**SYNTHESIS OF NEW CHIRAL 6-CARBONYL 2,3,8,8a-TETRAHYDRO-7H-
OXAZOLO[3,2-*a*]PYRIDINES**

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General information:

Unless otherwise specified, materials were purchased from commercial suppliers and used without further purification. THF was distilled from sodium/ benzophenone ketyl immediately prior to use. CH₂Cl₂ was distilled from calcium hydride. All reactions were carried out under argon. ¹H NMR spectra were recorded at 250 MHz and ¹³C NMR at 62.5 MHz. Chemical shifts (δ) were expressed in ppm relative to TMS at $\delta = 0$ for ¹H and to CDCl₃ at $\delta = 77.1$ for ¹³C and coupling constants (*J*) in Hertz.

2-Acetyl-5-oxo-pentanoic acid methyl ester (3a).

From **5a** (1.6 mL, 14.8 mmol) and **4a** (1 mL, 14.8 mmol) by general procedure A was obtained **3a**⁹ (1.52 g, 60%) as a colorless oil.

Rf (1:99 acetone-CH₂Cl₂) 0.28; ¹H NMR (CDCl₃) δ 2.10-2.19 (m, 2H), 2.26 (s, 3H), 2.54 (t, 2H, *J* = 7.25 Hz), 3.55 (t, 1H, *J* = 7.25 Hz), 3.75 (s, 3H), 9.75 (s, 1H); ¹³C NMR (CDCl₃) δ 20.1; 29.1, 41.0, 52.5, 57.8, 169.7, 200.9, 202.4.

2-Acetyl-5-oxo-hexanoic acid methyl esters (3b).

From **5a** (5 mL, 46.3 mmol) and **4b** (3.85 mL, 46.3 mmol) by general procedure B was obtained **3b**¹⁰ (8.18 g, 95%) as a colorless oil.

Rf (5:95 acetone-CH₂Cl₂) 0.43. ¹H NMR (CDCl₃) δ 2.07-2.11 (m, 2H), 2.14 (s, 3H), 2.25 (s, 3H), 2.51 (t, 2H, *J* = 7 Hz), 3.53 (t, 1H, *J* = 7 Hz), 3.74 (s, 3H); ¹³C NMR (CDCl₃) δ 21.6, 29.0, 29.9, 40.3, 52.4, 57.9, 169.9, 202.7, 207.4.

4-Acetyl-5-oxo-hexanal (3c).

From **5b** (5.1 mL, 49.7 mmol) and **4a** (3.35 mL, 49.7 mmol) by general procedure A was obtained **3c**¹¹ (4.95 g, 64%) as a colorless oil.

Rf (4:96 acetone-CH₂Cl₂) 0.43; ¹H NMR (CDCl₃) δ 2.11-2.18 (m, 2H), 2.21 (s, 6H), 2.50 (t, 2H, *J* = 7 Hz), 3.72 (t, 1H, *J* = 7 Hz), 9.95 (s, 1H); ¹³C NMR (CDCl₃) δ 19.9, 29.4, 41.1, 66.8, 201.0, 203.8.

3-Acetyl-heptane-2,6-dione (3d).

From **5b** (2 mL, 19.5 mmol) and **4b** (1.6 mL, 19.5 mmol) by general procedure B in dioxane (15mL) was obtained **3d**¹² (2.98 g, 90%) as a colorless oil. *Rf* (3:97 acetone-CH₂Cl₂) 0.29.

3-(2,6-Dioxo-cyclohexyl)-propionaldehyde (**3e**).

From **5c** (1.65 g, 14.7 mmol) and **4a** (1 mL, 14.7 mmol) by general procedure A was obtained **3e** (925 mg, 37%) as a solid.

In DMSO solution, **3e** appears totally in the form of hemiketal **3e''**.

R_f (4:96 MeOH-CH₂Cl₂) 0.23. Mp 140 °C. IR (nujol) 1590, 1705, 3280 cm⁻¹; ¹H NMR (DMSO-D₆) δ 1.68-1.80 (m, 2H), 1.86-1.96 (m, 2H), 2.15-2.20 (m, 2H), 2.27-2.32 (m, 2H), 2.36-2.41 (m, 2H), 5.41 (t, 1H, *J* = 3.50 Hz), 7.46 (br s, 1H); ¹³C NMR (DMSO-D₆) δ 14.7, 20.8, 27.3, 28.4, 36.5, 93.8, 110.7, 169.6, 197.2.

2-(3-Oxo-butyl)-cyclohexane-1,3-dione (**3f**).

From **5c** (3 g, 26.7 mmol) and **4b** (2.2 mL, 26.7 mmol) by general procedure B in benzene (20 mL) was obtained **3f**¹³ (2.77 g, 57%) as a colorless oil.

In DMSO solution, **3f** appears as an equilibrium between with the enol form **3f'** and the hemiketal **3f''** (in about 1:1 ratio).

R_f (7:93 acetone-CH₂Cl₂) 0.25. ¹H NMR (DMSO-D₆) δ 1.42 (s, 1.5H), 1.48-1.60 (m, 0.5H), 1.77-1.88 (m, 2.5H), 2.03-2.13 (m, 1.5H), 2.04 (s, 1.5H), 2.19-2.32 (m, 5.5H), 6.78 (br s, 0.5H), 10.44 (br s, 0.5H); ¹³C NMR (DMSO-D₆) δ 15.2, 16.4, 20.6, 27.0, 28.4, 29.4, 31.5, 36.3, 42.3, 54.9, 98.3, 110.0, 113.3, 169.1, 196.5, 208.7.

2-Benzoyl-5-oxo-pentanoic acid ethyl ester (**3g**).

From **5d** (8.5 mL, 46.3 mmol) and **4a** (3.3 mL, 49.1 mmol) by general procedure A was obtained **3g**¹⁴ (6.43 g, 53%) as a colorless oil.

R_f (1:99 acetone-CH₂Cl₂) 0.40. ¹H NMR (CDCl₃) δ 1.15 (t, 3H, *J* = 7.25 Hz), 2.24-2.37 (m, 2H), 2.59-2.66 (m, 2H), 4.13 (q, 2H, *J* = 7.25 Hz), 4.45 (t, 1H, *J* = 7.27 Hz), 7.45-8.04 (m, 5H), 9.75 (s, 1H); ¹³C NMR (CDCl₃) δ 13.8, 21.1, 40.9, 52.4, 61.3, 128.3, 128.6, 133.6, 135.7, 169.4, 194.7, 201.0.

2-Benzoyl-5-oxo-hexanoic acid ethyl ester (3h).

From **5d** (3.15 mL, 18.2 mmol) and **4b** (1.5 mL, 18.2 mmol) by general procedure B was obtained **3h**¹⁵ (2.83 g, 59%) as a colorless oil. *Rf* (2:98 acetone-CH₂Cl₂) 0.40.

2-(2-Chloro-acetyl)-5-oxo-pentanoic acid ethyl ester (3i).

From **5e** (1.7 mL, 14.7 mmol) and **4a** (1 mL, 14.8 mmol) by general procedure A was obtained crude **3i** (2.42 g) as a colorless oil. Due to its instability, this compound was immediately engaged without further purification in the next step.

2-(2-Chloro-acetyl)-5-oxo-hexanoic acid ethyl ester (3j).

From **5e** (0.5 mL, 4.33 mmol) and **4b** (0.36 mL, 4.35 mmol) by general procedure B was obtained **3j** (566 mg, 59%) as a colorless oil.

Rf (1:99 acetone-CH₂Cl₂) 0.29. IR (CHCl₃) 1705 cm⁻¹; ¹H NMR (CDCl₃) δ 2.14 (s, 3H), 2.10-2.21 (m, 2H), 2.54 (t, 2H, *J* = 7 Hz), 3.75 (s, 3H), 3.80-3.89 (m, 1H), 4.26 (s, 2H); ¹³C NMR (CDCl₃) δ 21.6, 30.0, 40.1, 48.0, 52.8, 53.8, 169.1, 197.3, 207.5.

5-methyl-3-phenyl-2,3,8,8a-tetrahydro-7H-oxazolo[3,2-*a*]pyridine-6-carboxylic acid methyl ester (2a).

From **3a** (2.1 g, 12.2 mmol) and (*S*)-phenylglycinol (842 mg, 6.14 mmol) by general procedure A was obtained **2a** (1.61 g, 96%).

From **3a** (256 mg, 1.49 mmol) and (*S*)-phenylglycinol (102 mg, 0.74 mmol) by general procedure B was obtained **2a** (164 mg, 81%).

(3*S*, 8*aS*)-**2a** (Major isomer):

Rf (15:85 AcOEt-Cyclohexane) 0.20; Colorless solid, mp 91°C; [α]_D²⁰ – 338 (*c* 0.995, CHCl₃); IR (CHCl₃) 1710 cm⁻¹; ¹H NMR (CDCl₃) δ 1.75-1.91 (m, 1H), 2.16 (s, 3H), 2.27-2.42 (m, 2H), 2.76 (dd, 1H, *J* = 5.75 and 15.25 Hz), 3.63 (s, 3H), 3.98 (d, 1H, *J* = 8.75 Hz), 4.25 (dd, 1H, *J* = 6.25 and 8.75 Hz), 4.76 (dd, 1H, *J* = 2.75 and 10 Hz), 4.86 (d, 1H, *J* = 6.25 Hz), 7.21-7.36 (m, 5H); ¹³C NMR (CDCl₃) δ 18.2, 22.1, 26.4, 50.4, 60.9, 74.4, 88.3, 93.6, 125.9, 127.6, 128.8, 142.8, 151.3, 168.2; HMRS (CI) *m/z* calcd for C₁₆H₂₀NO₃ (MH⁺) 274.1443, found 274.1446.

(3*S*, 8*aR*)-**2a** (Minor isomer):

R_f (15:85 AcOEt-Cyclohexane) 0.26; Colorless solid, mp 104°C; $[\alpha]_D^{20} + 277$ (*c* 0.995, CHCl₃); ¹H NMR (CDCl₃) δ 1.49-1.61 (m, 1H), 2.18 (s, 3H), 2.22-2.32 (m, 2H), 2.72-2.78 (m, 1H), 3.63 (s, 3H), 3.71 (dd, 1H, *J* = 7 and 8.75 Hz), 4.45 (dd, 1H, *J* = 7.25 and 8.75 Hz), 4.92 (t, 1H, *J* = 7.25 Hz), 5.02 (dd, 1H, *J* = 4 and 10 Hz), 7.22-7.38 (m, 5H); ¹³C NMR (CDCl₃) δ 18.0, 21.0, 26.6, 50.5, 61.7, 74.0, 89.3, 94.2, 125.7, 127.8, 129.1, 141.4, 152.3, 169.1.

5,8a-Dimethyl-3-phenyl-2,3,8,8a-tetrahydro-7H-oxazolo[3,2-*a*]pyridine-6-carboxylic acid methyl ester (2b).

From **3b** (117 mg, 0.63 mmol) and (*S*)-phenylglycinol (43 mg, 0.31 mmol) by general procedure A was obtained **2b** (78 mg, 87%).

From **3b** (489 mg, 2.63 mmol) and (*S*)-phenylglycinol (183 mg, 1.33 mmol) by general procedure B was obtained **2b** (235 mg, 61%).

(3*S*, 8*aR*)-**2b** (Major isomer):

R_f (10:90 AcOEt-Cyclohexane) 0.24; Colorless solid, mp 75°C (from cyclohexane); $[\alpha]_D^{20} + 384$ (*c* 1.025, CHCl₃); IR (CHBr₃) 1680 cm⁻¹; ¹H NMR (CDCl₃) δ 1.43 (s, 3H), 1.54 (dt, 1H, *J* = 5.75 and 12.75 Hz), 2.00 (ddd, 1H, *J* = 1.75, 5.75 and 12 Hz), 2.17 (s, 3H), 2.15-2.35 (m, 1H), 2.75 (dd, 1H, *J* = 5.5 and 16.75 Hz), 3.64 (s, 3H), 3.87 (dd, 1H, *J* = 6.25 and 9 Hz), 4.36 (dd, 1H, *J* = 7.25 and 9 Hz), 4.89 (t, 1H, *J* = 6.75 Hz), 7.21-7.37 (m, 5H); ¹³C NMR (CDCl₃) δ 18.9, 21.7, 22.9, 31.7, 50.2, 62.6, 71.3, 93.3, 94.9, 125.4, 127.3, 128.6, 141.7, 151.9, 168.9; HMRS (CI) *m/z* calcd for C₁₇H₂₂NO₃ (MH⁺) 288.1600, found 288.1595.

(3*S*, 8*aS*)-**2b** (Minor isomer, characteristic signals from a mixture):

R_f (10:90 AcOEt-Cyclohexane) 0.19; ¹H NMR (CDCl₃) δ 1.41 (s, 3H), 3.61 (s, 3H), 4.53 (dd, 1H, *J* = 6.5 and 9 Hz); ¹³C NMR (CDCl₃) δ 18.5, 20.7, 22.0, 31.6, 50.1, 61.0, 71.3, 91.9, 93.8, 125.6, 127.2, 128.6, 142.7, 150.5, 168.8

1-(5-Methyl-3-phenyl-2,3,8,8a-tetrahydro-7H-oxazolo[3,2-*a*]pyridin-6-yl)-ethanone (2c).

From **3c** (2.75, 17.6 mmol) and (*S*)-phenylglycinol (1.21 g, 8.80 mmol) by general procedure A was obtained **2c** (1.95 g, 86%).

From **3c** (153 mg, 0.98 mmol) and (*S*)-phenylglycinol (67 mg, 0.49 mmol) by general procedure B was obtained **2c** (109 mg, 86%).

(3*S*, 8*aS*)-**2c** (Major isomer):

R_f (40:60 AcOEt-Cyclohexane) 0.17; Colorless solid, mp 108°C; $[\alpha]_D^{20}$ - 304 (*c* 1.020, CHCl₃); IR (CHCl₃) 1650 cm⁻¹; ¹H NMR (CDCl₃) δ 1.83-2.00 (m, 1H), 2.13 (s, 3H), 2.17 (s, 3H), 2.38-2.53 (m, 2H), 2.62-2.72 (m, 1H), 3.94 (d, 1H, *J* = 8.5 Hz), 4.21 (dd, 1H, *J* = 6.5 and 8.5 Hz), 4.73 (dd, 1H, *J* = 2.75 and 10 Hz), 4.86 (d, 1H, *J* = 6.5 Hz), 7.20-7.33 (m, 5H); ¹³C NMR (CDCl₃) δ 18.9, 23.3, 26.2, 29.5, 60.5, 73.9, 87.7, 103.4, 125.5, 127.3, 128.5, 142.1, 151.2, 195.5; HMRS (CI) *m/z* calcd for C₁₆H₂₀NO₂ (MH⁺) 258.1494, found 258.1491.

(3*S*, 8*aR*)-**2c** (Minor isomer):

R_f (40:60 AcOEt-Cyclohexane) 0.26; Colorless solid, mp 138°C; $[\alpha]_D^{20}$ + 390 (*c* 1.015, CHCl₃); ¹H NMR (CDCl₃) δ 1.53-1.69 (m, 1H), 2.17 (s, 3H), 2.20 (s, 3H), 2.29-2.39 (m, 1H), 2.40-2.55 (m, 1H), 2.60-2.72 (m, 1H), 3.73 (dd, 1H, *J* = 7.25 and 9 Hz), 4.47 (dd, 1H, *J* = 7.25 and 9 Hz), 4.93 (t, 1H, *J* = 7.25 Hz), 5.01 (dd, 1H, *J* = 4 and 10 Hz), 7.20-7.40 (m, 5H); ¹³C NMR (CDCl₃) δ 18.9, 22.8, 26.8, 30.0, 61.6, 74.0, 89.1, 104.5, 125.7, 127.9, 129.2, 141.0, 152.5, 196.1.

1-(5,8a-Dimethyl-3-phenyl-2,3,8,8a-tetrahydro-7*H*-oxazolo[3,2-*a*]pyridin-6-yl)-ethanone (2d).

From **3d** (216 mg, 1.37 mmol) and (*S*)-phenylglycinol (194 mg, 1.41 mmol) by general procedure A was obtained **2d** (220 mg, 64%).

From **3d** (340 mg, 2.00 mmol) and (*S*)-phenylglycinol (309 mg, 2.25 mmol) by general procedure B was obtained **2d** (140 mg, 26%).

(3*S*, 8*aR*)-**2d** (Major isomer):

R_f (30:70 AcOEt-Cyclohexane) 0.23; Colorless solid, mp 138°C (from cyclohexane); $[\alpha]_D^{20}$ + 502 (*c* 1.015, CHCl₃); IR (CHBr₃) 1630 cm⁻¹; ¹H NMR (CDCl₃) δ 1.45 (s, 3H), 1.61 (dt, 1H, *J* = 6 and 12.75 Hz), 2.08 (dt, 1H, *J* = 1.75 and 6 Hz), 2.16 (s, 3H), 2.20 (s, 3H), 2.32-2.47 (m, 1H), 2.65 (dd, 1H, *J* = 5.75 and 16.25 Hz), 3.93 (dd, 1H, *J* = 6.75 and 9 Hz), 4.42 (dd, 1H, *J* = 7.5 and 9 Hz), 4.94 (t, 1H, *J* = 7.5 Hz), 7.25-7.40 (m, 5H); ¹³C NMR (CDCl₃) δ 20.1, 23.0, 23.6, 30.0, 32.4, 62.9, 71.6, 93.3, 105.1, 125.7, 127.7, 129.0, 141.4, 152.6, 196.8; HMRS (CI) *m/z* calcd for C₁₇H₂₂NO₂ (MH⁺) 272.1651, found 272.1648.

Phenyl-1,2,3a,4,5,7,8,9-octahydro-oxazolo[3,2-*a*]quinolin-6-one (2e).

From **3e** (67 mg, 0.40 mmol) and (*S*)-phenylglycinol (60 mg, 0.44 mmol) by general procedure A was obtained **2e** (87 mg, 81%).

From **3e** (80 mg, 0.47 mmol) and (*S*)-phenylglycinol (72 mg, 0.52 mmol) by general procedure B was obtained **2e** (118 mg, 91%).

The two isomers were not separated by column chromatography, *R_f* (30:70 MeOH-CH₂Cl₂) 0.21; Colorless solid; IR (CHCl₃) 1650 cm⁻¹; HMRS (CI) *m/z* calcd for C₁₇H₂₀NO₂ (MH⁺) 270.1494, found 270.1492.

(1*S*, 3*aS*)-**2e** (Major isomer, from a mixture):

¹H NMR (CDCl₃) δ 1.53-1.87 (m, 3H), 1.94-2.03 (m, 1H), 2.15-2.32 (m, 4H), 2.39-2.51 (m, 1H), 2.86 (dd, 1H, *J* = 5.5 and 16.5 Hz), 4.02 (d, 1H, *J* = 8.75 Hz), 4.29 (dd, 1H, *J* = 6.5 and 9 Hz), 4.81 (dd, 1H, *J* = 3.25 and 10.25 Hz), 4.86 (d, 1H, *J* = 6.25 Hz), 7.21-7.38 (m, 5H); ¹³C NMR (CDCl₃) δ 18.2, 21.3, 25.6, 27.3, 35.7, 60.4, 74.4, 88.5, 106.2, 125.7, 127.8, 128.8, 141.9, 156.4, 193.9.

(1*S*, 3*aR*)-**2e** (Minor isomer, characteristic signals from a mixture):

¹H NMR (CDCl₃) δ 3.73 (m, 1H), 4.53 (dd, 1H, *J* = 7.75 and 9 Hz), 4.92 (t, 1H, *J* = 7.5 Hz), 5.05 (dd, 1H, *J* = 4 and 10 Hz); ¹³C NMR (CDCl₃) δ 17.3, 26.1, 61.3, 89.5, 106.4, 140.0, 157.2.

3a-Methyl-1-phenyl-1,2,3a,4,5,7,8,9-octahydro-oxazolo[3,2-*a*]quinolin-6-one (2f).

From **3f** (165 mg, 0.90 mmol) and (*S*)-phenylglycinol (62 mg, 0.45 mmol) by general procedure A was obtained **2f** (110 mg, 86%).

From **3f** (2.52 mg, 13.8 mmol) and (*S*)-phenylglycinol (950 mg, 6.93 mmol) by general procedure B was obtained **2f** (1.76 mg, 90%).

(1*S*, 3*aR*)-**2f** (Major isomer):

R_f (AcOEt) 0.29; Colorless solid, mp 58°C; [α]_D²⁰ + 369 (*c* 0.995, CHCl₃); IR (CHCl₃) 1640 cm⁻¹; ¹H NMR (CDCl₃) δ 1.48 (s, 3H), 1.52-1.62 (m, 1H), 1.74-1.82 (m, 2H), 1.90-2.05 (m, 1H), 2.11-2.33 (m, 5H), 2.77-2.85 (m, 1H), 3.86-3.93 (m, 1H), 4.47 (dd, 1H, *J* = 7.75 and 9 Hz), 4.93 (t, 1H, *J* = 7.75 Hz), 7.26-7.43 (m, 5H); ¹³C NMR (CDCl₃) δ 18.3, 21.3, 23.0, 28.3,

32.2, 35.8, 62.5, 71.8, 93.9, 107.3, 125.6, 127.8, 129.1, 140.5, 157.0, 194.8. HMRS (CI) m/z calcd for $C_{18}H_{22}NO_2$ (MH^+) 284.1651, found 284.1651.

(1*S*, 3*aS*)-**2f** (Minor isomer, from a mixture):

R_f (AcOEt) 0.19; 1H NMR ($CDCl_3$) δ 1.33 (s, 3H), 1.63-2.04 (m, 4H), 2.15-2.29 (m, 5H), 2.80-2.86 (m, 1H), 3.95 (dd, 1H, $J = 1.25$ and 9 Hz), 4.58 (dd, 1H, $J = 6.75$ and 9 Hz), 4.89-4.92 (d, 1H, $J = 6.75$ Hz), 7.18-7.35 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 18.9, 21.2, 21.6, 28.1, 31.5, 36.1, 60.9, 71.9, 93.0, 106.9, 125.7, 129.1, 142.3, 156.2, 194.8.

(3,5-Diphenyl-2,3,8,8a-tetrahydro-7*H*-oxazolo[3,2-*a*]pyridine-6-carboxylic acid ethyl ester (2g).

From **3g** (179 mg, 0.72 mmol) and (*S*)-phenylglycinol (50 mg, 0.36 mmol) by general procedure A was obtained **2g** (76 mg, 60%).

From **3g** (356 mg, 1.43 mmol) and (*S*)-phenylglycinol (98 mg, 0.71 mmol) by general procedure B was obtained **2g** (225 mg, 91%).

The two isomers were not separated by column chromatography. Colorless solid. IR ($CHCl_3$) 1690 cm^{-1} ; HMRS (CI) m/z calcd for $C_{22}H_{24}NO_3$ (MH^+) 350.1756, found 350.1761.

(3*S*, 8*aS*)-**2g** (Major isomer, from a mixture):

R_f (15:85 AcOEt-Cyclohexane) 0.30; 1H NMR ($CDCl_3$) δ 0.75 (t, 3H, $J = 7$ Hz), 1.84-2.02 (m, 1H), 2.37-2.55 (m, 2H), 2.87-3.0 (m, 1H), 3.68-3.80 (m, 2H), 3.96 (d, 1H, $J = 8.75$ Hz), 4.18-4.26 (m, 1H), 4.48 (d, 1H, $J = 6$ Hz), 4.97 (dd, 1H, $J = 2.75$ and 9.75 Hz), 6.29-7.45 (m, 10H); ^{13}C NMR ($CDCl_3$) δ 13.8, 22.2, 26.9, 58.8, 61.7, 73.9, 88.5, 97.5, 126.6, 127.0, 127.2, 127.6, 128.1, 128.8, 137.3, 142.8, 151.7, 168.5.

(3*S*, 8*aR*)-**2g** (Minor isomer, from a mixture):

R_f (15:85 AcOEt-Cyclohexane) 0.32; 1H NMR ($CDCl_3$) δ 0.76 (t, 3H, $J = 7$ Hz), 1.63-1.78 (m, 1H), 2.26-2.48 (m, 2H), 2.86-2.96 (m, 1H), 3.69-3.80 (m, 3H), 4.19-4.28 (m, 1H), 4.32-4.37 (m, 1H), 5.24 (dd, 1H, $J = 4$ and 9.5 Hz), 6.68-7.30 (m, 10H); ^{13}C NMR ($CDCl_3$) δ 13.8, 21.0, 26.5, 58.9, 62.9, 73.0, 89.5, 98.3, 126.3, 127.2, 127.4, 127.6, 128.2, 128.3, 137.3, 142.4, 153.4, 168.3.

5-Chloromethyl-3-phenyl-2,3,8,8a-tetrahydro-7H-oxazolo[3,2-a]pyridine-6-carboxylic acid methyl ester (2i).

From **3i** (206 mg, 0.76 mmol) and (*S*)-phenylglycinol (53 mg, 0.38 mmol) by general procedure A was obtained **2i** (91 mg, 76%).

From **3i** (203 mg, 0.75 mmol) and (*S*)-phenylglycinol (52 mg, 0.38 mmol) by general procedure B was obtained **2i** (104 mg, 89%).

The two isomers are inseparable by column chromatography, *R_f* (15:85 AcOEt-Cyclohexane) 0.41; Colorless solid (mixture of isomers); IR (CHCl₃) 1680 cm⁻¹; HMRS (CI) *m/z* calcd for C₁₆H₁₉NO₃Cl (MH⁺) 308.1053, found 308.1055.

(3*S*, 8*aS*)-**2i** (Major isomer, from a mixture):

¹H NMR (CDCl₃) δ 1.79-1.91 (m, 1H), 2.33-2.49 (m, 2H), 2.73-2.85 (m, 1H), 3.55 (dd, 1H, *J* = 0.75 and 11.25 Hz), 3.67 (s, 3H), 4.01 (d, 1H, *J* = 8.75 Hz), 4.30 (dd, 1H, *J* = 6.5 and 8.75 Hz), 4.79 (dd, 1H, *J* = 2.5 and 9.75 Hz), 5.14 (d, 1H, *J* = 6.25 Hz), 5.56 (t, 1H, *J* = 11.25 Hz), 7.24-7.37 (m, 5H); ¹³C NMR (CDCl₃) δ 22.3, 26.4, 38.5, 51.0, 59.8, 74.7, 88.3, 97.6, 125.9, 128.1, 129.2, 142.7, 147.8, 168.1.

(3*S*, 8*aR*)-**2i** (Minor isomer, from a mixture):

¹H NMR(CDCl₃) δ 1.50-1.64 (m, 1H), 2.20-2.31 (m, 1H), 2.34-2.43 (m, 1H), 2.73-2.85 (m, 1H), 3.68 (s, 3H), 3.70-3.80 (m, 2H), 4.47 (dd, 1H, *J* = 7.25 and 8.75 Hz), 5.07 (dd, 1H, *J* = 4 and 9.75 Hz), 5.24 (t, 1H, *J* = 6.75 Hz), 5.56 (t, 1H, *J* = 11.25 Hz), 7.24-7.37 (m, 5H); ¹³C NMR(CDCl₃) δ 21.1, 25.7, 38.4, 51.0, 60.6, 74.1, 89.3, 97.9, 125.8, 128.2, 129.3, 141.3, 149.3, 167.9.

2-Hydroxy-4-oxo-2-phenyl-cyclohexanecarboxylic acid ethyl ester (6).

From **3h** (210 mg, 0.80 mmol) and (*S*)-phenylglycinol (121 mg, 0.88 mmol) by general procedure A was obtained **6** (147 mg, 70 %).

From **3h** (198 mg, 0.75 mmol) and (*S*)-phenylglycinol (114 mg, 0.83 mmol) by general procedure B was obtained **6** (175 mg, 89%).

R_f (30:70 AcOEt-Cyclohexane) 0.30; Colorless solid, mp 130°C (from water-EtOH 3:7) (lit^{17a,b}: 128-130°C); ¹H NMR (CDCl₃) δ 0.95 (t, 3H, *J* = 7.25 Hz), 2.10-2.67 (m, 6H), 3.42-

3.49 (m, 1H), 3.85-4.02 (m, 2H), 4.25 (br s, 1H), 7.13-7.49 (m, 5H); ^{13}C NMR (CDCl_3)
 δ 13.6, 25.0, 39.3, 48.9, 54.1, 60.8, 77.1, 124.3, 127.3, 128.2, 144.6, 174.9, 206.9.

Crystal data for C₁₇H₂₁NO₃ : (3*S*, 8*aR*)-2b

Formula	C₁₇H₂₁NO₃
Fw =	287.36
Crystal System	orthorhombic
a (Å) =	5.9101(4)
b (Å) =	14.4928 (14)
c (Å) =	17.659 (2)
α (°) =	90
β (°) =	90
γ (°) =	90
V (Å³) =	1512.6 (2)
Z =	4
Space group	P 2₁ 2₁ 2₁
Crystal shape	Parallelepiped
Crystal colour	colourless
Linear absorption coefficient μ (cm⁻¹)	1.26
Density ρ(g cm³)	0.86
Diffractometer	KAPPACCD-Enraf Nonius
Radiation	MoKα (λ = 0.71073 Å)
θ Limits (°)	2-35
Octants collected	-9, 6 ; -23, 23 ; -28, 22
Nb of data collected	15806
Nb of unique data collected	3743
Nb of unique data used for refinement	1785(Fo)² > 1.5σ(Fo)²
Merging R	0.08
R = Σ Fo - Fc / Σ Fo 	0.0656
Rw* = [Σw(Fo - Fc)² / ΣwFo²]^{1/2}	0.0582
Absorption correction	None
Secondary extinction coefficient	None
Goodness of fit	1.229
Nb of variables	191
Δρmin (e/ Å³)	-0.24
Δρmax (e/ Å³)	0.33

* Weighting scheme of the form $w=w'[1-((| Fo | - | Fc |) / 6\sigma(Fo))^2]^2$ with $w'=1/\Sigma_r A_r T_r(X)$ with coefficients 1.42, 1.66 and 1.01 for a Chebyshev serie for which $X=Fc/Fc(max)$

Table II : Fractional atomic coordinates for $C_{17}H_{21}NO_3$

Atom	x/a	y/b	z/c	U(eqv)
C(1)	1.4406(6)	0.2231(2)	0.06040(18)	0.0672
C(2)	1.1694(4)	0.19076(17)	0.15505(13)	0.0444
C(3)	0.9834(4)	0.23108(16)	0.19946(11)	0.0401
C(4)	0.9474(5)	0.33371(18)	0.19363(16)	0.0519
C(5)	0.7138(5)	0.36029(18)	0.22440(16)	0.0511
C(6)	0.8166(5)	0.3639(2)	0.36217(16)	0.0555
C(7)	0.6735(4)	0.31802(17)	0.30066(14)	0.0433
C(8)	0.8521(4)	0.17750(16)	0.24645(12)	0.0379
C(9)	0.8524(5)	0.07419(17)	0.24503(15)	0.0527
C(10)	0.3836(5)	0.2506(2)	0.36652(19)	0.0656
C(11)	0.5223(4)	0.16903(18)	0.33554(13)	0.0447
C(12)	0.5837(4)	0.10296(16)	0.39874(12)	0.0393
C(13)	0.7839(5)	0.1096(2)	0.43950(17)	0.0569
C(14)	0.8243(7)	0.0494(3)	0.49918(18)	0.0693
C(15)	0.6656(8)	-0.0146(2)	0.51955(18)	0.0736
C(16)	0.4674(7)	-0.0201(3)	0.4801(2)	0.0790
C(17)	0.4270(5)	0.03730(19)	0.41974(17)	0.0576
O(1)	1.2478(4)	0.25224(16)	0.10386(11)	0.0603
O(2)	1.2547(4)	0.11575(14)	0.16153(14)	0.0682
O(3)	0.4375(3)	0.32565(15)	0.31888(13)	0.0593
N(1)	0.7115(4)	0.21723(12)	0.29850(11)	0.0419

Table III : Interatomic distances (Å) for $C_{17}H_{21}NO_3$

C(1) - O(1)	1.437(4)	C(2) - C(3)	1.471(4)
C(2) - O(1)	1.352(3)	C(2) - O(2)	1.204(3)
C(3) - C(4)	1.506(3)	C(3) - C(8)	1.376(3)
C(4) - C(5)	1.533(4)	C(5) - C(7)	1.499(4)
C(6) - C(7)	1.529(4)	C(7) - O(3)	1.436(3)
C(7) - N(1)	1.478(3)	C(8) - C(9)	1.498(4)
C(8) - N(1)	1.366(3)	C(10) - C(11)	1.539(4)
C(10) - O(3)	1.412(4)	C(11) - C(12)	1.515(3)
C(11) - N(1)	1.472(3)	C(12) - C(13)	1.389(4)
C(12) - C(17)	1.379(4)	C(13) - C(14)	1.389(4)
C(14) - C(15)	1.368(5)	C(15) - C(16)	1.365(6)
C(16) - C(17)	1.374(5)		

Table IV : Bond angles (°) for $C_{17}H_{21}NO_3$

C(3) - C(2) - O(1)	110.5(2)	C(3) - C(2) - O(2)	128.4(2)
O(1) - C(2) - O(2)	121.0(2)	C(2) - C(3) - C(4)	117.5(2)
C(2) - C(3) - C(8)	121.2(2)	C(4) - C(3) - C(8)	121.3(2)
C(3) - C(4) - C(5)	110.5(2)	C(4) - C(5) - C(7)	111.0(2)
C(5) - C(7) - O(3)	111.9(2)	C(5) - C(7) - N(1)	108.9(2)
C(6) - C(7) - O(3)	110.2(2)	C(5) - C(7) - N(1)	110.9(2)
C(6) - C(7) - N(1)	111.4(2)	O(3) - C(7) - N(1)	103.3(2)
C(3) - C(8) - C(9)	123.6(2)	C(3) - C(8) - N(1)	120.7(2)
C(9) - C(8) - N(1)	115.7(2)	C(11) - C(10) - O(3)	105.1(2)
C(10) - C(11) - C(12)	110.6(2)	C(10) - C(11) - N(1)	101.4(2)
C(12) - C(11) - N(1)	116.47(19)	C(11) - C(12) - C(13)	122.8(2)
C(11) - C(12) - C(17)	118.3(2)	C(13) - C(12) - C(17)	118.7(2)
C(12) - C(13) - C(14)	119.7(3)	C(13) - C(14) - C(15)	120.6(3)
C(14) - C(15) - C(16)	119.6(3)	C(15) - C(16) - C(17)	120.7(3)
C(12) - C(17) - C(16)	120.7(3)	C(1) - O(1) - C(2)	115.8(2)
C(7) - O(3) - C(10)	107.1(2)	C(7) - N(1) - C(8)	121.7(2)
C(7) - N(1) - C(11)	110.0(2)	C(8) - N(1) - C(11)	124.1(2)

Table S1 : Anisotropic thermal parameters for $C_{17}H_{21}NO_3$

Atom	u(11)	u(22)	u(33)	u(23)	u(13)	u(12)
C(1)	0.0608(18)	0.0811(19)	0.0597(15)	-0.0098(15)	0.0214(15)	-0.0084(17)
C(2)	0.0468(12)	0.0496(12)	0.0368(10)	-0.0054(9)	-0.0041(9)	-0.0053(10)
C(3)	0.0472(13)	0.0398(10)	0.0334(10)	-0.0037(8)	-0.0058(9)	0.0013(10)
C(4)	0.0591(14)	0.0413(13)	0.0553(13)	0.0072(10)	0.0056(12)	0.0028(12)
C(5)	0.0587(16)	0.0427(12)	0.0518(12)	0.0055(10)	-0.0048(11)	0.0097(11)
C(6)	0.0581(17)	0.0536(13)	0.0550(14)	-0.0130(12)	-0.0066(12)	0.0106(12)
C(7)	0.0407(12)	0.0407(10)	0.0484(12)	-0.0060(10)	-0.0046(9)	0.0076(10)
C(8)	0.0427(10)	0.0369(10)	0.0341(9)	-0.0043(8)	-0.0078(8)	0.0006(10)
C(9)	0.0686(16)	0.0371(11)	0.0525(13)	-0.0026(10)	0.0075(12)	-0.0003(12)
C(10)	0.0435(16)	0.0656(18)	0.088(2)	0.0163(16)	0.0199(15)	0.0125(13)
C(11)	0.0401(11)	0.0499(13)	0.0443(12)	-0.0004(10)	-0.0030(9)	-0.0058(10)
C(12)	0.0418(12)	0.0414(10)	0.0346(10)	-0.0043(8)	0.0055(9)	-0.0002(9)
C(13)	0.0560(17)	0.0638(14)	0.0508(13)	0.0059(12)	-0.0090(12)	-0.0077(13)
C(14)	0.082(2)	0.0766(19)	0.0490(14)	0.0072(13)	-0.0154(15)	0.0120(18)
C(15)	0.097(3)	0.0703(18)	0.0533(16)	0.0178(14)	0.0233(17)	0.019(2)
C(16)	0.087(2)	0.0608(16)	0.090(2)	0.0205(17)	0.028(2)	-0.0071(18)
C(17)	0.0510(15)	0.0547(14)	0.0669(17)	0.0013(12)	0.0063(12)	-0.0101(12)
O(1)	0.0570(12)	0.0711(12)	0.0529(10)	0.0059(9)	0.0129(9)	0.0007(9)
O(2)	0.0742(15)	0.0557(10)	0.0747(14)	-0.0056(10)	0.0181(12)	0.0093(11)
O(3)	0.0396(9)	0.0594(11)	0.0790(12)	0.0117(10)	0.0055(9)	0.0144(9)
N(1)	0.0443(11)	0.0350(8)	0.0463(10)	0.0024(7)	0.0005(8)	0.0005(8)

Table S2 : Hydrogen atoms fractional atomic coordinates for $C_{17}H_{21}NO_3$

Atom	x/a	y/b	z/c	U(iso)
H(11)	1.4855	0.2730	0.0245	0.100(3)
H(12)	1.5695	0.2096	0.0955	0.100(3)
H(13)	1.4014	0.1660	0.0314	0.100(3)
H(41)	1.0653	0.3664	0.2236	0.100(3)
H(42)	0.9564	0.3528	0.1393	0.100(3)
H(51)	0.7044	0.4290	0.2291	0.100(3)
H(52)	0.5942	0.3382	0.1886	0.100(3)
H(61)	0.7849	0.3332	0.4121	0.100(3)
H(62)	0.7762	0.4305	0.3656	0.100(3)
H(63)	0.9803	0.3571	0.3497	0.100(3)
H(91)	0.7433	0.0509	0.2845	0.100(3)
H(92)	1.0067	0.0505	0.2565	0.100(3)
H(93)	0.8025	0.0520	0.1942	0.100(3)
H(101)	0.4256	0.2641	0.4198	0.100(3)
H(102)	0.2177	0.2366	0.3632	0.100(3)
H(111)	0.4321	0.1354	0.2963	0.100(3)
H(131)	0.9005	0.1569	0.4252	0.100(3)
H(141)	0.9691	0.0538	0.5283	0.100(3)
H(151)	0.6962	-0.0578	0.5630	0.100(3)
H(161)	0.3480	-0.0662	0.4952	0.100(3)
H(171)	0.2828	0.0320	0.3909	0.100(3)

Crystal structure of (3*S*, 8*aR*)-2b

Crystal data for C₁₆H₁₉NO₂ : (3*S*, 8*aR*)-2c

TABLE I CRYSTAL DATA

Formula	C₁₆H₁₉NO₂
Fw =	257.33
Crystal System	orthorhombic
a (Å) =	6.037 (2)
b (Å) =	7.533 (2)
c (Å) =	30.403 (5)
α (°) =	90
β (°) =	90
γ (°) =	90
V (Å³) =	1382.6 (6)
Z =	4
Space group	P 2₁2₁2₁
Crystal shape	Parallelepiped
Crystal colour	colourless
Linear absorption coefficient μ (cm⁻¹)	0.81
Density ρ(g cm³)	1.24
Diffractometer	KAPPACCD-Enraf Nonius
Radiation	MoKα (λ = 0.71073 Å)
θ Limits (°)	2-27.5
Octants collected	-7, 6 ; -9, 7 ; -39, 31
Nb of data collected	10896
Nb of unique data collected	1865
Nb of unique data used for refinement	1009(Fo)² > 1.5σ(Fo)²
Merging R	0.026
R = Σ Fo - Fc / Σ Fo 	0.049
Rw* = [Σw(Fo - Fc)² / ΣwFo²]^{1/2}	0.052
Absorption correction	None
Secondary extinction coefficient	400
Goodness of fit	1.073
Nb of variables	174
Δρmin (e/ Å³)	-0.226
Δρmax (e/ Å³)	0.226

* Weighting scheme of the form $w=w'[1-(|| Fo | - | Fc ||) / 6\sigma(Fo)]^2$ with $w'=1/\Sigma_r A_r T_r(X)$ with coefficients 1.73, -0.132 and 1.29 for a Chebyshev serie for which $X=Fc/Fc(max)$

Table II : Fractional atomic coordinates for $C_{16}H_{19}NO_2$

Atom	x/a	y/b	z/c	U(iso)
C(1)	0.9513(8)	1.2622(7)	0.95857(15)	0.0682
C(2)	0.7645(7)	1.2516(6)	0.92592(14)	0.0540
C(3)	0.6341(7)	1.0906(5)	0.92395(11)	0.0407
C(4)	0.6961(7)	0.9364(5)	0.95410(12)	0.0494
C(5)	0.5026(8)	0.8095(5)	0.96069(13)	0.0533
C(6)	0.4160(7)	0.7542(5)	0.91662(12)	0.0462
C(7)	0.4588(7)	1.0700(5)	0.89504(11)	0.0383
C(8)	0.3617(8)	1.2187(5)	0.86844(13)	0.0518
C(9)	0.1158(8)	0.6762(6)	0.87731(14)	0.0562
C(10)	0.1591(7)	0.8713(5)	0.86353(13)	0.0440
C(11)	0.1902(7)	0.8882(5)	0.81432(12)	0.0426
C(12)	0.3803(7)	0.8282(6)	0.79369(13)	0.0551
C(13)	0.4024(8)	0.8373(7)	0.74846(15)	0.0662
C(14)	0.2339(9)	0.9088(7)	0.72338(14)	0.0681
C(15)	0.0461(9)	0.9680(7)	0.74311(15)	0.0669
C(16)	0.0223(8)	0.9592(6)	0.78885(14)	0.0556
O(1)	0.7391(7)	1.3807(5)	0.90102(14)	0.0956
O(2)	0.2129(5)	0.6616(4)	0.92001(9)	0.0608
N(1)	0.3590(5)	0.9093(4)	0.88962(9)	0.0395

Table III : Interatomic distances (Å) for $C_{16}H_{19}NO_2$

C(1) - C(2)	1.505(6)	C(2) - C(3)	1.447(5)
C(2) - O(1)	1.242(5)	C(3) - C(4)	1.526(5)
C(3) - C(7)	1.384(5)	C(4) - C(5)	1.523(6)
C(5) - C(6)	1.497(5)	C(6) - O(2)	1.414(5)
C(6) - N(1)	1.469(5)	C(7) - C(8)	1.501(5)
C(7) - N(1)	1.363(5)	C(9) - C(10)	1.551(6)
C(9) - O(2)	1.428(5)	C(10) - C(11)	1.513(5)
C(10) - N(1)	1.472(5)	C(11) - C(12)	1.383(6)
C(11) - C(16)	1.383(6)	C(12) - C(13)	1.383(6)
C(13) - C(14)	1.380(7)	C(14) - C(15)	1.358(7)
C(15) - C(16)	1.400(6)		

Table IV : Bond angles (°) for $C_{16}H_{19}NO_2$

C(1) - C(2) - C(3)	118.6(4)	C(1) - C(2) - O(1)	116.9(4)
C(3) - C(2) - O(1)	124.4(4)	C(2) - C(3) - C(4)	118.7(3)
C(2) - C(3) - C(7)	122.4(3)	C(4) - C(3) - C(7)	118.9(3)
C(3) - C(4) - C(5)	111.6(3)	C(4) - C(5) - C(6)	109.0(3)
C(5) - C(6) - O(2)	112.0(3)	C(5) - C(6) - N(1)	111.1(3)
O(2) - C(6) - N(1)	103.3(3)	C(3) - C(7) - C(8)	123.9(3)
C(3) - C(7) - N(1)	121.0(3)	C(8) - C(7) - N(1)	115.1(3)
C(10) - C(9) - O(2)	104.4(3)	C(9) - C(10) - C(11)	111.6(3)
C(9) - C(10) - N(1)	100.2(3)	C(11) - C(10) - N(1)	114.5(3)
C(10) - C(11) - C(12)	121.6(4)	C(10) - C(11) - C(16)	119.7(4)
C(12) - C(11) - C(16)	118.7(3)	C(11) - C(12) - C(13)	121.0(4)
C(12) - C(13) - C(14)	119.8(4)	C(13) - C(14) - C(15)	120.0(4)
C(14) - C(15) - C(16)	120.6(4)	C(11) - C(16) - C(15)	119.9(4)
C(6) - O(2) - C(9)	104.6(3)	C(6) - N(1) - C(7)	122.4(3)
C(6) - N(1) - C(10)	109.8(3)	C(7) - N(1) - C(10)	126.9(3)

Table S1 : Anisotropic thermal parameters for C₁₆H₁₉NO₂

Atom	u(11)	u(22)	u(33)	u(23)	u(13)	u(12)
C(1)	0.067(3)	0.081(3)	0.057(2)	-0.004(2)	0.001(2)	-0.031(3)
C(2)	0.053(3)	0.049(2)	0.061(3)	-0.003(2)	0.007(2)	-0.008(2)
C(3)	0.046(2)	0.0377(18)	0.0382(19)	0.0039(16)	0.0061(18)	-0.003(2)
C(4)	0.052(3)	0.052(2)	0.044(2)	0.0018(18)	0.001(2)	-0.003(2)
C(5)	0.062(3)	0.051(2)	0.046(2)	0.019(2)	-0.001(2)	-0.005(2)
C(6)	0.056(2)	0.0343(19)	0.048(2)	0.0114(17)	-0.001(2)	-0.004(2)
C(7)	0.047(2)	0.0334(19)	0.0345(17)	0.0030(14)	0.0105(17)	-0.0027(19)
C(8)	0.068(3)	0.034(2)	0.054(2)	0.0074(17)	0.001(2)	-0.002(2)
C(9)	0.065(3)	0.048(2)	0.055(2)	0.010(2)	-0.004(2)	-0.019(2)
C(10)	0.046(2)	0.041(2)	0.045(2)	-0.0031(17)	0.0026(18)	-0.0027(19)
C(11)	0.049(2)	0.0368(19)	0.0416(19)	0.0005(17)	-0.0041(19)	-0.0015(19)
C(12)	0.053(3)	0.063(3)	0.049(2)	-0.003(2)	-0.003(2)	0.003(2)
C(13)	0.067(3)	0.081(3)	0.051(2)	-0.012(2)	0.009(3)	0.001(3)
C(14)	0.086(4)	0.071(3)	0.047(2)	0.003(2)	-0.004(3)	-0.010(3)
C(15)	0.075(3)	0.070(3)	0.055(3)	0.014(2)	-0.018(3)	0.003(3)
C(16)	0.052(3)	0.051(2)	0.064(3)	0.007(2)	-0.003(2)	0.009(2)
O(1)	0.091(3)	0.061(2)	0.134(3)	0.034(2)	-0.032(3)	-0.036(2)
O(2)	0.073(2)	0.0528(16)	0.0571(18)	0.0160(14)	-0.0035(16)	-0.0235(17)
N(1)	0.0526(19)	0.0306(15)	0.0352(15)	0.0053(13)	-0.0036(15)	-0.0050(17)

Table S2 : Hydrogen atoms fractional atomic coordinates for C₁₆H₁₉NO₂

Atom	x/a	y/b	z/c	U(iso)
H(11)	1.0291	1.3784	0.9563	0.082(4)
H(12)	0.8965	1.2433	0.9890	0.082(4)
H(13)	1.0639	1.1642	0.9516	0.082(4)
H(41)	0.7406	0.9848	0.9833	0.082(4)
H(42)	0.8226	0.8704	0.9407	0.082(4)
H(51)	0.3817	0.8697	0.9773	0.082(4)
H(52)	0.5533	0.7021	0.9769	0.082(4)
H(61)	0.5271	0.6788	0.9010	0.082(4)
H(81)	0.2354	1.1715	0.8500	0.082(4)
H(82)	0.3021	1.3129	0.8885	0.082(4)
H(83)	0.4760	1.2706	0.8487	0.082(4)
H(91)	-0.0467	0.6512	0.8784	0.082(4)
H(92)	0.1880	0.5921	0.8561	0.082(4)
H(101)	0.0346	0.9493	0.8734	0.082(4)
H(121)	0.5028	0.7744	0.8119	0.082(4)
H(131)	0.5424	0.7979	0.7339	0.082(4)
H(141)	0.2479	0.9120	0.6904	0.082(4)
H(151)	-0.0746	1.0210	0.7249	0.082(4)
H(161)	-0.1155	1.0034	0.8029	0.082(4)

Crystal structure of (3*S*, 8*aR*)-2c

Crystal data for C₁₆H₂₁NO₂ : (3*S*,8*aR*)-2d

TABLE I CRYSTAL DATA

Formula	C ₁₇ H ₂₁ NO ₂
Fw =	271.36
Crystal System	Orthorhombic
a (Å) =	7.9659 (14)
b (Å) =	10.1397 (15)
c (Å) =	18.360 (4)
α (°) =	90
β (°) =	90
γ (°) =	90
V (Å ³) =	1483.0 (5)
Z =	4
Space group	P 2 ₁ 2 ₁ 2 ₁
Crystal shape	Parallelepiped
Crystal colour	colourless
Linear absorption coefficient μ (cm ⁻¹)	0.791
Density ρ(g cm ³)	1.215
Diffractionmeter	KAPPACCD-Enraf Nonius
Radiation	MoKα (λ = 0.71073 Å)
θ Limits (°)	2-27.5
Octants collected	-10, 7 ; -12, 13 ; -23, 21
Nb of data collected	11336
Nb of unique data collected	1920
Nb of unique data used for refinement	1218(Fo) ² > 1.5σ(Fo) ²
Merging R	0.143
R = Σ Fo - Fc / Σ Fo	0.0604
Rw* = [Σw(Fo - Fc) ² / ΣwFo ²] ^{1/2}	0.0658
Absorption correction	None
Secondary extinction coefficient	None
Goodness of fit	1.129
Nb of variables	182
Δρmin (e/ Å ³)	-0.248
Δρmax (e/ Å ³)	0.382

* Weighting scheme of the form $w=w'[1-((| Fo | - | Fc |) / 6\sigma(Fo))^2]^2$ with $w'=1/\Sigma_r A_r T_r(X)$ with coefficients 0.635, 0.651 and 0.344 for a Chebyshev serie for which $X=Fc/Fc(max)$

Table II : Fractional atomic coordinates for C₁₇H₂₁NO₂

Atom	x/a	y/b	z/c	U(eqv)
C(1)	0.9172(5)	0.2775(4)	0.1877(2)	0.0324
C(2)	0.8343(5)	0.3138(4)	0.1253(2)	0.0333
C(3)	0.6450(5)	0.2987(5)	0.1218(2)	0.0452
C(4)	0.5826(5)	0.2026(5)	0.1797(2)	0.0459
C(5)	0.6500(5)	0.2422(4)	0.2528(2)	0.0367
C(6)	0.5737(6)	0.3720(5)	0.2800(3)	0.0543
C(7)	0.7453(7)	0.1454(5)	0.3561(3)	0.0496
C(8)	0.9062(6)	0.1780(4)	0.3129(2)	0.0372
C(9)	1.0312(6)	0.2549(4)	0.3590(2)	0.0393
C(10)	1.0049(7)	0.3860(5)	0.3760(3)	0.0578
C(11)	1.1190(10)	0.4519(6)	0.4200(3)	0.0745
C(12)	1.2525(10)	0.3870(7)	0.4482(3)	0.0790
C(13)	1.2823(8)	0.2575(7)	0.4317(3)	0.0715
C(14)	1.1709(7)	0.1908(5)	0.3861(3)	0.0531
C(15)	1.1061(5)	0.2670(5)	0.1926(2)	0.0440
C(16)	0.9203(6)	0.3622(4)	0.0612(2)	0.0405
C(17)	0.8147(7)	0.3931(6)	-0.0053(2)	0.0610
N(1)	0.8343(4)	0.2490(3)	0.25044(17)	0.0321
O(1)	0.6176(4)	0.1384(3)	0.30298(17)	0.0487
O(2)	1.0709(5)	0.3828(4)	0.05639(19)	0.0598

Table III : Interatomic distances (Å) for C₁₇H₂₁NO₂

C(1) - C(2)	1.372(6)	C(1) - C(15)	1.512(6)
C(1) - N(1)	1.360(5)	C(2) - C(3)	1.517(6)
C(2) - C(16)	1.449(6)	C(3) - C(4)	1.526(7)
C(4) - C(5)	1.498(6)	C(5) - C(6)	1.535(6)
C(5) - N(1)	1.470(5)	C(5) - O(1)	1.424(5)
C(7) - C(8)	1.542(7)	C(7) - O(1)	1.410(6)
C(8) - C(9)	1.520(6)	C(8) - N(1)	1.471(5)
C(9) - C(10)	1.382(6)	C(9) - C(14)	1.383(7)
C(10) - C(11)	1.385(8)	C(11) - C(12)	1.355(10)
C(12) - C(13)	1.369(10)	C(13) - C(14)	1.393(7)
C(16) - C(17)	1.516(7)	C(16) - O(2)	1.221(6)

Table IV : Bond angles (°) for C₁₇H₂₁NO₂

C(2) - C(1) - C(15)	123.3(4)	C(2) - C(1) - N(1)	122.1(4)
C(15) - C(1) - N(1)	114.7(4)	C(1) - C(2) - C(3)	119.1(4)
C(1) - C(2) - C(16)	122.9(4)	C(3) - C(2) - C(16)	118.0(4)
C(2) - C(3) - C(4)	110.9(4)	C(3) - C(4) - C(5)	109.7(4)
C(4) - C(5) - C(6)	112.2(4)	C(4) - C(5) - N(1)	110.1(3)
C(6) - C(5) - N(1)	111.5(4)	C(4) - C(5) - O(1)	108.5(3)
C(6) - C(5) - O(1)	110.6(3)	N(1) - C(5) - O(1)	103.5(3)
C(8) - C(7) - O(1)	104.8(3)	C(7) - C(8) - C(9)	111.5(4)
C(7) - C(8) - N(1)	100.5(3)	C(9) - C(8) - N(1)	116.0(3)
C(8) - C(9) - C(10)	121.2(4)	C(8) - C(9) - C(14)	119.1(4)
C(10) - C(9) - C(14)	119.7(4)	C(9) - C(10) - C(11)	119.6(5)
C(10) - C(11) - C(12)	120.2(5)	C(11) - C(12) - C(13)	121.3(5)
C(12) - C(13) - C(14)	119.1(6)	C(9) - C(14) - C(13)	120.0(5)
C(2) - C(16) - C(17)	117.6(4)	C(2) - C(16) - O(2)	125.5(4)
C(17) - C(16) - O(2)	116.9(4)	C(1) - N(1) - C(5)	121.3(3)
C(1) - N(1) - C(8)	125.2(3)	C(5) - N(1) - C(8)	110.1(3)
C(5) - O(1) - C(7)	106.3(3)		

Table S1 : Anisotropic thermal parameters for $C_{17}H_{21}NO_2$

Atom	u(11)	u(22)	u(33)	u(23)	u(13)	u(12)
C(1)	0.034(2)	0.0264(18)	0.037(2)	-0.0054(16)	0.0022(18)	0.0003(17)
C(2)	0.036(2)	0.034(2)	0.0301(19)	-0.0023(16)	0.0012(17)	-0.0022(19)
C(3)	0.031(2)	0.063(3)	0.041(2)	0.003(2)	-0.005(2)	-0.008(2)
C(4)	0.030(2)	0.060(3)	0.047(2)	0.001(2)	-0.0035(19)	-0.004(2)
C(5)	0.032(2)	0.042(2)	0.037(2)	0.006(2)	0.0070(17)	0.0000(19)
C(6)	0.048(3)	0.050(3)	0.065(3)	-0.001(2)	0.009(2)	0.015(2)
C(7)	0.059(3)	0.044(2)	0.045(2)	0.010(2)	-0.002(2)	-0.010(2)
C(8)	0.044(2)	0.0270(17)	0.041(2)	0.0022(17)	-0.0036(19)	-0.0030(18)
C(9)	0.053(3)	0.034(2)	0.0308(18)	0.0047(17)	-0.0018(19)	-0.0103(19)
C(10)	0.073(3)	0.039(3)	0.061(3)	-0.002(2)	-0.006(3)	-0.010(3)
C(11)	0.113(6)	0.054(3)	0.057(3)	-0.017(3)	0.010(4)	-0.036(4)
C(12)	0.101(5)	0.100(5)	0.035(2)	-0.001(3)	-0.007(3)	-0.069(5)
C(13)	0.066(4)	0.093(5)	0.055(3)	0.016(3)	-0.024(3)	-0.022(4)
C(14)	0.059(3)	0.051(3)	0.050(2)	0.008(2)	-0.012(2)	-0.008(3)
C(15)	0.033(2)	0.057(3)	0.042(2)	0.001(2)	0.001(2)	0.001(2)
C(16)	0.045(3)	0.034(2)	0.042(2)	-0.0019(19)	-0.001(2)	-0.003(2)
C(17)	0.074(4)	0.072(4)	0.037(2)	0.009(2)	-0.004(2)	-0.014(3)
N(1)	0.0307(17)	0.0304(15)	0.0350(17)	0.0021(16)	-0.0025(14)	0.0014(15)
O(1)	0.0494(19)	0.0487(17)	0.0481(16)	0.0115(15)	0.0018(15)	-0.0111(15)
O(2)	0.052(2)	0.072(2)	0.0553(19)	0.0154(18)	0.0076(17)	-0.012(2)

Table S2 : Hydrogen atoms fractional atomic coordinates for

 $C_{17}H_{21}NO_2$

Atom	x/a	y/b	z/c	U(iso)
H(31)	0.5914	0.3857	0.1301	0.091(5)
H(32)	0.6130	0.2638	0.0727	0.091(5)
H(41)	0.6217	0.1114	0.1672	0.091(5)
H(42)	0.4568	0.2038	0.1803	0.091(5)
H(61)	0.6227	0.3942	0.3286	0.091(5)
H(62)	0.6015	0.4437	0.2445	0.091(5)
H(63)	0.4499	0.3621	0.2840	0.091(5)
H(71)	0.7222	0.2163	0.3922	0.091(5)
H(72)	0.7584	0.0590	0.3819	0.091(5)
H(81)	0.9748	0.0999	0.2979	0.091(5)
H(101)	0.9033	0.4336	0.3571	0.091(5)
H(111)	1.1032	0.5472	0.4305	0.091(5)
H(121)	1.3298	0.4351	0.4821	0.091(5)
H(131)	1.3839	0.2103	0.4512	0.091(5)
H(141)	1.1922	0.0973	0.3727	0.091(5)
H(151)	1.1392	0.2389	0.2426	0.091(5)
H(152)	1.1587	0.3535	0.1808	0.091(5)
H(153)	1.1462	0.1988	0.1565	0.091(5)
H(171)	0.8884	0.4266	-0.0454	0.091(5)
H(172)	0.7308	0.4640	0.0075	0.091(5)
H(173)	0.7535	0.3129	-0.0217	0.091(5)

Crystal structure of (3*S*, 8*aR*)-2c

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2d - Major