Bioorganic Chemistry 38 (2010) 275–278

Contents lists available at ScienceDirect

Bioorganic Chemistry

journal homepage: www.elsevier.com/locate/bioorg

# Synthesis of the natural enantiomer of neplanocin B

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ARTICLE INFO

*Article history:*
Received 29 June 2010
Available online 16 August 2010

*Keywords:*
Neplanocin derivatives
Natural product
Carbocyclic nucleosides

ABSTRACT

(−)-Neplanocin B, the natural isomer of a component of the neplanocin family was diasteroselectively synthesized from 2,3-*O*-isopropylidene-D-1,4-ribonolactone. However, when evaluated against several DNA and RNA viruses in cell culture experiments, it did not show any antiviral activity.



## 1. Introduction

The neplanocin derivatives are an important class of naturally occurring carbocyclic nucleosides isolated from the culture filtrate of soil fungus *Ampullariella regularis* [1,2]. Five distinct components were identified: (−)-neplanocin A (**1**), (−)-neplanocin B (**2**), (−)-neplanocin C (**3**), (−)-neplanocin D (**4**) and (−)-neplanocin F (**5**) (Fig. 1). Among these compounds, (−)-neplanocin A received great attention due to its interesting biological properties [3,4] and numerous syntheses of neplanocin A as well as of their analogs were reported [5]. Indeed, (−)-neplanocin A, the lead compound of the neplanocin family has been recognized as a potent inhibitor of *S*-adenosyl-L-homocysteine (AdoHcy) hydrolase. Inhibition of AdoHcy hydrolase leads to the accumulation of AdoHcy which is a feedback inhibitor of *S*-adenosyl methionine-dependent methyltransferases. Such methylation reactions are required for the 5′-capping of viral mRNAs leading to the inhibition of the maturation of mRNAs which provides an antiviral effect [3].

In a less extent, only few syntheses of the other natural components of the neplanocin family have been reported in the literature. The total synthesis of (+/−)-neplanocin F as a racemate [6] as well as the enantioselective synthesis of its unnatural (+) [7] and natural (−)-enantiomer [8] were described. The synthesis of (+) and (−)-neplanocin D [9] as well as (−)-neplanocin C [10] were also reported. In contrast, only the synthesis of the unnatural (+)-enantiomer of neplanocin B was published [11].

In this context, and in order to evaluate its antiviral properties, we report herein the first synthesis of the natural enantiomer.

## 2. Experimental

### 2.1. General methods

Evaporation of solvents was carried out on a rotary evaporator under reduced pressure. Melting points were determined in open capillary tubes on a Gallenkamp MFB-595-010 M apparatus and are uncorrected. UV spectra were recorded on an Uvikon 931 (Kontron) spectrophotometer. $^{1}$H NMR spectra were recorded at 300 MHz, $^{13}$C NMR spectra at 100 MHz and $^{19}$F NMR at 235 MHz in $(CD_3)_2SO$ at ambient temperature with a Brüker DRX 400. Chemical shifts ($\delta$) are quoted in parts per million (ppm) referenced to the residual solvent peak, $(CD_3)CD_2H)SO$ being set at $\delta$-$_H$ 2.49 and $\delta$-$_C$ 39.5 relative to tetramethylsilane (TMS). Deuterium exchange and COSY experiments were performed in order to confirm proton assignments. Coupling constants, *J*, are reported in Hertz. 2D $^{1}$H–$^{13}$C heteronuclear COSY were recorded for the attribution of $^{13}$C signals. Specific rotations were measured on a Perkin–Elmer Model 241 spectropolarimeter (path length 1 cm), and are given in units of $10^{-1}$ deg cm$^2$ g$^{-1}$. Elemental analyses were carried out by the Service de Microanalyses du CNRS, Division de Vernaison (France). Thin layer chromatography was performed on precoated aluminum sheets of Silica Gel 60 $F_{254}$ (Merck, Art. 5554), visualization of products being accomplished by UV absorbency followed by charring with 5% ethanolic sulfuric acid and heating. Column chromatography was carried out on Silica Gel 60 (Merck, Art. 9385). All moisture-sensitive reactions were carried out under rigorous anhydrous conditions under an argon atmosphere using oven-dried glassware. Solvents were dried and distilled prior to use and solids were dried over $P_2O_5$ under reduced pressure.

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0045-2068/$ - see front matter © 2010 Elsevier Inc. All rights reserved.
doi:10.1016/j.bioorg.2010.07.003

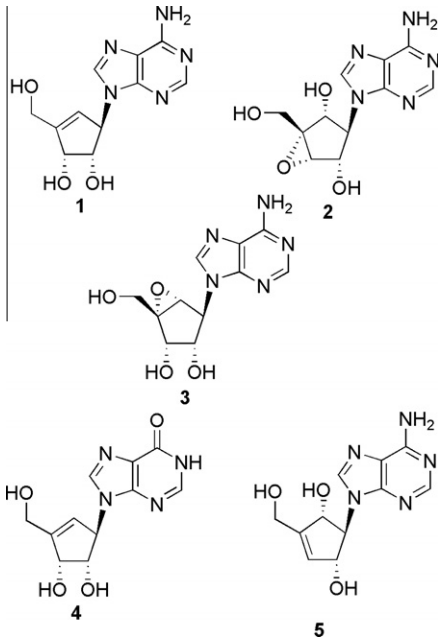


**Fig. 1.** Naturally occurring carbocyclic nucleosides from the neplanocin family.

### 2.2. (−)-9-[(1S,2S,5R)-5-(benzyloxy)-3-[(benzyloxy)methyl]-2-(methoxymethoxy)-3-cyclopenten-1-yl]-6-chloro-9H-purine (**7**)

To a solution of **6** (1.12 g, 2.23 mmol) in dry DMF (22.5 mL) was added successively $K_2CO_3$ (971 mg, 7.03 mmol), 6-chloropurine (776 mg, 5.02 mmol) and 18-crown-6 ether (206 mg, 0.78 mmol). The mixture was heated at 60 °C for 3 h and poured into brine (125 mL) and ethyl acetate (125 mL). The aqueous phase was extracted with ethyl acetate (3 × 125 mL) and the combined organic phases were washed with brine (2 × 125 mL), dried ($MgSO_4$), and evaporated under reduced pressure. Purification by column chromatography using diethyl ether/methylene chloride (5/95 to 10/90, v/v) gave **7** as a yellow solid (686 mg, 60% yield): mp 93–94 °C; $R_f$: (diethyl ether) 0.41; UV (EtOH, 96%) $\lambda_{max}$ = 266.0 nm ($\epsilon$ 9000); $[\alpha]_D^{20}$ −23 (c 1.0, $CHCl_3$); $^1$H NMR ($CDCl_3$, 300 MHz) $\delta$ 8.62 (s, 1H), 7.96 (s, 1H), 7.31–7.38 (m, 5H), 7.10–7.13 (m, 3H), 7.01–7.04 (m, 2H), 6.12 (s, 1H), 5.13 (d, $J$ = 6.0 Hz, 1H), 5.02 (d, $J$ = 5.7 Hz, 1H), 4.72 (t, $J$ = 6.0 Hz, 1H), 4.63 (d, $J$ = 12.0 Hz, 1H), 4.59 (d, $J$ = 12.0 Hz, 1H), 4.55 (d, $J$ = 12.0 Hz, 1H), 4.45 (s, 2H), 4.37 (d, $J$ = 12.0 Hz, 1H), 4.19 (d, $J$ = 5.4 Hz, 2H), 3.07 (s, 3H); $^{13}$C NMR ($CDCl_3$, 75 MHz) $\delta$ 151.3, 151.0, 145.8, 143.0, 137.7, 137.3, 132.3, 128.4, 128.2, 127.9, 127.8, 127.8, 127.7, 127.6, 97.0, 82.5, 81.5, 72.8, 72.0, 71.1, 65.9, 55.5; FAB-MS (>0) $m/z$ 507 $[M + H]^+$. HRMS TOF MS $E^+$ for $C_{27}H_{28}ClN_4O_4$: calculated: 507.1799 found: 507.1800. Anal. Calcd for $C_{27}H_{27}ClN_4O_4$. 0.1 $Et_2O$: C, 63.98; H, 5.49, N, 10.99. Found: C, 63.63; H, 5.28; N, 10.97.

### 2.3. (−)-(1S,4R,5R)-4-(benzyloxy)-2-[(benzyloxy)methyl]-5-(6-chloro-9H-purin-9-yl)-2-cyclopenten-1-ol (**8**)

To a solution of **7** (1.5 g, 2.96 mmol) in $CH_2Cl_2$ (59 mL) was added dropwise TFA (14.8 mL). The solution was stirred at room temperature for 28 h, then poured into an aqueous saturated $NaHCO_3$ solution (400 mL) and extracted with $CH_2Cl_2$ (3 × 200 mL). The combined organic layers were dried ($MgSO_4$) and evaporated to dryness. A purification by column chromatography using diethyl ether gave **8** as a white foam (832 mg, 60% yield): $R_f$ (diethyl ether) 0.36; UV (EtOH, 96%) $\lambda_{max}$ = 266.0 nm ($\epsilon$ = 7200); $[\alpha]_D^{20}$ −50 (c 1.0, $CHCl_3$); $^1$H NMR ($CDCl_3$, 300 MHz) $\delta$ 8.67 (s, 1H), 8.04 (s, 1H), 7.33–7.39 (m, 5H), 7.21–7.25 (m, 3H), 7.17–7.20 (m, 2H), 6.11 (d, $J$ = 1.2 Hz, 1H), 5.19 (m, 1H), 5.07 (m, 1H), 4.71 (d, $J$ = 12.0 Hz, 1H), 4.65 (d, $J$ = 6.3 Hz, 1H), 4.62 (s, 2H), 4.49 (d, $J$ = 12.0 Hz, 1H), 4.30 (s, 2H), 4.17 (d, $J$ = 4.2 Hz, 1H); $^{13}$C NMR ($CDCl_3$, 75 MHz) $\delta$ 151.3, 151.2, 150.9, 145.4, 143.6, 137.6, 137.6, 131.9, 128.5, 128.3, 128.0, 127.9, 127.8, 126.4, 80.6, 75.9, 73.1, 72.6, 71.7, 66.5; FAB-MS (>0) $m/z$ 463 $[M + H]^+$. HRMS TOF MS $E^+$ for $C_{25}H_{24}ClN_4O_3$: calculated: 463.1537 found: 463.1547. Anal. Calcd for $C_{25}H_{23}ClN_4O_3$. 0.35 $Et_2O$: C, 64.86; H, 5.46; N, 11.46. Found: C, 64.64; H, 5.86; N, 11.35.

### 2.4. (−)-(1S,2R,3R,4S,5R)-4-(benzyloxy)-1-[(benzyloxy)methyl]-3-(6-chloro-9H-purin-9-yl)-6-oxabicyclo[3.1.0]hexan-2-ol (**9**)

To as solution of **8** (158 mg, 0.34 mmol) in $CH_2Cl_2$ (7.2 mL) at 0 °C was added a solution of *m*-CPBA (124 mg, 0.72 mmol) in $CH_2Cl_2$ (3.6 mL). The reaction mixture was stirred at room temperature for 21 h and solvent was evaporated under reduced pressure. The residue was purified by column chromatography using diethyl ether to afford **9** as a white foam (503 mg, 93% yield): $R_f$ (diethyl ether) 0.36; UV (EtOH, 96%) $\lambda_{max}$ = 266.0 nm ($\epsilon$ = 9500); $[\alpha]_D^{20}$ −41 (c 1.0, $CHCl_3$); $^1$H NMR ($CDCl_3$, 300 MHz) $\delta$ 8.44 (s, 1H), 8.07 (s, 1H), 7.27–7.38 (m, 5H), 6.89–6.98 (m, 5H), 5.05 (m, 1H), 5.02 (m, 1H), 4.71 (dd, $J$ = 1.2, 8.1 Hz, 1H), 4.65 (s, 2H), 4.64 (d, $J$ = 12.3 Hz, 1H), 4.37 (t, $J$ = 7.6 Hz, 1H), 4.32 (d, $J$ = 12.2 Hz, 1H), 4.08 (d, $J$ = 11.4 Hz, 1H), 3.85 (d, $J$ = 11.4 Hz, 1H), 3.78 (d, $J$ = 0.9 Hz, 1H); $^{13}$C NMR ($CDCl_3$, 75 MHz) $\delta$ 151.2, 150.7, 150.1, 146.9, 137.4, 136.7, 131.4, 128.5, 127.9, 127.9, 127.8, 127.6, 75.1, 73.8, 71.8, 70.5, 66.9, 56.4, 63.5, 57.6; FAB-MS (>0) $m/z$ 479 $[M+H]^+$. Anal. Calcd for $C_{25}H_{23}ClN_4O_4$. 0.35 $Et_2O$: C, 62.80; N, 11.10; Found: C, 62.97; N, 10.72.

### 2.5. (−)-(1S,2S,3R,4R,5R)-3-(6-amino-9H-purin-9-yl)-4-(benzyloxy)-1-[(benzyloxy)methyl]-6-oxabicyclo[3.1.0]hexan-2-ol (**10**)

A solution of **9** (716 mg, 13.9 mmol) in saturated methanolic ammonia (35 mL) in a steel bomb was heated at 70 °C for 14 h. The reaction mixture was cooled and the solvent removed under reduced pressure. The residue was purified by column chromatography using ethyl acetate/methanol (96:4; v/v) to afford **10** as a white foam (375 mg, 50% yield); $R_f$ (ethyl acetate/methanol, 9:1) 0.37; UV (EtOH, 96%) $\lambda_{max}$ = 260.0 nm ($\epsilon$ = 12,500); $[\alpha]_D^{20}$ −37 (c 1.0, DMSO); $^1$H NMR ($CDCl_3$, 300 MHz) $\delta$ 8.13 (s, 1H), 7.69 (s, 1H), 7.38–7.15 (m, 10H), 5.65 (br s, 2H), 5.43 (br s, 1H), 4.87 (d, $J$ = 7.2 Hz, 1H), 4.72 (d, $J$ = 12.0 Hz, 1H), 4.58–4.67 (m, 3H), 4.54 (d, $J$ = 12.0 Hz, 1H), 4.29 (t, $J$ = 7.3 Hz, 1H), 4.03 (d, $J$ = 11.4 Hz, 1H), 3.83 (d, $J$ = 11.1 Hz, 1H), 3.73 (s, 1H); $^{13}$C NMR ($CDCl_3$, 75 MHz) $\delta$ 155.1, 151.9, 149.5, 141.4, 137.5, 136.9, 128.5, 128.3, 127.9, 127.9, 127.7, 119.9, 76.4, 73.8, 71.5, 70.7, 66.9, 64.2, 63.4, 57.6; FAB-MS (>0) $m/z$ 460 $[M + H]^+$. HRMS TOF MS $E^+$ for $C_{25}H_{26}N_5O_4$: calculated: 460.1985 found: 460.1986. Anal. Calcd for $C_{25}H_{25}N_5O_4$. 0.75 MeOH: C, 63.96; H, 5.84; N, 14.48; Found: C, 64.17; H, 6.24; N, 14.87.

### 2.6. (−)-(1S,2S,3S,4R,5R)-3-(6-amino-9H-purin-9-yl)-1-(hydroxymethyl)-6-oxabicyclo[3.1.0]hexane-2,4-diol (**2**)

To a solution of **10** (375 mg, 0.82 mmol) in methanol (9.1 mL) was added ammonium formate (772 mg, 12.3 mmol) and 10% Pd/C (390 mg). The mixture was stirred at reflux for 18 h and filtrated through a pad of Celite before evaporation of solvent. The residue was purified by column chromatography using ethyl acetate/methanol (3:1, *v/v*) to give **2** as a white solid (44 mg, 20% yield); mp 270 °C; $R_f$ (iPrOH, $H_2O$, $NH_4OH$ 5:1:1) 0.61; UV ($H_2O$) $\lambda_{max}$ = 260.0 nm; $[\alpha]_D^{20}$ −3.0 (c 1.0, DMSO); $^1$H NMR (DMSO-$d_6$, 300 MHz): $\delta$ 8.05 (br s, 1H), 8.02 (s, 1H) 7.21 (s, 2H, $NH_2$), 5.50

(br s, 2H), 4.93 (br s, 1H), 4.70 (m, 1H), 4.64 (m, 1H), 4.06 (t, $J$ = 7.8 Hz, 1H), 3.88 (dd, $J$ = 4.2 Hz, 12.3 Hz, 1H), 3.52 (m, 2H); $^{13}$C NMR (DMSO-$d_6$, 75 MHz) $\delta$ 156.1, 152.0, 149.8, 141.9, 119.7, 69.74, 69.7, 64.7, 64.69, 59.3, 57.6; FAB-MS (>0) $m/z$ 280 $[M+H]^+$. HRMS TOF MS $E^+$ for $C_{11}H_{14}N_5O_4$: calculated: 280.1046 found: 280.1041.

## 3. Results and discussion

The synthesis of (−)-neplanocin B was achieved from the preparation of a suitable carbocyclic precursor (**6**) bearing appropriate protective groups (Fig. 1). Such an intermediate can be obtained in several steps from commercially available D-γ-ribonolactone [8]. Thus, treatment of **6** with $K_2CO_3$, 6-chloropurine and a catalytic amount of 18-crown-6 ether in dry DMF at 60 °C afforded only the N-9 alkylated compound **7**. The structure of **7** was fully established from $^1$H, $^{13}$C NMR and UV spectra. The selective removal of MOM group was carried out with diluted TFA in methylene chloride. Compound **8** was treated with *m*-chloroperbenzoic acid to give stereoselectively the epoxy derivative **9** in 93% yield. The epoxidation reaction was directed by the allylic alcohol and gave a single diastereoisomer [12]. Treatment with methanolic ammonia afforded the desired carbocyclic analog **10** (see Fig. 2).

Once the synthesis of the carbocyclic intermediate **10** was achieved, removal of the benzyl groups was envisioned via a catalytic hydrogenation [13]. Different reaction conditions were attempted (Table 1).

Catalytic hydrogenation of compound **10** in the presence of Pd/C was not as effective as previously reported [11]. The influence of pressure, temperature, solvent and the catalyst on the hydrogenation reaction have been investigated. Whatever the conditions used, no debenzylation occured. Nevertheless, compound **10** was finally deprotected (entry 9) by treatment with ammonium formate and Pd/C in MeOH to afford the target molecule (−)-neplanocin B (**2**). The $^1$H NMR and $^{13}$C NMR were identical with those previously reported for the unnatural enantiomer. The optical rotation value was in agreement with the one previously reported [2].

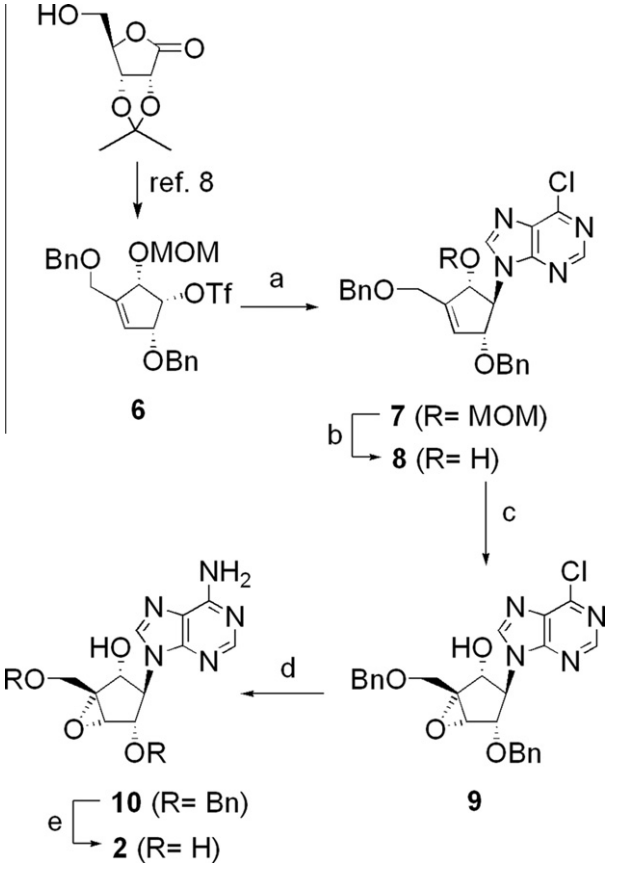


**Fig. 2.** Reagents and conditions: (a) $K_2CO_3$, 18-crown-6 ether, DMF, 60 °C; (b) TFA (18%)/$CH_2Cl_2$, r.t.; (c) m-CPBA/$CH_2Cl_2$, r.t.; (d) $NH_3$/MeOH, 70 °C; (e) see Table 1.

**Table 1**
Catalytic hydrogenation conditions.

| Entry | Conditions | Compound **2** (yield) |
|---|---|---|
| 1 | $H_2$, 5%Pd/C, MeOH, r.t., 1 atm | No deprotection |
| 2 | $H_2$, 10%Pd/C, MeOH, r.t., 1 atm | No deprotection |
| 3 | $H_2$, 5%Pd/C, $HCO_2H$, MeOH, r.t., 3 atm | No deprotection |
| 4 | $H_2$, 5% Pd black, $HCO_2H$, MeOH, r.t., 3 atm | No deprotection |
| 5 | $H_2$, $Pd(OH)_2$, EtOAc/MeOH, r.t., 1 atm | No deprotection |
| 6 | $H_2$, Ni Raney, EtOH, r.t., 1 atm. | No deprotection |
| 7 | $HCO_2NH_4$ (28 eq.), $Pd(OH)_2$, EtOH, reflux | Trace |
| 8 | $HCO_2NH_4$ (5 eq.), 10% Pd/C, MeOH, reflux | Trace |
| 9 | $HCO_2NH_4$ (10 eq.), 10% Pd/C, MeOH, reflux | 20% |

## 4. Conclusion

In summary, the synthesis of the natural isomer (−)-neplanocin B (**2**) was carried out from 2,3-*O*-isopropylidene-D-1,4-ribonolactone. However, when the carbocyclic nucleoside **2** was evaluated against several DNA and RNA viruses in cell culture experiments, compound **2** did not showed any antiviral activity nor cytotoxicity at the highest concentration tested (usually 100 μM).

## Acknowledgments

One of us N.H. is particularly grateful to the Ministère de l'Education Nationale, de la Recherche et de la Technologie (France) for a doctoral fellowship. We gratefully acknowledge Pr. J. Balzarini (Laboratory of virology and chemotherapy, Katholieke Universiteit, Leuven) for the biological results.

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