Practical Biocatalytic Desymmetrization of Meso N-Heterocyclic Dicarboxamides and Their Application in the Construction of Aza-sugar Containing Nucleoside Analogs

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1. General Information

¹H and ¹³C NMR spectra were recorded on 300MHz and 400MHz spectrometers. Chemical shifts are reported in ppm versus tetramethylsilane or the residual solvent resonance used as an internal standard. Melting points are uncorrected. Enantiomeric excess (ee) values of all compounds were obtained from HPLC analyses using chiral stationary phases.
2. General Procedure for Biocatalytic Desymmetrization

To an Erlenmeyer flask (150 mL) with a screw cap were added aqueous potassium phosphate buffer (0.1 M, pH 7.0, 250 mL) and *Rhodococcus erythropolis* AJ270 cells (2 g wet weight). The resting cells were activated at 30 °C for 0.5 h with orbital shaking. Dicarboxamide substrates 1 (1 mmol) was added in one portion to the flask, and the resulting mixture was incubated at 30 °C using an orbital shaker (200 rpm). The reaction, monitored TLC, was quenched after a period of time (see Table 1 and Scheme 1) by removing the biomass by filtration through a Celite pad, and the filtrate was freeze-dried. The residue was mixed with DMF (2 mL), BnBr (1 mL) and K₂CO₃ (1.38 g, 10 mmol). After stirring for 24 h, water (10 mL) was added and the resulting mixture was extracted with ethyl acetate (3 × 100 mL), washed with brine (3 × 2 mL) and dried with anhydrous Na₂SO₄. After removal of solvent, the residue was chromatographed using a silica gel column with ethyl acetate as eluent to give pure products 2.

![Chemical structure](image)

**Benzyl N-benzyl-5-carbamoylpyrrolidine-2-carboxylate (2a):** white solid; mp 58 °C; [α]₂⁰⁰ +19.2° (c 1, CH₂Cl₂); ee >99.5% (HPLC analysis using a Diacel-ADH column); IR (KBr) ν 3422, 3369, 1740, 1663 cm⁻¹; ¹H NMR (300 MHz/CDCl₃) δ 7.89 (br, s, 1H), 7.37-7.22 (m, 10H), 5.90 (br, s, 1H), 4.98 (s, 2H), 3.88 (d, J = 13.2 Hz, 1H), 3.79 (d, J = 13.2 Hz, 1H), 3.63 (t, J = 7.4 Hz, 1H), 3.54-3.50 (m, 1H), 2.19-1.88 (m, 4H); ¹³C NMR (75MHz/CDCl₃) δ 177.1, 174.1, 136.4, 134.7, 128.5, 127.8, 127.6, 127.56, 127.3, 126.8, 67.1, 65.9, 65.5, 58.2, 29.8, 29.5; MS (ESI) m/z 339.1 (M+1, 86), 361.2 (M+23, 100%). Anal. Calcd. for C₂₀H₂₂N₂O₃: C, 70.99; H, 6.55; N, 8.28. Found: C, 70.65; H, 6.71; N, 8.53.
Benzyl N-benzyl-5-carbamoyl-2,5-dihydro-1H-pyrrole-2-carboxylate (2b): mp 127-128 °C; [α]$^D_{25}$ -36.4° (c 1, CH$_2$Cl$_2$); ee >99.5% (HPLC analysis using a Diacel-ADH column); IR (KBr) ν 3361, 3344, 3223, 2527, 1707, 1682, 1627, 1177 cm$^{-1}$; $^1$H NMR (300 MHz/Acetone-$d_6$) δ 7.88 (br, s, 1H), 7.41-7.26 (m, 10H), 6.43 (br, s, 1H), 5.84-5.82 (m, 1H), 5.07 (d, $J$ = 12.3 Hz, 1H), 5.04 (d, $J$ = 12.3 Hz, 1H), 4.63-4.61 (m, 1H), 4.27-4.25 (m, 1H), 4.05 (d, $J$ = 13.5 Hz, 1H), 3.97 (d, $J$ = 13.5 Hz, 1H); $^{13}$C NMR (75 MHz/Acetone-$d_6$) δ 173.9, 172.7, 137.9, 136.0, 130.9, 129.2, 128.5, 128.3, 128.1, 128.0, 127.3, 126.6, 76.2, 73.6, 66.4, 58.4; MS (ESI) m/z 338.3 (M+D, 90), 360.3 (M(D)+23, 100%). Anal. Calcd. for C$_{20}$H$_{20}$N$_2$O$_3$: C, 71.41; H, 5.99; N, 8.33. Found: C, 71.15; H, 5.98; N, 8.17.

Benzyl N-benzyl-6-carbamoylpiperidine-2-carboxylate (2c): mp 105 °C; [α]$^D_{25}$ -25.6° (c 1, CH$_2$Cl$_2$); ee >99.5% (HPLC analysis using a Diacel-ADH column); IR (KBr) ν 3420, 1744, 1632 cm$^{-1}$; $^1$H NMR (300 MHz/CDCl$_3$) δ 7.36-7.17 (m, 11 H), 5.71 (br, s, 0.5 H), 5.64 (br, s, 0.5H), 5.07 (s, 2 H), 3.77 (s, 2 H), 3.23 (dd, 1H, $J$ = 10.3, 3.7 Hz), 3.09 (dd, 1H, $J$ = 9.4, 3.7 Hz), 2.00-1.84 (m, 2H), 1.73-1.63 (m, 3H), 1.26-1.17 (m, 2H); $^{13}$C NMR (75 MHz/CDCl$_3$) δ 177.3, 174.0, 135.6, 134.9, 130.3, 128.6, 128.5, 128.4, 128.2, 127.6, 66.7, 63.3, 61.6, 58.3, 28.2, 28.1, 20.7; MS (ESI) m/z 353.2 (M+1,100%), 375.2 (M(D)+23, 100%). Anal. Calcd. for C$_{21}$H$_{24}$N$_2$O$_3$: C, 71.57; H, 6.86; N, 7.95. Found: C, 71.58; H, 6.84; N, 7.96.

Benzyl 1-benzyl-7-carbamoylazepane-2-carboxylate (2d): oil; [α]$^D_{25}$ -25.6 ° (c 1,
CH$_2$Cl$_2$); IR (KBr) ν 3410, 1737, 1670 cm$^{-1}$; $^1$H NMR (300 MHz/CDCl$_3$) δ 8.42 (br, s, 1 H), 7.38-7.23 (m, 10 H), 5.88 (br, s, 1 H), 5.15 (d, 1 H, J = 12.1 Hz), 5.07 (d, 1 H, J = 12.1 Hz), 3.75 (d, 1 H, J = 8.7 Hz), 3.72 (d, 1 H, J = 8.7 Hz), 3.62 (dd, 1 H, J = 8.7, 1.8 Hz), 3.42 (d, 1 H, J = 8.1 Hz), 2.02-1.46 (m, 8 H); $^{13}$C NMR (75 MHz/CDCl$_3$) δ 178.4, 176.3, 138.0, 135.5, 129.0, 128.7, 128.54, 128.50, 128.3, 127.6, 67.0, 66.3, 60.6, 29.1, 28.2, 28.1, 26.8; MS (ESI) m/z 367.2 (M+1, 20%), 389.3 (M+23, 100). Anal. Calcd. for C$_{22}$H$_{26}$N$_2$O$_3$: C, 72.11; H, 7.15; N, 7.64. Found: C, 72.07; H, 7.10; N, 7.79.

To determine the ee value of the product, 2d was converted into 2d'.

**Preparation of 2d'.** To a solution of 2d (70 mg, 0.2 mmol) in a mixture of EtOH (5 mL) and THF (5 mL) was added consecutively NaBH$_4$ (15.2 mg, 0.4 mmol) and LiCl (15.2 mg, 0.4 mmol). After the resulting mixture was stirred for 3 h at ambient temperature, water (20 mL) was added and the resulting mixture was extracted with ethyl acetate (3 × 15 mL). Combined organic phase was washed with brine (3 × 5 mL) and dried with anhydrous Na$_2$SO$_4$. After removal of solvent, the residue was chromatographed using a silica gel column with ethyl acetate as an eluent to give pure products 2d' (50 mg, 95%): mp 146 °C; [$\alpha$]$^25_D$ +22.9° (c 0.35, CH$_2$Cl$_2$); ee 63% (HPLC analysis using a Diacel-OJH column); IR (KBr) ν 3364, 3274, 2943, 2919, 1664 cm$^{-1}$; $^1$H NMR (300 MHz/ DMSO-$_d_6$) δ 7.86 (br, s, 0.5 H), 7.85 (br, s, 0.5 H), 7.38-7.21 (m, 5 H), 7.02 (br, s, 1 H), 7.02 (br, s, 1 H), 4.98 (t, 1 H, J = 4.8 Hz), 3.81 (d, 1 H, J = 14.4 Hz), 3.67 (d, 1 H, J = 14.4 Hz), 3.53-3.47 (m, 2H), 3.17 (dd, 1 H, J = 15.3, 2.4 Hz), 2.67 (t, 1 H, J = 4.2 Hz), 1.98-1.91 (m, 1 H), 1.72-1.46 (m, 6 H), 1.34-1.17 (m, 1 H); $^{13}$C NMR (75 MHz/ DMSO-$_d_6$) δ 176.4, 140.5, 128.4, 128.1, 126.8, 66.6, 65.0, 63.2, 58.0, 29.2, 28.0, 26.3, 25.2; MS (ESI) m/z 263.4 (M+1, 75%), 285.3(M+23,100). Anal. Calcd. for C$_{15}$H$_{22}$N$_2$O$_2$: C, 68.67; H, 8.45; N, 10.68. Found: C, 68.60; H, 8.38; N, 10.72.
3. Procedure for large scale biocatalytic desymmetric hydrolysis of 1a. To an Erlenmeyer flask (500 mL) with a screw cap *Rhodococcus erythropolis* AJ270 cells (2 g wet weight) were suspended in aqueous potassium phosphate buffer (0.1 M, pH 7.0, 250 mL), and activated at 30 °C for 0.5 hour with orbital shaking. Dicarboxamide 1a (20 g) was added in one portion to the flask, and the resulting mixture was incubated at 30 °C using an orbital shaker (200 rpm). The reaction, monitored by HPLC, was quenched after 12 h by removing the biomass by filtration through a Celite pad. The filtrate was concentrated to 100 mL using a rotary evaporator under reduced pressure, and product 3 (8.0 g) precipitated from solution. The mother liquid was subjected to an ion-exchange column and eluted with aqueous ammonia solution (1%) to give another portion of product 3 (11.0 g).

![Product 3](image)

Product 3 (19.0 g, 94%): mp 234-233 °C; [α]$_D$^25$ -9.6°(c 1, H$_2$O); IR(KBr) $\tilde{\nu}$ 3308, 3153, 2361, 1696, 1642, 1576 cm$^{-1}$; $^1$H NMR (D$_2$O/300MHz) 4.34 (t, $J = 7.2$ Hz, 1 H), 4.11 (t, $J = 6.3$ Hz, 1 H), 2.39-2.25 (m, 2 H), 2.05-1.91 (m, 2 H); $^{13}$C NMR (75MHz /D$_2$O) 173.8, 171.4, 61.8, 60.31, 29.8, 29.2; MS (ESI) m/z 159 (M+1, 7), 181 (M+23, 100%). Anal. Calcd. for C$_6$H$_{10}$N$_2$O$_3$: C, 45.57; H, 6.37; N, 17.71. Found: C, 45.54; H, 6.53; N, 17.78.

To determine the enantiomeric excess (ee) value and the absolute configuration of product 3, transformation of 3 into 2a was conducted. Thus, a mixture of 3 (158 mg), K$_2$CO$_3$ (1.38 g), benzyl bromide (1 mL) in dry DMF (2 mL) was stirred at ambient temperature for 24 h. Water (10 mL) was then added, and the mixture was extracted with ethyl acetate (3×10 mL). The combined organic layer was washed with brine (3×10 mL), and dried with anhydrous Na$_2$SO$_4$. After removal of organic solvent, the residue was chromatographed on a silica gel column eluted with ethyl acetate to give 2a (320 mg, 95%). Slow vapor diffusion of diethyl ether into a mixture of 2a (15.7
mg), concentrated hydrochloric acid (15 µL) and ethanol (0.5 mL) at 5 °C for 2 days led to the formation of a single crystal of 2a·HCl.

X-ray structure:

4. Synthesis of Aza sugar containing nucleoside Analogs

Preparation of 4. To a mixture of 3 (4.0 g, 25 mmol) in ethanol (20 mL) and saturated NaHCO₃ aqueous solution (20 mL) was added drop-wise CbzCl (6 mL) at room temperature while stirring. The resulting mixture was kept stirring overnight. The reaction was quenched by adding hydrochloric acid (2 N) until the pH was adjusted to 7. Under reduced pressure, the solvent was completely removed. The residue was mixed with methanol (100 mL) and SOCl₂ (4 mL) at ambient temperature. After stirring for another 4 h at ambient temperature, saturated NaHCO₃ aqueous solution (200 mL) was added. The mixture was extracted with ethyl acetate (3×100 mL). The combined organic layer was washed with brine (3×20 mL) and dried with anhydrous Na₂SO₄. After removal of organic solvent, the residue was chromatographed on a silica gel column eluted with ethyl acetate to give product 4 (6.9 g, 91%): mp 104 °C; [α]²⁵ᵇ +46.6° (c 0.6, CHCl₃); IR (KBr) ν 3435, 3401, 1734, 1700, 1595, 1545, 1490, 1460, 1380, 1295, 1205, 1180, 1165, 1110, 1050, 1000, 970, 920, 830, 800, 760, 700, 670, 600; 1H-NMR (DMSO-d₆) δ 8.60 (d, 1H, J=16 Hz), 8.20 (d, 1H, J=16 Hz), 7.00 (s, 1H), 6.70 (d, 1H, J=8 Hz), 5.80 (d, 1H, J=8 Hz), 5.50 (d, 1H, J=8 Hz), 5.00 (d, 1H, J=8 Hz), 4.00 (s, 3H), 3.80 (s, 3H), 3.30 (s, 3H), 3.00 (s, 3H), 2.80 (s, 3H), 2.60 (s, 3H), 2.50 (s, 3H), 2.30 (s, 3H), 2.00 (s, 3H), 1.80 (s, 3H), 1.50 (s, 3H), 1.20 (s, 3H); 13C-NMR (DMSO-d₆) δ 170.0, 169.0, 168.0, 165.0, 163.0, 162.0, 161.0, 160.0, 159.0, 158.0, 157.0, 156.0, 155.0, 154.0, 153.0, 152.0, 151.0, 150.0, 149.0, 148.0, 147.0, 146.0, 145.0, 144.0, 143.0, 142.0, 141.0, 140.0, 139.0, 138.0, 137.0, 136.0, 135.0, 134.0, 133.0, 132.0, 131.0, 130.0, 129.0, 128.0, 127.0, 126.0, 125.0, 124.0, 123.0, 122.0, 121.0, 120.0, 119.0, 118.0, 117.0, 116.0, 115.0, 114.0, 113.0, 112.0, 111.0, 110.0, 109.0, 108.0, 107.0, 106.0, 105.0, 104.0, 103.0, 102.0, 101.0, 100.0, 99.0, 98.0, 97.0, 96.0, 95.0, 94.0, 93.0, 92.0, 91.0, 90.0, 89.0, 88.0, 87.0, 86.0, 85.0, 84.0, 83.0, 82.0, 81.0, 80.0, 79.0, 78.0, 77.0, 76.0, 75.0, 74.0, 73.0, 72.0, 71.0, 70.0, 69.0, 68.0, 67.0, 66.0, 65.0, 64.0, 63.0, 62.0, 61.0, 60.0, 59.0, 58.0, 57.0, 56.0, 55.0, 54.0, 53.0, 52.0, 51.0, 50.0, 49.0, 48.0, 47.0, 46.0, 45.0, 44.0, 43.0, 42.0, 41.0, 40.0, 39.0, 38.0, 37.0, 36.0, 35.0, 34.0, 33.0, 32.0, 31.0, 30.0, 29.0, 28.0, 27.0, 26.0, 25.0, 24.0, 23.0, 22.0, 21.0, 20.0, 19.0, 18.0, 17.0, 16.0, 15.0, 14.0, 13.0, 12.0, 11.0, 10.0, 9.0, 8.0, 7.0, 6.0, 5.0, 4.0, 3.0, 2.0, 1.0, 0.0; MS m/z 1056 (M+).
1700, 1683 cm$^{-1}$; $^1$H NMR (300 MHz/DMSO-$d_6$, 375 K) $\delta$ 7.35-7.25 (m, 5H), 6.97 (br, s, 2H), 5.09 (d, $J$ = 12.6 Hz, 1H), 5.05 (d, $J$ = 12.9 Hz, 1H), 4.42 (t, $J$ = 7.2 Hz, 1H), 4.20-4.16 (m, 1H), 3.66 (s, 3H), 2.31-2.17 (m, 2H), 1.98-1.87 (m, 2H); $^{13}$C NMR (75 MHz/DMSO-$d_6$) $\delta$ 175.0, 174.8, 174.3, 174.0, 154.4, 154.1, 136.9, 136.7, 128.8, 128.4, 128.3, 127.7, 127.6, 67.2, 67.1, 62.5, 62.1, 60.5, 60.1, 53.1, 53.0, 30.6, 29.7, 29.6, 28.8; MS (ESI) $m/z$ 307 (M+1, 20), 329 (M+23, 100%). Anal. Calcd. for C$_{15}$H$_{18}$N$_2$O$_5$: C, 58.82; H, 5.92; N, 9.15. Found: C, 58.87; H, 5.99; N, 9.10.

**Synthesis of 5.**

To a solution of 4 (3.06 g, 10 mmol) in dry DMF (4 mL) was added SOCl$_2$ (1 mL) at 0°C. After stirring for another 1 h at room temperature, the reaction was quenched by the addition of saturated NaHCO$_3$ aqueous solution (10 mL). The mixture was extracted with ethyl acetate (3×20 mL). The combined organic layer was washed with brine (3×4 mL) and dried with anhydrous Na$_2$SO$_4$. After removal of organic solvent, the residue was chromatographed on a silica gel column eluted with a mixture of ethyl acetate and petroleum ether (1:2) to give compound 5 (2.6 g, 90%): oil; $[\alpha]_{25}^{25}$ -14.6$^\circ$ (c 1, CHCl$_3$); IR (KBr) ν 1746, 1715 cm$^{-1}$; $^1$H NMR (300 MHz/CDCl$_3$) $\delta$ 7.40-7.33 (m, 5H), 5.28-5.09 (m, 2H), 4.72-4.63 (m, 1H), 4.47-4.37 (m, 1H), 3.79 (s, 1.5H), 3.64 (s, 1.5H), 2.44-2.21 (m, 4H); $^1$H NMR (300 MHz/DMSO-$d_6$, 375K) $\delta$ 7.39-7.28 (m, 5H), 5.18-5.06 (m, 2H), 4.84-4.80 (m, 1H), 4.42-4.37 (m, 1H), 3.64 (s, 3H), 2.42-2.30 (m, 2H), 2.24-2.12 (m, 1H), 2.10-2.02 (m, 1H); $^{13}$C NMR (75 MHz/DMSO-$d_6$) $\delta$ 171.6, 171.4, 153.3, 135.6, 128.6, 128.4, 128.2, 128.1, 128.0, 127.8, 118.0, 117.8, 68.3, 68.0, 67.5, 59.6, 59.3, 52.7, 52.5, 48.1, 47.5, 30.6, 29.8, 29.7, 28.7; MS (ESI) $m/z$ 311 (M+23, 100%). Anal. Calcd. for C$_{15}$H$_{16}$N$_2$O$_4$: C, 62.49; H, 5.59; N, 9.72. Found: C, 62.70; H, 5.62; N, 9.57.
**Synthesis of 6.** A mixture of 5 (576 mg, 2 mmol), NaN₃ (260 mg, 4 mmol), ZnBr₂ (225 mg, 1 mmol) in water (6 mL) and 2-propanol (3 mL) was refluxed for 16 h. After cooling down to room temperature, hydrochloric acid (1 N, 10 mL) was added. The mixture was extracted with ethyl acetate (3×20 mL). The combined organic layer was washed with brine (3×4 mL) and dried with anhydrous Na₂SO₄. After removal of organic solvent, the residue was chromatographed on a silica gel column eluted with a mixture of ethyl acetate and methanol (20:1) to give compound 6 (367 mg, 58%): mp 183-185 °C; oil; [α]₂₅° -22.0° (c 0.5, CHCl₃); IR (KBr) ν 2960, 1707, 1451, 1422 cm⁻¹; ¹H NMR (300 MHz/DMSO-d₆, 373K) δ 7.31-7.23 (m, 5H), 5.33-5.31 (m, 1H), 5.05 (s, 2H), 4.41 (t, 1H, J = 8.0 Hz), 2.46-2.35 (m, 2H), 2.19-2.05 (m, 2H); ¹³C NMR (75 MHz/ DMSO-d₆) δ 177.53, 177.05, 157.7, 157.3, 152.8, 134.4, 134.1, 127.6, 127.4, 126.9, 67.2, 67.1, 58.97, 58.5, 52.5, 52.1, 31.6, 30.5, 29.9, 28.7, 27.8; MS (ESI) m/z 340 (M⁺+23, 100%). Anal. Calcd. for C₁₄H₁₆N₅O₄ [M⁺H]: 318.1202. Found: 318.1192.

**Synthesis of 7.** Under protection of argon, a mixture of 6 (317 mg, 1 mmol) and LiAlH₄ (380 mg, 10 mmol) in dry THF (10 mL) was refluxed for 24 h. While the mixture was kept at -20°C, NaOH aqueous solution (2 N, 1 mL) was slowly injected through a syringe. The resulting mixture was filtrate through a Celite pad and washed thoroughly with a mixture of CH₂Cl₂ and MeOH (20:1). After removal of solvent, the
residue was mixed with hydrochloric acid (1 N) with pH value of the mixture being adjusted to pH 2. The resulting mixture was subjected to an ion-exchange column (nk 001×7) and then eluted with pure water. After the pH of the fraction was around 7, the column was eluted with aqueous ammonia solution (1%). After removal of solvent, product 7 was obtained. 7 (152 mg, 83%): mp 185 °C; [α]$_D^{25}$ +10.3° (c 0.78, H$_2$O); IR (KBr) v 3447, 2959, 2922, 1578, 1411, 1075, 1030 cm$^{-1}$; $^1$H NMR (300 MHz/D$_2$O) δ 4.53 (t, $J$ = 8.7 Hz, 1H), 3.77-3.64 (m, 2H), 3.54-3.45 (m, 1H), 2.72 (s, 3H), 2.38-2.26 (m, 2H), 2.23-2.14 (m, 1H), 1.94-1.83 (m, 1H); $^{13}$C NMR (75MHz/D$_2$O) δ 157.1, 70.3, 64.2, 59.6, 39.1, 27.7, 24.9; MS (ESI) m/z 184.1 (M+1, 15%), 206.1 (M+23, 100%). Anal. Calcd. for C$_7$H$_{14}$N$_5$O [M+H]: 184.1198. Found: 184.1193.

Preparation of 8. A mixture of 5 (100 mg, 0.33 mmol) and LiOH·H$_2$O (23 mg, 0.55 mmol) in THF (3 mL) and water (3 mL) was stirring at ambient temperature for 2 h. The mixture was then treated with hydrochloric acid (1 N) until the pH of the mixture was adjusted to 2, and extracted with ethyl acetate (3×10 mL). The combined organic layer was washed with brine (3×3 mL) and dried with anhydrous Na$_2$SO$_4$. After removal of organic solvent, the residue was chromatographed on a silica gel column eluted with a mixture of petroleum ether, ethyl acetate and acetic acid (200:100:0.5). The collected fractions were concentrated to about 10 mL at 30-40 °C under reduced pressure using a rotary evaporator. The resulting solution was mixed with ethyl acetate (50 mL) and washed with brine (3×10 mL). After drying with anhydrous Na$_2$SO$_4$ and removing solvent, pure 8 was obtained. 8 (90 mg, 95%): oil; [α]$_D^{25}$ -24.6° (c 4.6, CHCl$_3$); IR (KBr) v 3065, 3038, 2960, 1715, 1414, 1358 cm$^{-1}$; $^1$H NMR (300MHz/DMSO-$d_6$, 375K) δ 7.36-7.28 (m, 5H), 5.14 (s, 2H), 4.82-4.78 (m, 1H), 4.34-4.30 (m, 1H), 2.42-2.29 (m, 2H), 2.24-2.11 (m, 1H), 2.09-2.00 (m, 1H); $^{13}$C
NMR (75 MHz/DMSO-\textit{d}_6) \delta 173.3, 173.0, 153.7, 153.4, 136.7, 128.8, 128.4, 128.3, 127.9, 127.7, 119.7, 119.4, 67.4, 67.3, 60.1, 59.6, 48.4, 47.8, 30.7, 29.9, 29.7, 28.9; MS (ESI) \textit{m/z} 273.1 (M-1, 100%). Anal. Calcd. for C_{14}H_{15}N_{2}O_{4} [M+H]: 275.1032. Found: 275.1022.

9: To a solution of 8 (66.1 mg, 0.24 mmol) in CH\textsubscript{2}Cl\textsubscript{2} (4 mL) was added consecutively 2-chloro-4,6-dimethoxy-1,3,5-triazine (CDMT, 50.5 mg, 0.29 mmol) and \textit{N}-methylmorpholine (NMM, 83 \textmu L, 0.72 mmol). After stirring at room temperature for 2.5 h, an ammonia gas balloon (250 mL) was connected, and the reaction mixture was stirred for 1 h. After removal of the ammonia gas balloon, the mixture was kept stirring for another 1 h. The reaction was quenched by adding hydrochloric acid (1 N, 20 mL). After extraction with ethyl acetate (3×10 mL), the combined organic layer was dried with anhydrous Na\textsubscript{2}SO\textsubscript{4}. After removal of organic solvent, the residue was chromatographed on a silica gel column eluted with a mixture of petroleum ether and ethyl acetate (1:2) to afford 9 (44 mg, 67%): mp 44 °C; [\alpha]^{25}\textsubscript{D} -7.0° (c 0.58, CHCl\textsubscript{3}); IR (KBr) \nu 3428, 3352, 3200, 1708, 1412, 1356, 1119 cm\textsuperscript{-1}; \textsuperscript{1}H NMR (300 MHz/DMSO-\textit{d}_6, 375K) \delta 7.38-7.29 (m, 5H), 6.87 (s, br, 1H), 5.13 (d, \textit{J} = 12.9 Hz, 1H), 5.11 (d, \textit{J} = 12.9 Hz, 1H), 4.76 (dd, \textit{J} = 7.4, 4.8 Hz, 1H), 4.29 (dd, \textit{J} = 7.4, 5.1 Hz, 1H), 2.32-2.14 (m, 3H), 2.02-1.96 (s, br, 1H); \textsuperscript{13}C NMR (75MHz/DMSO-\textit{d}_6) \delta 173.0, 172.8, 153.9 153.5, 136.8, 128.8, 128.4, 128.2, 127.9, 127.6, 119.8, 119.5, 67.2, 67.1, 60.7, 60.2, 48.6, 48.0, 30.6, 29.7, 29.5; MS (ESI) \textit{m/z} 296.2 (M+23, 100%). Anal. Calcd. for C_{14}H_{16}N_{3}O_{3} [M+H]: 274.1192. Found: 274.1186. Anal. Calcd. for C_{14}H_{15}N_{3}NaO_{3} [M+Na]: 296.1011. Found: 296.1004.
Synthesis of ent-4. To a solution of 9 (155mg, 0.57 mmol) in MeOH (4 mL) at -15 °C was bubbled with dry HCl (g) for 1 h. After the resulting solution was kept at -20 °C for 12 h, hydrochloric acid (6 N, 10 mL) was added. After extraction with ethyl acetate (3×100mL) and washing with brine (3×10 mL), the combined organic phase was dried with anhydrous Na₂SO₄. The solvent was removed and the residue was chromatographed on a silica gel column eluted with ethyl acetate to give ent-4 (133 mg, 76%): [α]_25^D -47.3° (c 1.7, CHCl₃). Identical IR and ¹H NMR spectra as that of 4 were obtained.

Ent-5 was synthesized from ent-4 following the same procedure for the conversion of 4 to 5. Ent-5: [α]_25^D +14.7° (c 3.0, CHCl₃). Identical IR and ¹H NMR spectra as that of 5 were obtained.

Ent-6 was synthesized from ent-5 following the same procedure for the transformation of 5 into 6. Ent-6: [α]_25^D +24.0° (c 1.0, MeOH). Identical IR and ¹H NMR spectra as that of 6 were obtained.
Ent-7 was synthesized from ent-6 following the same procedure for the transformation from 6 into 7. Ent-7: mp 187 °C; [α]$_D^{25}$ -11.0° (c 2.0, H$_2$O). Following identical IR, $^1$H and $^{13}$C NMR spectra as that of 7 were obtained. IR (KBr) ν 3447, 2958, 2921, 1577, 1409, 1077, 1025 cm$^{-1}$; $^1$H NMR (300 MHz/D$_2$O) δ 4.53 (t, $J$ = 8.7 Hz, 1H), 3.77-3.64 (m, 2H), 3.54-3.45 (m, 1H), 2.72 (s, 3H), 2.38-2.26 (m, 2H), 2.23-2.14 (m, 1H), 1.94-1.83 (m, 1H); $^{13}$C NMR (75 MHz/D$_2$O) δ 157.1, 70.3, 64.2, 59.6, 39.1, 27.7, 24.9; MS (ESI) m/z 184.1 (M+1, 100%). Anal. Calcd. for C$_7$H$_{14}$N$_5$O [M+H]: 184.1198. Found: 184.1190.

4. HPLC analysis of products.

A Shimadzu LC-10AVP HPLC system was used to analyze enantiomeric excess values of all products.

Table S1: HPLC analysis of amides 2a-d

<table>
<thead>
<tr>
<th>Compound</th>
<th>Hexane / 2-propanol</th>
<th>Temperature (°C)</th>
<th>Rate (mL/min)</th>
<th>$t_1$ (min)</th>
<th>$t_2$ (min)</th>
<th>Column</th>
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</thead>
<tbody>
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<td>2a</td>
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<td>41.9</td>
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<td>2b</td>
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<td>57.2</td>
<td>Chiralcel ADH</td>
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<tr>
<td>2c</td>
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<td>15</td>
<td>0.5</td>
<td>26.4</td>
<td>27.9</td>
<td>Chiralcel ADH</td>
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Method Name: D:\HPLC\yangluo\2006\method\\%p Gaoming212nm.met
Data Name: E:\chenpeng\211b\rac-211b001
User: System
Acquired: 2011-9-10 18:16:06

Detector A-213 nm

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Data Name: E:\chenpeng\2011-9-10 211b-16h001
User: System

Detector A-213 nm

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Acquired: 2009-12-19 19:12:10
Printed: 2010-4-1 20:53:21

Detector
A-207 nm

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CONH$_2$

N–Bn

COOBn

cShimadzu CLASS-VP V6.13 SP2

Method Name: D:\HPLC\yangluo\2006\method\Yangluo\Gaoming212nm.met
Data Name: E:\chenpeng\bio-d5-15c-0001.5-adh
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Acquired: 2009-12-31 12:38:04
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Detector
A-207 nm

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Racemic

Shimadzu CLASS-VP V6.13 SP2

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Data Name: E:chenpeng\hexdiamide\rac-h-3-15c-0.5-adh'
User: System
Acquired: 2008-7-9 20:33:29

1: 254 nm, 8 nm

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User: System
Acquired: 2010-6-25 17:25:06

Detector A-216 nm

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Data Name: E:\chenpeng\q\RAC-Q5-0001.5-20C-OJH'
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Detector A-215 nm

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Detector A-215  

\[ \text{nm} \]

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5. $^1$H and $^{13}$C NMR spectra of all products
Current Data Parameters
NAME               cp-2d
EXPNO                10
PROCNO                1

F2 - Acquisition Parameters
Date_          20090821
Time                 9.41
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                92
DS                  4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ               1.8219508 sec
RG                1625.5
DW                27.800 usec
DE                6.00 usec
TE                301.2 K
D1        2.000000000 sec
D11          0.030000000 sec
DELTA       1.889999998 sec
MCREST       0.000000000 sec
MCWRK       0.015000000 sec

------- CHANNEL f1 ------
NUC1          13C
P1           12.50 usec
PL1          2.00 dB
SFO1       75.4752953 MHz

------- CHANNEL f2 ------
CPDPRG2     waltz16
NUC2          1H
PCPD2       80.00 usec
PL2        -1.00 dB
PL12       20.16 dB
PL13       16.98 dB
SFO2    300.1312005 MHz

F2 - Processing parameters
SI                32768
SF     75.4678131 MHz
WDW             EM
SSB                0
LB                1.00 Hz
GB                0
PC                1.40
Current Data Parameters
NAME             cp-h3
EXPN0            20
PROCNO           1

F2 - Acquisition Parameters
Date_            20090717
Time              11.24
INSTRUM           spect
PROBHD     5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                16
DS                0
SWH       8992.806 Hz
AQ            3.6438515 sec
RG                101.6
DW                55.600 usec
DE                6.00 usec
TE                302.0 K
D1           1.00000000 sec
MCREST   0.00000000 sec
MCWRK        0.01500000 sec

------ CHANNEL f1 ------
NUC1                 1H
P1                7.00 usec
PL1               -1.00 dB
SF01           300.1324010 MHz

F2 - Processing parameters
SI                32768
SF            300.1300067 MHz
WDW             EM
SSB              0
LB                0.30 Hz
GB              0
PC                1.00
Current Data Parameters

NAME          cp-h3
EXPN0          21
PROCNO          1

F2 - Acquisition Parameters
Date_          20090717
Time            11.38

INSTRUM       spect
PROBHD           5 mm DUL 13C-1
PULPROG         zgpg30
TD            65536
SOLVENT       CDCl3
NS             261
DS              4

SWH        17985.611 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG           1140.4
DW        27.800 usec
DE          6.00 usec
TE          302.1 K

D1         2.00000000 sec
d11        0.03000000 sec
DELTA        1.89999998 sec
DELTA         0.00000000 sec
MCREST        0.01500000 sec
MCPG          0.03000000 sec

= CHANNEL f1 =====
NUC1        13C
P1          12.50 usec
PL1         2.00 dB
SFO1       75.4752953 MHz

= CHANNEL f2 =====
CPDPRG2     waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -1.00 dB
PL12        20.16 dB
PL13        16.98 dB
SFO2       300.1312005 MHz

F2 - Processing parameters
SI           32768
SF        75.4677487 MHz
WDM         EM
SSB          0
LB          1.00 Hz
GB          0
PC          1.40

Electronic Supplementary Material (ESI) for Chemical Communications
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Current Data Parameters

NAME              cp-q4
EXPNO                60
PROCNO                1

F2 - Acquisition Parameters
Date_          20090630
Time              14.12
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8992.806 Hz
FIDRES    0.137219 Hz
AQ            3.6438515 sec
RG                57
DN      55.600 usec
DE                6.00 usec
TE                299.7 K
DI   1.00000000 sec
MCREST       0.00000000 sec
MCWRK         0.01500000 sec

----- CHANNEL f1 -----  
NUC1                 1H
P1                 7.00 usec
PL1               -1.00 dB
SF01        300.1324010 MHz

F2 - Processing parameters
SI                32768
SF     300.1300086 MHz
WDW                EM
SSB                    0
LB                 0.30 Hz
GB                    0
PC                1.00
Current Data Parameters
NAME              cp-q4
EXPN0              60
PROCNO              1

F2 - Acquisition Parameters
Date              20090630
Time              14.12
INSTRUM           spect
PROBHD             5 mm DUL 13C-1
PULPROG            zg30
TD                  65536
SOLVENT           CDCl3
NS                16
DS                   0
SNH             8992.806 Hz
FIDRES            0.137219 Hz
AQ              3.6438515 sec
RG               57
DW              55.600 usec
DE               6.00 usec
TE               299.7 K
D1              1.00000000 sec
MCREST           0.00000000 sec
MCREST        0.01500000 sec

F2 - Processing parameters
SI                32768
SF            300.1300086 MHz
WDW               EM
SSB              0
LB              0.30 Hz
GB                 0
PC               1.00
Electronic Supplementary Material (ESI) for Chemical Communications
This journal is © The Royal Society of Chemistry 2012
Current Data Parameters
NAME:  cp-p13 375K
EXPNO:  397
PROCNO:  1

F2 - Acquisition Parameters
Date:  20110326
Time:  18.48
INSTRUM:  spect
PROBHD:  5 mm BBO BB-1H
PULPROG:  zg30
TD:  65536
SOLVENT:  DMSO
NS:  16
DS:  0
SWH:  6188.119 Hz
FIDRES:  0.094423 Hz
AQ:  5.2953587 sec
RG:  8192
DW:  80.000 usec
DE:  6.000 usec
TE:  375.1 K
D1:  1.0000000 sec
MCREST:  0.0000000 sec
MCWRK:  0.0150000 sec

====== CHANNEL f1 ======
NUC1:  1H
P1:  13.80 usec
PL1:  3.00 dB
SFO1:  300.1318534 MHz

F2 - Processing parameters
SI:  32768
SF:  300.1300114 MHz
WDW:  EM
SSB:  0
LB:  0.20 Hz
GB:  0
PC:  1.00
NAME             cp-p13
EXPNO                12
PROCNO                1
Date_          20101111
Time              12.30
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgig30TD                32768
SOLVENT            DMSO
NS                  243
DS                    0
SWH           18832.393 Hz
FIDRES             0.574719 Hz
AQ            0.8700404 sec
RG                16384
DW                  26.550 usec
DE                 8.00 usec
TE                297.4 K
D1                2.00000000 sec
D11              0.03000000 sec
TD0                   1

= = = = = = = = CHANNEL f1 = = = = = =
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

= = = = = = = = CHANNEL f2 = = = = = =
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.33 dB
SFO2        300.1312005 MHz
SI                32768
SP                75.4677490 MHz
WDW             EM
SSB                0
LB                1.00 Hz
GB                0
PC                1.40
Current Data Parameters
NAME           shengwen
EXPNO               367
PROCNO                1

F2 - Acquisition Parameters
Date_          20110226
Time              16.54
INSTRUM           spect
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   27
DS                        0
SWH            6188.119 Hz
FIDRES        0.094423 Hz
AQ            5.2953587 sec
RG                16384
DW                80.800 usec
DE                6.00 usec
TE                375.8 K
D1      1.00000000 sec
MCREST       0.00000000 sec
MCWRK      0.01500000 sec

---------- CHANNEL f1 ----------
NUC1                 1H
P1                13.60 usec
PL1             3.00 dB
SF01        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300075 MHz
NDW                 EM
SSB                        0
LB             0.20 Hz
GB                        0
PC                        1.00
Current Data Parameters
NAME           shengwen
EXPNO               367
PROCNO                1

F2 - Acquisition Parameters
Date_          20110226
Time              16.54
STRUM           spect
PROBHD   5 mm BBO BB-1H
.PROG            zg30
.SOLVENT         DMSO
NS                   27
DS                    0
SWH            6188.119 Hz
FIDRES         0.094423 Hz
AQ            5.2953587 sec
RG                16384
DW               80.800 usec
DE                 6.00 usec
TE                375.8 KD
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

------- CHANNEL f1 -------
NUC1                 1H
P1                13.80 usec
PL1                3.00 dB
SFO1        300.1318534 MHz

F2 - Processing parameters
SI                32768
SF          300.1300075 MHz
WDW                  EMSSB                   0
LB                 0.20 Hz
GB                0
PC                1.00
NAME           cp-p35
EXPNO          11
PROCNO          1
Date_          20100818
Time              20.20
INSTRUM         spect
PROBHD         5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT        CDCl3
NS                398
DS                4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ                1.8219508 sec
RG             16384
DW              27.800 usec
DE                8.00 usec
TF            301.4 K
D1             2.00000000 sec
D11           0.03000000 sec
WD             27.800 usec
D              8.00 usec
TF             301.4 K
D1             2.00000000 sec
D11           0.03000000 sec
TD0                 1

--- CHANNEL f1 ---
NUC1           13C
P1            12.50 usec
PL1          2.00 db
SFO1          75.4752953 MHz

--- CHANNEL f2 ---
CPDPRG2       waltz16
NUC2           1H
PCPD2         100.00 usec
PL2           3.00 db
PL12          22.33 dB
PL13         23.00 db
SFO2        300.1312005 MHz
SI              32768
SF           75.4677490 MHz
WDW              EM
SSB                 0
LB            1.00 Hz
GB                0
PC             1.40
Current Data Parameters
NAME cp-p90
EXPN0 1
PROCNO 1

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Time 10.55
INSTRUM spect
PROBHD 5 mm DUL 13C-1
FULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SNH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 256
DW 55.600 usec
DE 8.00 usec
TE 297.5 K
D1 1.00000000 sec
TD0 1

-------- CHANNEL f1 --------
NUC1 1H
P1 10.80 usec
PL1 3.00 dB
SF01 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300012 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Current Data Parameters
NAME           shengwen
EXPNO          424
PROCNO          1

F2 - Acquisition Parameters
Date              20111110
Time              8.35
INSTRUM           spect
PROBHD     5 mm BBO BB-1H
PULPROG        zg30
TD               32768
SOLVENT         DMSO
NS               16
DS               0
SWH             8992.806 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG              2048
DW              55.600 usec
DE              6.00  usec
TE            372.9 K
DI            2.00000000 sec
MCREST     0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1              1H
P1               13.80 usec
PL1              3.00 dB
SFO1         300.1315007 MHz

F2 - Processing parameters
SI              32768
SF          300.1300071 MHz
WDW             EM
SSB            0
LB              0.30 Hz
GB              0
Current Data Parameters
NAME           shengwen
EXPNO               424
PROCNO                1

F2 - Acquisition Parameters
Date_          20111110
Time               8.35
INSTRUM           spectPROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                32768
SOLVENT            DMSO
NS                   16
DS                    0
SWH            8992.806 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 secRG                 2048
RG               55.600 usec
D0                  6.00 usec
TE                372.9 KD1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

== CHANNEL f1 =====
NUC1                 1H
P1                13.80 usec
PL1                3.00 dB
SFO1        300.1315007 MHz

F2 - Processing parameters
SI                32768
SF          300.1300071 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                  0

--------- CHANNEL f1 ---------
NUC1                 1H
P1                13.80 usec
PL1                3.00 dB
SFO1        300.1315007 MHz

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Current Data Parameters
NAME             cp-p91
EXPNO                13
PROCNO                1

F2 - Acquisition Parameters
Date_          20111103
Time              11.51
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgig30
TD                32768
SOLVENT            DMSO
NS                 1119
DS                0
SNH           18832.393 Hz
FIDRES         0.574719 Hz
AQ                  0.8700404 sec
RG                  256
WM                26.550 usec
DE                  8.00 usec
TE                297.8 K
D1                2.00000000 sec
D11                0.03000000 sec
TD0                  1

== CHANNEL f1 ==
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

== CHANNEL f2 ==
CPDPRG2         waltz16
NUC2                 1H
PCPD2             100.00 usec
PL2                3.00 dB
PL12               22.33 dB
SFO2        300.1312005 MHz

F2 - Processing parameters
SI                32768
SF          75.4677490 MHz
WDW             EM
SSB                  0
LB                1.00 Hz
GB                0
PC                1.40
Current Data Parameters
NAME          cp-p80-3e
EXPNO                12
PROCNO                1

F2 - Acquisition Parameters
Date_          20111105
Time               1.57
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   16
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ            3.6438515 sec
RG                  256
DW               55.600 usec
DE                8.00 usec
TE                296.5 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1                10.80 usec
PL1                3.00 dB
SFO1        300.1324010 MHz

F2 - Processing parameters
SI                32768
SF          300.1299984 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                   0
PC                1.00
Current Data Parameters
NAME cp-p82e
EXPNO 14
PROCNO 1

F2 - Acquisition Parameters
t_e 20111105
me 0.32
STRUM spect
DOBHD 5 mm DUL 13C-1
LPREG zg30
\delta 65536
\delta VENT D20
16

\delta H 8992.806 Hz
DRES 0.137219 Hz
\sigma 3.6438515 sec
256
55.600 usec
8.00 usec
296.9 K
1.00000000 sec
0

== CHANNEL f1 ==

\delta C1 1H
PL1 3.00 dB
SF01 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300268 MHz
WDM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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