *m/z* 443 ($M^+$, 12), 232 (100), 215 (70), 203(61), 138 (24). Anal. Calcd for $C_{27}H_{25}NO_5$: C, 73.12; H, 5.68; N, 3.16. Found: C, 72.50; H, 5.69; N, 3.16. Major diastereomer: $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.34 (t, 3H, *J* = 7.1 Hz, $CH_3$), 1.42–1.76 (m, 4H), 1.96–2.21 (m, 1H), 2.32–2.51 (m, 1H), 2.52–2.74 (m, 1H), 3.99–4.08 (m, 1H), 4.33 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 5.14 (s, 1H, CH), 5.26 (s, 2H, $OCH_2$Py), 7.84–8.24 (m, 8H), 8.37 (d, 1H, *J* = 9.2 Hz). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 22.0 ($CH_2$), 24.8 ($CH_2$), 37.2 ($CH_2$), 40.2 ($CH_2$), 40.4 ($CH_2$), 62.6 ($CH_2$), 63.6 ($CH_2$), 78.9 (CH), 112.7 (*quat-C*), 123.7 (CH), 126.7 (CH), 127.5 (CH), 127.6 (CH), 128.0 (CH) 128.2 (CH), 128.6 (CH), 130.0 (*quat-C*), 131.4 (*quat-C*), 131.9 (*quat-C*), 132.1 (*quat-C*), 167.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 5.08 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.8 ($CH_3$), 37.3 ($CH_2$), 63.2 ($CH_2$), 78.4 (CH),

112.4 (*quat-C*), 123.6 (CH), 125.9 (CH), 126.6 (CH) 128.4 (CH), 128.5 (*quat-C*), 130.1 (*quat-C*), 131.6 (*quat-C*), 132.0 (*quat-C*), 164.1 (*quat-C*).

**Ethyl 3-oxo-9a-(pyren-1-ylmethoxy)-2,3,5,6,7,8,9,9a-octahydro[1,3]oxazolo[3,2-*a*]azepine-2-carboxylate (2p).** Title compound was synthesized by following the method A. Yield: 97 mg (83%); Yellow solid. mp 63-65 $^0$C (hexane/EtOAc). Obtained as 41:59 mixture of diastereomers. IR (KBr): 2927, 1751, 1717, 1415 cm$^{-1}$. MS (FD$^+$), *m/z* 457 (M$^+$). Anal. Calcd for $C_{28}H_{27}NO_5$: C, 73.51; H, 5.95; N, 3.06. Found: C, 73.73; H, 5.97; N, 3.07. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.20–1.37 (m, 4H), 1.56–1.73 (m, 4H), 1.99–2.37 (m, 1H), 2.36–2.51 (m, 2H), 2.52–2.83 (m, 1H), 3.58–3.76 (m, 1H), 4.35 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 5.09 (s, 1H, CH), 5.20 (s, 2H, $OCH_2$Py), 7.77–8.16 (m, 8H), 8.31 (d, 1H, *J* = 9.4 Hz). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.8 ($CH_3$), 22.9 ($CH_2$), 26.6 ($CH_2$), 27.8 ($CH_2$), 29.6 ($CH_2$), 30.1 ($CH_2$), 41.3 ($CH_2$), 41.7 ($CH_2$), 63.2 ($CH_2$), 64.2 ($CH_2$), 78.0 (CH), 117.1 (*quat-C*), 123.5 (CH), 123.6 (CH), 125.8 (CH), 126.0 (CH) 126.4 (CH), 126.5 (CH), 126.7 (CH), 127.6 (CH) 128.0 (CH), 128.3 (CH), 128.4 (*quat-C*), 128.6 (CH), 130.0 (*quat-C*), 131.4 (*quat-C*), 131.9 (*quat-C*), 134.6 (*quat-C*), 167.1 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 5.08 (s, 1H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 23.2 ($CH_2$), 41.4 ($CH_2$), 41.8 ($CH_2$), 62.9 ($CH_2$), 77.4 (CH), 116.3 (*quat-C*), 125.3 (CH), 128.2 (CH), 130.2 (*quat-C*), 132.1 (*quat-C*), 132.8 (*quat-C*), 167.0 (*quat-C*).

**Ethyl 2-hydroxy-3-oxo-3-(2-oxopyrrolidin-1-yl)propanoate (3).** Title compound was synthesized by following the method A. Yield: 235 mg (55%); Colorless oil. Obtained as 46:54 mixture of diastereomers. IR (neat, NaCl): 3450 (OH), 2981, 1741, 1370 cm$^{-1}$. MS (EI, 70 eV), *m/z* 215 (M$^+$, 1), 197 (10), 142 (26), 125 (49), 85 (51), 69 (28), 41 (88), 29 (100). Anal. Calcd for $C_9H_{13}NO_5$: C, 50.23; H, 6.09; N, 6.51. Found: C, 50.15; H, 6.07; N, 6.47. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.30 (t, 3H, *J* = 7.1 Hz, $CH_3$), 1.85 (br s, 1H, OH), 2.04–2.23 (m, 2H), 2.53–2.67 (m, 2H), 3.81–3.94 (m, 2H), 4.26 (q, 2H, *J* = 7.2 Hz, $OCH_2$), 5.39 (d, 1H, *J* = 5.6 Hz, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ

14.7 ($CH_3$), 18.3 ($CH_2$), 33.4 ($CH_2$), 47.0 ($CH_2$), 62.4 ($CH_2$), 73.9 (CH), 161.8 (*quat-C*), 170.0 (*quat-C*), 177.0 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.8 ($CH_2$), 46.0 ($CH_2$), 62.3 ($CH_2$), 161.4 (*quat-C*), 168.6 (*quat-C*), 174.9 (*quat-C*).

**Ethyl 7a,7a'-[ethane-1,2-diylbis(oxy)]bis(3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate) (5a).** Title compound was synthesized by following the method B. Yield: 156 mg (82%); Colorless oil. Obtained as 43:57 mixture of diastereomers. IR (neat, NaCl): 2983, 1735, 1638, 1456, cm$^{-1}$. MS (EI, 70 eV), *m/z* 456 ($M^+$, 0.4), 198 (100), 86 (23). Anal. Calcd for $C_{20}H_{28}N_2O_{10}$: C, 52.63; H, 6.18; N, 6.14. Found: C, 52.51; H, 6.19; N, 6.12. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.32 (t, 6H, *J* = 7.1 Hz, $CH_3$), 2.16–2.26 (m, 8H), 3.13–3.25 (m, 2H), 3.59–3.77 (m, 6H), 4.18–4.35 (m, 4H), 4.87 (s, 2H, CH), $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 13.7 ($CH_3$), 23.8 ($CH_2$), 34.6 ($CH_2$), 41.5 ($CH_2$), 49.8 ($OCH_2$), 62.3 ($CH_2$), 81.3 (CH), 121.7 (*quat-C*), 166.5 (*quat-C*), 167.8 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1$H NMR (200 MHz, $CDCl_3$): δ 5.18 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 23.6 ($CH_2$), 34.5 ($CH_2$), 41.4 ($CH_2$), 50.2 ($OCH_2$), 62.2 ($CH_2$), 79.5 (CH), 120.6 (*quat-C*).

**Ethyl 7a,7a'-[propane-1,3-diylbis(oxy)]bis(3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate) (5b).** Title compound was synthesized by following the method B. Yield: 199 mg (87%); Colorless thick oil. Obtained as 36:64 mixture of diastereomers. IR (neat, NaCl): 2941, 1744, 1696, 1371 cm$^{-1}$. MS (EI, 70 eV), *m/z* 470 ($M^+$, 0.5), 397 (3), 256 (24), 198 (100), 142 (16), 126 (45), 86 (30) 41 (38). Anal. Calcd for $C_{21}H_{30}N_2O_{10}$: C, 53.61; H, 6.43; N, 5.95. Found: C, 53.84; H, 6.42; N, 6.42. Major diastereomer: $^1$H NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 6H, *J* = 7.1 Hz, $CH_3$), 1.54–1.87 (m, 2H), 2.03–2.26 (m, 8H), 3.12–3.23 (m, 2H), 3.56–3.72 (m, 4H), 3.86–4.02 (m, 2H), 4.27 (q, 4H, *J* = 7.2 Hz, $OCH_2$), 5.19 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.3 ($CH_3$), 24.2 ($CH_2$), 26.6 ($CH_2$), 35.3 ($CH_2$), 41.9 ($CH_2$), 58.6 ($CH_2$), 62.5 ($CH_2$), 81.5 (CH), 121.7 (*quat–C*), 166.9 (*quat-C*), 168.2 (*quat-C*).

Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 4.88 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 24.1 ($CH_2$), 35.1 ($CH_2$), 42.0 ($CH_2$), 58.4 ($CH_2$), 62.4 ($CH_2$), 79.8 (CH), 120.7 (*quat-C*), 165.7 (*quat-C*), 168.0 (*quat-C*).

**Ethyl 7a,7a'-[butane-1,4-diylbis(oxy)]bis(3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate) (5c).** Title compound was synthesized by following the method B. Yield: 80 mg (89%); Colorless viscous oil. Obtained as a 39:61 mixture of diastereomers. IR (neat, NaCl): 2960, 1738, 1730, 1372 cm$^{-1}$. MS (FD$^{+}$), *m/z* 484 ($M^{+}$). Anal. Calcd for $C_{22}H_{32}N_2O_{10}$: C, 54.54; H, 6.66; N, 5.78. Found: C, 54.78; H, 6.67; N, 5.77. Major diastereomer: $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 6H, *J* = 7.1 Hz, $CH_3$), 1.52–1.76 (m, 4H), 2.04–2.26 (m, 8H), 3.14–3.25 (m, 2H), 3.48–3.58 (m, 4H), 3.68–3.94 (m, 2H), 4.25 (q, 4H, *J* = 7.2 Hz, $OCH_2$), 4.87 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.6 ($CH_3$), 24.4 ($CH_2$), 26.6 ($CH_2$), 35.6 ($CH_2$), 42.2 ($CH_2$), 62.0 ($CH_2$), 63.0 ($CH_2$), 81.8 (CH), 122.0 (*quat-C*), 167.2 (*quat-C*), 168.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 5.19 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 24.3 ($CH_2$), 35.5 ($CH_2$), 42.0 ($CH_2$), 62.9 ($CH_2$), 80.0 (CH), 120.9 (*quat-C*), 167.1 (*quat-C*), 168.3 (*quat-C*).

**Ethyl 7a,7a'-[hexane-1,6-diylbis(oxy)]bis(3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate) (5d).** Title compound was synthesized by following the method B. Yield: 96 mg (82%); Yellow oil. Obtained as 40:60 mixture of diastereomers. IR (neat, NaCl): 2941, 1752, 1731, 1461, 1373 cm$^{-1}$. Major diastereomer: MS (FD$^{+}$), *m/z* 512 ($M^{+}$). Anal. Calcd for $C_{24}H_{36}N_2O_{10}$: C, 56.24; H, 7.08; N, 5.47. Found: C, 56.11; H, 7.07; N, 5.46. $^{1}$H NMR (200 MHz, $CDCl_3$): δ 1.22–1.36 (m, 9H), 1.56–1.78 (m, 6H), 2.06–2.31 (m, 7H), 3.08–3.25 (m, 2H), 3.38–3.55 (m, 3H), 3.59–3.97 (m, 3H), 4.19–4.33 (m, 4H), 5.16 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 14.6 ($CH_3$), 24.5 ($CH_2$), 26.4 ($CH_2$), 29.8 ($CH_2$), 35.6 ($CH_2$), 42.1 ($CH_2$), 62.2 ($CH_2$), 63.0 ($CH_2$), 81.8 (CH), 122.0 (*quat-C*), 167.2 (*quat-C*), 168.3 (*quat-C*), 168.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^{1}$H NMR (200 MHz, $CDCl_3$): δ 4.86 (s, 2H, CH). $^{13}$C NMR (50.3 MHz, $CDCl_3$): δ 24.3 ($CH_2$), 35.5 ($CH_2$),

42.0 ($CH_2$), 62.8 ($CH_2$), 80.1 (CH), 121.8 (*quat-C*), 165.9 (*quat-C*).

**Ethyl 7a,7a'-[but-2-ynyl-1,4-diylbis(oxy)]bis(3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate) (5e).** Title compound was synthesized by following the method B. Yield: 88 mg (79%); yellow oil. Obtained as 44:56 mixture of diastereomers. IR (neat, NaCl): 2984, 1730, 1695, 1371 $cm^{-1}$. MS ($FD^+$), *m/z* 480 ($M^+$). Anal. Calcd for $C_{22}H_{28}N_2O_{10}$: C, 55.00; H, 5.87; N, 5.83. Found: C, 55.29; H, 5.84; N, 5.82. Major diastereomer: $^1H$ NMR (200 MHz, $CDCl_3$): δ 1.27–1.38 (m, 6H, $CH_3$), 2.08–2.28 (m, 8H), 3.20–3.25 (m, 2H), 3.68–3.76 (m, 2H), 4.15–4.43 (m, 8H), 4.87 (s, 2H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.6 ($CH_3$), 24.4 ($CH_2$), 35.6 ($CH_2$), 42.4 ($CH_2$), 51.1 ($CH_2$), 51.1 ($CH_2$), 63.1 ($CH_2$), 81.8 (CH), 82.0 (*quat-C*), 122.3 (*quat-C*), 166.9 (*quat-C*), 170.0 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1H$ NMR (200 MHz, $CDCl_3$): δ 5.20 (s, 2H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 24.2 ($CH_2$), 42.3 ($CH_2$), 62.6 ($CH_2$), 80.2 (CH), 121.1 (*quat-C*), 165.9 (*quat-C*), 168.6 (*quat-C*).

**Ethyl 7a,7a'-[1,4-phenylenebis(methyleneoxy)]bis(3-oxo-2,3,5,6,7,7a-hexahydropyrrolo[2,1-*b*][1,3]oxazole-2-carboxylate) (5f).** Title compound was synthesized by following the method B. Yield: 130 mg (81%); Thick yellow oil. Obtained as 38:62 mixture of diastereomers. IR ($CHCl_3$): 2982, 1749, 1731, 1371 $cm^{-1}$. MS ($FD^+$), *m/z* 532 ($M^+$). Anal. Calcd for $C_{26}H_{32}N_2O_{10}$: C, 58.64; H, 6.06; N, 5.26. Found: C, 58.75; H, 6.05; N, 5.26. Major diastereomer: $^1H$ NMR (200 MHz, $CDCl_3$): δ 1.33 (t, 6H, $J$ = 7.1 Hz, $CH_3$), 1.68–1.75 (m, 1H), 2.12–2.23 (m, 7H), 3.01–3.15 (m, 2H), 3.66–3.75 (m, 2H), 4.18–4.34 (m, 4H), 4.37–4.68 (m, 4H), 5.22 (s, 2H, CH), 7.27–7.33 (m, 4H). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 14.7 ($CH_3$), 24.5 ($CH_2$), 35.7 ($CH_2$), 42.3 ($CH_2$), 63.1 ($CH_2$), 64.5 ($CH_2$), 65.1 ($CH_2$), 81.9 ($CH_2$), 122.3 (*quat-C*), 128.4 (CH), 137.4 (*quat-C*), 137.7 (*quat-C*), 137.9 (*quat-C*), 167.0 (*quat-C*), 168.4 (*quat-C*). Minor diastereomer (selected signals from the mixture of diastereomers): $^1H$ NMR (200 MHz, $CDCl_3$): δ 4.91 (s, 2H, CH). $^{13}C$ NMR (50.3 MHz, $CDCl_3$): δ 24.4 ($CH_2$), 35.6 ($CH_2$), 42.2 ($CH_2$), 63.0 ($CH_2$), 80.3 (CH), 121.2 (*quat-C*), 128.6 (CH), 137.2 (*quat-C*), 166.0 (*quat-C*), 168.3 (*quat-C*).

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