Synthetic Strategy Toward 1,9-Functionalized Pyrido[2,3-*d*:6,5-*d*′]dipyrimidine-2,4,6,8-tetrones

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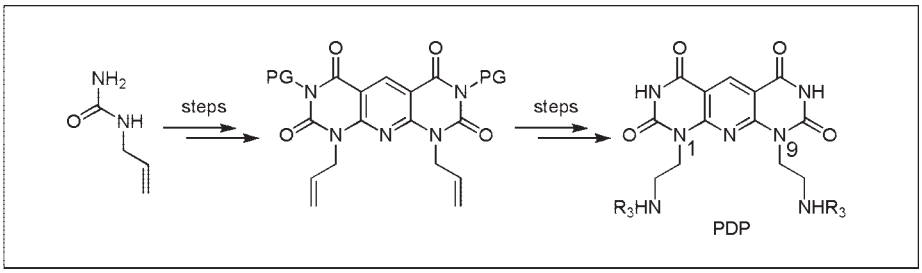

A synthetic approach involving a solubilizing protecting group strategy is described to generate pyrido[2,3-*d*:6,5-*d*′]dipyrimidine-2,4,6,8-tetrones functionalized at positions 1 and 9 with alkylamine substitutents.

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INTRODUCTION

Pyrido[2,3-*d*:6,5-*d*′]dipyrimidine-2,4,6,8-tetrones (PDP) **4** illustrated in Scheme 1 are an interesting class of biannulated pyridine compounds. Derivatives of **4** have been shown to exhibit antibacterial and antiviral properties [1] as well as NAD-type redox catalytic activity [2]. Solid-state ribbons have also been described from the self-assembly of an amine derivative of **4**, which demonstrates the potential of these heterocyclic compounds in supramolecular synthesis applications [3].

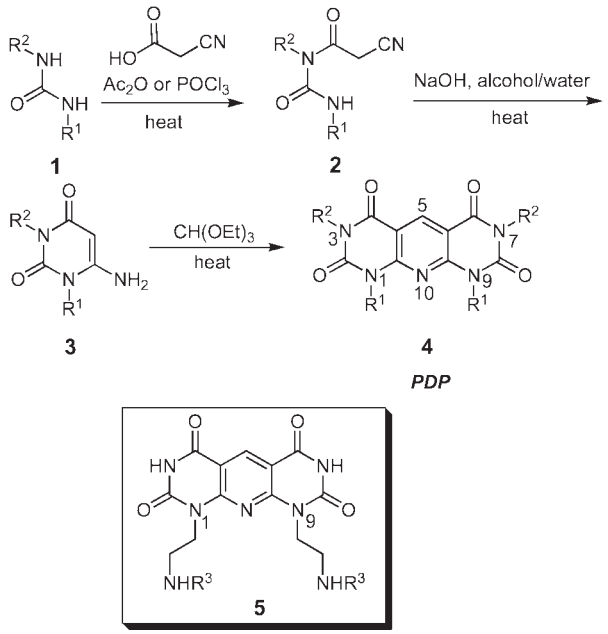
A typical synthetic scheme for these nitrogen-containing tricycles **4** is shown in Scheme 1 and begins with an often low-yielding condensation reaction of functionalized urea **1** with cyanoacetic acid under thermal conditions, followed by a base induced cyclization of **2** to generate 6-amino-uracil **3** [4]. A second condensation reaction between **3** and an electrophile such as triethyl orthoformate [3] (or an aromatic aldehyde [5a], or dimethyl sulfoxide [5b]), generates the target compound **4** (Scheme 1).

As part of our research program on the hierarchical self-assembly of a DNA-based G^C hybrid molecule into rosette nanotubes in aqueous media [6], we required PDP substrates **5** shown in Scheme 1, which were selectively functionalized at positions 1 and 9 with a variety of NHR^3 substituents such as L-Lysine. Given the harsh conditions required to construct the intermediate 6-aminouracil **3** and **4** (Scheme 1), we reasoned that it would be best to introduce these amine substituents (some of which would be incompatible with the acidic/basic conditions) after the PDP core **4** was constructed. A convergent strategy was therefore proposed as shown in Scheme 2, whereby the desired PDPs **5** would be derived from a common intermediate aldehyde **6** *via* a reductive amination reaction with the requisite amines. Aldehyde **6** would originate from an oxidative cleavage of di-alkene **7**, which in turn would be prepared from urea **8** containing a robust allyl group. Herein we describe the synthesis of **5** in detail, which required overcoming some challenging solubility constraints.

RESULTS AND DISCUSSION

The synthesis of **5** commenced according to Scheme 3, by treating commercially available *N*-allyl urea (**8**) with cyanoacetic acid and acetic anhydride at 85°C to provide the condensed adduct **9**. Cyclization of **9** in the presence of 10% sodium hydroxide in a 2:1 mixture of water and ethanol at 85°C generated uracil **10** with a moderate 57% yield [4c]. Finally, treatment of **10** with triethyl orthoformate in acetic acid under refluxing conditions for 2 h provided the desired allyl functionalized PDP adduct **7** in 80% yield [3]. Unfortunately, this adduct was found to be insoluble in many common organic solvents such as dichloromethane, ether, tetrahydrofuran, acetone, and methanol and could only be maintained in solution using dimethyl sulfoxide, refluxing *N*,*N*-dimethylformamide (DMF), or pyridine. This restricted our solvent choices for the subsequent two-step dihydroxylation/sodium periodate oxidative

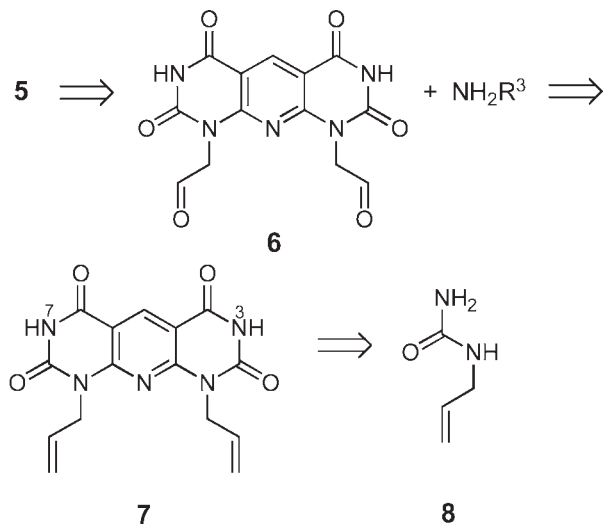
Scheme 1



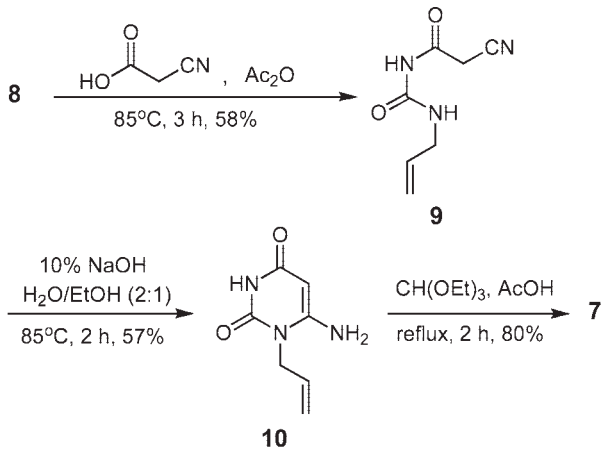
cleavage reaction for the preparation of **6**. Diol formation was therefore attempted using catalytic osmium tetroxide and an excess of 4-methyl-morpholine *N*-oxide (NMO) co-oxidant in a mixture of dimethyl sulfoxide and water, but only starting material was recovered.

To confer the required solubility to the PDP core **7**, various protecting groups (PGs) were installed on the nitrogen atoms at positions 3 and 7. The benzoyl (Bz) group was initially chosen and although it did afford the required solubility to conduct the oxidative cleavage reaction as illustrated in Scheme 4, the following reductive amination step of aldehyde **12** with protected lysine

Scheme 2

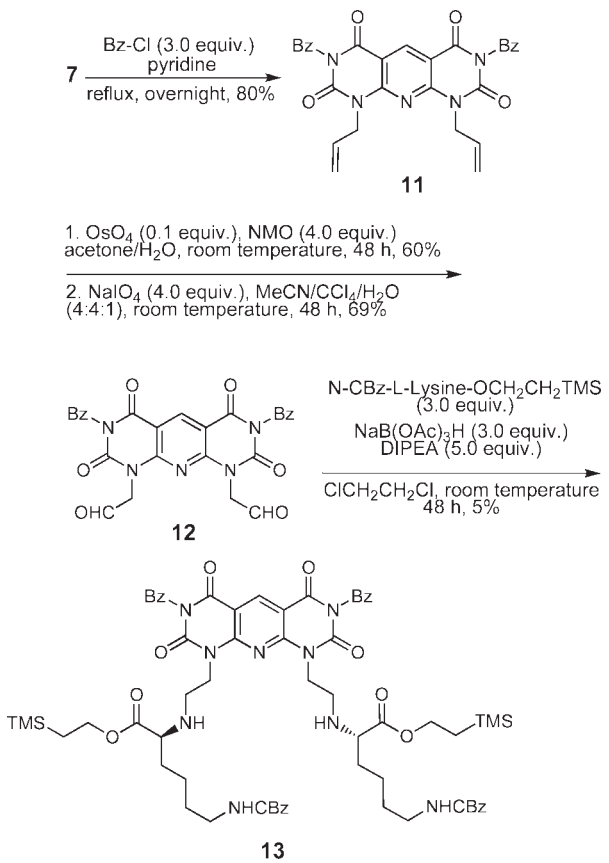


Scheme 3

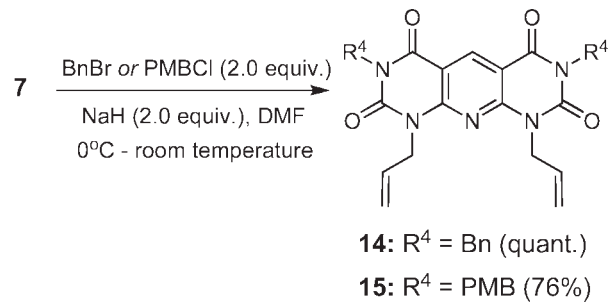


in the presence of sodium triacetoxyborohydride and *N*,*N*-diisopropylethylamine was very low yielding (5%). This was attributed to the removal of the Bz group(s) on **12** under the basic conditions, which caused the resulting material to precipitate out of the solution prior to undergoing the coupling reaction.

Scheme 4



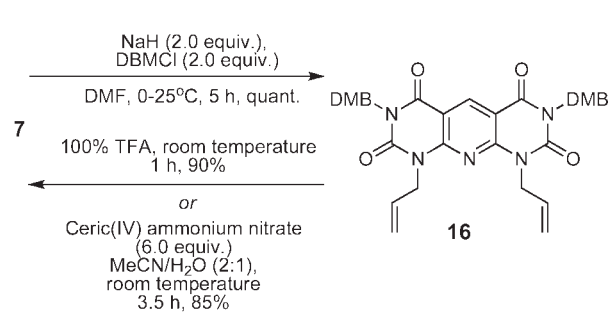
Scheme 5



Base-stable benzyl (Bn) and *p*-methoxy benzyl (PMB) PGs were subsequently installed on the PDP core **7** under standard conditions (Scheme 5). To ensure that these groups could also be easily removed, the deprotection of **14** and **15** was attempted under radical (2,3-dichloro-5,6-dicyano-*p*-benzoquinone, wet dichloromethane, room temperature, 1 day), acidic (98% trifluoroacetic acid (TFA), 2 days) and hydrogenation (H_2/Pd(C), acetic acid, 20 psi) conditions. Surprisingly, only starting material was recovered in all cases, except for the latter, in which the allyl group was reduced. Only in the presence of ceric (IV) ammonium nitrate in a 2:1 mixture of acetonitrile-water at room temperature for 14 h was the desired deprotected product **7** isolated, although in a mere 50% and 68% yield from **14** and **15**, respectively.

As a result of these unsatisfactory deprotection yields and limited conditions in which to remove the PMB/Bn groups, a fourth *N*-PG, DMB (di-(4-methoxyphenyl)-methyl) was also examined. Although it is less common than other *N*-PGs [7], examples of its applications can be found in amino acid [8a], allylic amine [8b], β-lactam [8c], and urethane/uridine [8d] synthesis to name a few. Gratifyingly, installation of DMB on compound **7** proceeded smoothly in the presence of sodium hydride and di-(4-methoxyphenyl)methyl chloride [9] in *N*,*N*-DMF to afford the bis-protected product **16** in quantitative yield (Scheme 6). Solubility tests also confirmed that **16** was soluble in a range of solvents such as acetone, dichloromethane, dimethylsulfoxide, *N*,*N*-DMF and ether with heating. Furthermore, the DMB group was easily removed under both acidic (100% TFA, room temperature, 1 h) and oxidative conditions (ceric (IV) ammonium nitrate, acetonitrile:water (2:1), room temperature, 3.5 h) in excellent yields of 90% and 85%, respectively (Scheme 6).

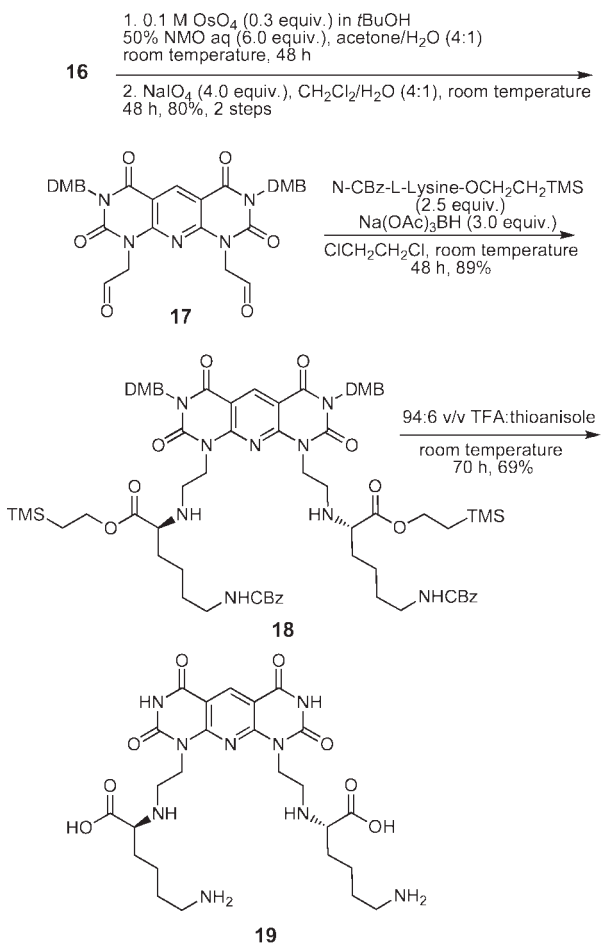
Scheme 6



With this PG strategy in hand, the synthesis was continued from compound **16**. Oxidative cleavage in the presence of catalytic osmium tetroxide and NMO followed by treatment with sodium periodate provided aldehyde **17** in 80% yield for the two steps. Double reductive amination of **17** with L-Lysine furnished the coupled adduct **18** in 89% yield. Finally, treatment of **18** with a 94:6 *v*/*v* TFA:thioanisole solution ensured complete removal of all six PGs (*i.e.*, benzyloxycarbonyl (Cbz), trimethylsilyl (TMS) and DMB) and the desired water-soluble PDP adduct **19** was obtained in 69% yield (~94% yield per PG).

In conclusion, a convergent synthetic strategy has been developed to access PDP substrates functionalized at positions 1 and 9 with amines such as L-lysine (*i.e.*,

Scheme 7



19) from the common intermediate aldehyde **17**. Central to this strategy is the protection of the nitrogen atoms at positions 3 and 7 on compound **7** with the base-stable DMB group. This PG provides the necessary solubility to the parent compound for the alkene cleavage and double reductive amination reactions and yet is sufficiently labile on the PDP core under both oxidative and acidic conditions for its eventual removal (Scheme 7).

EXPERIMENTAL

General. All commercial reagents and solvents were used without further purification except for dichloroethane which was purified on an MBraun solvent purification system. Reactions were monitored by TLC analysis using silica-coated TLC plates (Merck F 60254) and visualized under UV light. Flash chromatography was carried out using Merck 60 (0.040–0.063 mm) or Merck 60 (0.063–0.2 mm) silica. ^{1}H and ^{13}C NMR spectra were recorded on Varian Inova NMR spectrometers (300, 400, 500, or 600 MHz) with the solvent as the internal reference. The NMR data is presented as follows: chemical shift, multiplicity, coupling constant, integration. The mass spectra were performed at the Mass Spectrometry Laboratory at the Department of Chemistry, University of Alberta, or at the Analytical Laboratory of The National Institute for Nanotechnology, University of Alberta.

N-(Allylcarbamoyl)-2-cyanoacetamide (9). A mixture of allylurea (104.8 g, 1.046 mol), cyanoacetic acid (89.03 g, 1.046 mol) and acetic anhydride (197.4 mL, 2.092 mol) was heated for 3 h at 85°C. On cooling to room temperature, ether (530 mL) was added and the mixture was placed in an ice-bath for 2 h. The resulting solid was isolated, washed with ether and dried on the high vacuum to yield **9** as a white powder ($C_7H_9N_3O_2$, 98 g, 58%). R_f = 0.6 (SiO_2, 75% ethyl acetate/hexane); mp 140.1–141.9°C; ^{1}H NMR (300 MHz, DMSO-d_6) δ (ppm): 10.56 (brs, 1H), 8.03 (brs, 1H), 5.83 (ddt, J = 17.1, 10.2, 5.1 Hz, 1H), 5.16–5.05 (m, 2H), 3.90 (s, 2H), 3.78 (ddd, J = 5.1, 2.4, 1.5 Hz, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 165.0, 152.2, 134.9, 115.2, 115.0, 41.3, 26.7; MS: m/z 168.40 (M^+ + 1). *Anal*. Calcd. for $C_7H_9N_3O_2$: C, 50.29; H, 5.43; N, 25.14. Found: C, 49.90, H, 5.35, N, 24.83.

1-Allyl-6-aminopyrimidine-2,4(1H,3H)-dione (10). A mixture of **9** (91 g, 0.54 mol) in water (248 mL) and ethanol (124 mL) was warmed to 85°C and slowly treated with 10% aqueous sodium hydroxide until a pH of 10 was obtained and the starting material was fully dissolved. After 10 min, the desired product **10** began to precipitate out of solution. The reaction mixture was stirred for an additional 2 h at 85°C and then treated with 1 *N* hydrochloric acid until the solution was slightly acidic. On cooling to room temperature, the resulting solid was isolated and dried on the high vaccum to furnish **10** as a white powder ($C_7H_9N_3O_2$, 51.5 g, 57%). R_f = 0.6 (SiO_2, 20% methanol/dichloromethane); mp 271.2–273.1°C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.38 (s, 1H), 6.71 (s, 2H), 5.79 (ddt, J = 17.2, 10.2, 4.5 Hz, 1H), 5.12–5.03 (m, 2H), 4.55 (s, 1H), 4.39–4.38 (m, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 162.5, 155.7, 151.0, 132.4, 115.7, 75.2, 42.5; HRMS: m/z 167.0699 (M^+). *Anal*. Calcd. for $C_7H_9N_3O_2$: C, 50.29; H, 5.43; N, 25.14. Found: C, 50.40, H, 5.39, N, 25.15.

1,9-Diallylpyrido[2,3-d:6,5-d′]dipyrimidine-2,4,6,8 (1H, 3H,-7H,9H)-tetrone (7). A mixture of **10** (51.5 g, 0.308 mol), triethyl orthoformate (118 mL, 0.709 mol) and glacial acetic acid (1.2 L, 21 mol) was heated to reflux for 2 h. After cooling to room temperature, the precipitate was washed with glacial acetic acid, isolated by filtration and dried under high vacuum to provide **7** as a white solid ($C_{15}H_{13}N_5O_4$, 41.2 g, 80%). mp = 351.6–353.2°C (decomposes); ^{1}H NMR (500 MHz, DMSO-d_6) δ (ppm): 11.87 (s, 2H), 8.58 (s, 1H), 5.95–5.87 (m, 2H), 5.19–5.12 (m, 4H), 4.72–4.71 (m, 4H); ^{13}C NMR (125 MHz, DMSO-d_6) δ (ppm): 160.1, 154.0, 150.1, 137.6, 132.3, 116.9, 106.7, 43.8; HRMS: m/z 327.0971 (M^+). *Anal*. Calcd. for $C_{15}H_{13}N_5O_4$: C, 55.05; H, 4.00; N, 21.40. Found: C, 55.11, H, 4.03, N, 21.53.

1,9-Diallyl-3,7-(bis(4-methoxyphenyl)methyl)pyrido[2,3-d:6,5-d′] dipyrimidine-2,4,6,8(1H,3H,7H,9H)-tetrone (16). Compound **7** (1.3 g, 3.8 mmol) was slowly added to a mixture of NaH (183 mg, 7.63 mmol) in *N*,*N*-DMF (26 mL) at 0°C. After stirring for 1 h, di-(4-methoxyphenyl)methyl chloride (2.0 g, 7.6 mmol) was added and the reaction was slowly warmed to room temperature and stirred for 5 h. After re-cooling to 0°C, saturated aqueous ammonium chloride (50 mL) was added and the product was extracted with dichloromethane (3×). The combined organic phases were washed with water, dried over anhydrous sodium sulfate, filtered and concentrated *in vacuo* to provide **16** as a white foam ($C_{45}H_{41}N_5O_8$, 3.09 g, quantitative yield). R_f = 0.8 (50% ethyl acetate/hexane). ^{1}H NMR (600 MHz, CD_2Cl_2) δ (ppm): 9.06 (s, 1H), 7.36 (s, 2H), 7.33–7.30 (m, 8H), 6.87–6.86 (m, 8H), 5.95 (ddt, J = 22, 10.8, 5.4 Hz, 2H), 5.24–5.20 (m, 4H), 4.88 (d, J = 5.5 Hz, 4H), 3.80 (s, 12H); ^{13}C NMR (125 MHz, CD_2Cl_2) δ (ppm): 160.2, 159.3, 153.7, 150.6, 141.9, 132.0, 130.7, 130.3, 118.0, 113.7, 107.3, 59.9, 55.6, 45.7; MS: m/z 752.6 ((M^+ + 1)-C_2H_3). *Anal*. Calcd. for $C_{45}H_{41}N_5O_8$: C, 69.31; H, 5.30; N, 8.98. Found: C, 69.68, H, 5.53, N, 8.74.

1,9-Diacetaldehyde-3,7-(bis(4-methoxyphenyl)methyl)pyrido [2,3-d:6,5-d′]dipyrimidine-2,4,6,8(1H,3H,7H,9H)-tetrone (17). A solution of **16** (1 g, 1.3 mmol) in acetone/water (4:1, 50 mL) was treated with 50% aqueous *N*-methylmorpholine *N*-oxide (1.9 mL, 7.8 mmol) at room temperature. After stirring for 5 min, OsO_4 (4 mL, 0.1*M* solution in *t*-BuOH, 0.4 mmol) was added over a period of 5 min and the resulting solution was stirred at room temperature for 48 h. Sodium sulfite (∼500 mg) was then added to quench the reaction and stirring was continued for an additional 1 h. The solvent was subsequently removed under reduced pressure, water was added and the product was extracted with dichloromethane (3×). The combined organic phases were washed with brine, dried over anhydrous sodium sulfate, filtered and concentrated *in vacuo* to provide 1.1 g of crude tetrol. R_f = 0.3 (5% methanol/dichloromethane). A solution of the tetrol (1.1 g, 1.3 mmol) and sodium periodate (1.11 g, 5.2 mmol) in dichloromethane:-water (4:1, 200 mL) was stirred at room temperature for 48 h. The organic layer was then separated, washed with water, dried over anhydrous sodium sulfate, filtered, and concentrated. Purification by flash chromatography on silica gel (0–2% methanol in dichloromethane) provided the desired product which was subsequently re-dissolved in dichloromethane and precipitated with hexane, filtered and dried under reduced

pressure to provide **17** ($C_{43}H_{37}N_5O_{10}$, 812 mg, 80%) as a white solid. $R_f = 0.2$ (90% ethyl acetate/hexane); mp 172.8–173.9°C; ^{1}H NMR (500 MHz, acetone-d_6) δ (ppm): 9.63 (s, 2H), 8.94 (s, 1H), 7.33–7.30 (m, 10H), 6.87–6.85 (m, 8H), 5.11 (s, 4H), 3.77 (s, 12H); ^{13}C NMR (125 MHz, acetone-d_6) δ (ppm): 196.8, 161.1, 160.5, 154.8, 151.9, 142.3, 131.8, 131.4, 114.8, 108.6, 60.8, 56.1, 53.4; MS: *m/z* 784.6 ($M^+ + 1$). *Anal*. Calcd. for $C_{43}H_{37}N_5O_{10}$: C, 65.89; H, 4.76; N, 8.94. Found: C, 65.98, H, 4.93, N, 8.60.

1,9-Di((S)-2-(trimethylsilyl)ethyl-2-amino-6-(benzyloxy-carbonylamino)hexanoate)-3,7-(bis(4-methoxyphenyl)methyl)pyrido[2,3-d:6,5-d′]dipyrimidine-2,4,6,8(1H,3H,7H,9H)-tetrone (18). A solution of **17** (200 mg, 0.26 mmol) in dichloroethane (20 mL) was treated with *N*-Cbz-L-Lysine-OCH_2CH_2TMS (250 mg, 0.65 mmol) at room temperature. After stirring for 15 min, $Na(AcO)_3BH$ (165 mg, 0.78 mmol) was added and stirring was continued for an additional 48 h. The reaction was then quenched with water and the product was extracted with dichloromethane (2×). The combined organic layers were washed successively with 10% aqueous citric acid, water, 5% aqueous sodium bicarbonate and brine, dried over anhydrous sodium sulfate. Concentration under reduced pressure followed by purification by silica gel preparative thin layer chromatography (90% ethyl acetate/hexane) yielded compound **18** as white foam ($C_{81}H_{101}N_9O_{16}Si_2$, 346 mg, 89%). $R_f = 0.8$ (90% ethyl acetate/hexane); ^{1}H NMR (600 MHz, CD_2Cl_2) δ (ppm): 9.02 (s, 1H), 7.34–7.29 (m, 20H), 6.85–6.84 (m, 8H), 5.03 (s, 4H), 4.98 (s, 2H), 4.35 (dt, $J = 13.2$, 6.6 Hz, 4H), 4.16–4.13 (m, 4H), 3.77 (s, 12H), 3.15 (t, $J = 6.6$ Hz, 2H), 3.06 (dt, $J = 13.2$, 6.6 Hz, 4H), 2.97 (dt, $J = 13.0$, 6.0 Hz, 2H), 2.80–2.76 (m, 2H), 2.0 (broad s, 2H), 1.60–1.25 (m, 12H), 0.96–0.93 (m, 4H), 0.02 (s, 18H) ^{13}C NMR (150 MHz, CD_2Cl_2) δ (ppm): 175.8, 160.4, 159.6 , 156.8, 154.2, 151.4, 141.9, 137.7, 131.0, 130.9, 130.5, 128.9, 128.5, 113.9, 107.5, 66.8, 63.4, 61.7, 60.0, 55.8, 46.1, 43.6, 41.4, 33.6, 30.2, 23.6, 17.9, −1.2; MS: *m/z* 1512.8 ($M^+ + 1$). *Anal*. Calcd. for $C_{81}H_{101}N_9O_{16}Si_2$: C, 64.31; H, 6.73; N, 8.33. Found: C, 64.05, H, 6.78, N, 7.93.

1,9-Di((S)-2,6-diaminohexanoic acid)pyrido[2,3-d:6,5-d′]dipyrimidine-2,4,6,8 (1H,3H,7H,9H)-tetrone (19). A 94:6 *v/v* solution of TFA:thioanisole (10 mL) was added to **18** (70 mg, 0.046 mmol) at room temperature. After stirring for 70 h, ether (50 mL) was added and the resulting precipitate was filtered, washed with ether and dried under vacuum to provide **19** as a white solid (31 mg, 69%). ^{1}H NMR (500 MHz, D_2O) δ (ppm): 8.93 (s, 1H), 4.69–4.65 (m, 4H), 3.82 (t, $J = 6.5$ Hz, 2H), 3.58–3.44 (m, 4H), 3.01 (t, $J = 7.5$ Hz, 4H), 2.02–1.90 (m, 4H), 1.75–1.69 (m, 4H), 1.57–1.45 (m, 4H); ^{13}C NMR (150 MHz, D_2O) δ (ppm): 173.1, 162.1, 154.8, 152.1, 140.2, 108.0, 62.6, 44.5, 39.3, 39.0, 29.6, 26.7, 21.8; HRMS: *m/z* 592.2838 (M^+). *Anal*. Calcd. for $C_{25}H_{37}N_9O_8 \cdot 3.2CF_3COOH \cdot 0.5H_2O$: C, 39.06; H, 4.30; N, 13.06. Found: C, 38.66, H, 4.69, N, 13.39.

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