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Synthesis of benzamide-*C*-ribonucleosides by Pd-catalyzed aminocarbonylations

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ABSTRACT

A novel modular, efficient and practical methodology for preparation of *p*- and *m*-substituted benzamide-*C*-ribonucleosides was developed. Reaction of TBS-protected 3- and 4-bromophenyl-*C*-ribonucleosides **1** and **4** with various primary and secondary amines or NH_4Cl under atmospheric pressure of carbon monoxide and in the presence of $Pd(OAc)_2$ and Xantphos lead to the corresponding amides **2a–j** and **5a–j** in high yields. Subsequent deprotection of silylated nucleosides by $Et_3N{\cdot}3HF$ or TFA afforded a series of free *C*-ribonucleosides **3a–j** or **6a–j** in excellent yields (20 examples).

1. Introduction

C-nucleosides are important class of stable analogues of biogenic nucleosides that find useful applications in chemical biology[1] and medicinal chemistry.[2] Many of the current synthetic approaches[3] to these compounds are inefficient and nonselective and even the efficient procedures[4] are not truly general. We have recently developed a new modular approach[5] to C-nucleosides consisting in the synthesis of halogenated aryl-C-nucleosides and their follow-up functionalizations by cross-couplings, aminations, etc. and it is now applied to the synthesis of diverse classes of nucleosides.

Carboxamide derivatives of C-nucleosides are analogues of biogenic nicotinamine ribonucleoside (Fig. 1) and some of them exhibit cytostatic effect.[6] Apart from five-membered heterocyclic carboxamides[7] (tiazofurin and analogues), also simple benzamide-3-*C*-ribonucleoside (BR) was found[8] to be a strong cytostatic agent inducing apoptosis in cancer cells.[9] General mechanism of action of these compounds is their biotransformation to analogues of NAD, which then inhibit IMP dehydrogenase.[10] Syntheses of these C-nucleosides are[7,8] quite difficult and low yielding and do not allow access to large series of derivatives for SAR studies. We have envisaged a new approach to a series of analogues and positional isomers of important cytostatic BR via Pd-catalyzed aminocarbonylation of haloaryl C-nucleosides (Fig. 1).

Palladium-catalyzed aminocarbonylations of aryl halides (or halide equivalents) are a general tool[11] for the introduction of carboxamide function. There is only one single known example[12] of the use of aminocarbonylation in nucleoside chemistry (modification of position 7 of 7-deazapurine nucleoside) in the total synthesis of echiguanine B. Originally, the carbonylation reactions required higher pressure of CO and more reactive aryl iodides. Only recently, Buchwald et al.[13] reported a highly efficient method for direct synthesis of *N*-substituted benzamides from aryl bromides under atmospheric pressure of CO in presence of $Pd(OAc)_2$ and Xantphos. Here we report a general and practical method for the Pd-catalyzed aminocarbonylation of easily available 3-bromophenyl-*C*-ribonucleoside **1** and 4-bromophenyl-*C*-ribonucleoside **4**[14] at atmospheric pressure of CO under mild conditions, to efficiently prepare wide range of diverse *N*-substituted benzenecarboxamide C-nucleosides.

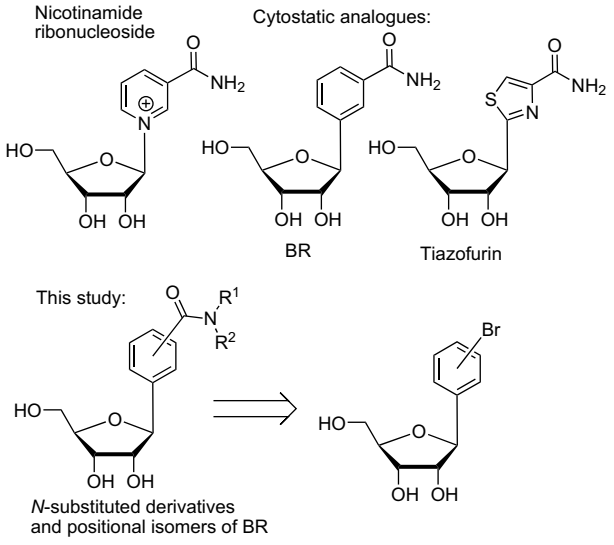

Figure 1. Structure of nicotinamide ribonucleoside, its cytostatic C-nucleoside analogues and rationale for this study.

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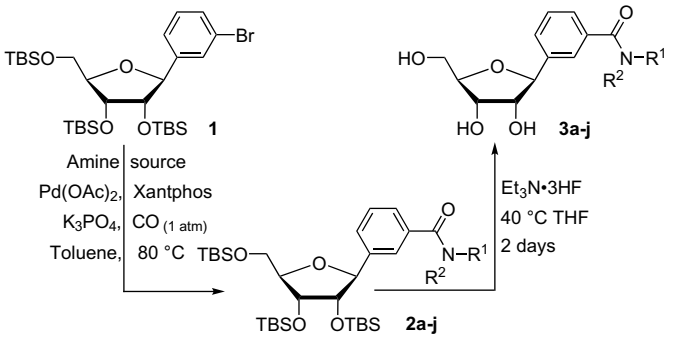
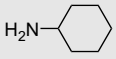
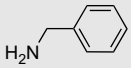
2. Results and discussion

The first target series of compounds were benzene-3-carboxamide-*C*-nucleosides. The aminocarbonylation reactions of 3-bromophenyl-*C*-ribonucleoside **1** with CO (1 atm) and amines were performed under conditions analogous to the Buchwald procedure[13] in presence of 5 mol % of $Pd(OAc)_2$, Xantphos and K_3PO_4 in toluene (Scheme 1, Table 1). In all cases, short reaction times (2–4 h) were sufficient for the complete conversion of starting material. Reactions with secondary amines proceeded very smoothly to give the corresponding amides **2a–d** in excellent yields (Table 1, entry 1–4). Dimethyl amine hydrochloride was used as a solid equivalent of gaseous dimethyl amine in the preparation of *N*,*N*-dimethylcarboxamide **2e** in 84% yield (entry 5). Reactions with primary amines were more sensitive to strictly anhydrous conditions and special precautions (i.e., finely grounded freshly dried K_3PO_4 and extra dry solvent) had to be applied to get good conversions. In this way, cyclopropyl, cyclohexyl and benzyl carboxamides **2f–h** were prepared in good yields of 86%, 84% and 79%, respectively (entries 6–8). Analogous reaction of methylamine hydrochloride gave the *N*-methylcarboxamide **2i** in 88% yield (entry 9).



Scheme 1. Aminocarbonylation of 3-bromophenyl-*C*-ribonucleoside **1**.

Table 1
Aminocarbonylation of 3-bromophenyl-*C*-ribonucleoside **1**[a]

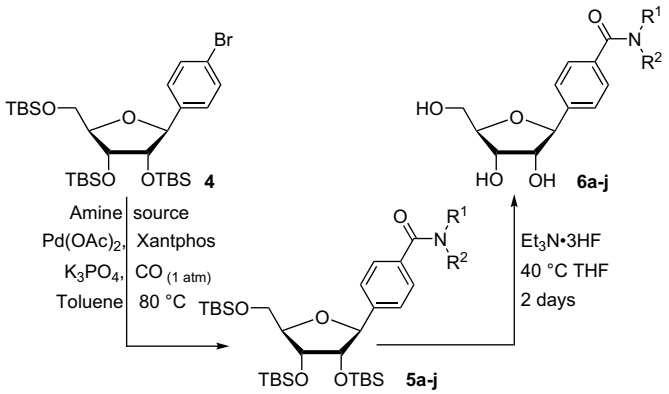
Entry	Amine	Time (h)	Aminocarbonylation product			Deprotect. product (yield)
			R^1	R^2	Yield	
1		4	–$(CH_2)_4$–		**2a** (95%)	**3a** (86%)
2		2	–$(CH_2)_5$–		**2b** (92%)	**3b** (93%)
3		2	–$(CH_2)_2O(CH_2)_2$–		**2c** (89%)	**3c** (89%)
4	HN(*n*-Bu)$_2$	4	*n*-Bu	*n*-Bu	**2d** (95%)	**3d** (83%)
5	$HNMe_2 \cdot HCl$	2	Me	Me	**2e** (84%)[b]	**3e** (95%)
6		3.5	H	*c*-Pr	**2f** (86%)	**3f** (89%)
7		2	H	*c*-Hex	**2g** (84%)	**3g** (91%)
8		4	H	Bn	**2h** (79%)	**3h** (88%)
9	$H_2NMe \cdot HCl$	2	H	Me	**2i** (88%)	**3i** (93%)
10	NH_4Cl	3.5	H	H	**2j** (68%)[c]	**3j** (90%)[d]

[a] Reaction conditions: 5 mol % of $Pd(OAc)_2$, 10 mol % Xantphos, 3 equiv amine, 4 equiv K_3PO_4, toluene (2.5 mL/mmol), 80 °C, CO (1 atm).
[b] $Me_2NH \cdot HCl$ (2 equiv) was used.
[c] Toluene/DMSO 1:1 (3 mL/mmol) was used.
[d] TFA/H_2O 9:1, rt, 1.5 h.

Final deprotection of the silylated nucleosides **2a–i** by treatment with $Et_3N \cdot 3HF$ at 40 °C for 2 days followed by treatment with K_2CO_3 resulted in formation of the desired free nucleosides **3a–i**. Chromatographic purification of the resulting crude nucleosides on silica gel was not practical due to significant losses of the hydrophilic products on the column. Therefore, reverse-phase flash chromatography was used for the final purification to give the free nucleosides **3a–i** in excellent yields (83–95%).

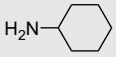
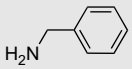
An analogous two-step procedure was used for the preparation of isomeric 4-substituted-benzamide-*C*-ribonucleosides. Also in this case all the amines or amine hydrochlorides generally show good reactivity under the same conditions of the aminocarbonylation to give the carboxamide nucleosides **5a–i** (Scheme 2, Table 2). Desilylation of the TBS-protected nucleosides **5a–i** by treatment with $Et_3N \cdot 3HF$ has been performed analogously to furnish the series of free *C*-ribonucleosides **6a–i** in excellent yields (85–95%, Table 2), after purification by reversed-phase flash chromatography.

In order to prepare unsubstituted benzamide nucleoside **2j** and **5j**, several sources of ammonia have been tested in the reaction of 3-bromophenyl-*C*-ribonucleoside **1** under analogous conditions (Table 3) using higher loading (10 mol %) of $Pd(OAc)_2$. When using LiHMDS (entry 1), no reaction occurred. The use of $HCOONH_4$ resulted in the formation of an inseparable reaction mixture (entry 2), while the use of HMDS furnished desired amide **2j** but in low hardly reproducible yields of 5–20% (entry 3). Finally with $AcONH_4$, the desired 3-(carbamoylphenyl)-*C*-ribonucleoside **2j** was prepared in 76% yield (entry 4). As an alternative, gaseous ammonia was used as a ca. 1:1 mixture with CO and the desired amide **2j** was prepared in an acceptable 62% yield (entry 5). With NH_4Cl as an ammonia surrogate, **2j** was prepared in low yield of 20% but much cleaner mixture was obtained making the isolation of the product more straightforward (entry 6). Revising the former conditions lead us to the use of solvent mixture toluene/DMSO (1:1). This modification increased the yield and enabled us to reduce the reaction time or palladium loading (to 5 mol %) (entries 7 and 8). The optimized conditions then employed 5 mol % of $Pd(OAc)_2$ and a reaction time of 3.5 h to give **2j** in 68% yield. For the aminocarbonylation of 4-bromophenyl-*C*-nucleoside **4** to the corresponding amide **5j**, the use of gaseous ammonia and NH_4OAc gave good yields of the product only after 14–16 h (entries 9 and 10), while the use of



Scheme 2. Aminocarbonylation of 4-bromophenyl-*C*-ribonucleoside **4**.

Table 2
Aminocarbonylation of 4-bromophenyl-*C*-ribonucleoside **4**[a]

Entry	Amine	Time (h)	Aminocarbonylation product			Deprotect. product (yield)
			R^1	R^2	Yield	
1		3	–$(CH_2)_4$–		**5a** (88%)	**6a** (90%)
2		3	–$(CH_2)_5$–		**5b** (92%)	**6b** (91%)
3		2	–$(CH_2)_2O(CH_2)_2$–		**5c** (89%)	**6c** (87%)
4	HN(*n*-Bu)$_2$	3	*n*-Bu	*n*-Bu	**5d** (95%)	**6d** (87%)
5	$HNMe_2 \cdot HCl$	4	Me	Me	**5e** (87%)[b]	**6e** (95%)
6		3	H	*c*-Pr	**5f** (81%)	**6f** (89%)
7		2	H	*c*-Hex	**5g** (87%)	**6g** (85%)
8		3	H	Bn	**5h** (86%)	**6h** (93%)
9	$H_2NMe \cdot HCl$	3.5	H	Me	**5i** (81%)	**6i** (90%)
10	NH_4Cl	3.5	H	H	**5j** (65%)[c]	**6j** (90%)[d]

[a] Reaction conditions: 5 mol % of $Pd(OAc)_2$, 10 mol % Xantphos, 3 equiv amine, 4 equiv K_3PO_4, toluene (2.5 mL/mmol), 80 °C, CO (1 atm).
[b] $Me_2NH \cdot HCl$ (2 equiv) was used.
[c] Toluene/DMSO 1:1 (3 mL/mmol) was used.
[d] TFA/H_2O 9:1, rt, 1.5 h.

NH_4Cl proved to be the best, yielding the nucleoside **5j** in 65% yield within 3.5 h (entry 11).

Attempted deprotection of amides **2j** and **5j** by treatment with $Et_3N \cdot 3HF$ and subsequent treatment with K_2CO_3 resulted in degradation of final products. Therefore, an acidic cleavage by TFA/water (9:1) mixture was used. The reaction was finished within 1.5 h at room temperature and crude nucleosides were purified by reversed-phase chromatography to give free nucleosides **3j** and **6j** in excellent 90% yields.

Table 3
Conversion of nucleosides **1** and **4** to carboxamides **2j** and **5j**[a]

Entry	Starting comp.	Ammonia source	Reaction time (h)	Product (yield)
1	**1**	LiHMDS	24	**2j** N.R.[b]
2	**1**	$HCOONH_4$	24	**2j** N.R.[c]
3	**1**	HMDS	8	**2j** (5–20%)[d]
4	**1**	$AcONH_4$	15	**2j** (76%)[a]
5	**1**	$NH_{3\ (g)}$	16	**2j** (62%)[e]
6	**1**	NH_4Cl	8	**2j** (20%)[a]
7	**1**	NH_4Cl	1.5	**2j** (69%)[f]
8	**1**	NH_4Cl	3.5	**2j** (68%)[f,g]
9	**4**	NH_3	14	**5j** (69%)[e]
10	**4**	$AcONH_4$	16	**5j** (60%)[a]
11	**4**	NH_4Cl	3.5	**5j** (65%)[f,g]

[a] Reaction conditions: 10 mol % of $Pd(OAc)_2$, 20 mol % Xantphos, 3 equiv of ammonia source, 4 equiv K_3PO_4, toluene (2.5 mL/mmol), 80 °C, CO (1 atm).
[b] No base was used.
[c] Inseparable reaction mixture.
[d] Yields were not reproducible.
[e] 1:1 Mixture of $NH_{3(g)}$ and $CO_{(g)}$ was used.
[f] Toluene/DMSO 1:1 (3 mL/mmol) was used.
[g] $Pd(OAc)_2$ (5 mol %), 10 mol % Xantphos.

In conclusion, the presented methodology gives a facile access to primary and secondary benzamide-*C*-ribonucleosides by $Pd(OAc)_2$/Xanthphos catalyzed aminocarbonylation in presence of primary or secondary amines and represents the first systematic study and application of this reaction in nucleoside chemistry. An analogous reaction using NH_4Cl gives unsubstituted benzamides in somewhat lower (but still acceptable) yields. In contrast to know cytostatic unsubstituted benzamide **3j** (BR),[8] the *N*-substituted benzamide-*C*-ribonucleosides **3a–3i** and **6a–6i** did not exert any significant cytostatic activity. However, this powerful and efficient methodology extends the repertoire of reactions for functionalization of C-nucleosides in our modular approach[5] and will now be used for the synthesis of heterocyclic benzamide ribonucleosides with potential biological activity.

3. Experimental

3.1. General

NMR spectra were recorded on 600 MHz (^1H at 600.1 MHz, ^{13}C at 150.9 MHz) and 500 MHz (499.8 and 500.0 MHz for ^1H and 125.7 MHz for ^{13}C) spectrometers. Chemical shifts (in ppm, δ scale) were referenced to TMS as internal standard. Coupling constants (J) are given in hertz. The assignment of carbons was based on C,H-HSQC and C,H-HMBC experiments. Melting points were determined on a Kofler block and are uncorrected. Mass spectra were measured using FAB (ionization by Xe, accelerating voltage 8 kV, glycerol+thioglycelor matrix) or ESI. $Pd(OAc)_2$, $Pd(PPh_3)_4$, Xantphos and all amines were purchased from commercial suppliers and used without any further treatment. All reactions were carried out in flame-dried glassware with magnetic stirring. K_3PO_4 was finely ground and dried (630 °C, 4 Torr, 1 h) prior to each use. Toluene was dried by refluxing with sodium/benzophenone and freshly distilled prior to use. DMSO was stored over molecular sieves under argon.

3.2. General procedure for aminocarbonylation

A flame-dried septum-sealed flask containing 3- or 4-bromophenyl-*C*-ribonucleoside **1** or **4** (1 mmol), $Pd(OAc)_2$ (0.05 mmol), Xantphos (0.1 mmol), K_3PO_4 (4 mmol) [and an amine hydrochloride (3 mmol) in cases when solid hydrochlorides were used] was evacuated and backfilled with $CO_{(g)}$. Then, toluene (2.5 mL) and an amine (3 mmol) [in cases when liquid amines were used] were added via syringe. The reaction mixture was stirred at room temperature for 5 min and then immersed into a preheated oil bath (80 °C) and vigorously stirred until the starting material had been completely consumed (TLC analysis, hexanes/EA 8:2). The reaction mixture was then allowed to cool to room temperature, diluted with Et_2O, filtered through a plug of Celite (eluting with Et_2O) and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel.

*3.2.1. 1β-{3-[(Pyrrolidine-1-yl)carbonyl]phenyl}-1-deoxy-2,3,5-tri-O-(tert-butyldimethylsilyl)-D-ribofuranose (**2a**)*

According to the general procedure, a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.502 mmol, 318 mg), $Pd(OAc)_2$ (5 mol %, 0.025 mmol, 5.6 mg), Xantphos (10 mol %, 0.050 mmol, 29.1 mg), pyrrolidine (1.51 mmol, 3 equiv, 0.13 mL), K_3PO_4 (2.01 mmol, 4 equiv, 427 mg) and toluene (1.3 mL) was heated at 80 °C for 4 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:16 to provide the title compound as a colourless light oil (311 mg, 95%). HRMS (FAB) for $C_{34}H_{64}NO_5Si_3$: [M+H] calculated, 650.4092; found, 650.4108. ^1H NMR (600.1 MHz, $CDCl_3$): −0.43, −0.13, 0.09, 0.11 and 0.12 (5×s, 18H, CH_3Si), 0.80, 0.93 and 0.94

(3×s, 3×9H, $(CH_3)_3C$), 1.81–1.87 and 1.92–1.97 (2×m, 2×2H, CH_2-pyr), 3.37 and 3.40 (2×dt, 2H, J_{gem}=10.6, J_{vic}=6.8, CH_2N-pyr), 3.63 (t, 2H, J_{vic}=7.0, CH_2N-pyr), 3.77 and 3.79 (2×dd, 2H, J_{gem}=11.1, $J_{5',4'}$=3.5, H-5′), 3.85 (dd, 1H, $J_{2',1'}$=7.9, $J_{2',3'}$=4.4, H-2′), 4.04 (td, 1H, $J_{4',5'}$=3.5, $J_{4',3'}$=1.7, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.7, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.9, H-1′), 7.33 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.41 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.7, $J_{4,6}$=1.4, H-4), 7.48 (ddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.7, $J_{6,4}$=1.4, H-6), 7.56 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.7, H-2). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.52, −5.48, −5.31, −4.50, −4.48 and −4.38 (CH_3Si), 17.93, 18.07 and 18.36 ($C(CH_3)_3$), 24.49 (CH_2-pyr), 25.83, 25.87 and 25.98 ($(CH_3)_3C$), 26.37 (CH_2-pyr), 46.06 and 49.59 (CH_2N-pyr), 63.76 (CH_2-5′), 73.95 (CH-3′), 79.48 (CH-2′), 82.49 (CH-1′), 86.07 (CH-4′), 125.41 (CH-2), 126.48 (CH-4), 127.91 (CH-5), 128.33 (CH-6), 137.15 (C-3), 140.82 (C-1), 169.71 (CO). IR spectrum (CCl_4): 1636, 1607, 1587, 1485, 1472, 1463, 1446, 1409, 1390, 1361, 1343, 1308, 1255, 1218, 1163, 1111, 1095, 1081, 940, 837, 703, 671 cm^{-1}.

3.2.2. 1β-{3-[(Piperidine-1-yl)carbonyl]phenyl}-1-deoxy-2,3,5-tri-O-(tert-butyldimethylsilyl)-D-ribofuranose (**2b**)

According to the general procedure, a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.508 mmol, 321 mg), $Pd(OAc)_2$ (5 mol %, 0.025 mmol, 5.7 mg), Xantphos (10 mol %, 0.050 mmol, 29.4 mg), piperidine (1.52 mmol, 3 equiv, 0.15 mL), K_3PO_4 (2.03 mmol, 4 equiv, 432 mg) and toluene (1.3 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:8 to provide the title compound as a colourless light oil (310 mg, 92%). HRMS (FAB) for: $C_{35}H_{66}NO_5Si_3$: [M+H] calculated, 664.4249; found, 664.4235. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.42, −0.13, 0.09, 0.11 and 0.12 (5×s, 18H, CH_3Si), 0.80 and 0.93 (2×s, 27H, $(CH_3)_3C$), 1.45–1.52 and 1.63–1.70 (2×br m, 6H, CH_2-pip), 3.63–3.69 and 3.69–3.77 (2×br m, 2×2H, CH_2N-pip), 3.77 (dd, 1H, J_{gem}=10.8, $J_{5'b,4'}$=4.0, H-5′b), 3.79 (dd, 1H, J_{gem}=10.8, $J_{5'a,4'}$=3.5, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.8, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=4.0, 3.5, $J_{4',3'}$=1.8, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.8, H-3′), 4.79 (d, 1H, $J_{1',2'}$=7.8, H-1′), 7.28 (dt, 1H, $J_{4,5}$=7.6, $J_{4,2}$=$J_{4,6}$=1.7, H-4), 7.33 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.6, H-5), 7.42 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.7, H-2), 7.48 (dt, 1H, $J_{6,5}$=7.6, $J_{6,2}$=$J_{6,4}$=1.7, H-6). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.52, −5.42, −5.31, −4.53, −4.48 and −4.40 (CH_3Si), 17.92, 18.06 and 18.37 ($C(CH_3)_3$), 24.61 and 25.57 (CH_2-pip), 25.83, 25.87 and 26.00 ($(CH_3)_3C$), 26.55 (CH_2-pip), 43.03 and 48.72 (CH_2N-pip), 63.76 (CH_2-5′), 73.91 (CH-3′), 79.46 (CH-2′), 82.50 (CH-1′), 86.01 (CH-4′), 125.29 (CH-2), 126.22 (CH-4), 127.98 and 127.99 (CH-5,6), 136.32 (C-3), 141.03 (C-1), 170.18 (CO). IR spectrum (CCl_4): 1636, 1607, 1587, 1485, 1472, 1463, 1446, 1409, 1390, 1361, 1343, 1308, 1255, 1218, 1163, 1111, 1095, 1081, 940, 837, 703, 671 cm^{-1}.

3.2.3. 1β-{3-[(Morpholine-4-yl)carbonyl]phenyl}-1-deoxy-2,3,5-tri-O-(tert-butyldimethylsilyl)-D-ribofuranose (**2c**)

According to the general procedure, a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.576 mmol, 364 mg), $Pd(OAc)_2$ (5 mol %, 0.029 mmol, 6.5 mg), Xantphos (10 mol %, 0.058 mmol, 33.3 mg), morpholine (1.73 mmol, 3 equiv, 0.15 mL), K_3PO_4 (2.30 mmol, 4 equiv, 489 mg) and toluene (1.4 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:16 to provide the title compound as a colourless light oil (340 mg, 89%). HRMS (FAB) for $C_{34}H_{64}NO_6Si_3$: [M+H] calculated, 666.4042; found, 666.4053. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.45, −0.12, 0.096, 0.098, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.80, 0.93 and 0.94 (3×s, 3×9H, $(CH_3)_3C$), 3.35–3.50, 3.53–3.67 and 3.67–3.90 (3×br m, 8H, H-morph), 3.78 (d, 2H, $J_{5',4'}$=3.6, H-5′), 3.85 (dd, 1H, $J_{2',1'}$=8.0, $J_{2',3'}$=4.4, H-2′), 4.04 (td, 1H, $J_{4',5'}$=3.6, $J_{4',3'}$=1.6, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.6, H-3′), 4.79 (d, 1H, $J_{1',2'}$=8.0, H-1′), 7.29 (dt, 1H, $J_{4,5}$=7.6, $J_{4,2}$=$J_{4,6}$=1.7, H-4), 7.35 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.6, H-5), 7.46 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.7, H-2), 7.51 (dt, 1H, $J_{6,5}$=7.6, $J_{6,2}$=$J_{6,4}$=1.7, H-6). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.50, −5.41, −5.34, −4.50, −4.47 and −4.37 (CH_3Si), 17.93, 18.06 and 18.36 ($C(CH_3)_3$), 25.83, 25.87 and 26.00 ($(CH_3)_3C$), 42.46 and 48.28 (CH_2N-morph), 63.79 (CH_2-5′), 66.90 (CH_2O-morph), 74.04 (CH-3′), 79.52 (CH-2′), 82.33 (CH-1′), 86.26 (CH-4′), 125.59 (CH-2), 126.44 (CH-4), 128.14 (CH-5), 128.51 (CH-6), 135.16 (C-3), 141.37 (C-1), 170.33 (CO). IR spectrum (CCl_4): 1647, 1607, 1588, 1487, 1472, 1462, 1439, 1419, 1389, 1362, 1330, 1300, 1257, 1211, 1153, 1117, 1111, 1098, 1080, 1026, 940, 888, 837, 698, 673 cm^{-1}.

3.2.4. 1β-[3-(Dibutylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2d**)

According to the general procedure a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.237 mmol, 150 mg), $Pd(OAc)_2$ (5 mol %, 0.012 mmol, 2.7 mg), Xantphos (10 mol %, 0.024 mmol, 13.7 mg), dibutylamine (0.712 mmol, 3 equiv, 0.12 mL), K_3PO_4 (0.949 mmol, 4 equiv, 202 mg) and toluene (0.60 mL) was heated at 80 °C for 4 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:6 to provide the title compound as a colourless light oil (158 mg, 95%). HRMS (FAB) for $C_{38}H_{74}NO_5Si_3$: [M+H] calculated, 708.4875; found, 708.4864. ^{1}H NMR (500.0 MHz, $CDCl_3$): −0.42, −0.13, 0.09, 0.11 and 0.12 (5×s, 18H, CH_3Si), 0.78–0.81 (br m, 3H, $CH_3CH_2CH_2CH_2N$), 0.81, 0.93 and 0.93 (3×s, 3×9H, $(CH_3)_3C$), 0.95–0.99 (br m, 3H, $CH_3CH_2CH_2CH_2N$), 1.08–1.16, 1.35–1.43, 1.43–1.50 and 1.59–1.67 (4×br m, 4×2H, $CH_3CH_2CH_2CH_2N$), 3.12–3.20 and 3.40–3.53 (2×br m, 2×2H, $CH_3CH_2CH_2CH_2N$), 3.76 (dd, 1H, J_{gem}=10.9, $J_{5'b,4'}$=4.1, H-5′b), 3.79 (dd, 1H, J_{gem}=10.9, $J_{5'a,4'}$=3.5, H-5′a), 3.84 (dd, 1H, $J_{2',1'}$=7.8, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=4.1, 3.5, $J_{4',3'}$=1.9, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.9, H-3′), 4.79 (d, 1H, $J_{1',2'}$=7.8, H-1′), 7.26 (dt, 1H, $J_{4,5}$=7.6, $J_{4,2}$=$J_{4,6}$=1.7, H-4), 7.32 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.6, H-5), 7.36 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.7, H-2), 7.47 (dt, 1H, $J_{6,5}$=7.6, $J_{6,2}$=$J_{6,4}$=1.7, H-6). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.51, −5.39, −5.27, −4.54, −4.46 and −4.39 (CH_3Si), 13.70 and 13.96 ($CH_3CH_2CH_2CH_2N$), 17.93, 18.07 and 18.37 ($C(CH_3)_3$), 19.80 and 20.34 ($CH_3CH_2CH_2CH_2N$), 25.86, 25.89 and 26.00 ($(CH_3)_3C$), 29.69 and 30.87 ($CH_3CH_2CH_2CH_2N$), 44.45 and 48.83 ($CH_3CH_2CH_2CH_2N$), 63.75 (CH_2-5′), 73.89 (CH-3′), 79.44 (CH-2′), 82.54 (CH-1′), 85.99 (CH-4′), 124.77 (CH-2), 125.80 (CH-4), 127.52 (CH-6), 127.97 (CH-5), 137.25 (C-3), 140.96 (C-1), 171.47 (CO). IR spectrum (CCl_4): 1638, 1607, 1588, 1487, 1471, 1463, 1421, 1389, 1379, 1362, 1308, 1257, 1211, 1152, 1110, 1098, 1082, 940, 837, 701, 673 cm^{-1}.

3.2.5. 1β-[3-(Dimethylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2e**)

According to the general procedure, a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.339 mmol, 214 mg), $Pd(OAc)_2$ (5 mol %, 0.017 mmol, 3.8 mg), Xantphos (10 mol %, 0.034 mmol, 19.6 mg), $Me_2NH{\cdot}HCl$ (0.677 mmol, 2 equiv, 55.2 mg), K_3PO_4 (1.35 mmol, 4 equiv, 288 mg) and toluene (0.85 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:10 to provide the title compound as a colourless light oil (178 mg, 84%). HRMS (FAB) for $C_{32}H_{62}NO_5Si_3$: [M+H] calculated, 624.3936; found, 624.3940. ^{1}H NMR (500.0 MHz, $CDCl_3$): −0.43, −0.12, 0.09, 0.11 and 0.12 (5×s, 18H, CH_3Si), 0.80, 0.931 and 0.935 (3×s, 3×9H, $(CH_3)_3C$), 2.90–3.02 and 3.02–3.14 (2×br s, 2×3H, CH_3N), 3.78 (d, 2H, $J_{5',4'}$=3.7, H-5′), 3.85 (dd, 1H, $J_{2',1'}$=7.8, $J_{2',3'}$=4.4, H-2′), 4.04 (td, 1H, $J_{4',5'}$=3.7, $J_{4',3'}$=1.8, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.8, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.8, H-1′), 7.31 (dt, 1H, $J_{4,5}$=7.6, $J_{4,2}$=$J_{4,6}$=1.7, H-4), 7.34 (td, 1H, $J_{5,4}$=$J_{5,6}$=7.6, $J_{5,2}$=0.6, H-5), 7.46 (tt, 1H, $J_{2,4}$=$J_{2,6}$=1.7, $J_{2,5}$=$J_{2,1'}$=0.6, H-2), 7.48 (dtd, 1H, $J_{6,5}$=7.6, $J_{6,2}$=$J_{6,4}$=1.7, $J_{6,1'}$=0.6, H-6). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.50, −5.47, −5.28, −4.49, −4.46 and −4.37 (CH_3Si), 17.94, 18.08 and 18.37 ($C(CH_3)_3$), 25.84, 25.89 and 26.00 ($(CH_3)_3C$), 35.26 and 39.56 (CH_3N), 63.75 (CH_2-5′), 73.95 (CH-3′), 79.51 (CH-2′), 82.50 (CH-1′),

86.08 (CH-4′), 125.41 (CH-2), 126.43 (CH-4), 128.00 (CH-5), 128.11 (CH-6), 136.24 (C-3), 141.03 (C-1), 171.59 (CO). IR spectrum (CCl_4): 1646, 1608, 1587, 1499, 1472, 1463, 1434, 1406, 1391, 1391, 1362, 1308, 1257, 1218, 1152, 1111, 1093, 1080, 939, 837, 699, 681, 673 cm^{-1}.

3.2.6. 1β-[3-(Cyclopropylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2f**)

According to the general procedure a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.185 mmol, 120 mg), $Pd(OAc)_2$ (5 mol %, 0.009 mmol, 2.1 mg), Xantphos (10 mol %, 0.018 mmol, 11.0 mg), cyclopropylamine (0.57 mmol, 3 equiv, 0.04 mL), K_3PO_4 (0.76 mmol, 4 equiv, 161 mg) and toluene (0.46 mL) was heated at 80 °C for 3.5 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 25:1 to 8:1 to provide the title compound as a white foam (100 mg, 86%). HRMS (ESI) for $C_{33}H_{61}NO_5Si_3Na$: [M+Na] calculated, 658.3756; found, 658.3748. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.53, −0.14, 0.097, 0.102, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.58–0.62 (m, 2H, CH_2-cycloprop), 0.82–0.92 (s, 9H, $(CH_3)_3C$), 0.87 (m, 2H, CH_2-cycloprop), 0.93 and 0.94 (2×s, 2×9H, $(CH_3)_3C$), 2.89 (ttd, 1H, J=7.3, 3.9, 2.5, CH-cycloprop), 3.77–3.82 (m, 3H, H-2′,5′), 4.05 (td, 1H, $J_{4',5'}$=3.2, $J_{4',3'}$=1.5, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.5, $J_{3',4'}$=1.5, H-3′), 4.80 (d, 1H, $J_{1',2'}$=8.3, H-1′), 6.20 (d, 1H, J=2.5, NH), 7.38 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.59 (ddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.3, H-6), 7.62 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.8, H-2), 7.73 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.57, −5.53, −5.38, −4.48 and −4.35 (CH_3Si), 6.67 and 6.82 (CH_2-cycloprop), 17.91, 18.06 and 18.31 ($C(CH_3)_3$), 23.04 (CH-1-cycloprop), 25.80, 25.88 and 25.95 ($(CH_3)_3C$), 63.77 (CH_2-5′), 74.14 (CH-3′), 79.45 (CH-2′), 82.06 (CH-1′), 86.59 (CH-4′), 124.43 (CH-2), 126.89 (CH-4), 128.49 (CH-5), 129.83 (CH-6), 134.15 (C-3), 141.01 (C-1), 168.79 (CO). IR spectrum (CCl_4): 3454, 3346, 3094, 3011, 1678, 1663, 1608, 1588, 1524, 1504, 1472, 1463, 1436, 1425, 1406, 1389, 1362, 1305, 1285, 1257, 1218, 1152, 1112, 1098, 1081, 940, 837, 711, 688, 671 cm^{-1}.

3.2.7. 1β-[3-(Cyclohexylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2g**)

According to the general procedure, a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.392 mmol, 248 mg), $Pd(OAc)_2$ (5 mol %, 0.019 mmol, 4.4 mg), Xantphos (10 mol %, 0.039 mmol, 22.7 mg), cyclohexylamine (1.18 mmol, 3 equiv, 0.14 mL), K_3PO_4 (1.57 mmol, 4 equiv, 333 mg) and toluene (1.0 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 50:1 to 8:1 to provide the title compound as a white foam (224 mg, 84%). HRMS (ESI) for $C_{36}H_{67}NO_5Si_3Na$: [M+Na] calculated, 700.4225; found, 700.4219. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.55, −0.13, 0.10, 0.11, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.79, 0.93 and 0.95 (3×s, 3×9H, $(CH_3)_3C$), 1.15–1.27, 1.38–1.47, 1.63–1.69, 1.71–1.79 and 1.99–2.07 (5×m, 10H, H-2,3,4,5,6-cyclohex), 3.80 (d, 2H, $J_{5',4'}$=3.8, H-5′), 3.81 (dd, 1H, $J_{2',1'}$=8.3, $J_{2',3'}$=4.4, H-2′), 3.98 (tdt, 1H, J=10.8, 8.2, 3.9, H-1-cyclohex), 4.05 (td, 1H, $J_{4',5'}$=3.8, $J_{4',3'}$=1.4, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.4, H-3′), 4.81 (d, 1H, $J_{1',2'}$=8.3, H-1′), 5.92 (d, 1H, J=8.2, NH), 7.39 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.60–7.63 (m, 2H, H-2,6), 7.73 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.56, −5.53, −5.34, −4.48 and −4.36 (CH_3Si), 17.92, 18.07 and 18.33 ($C(CH_3)_3$), 24.98 and 25.58 (CH_2-3,4,5-cyclohex), 25.84, 25.89 and 25.97 ($(CH_3)_3C$), 33.27 and 33.30 (CH_2-2,6-cyclohex), 48.65 (CH-1-cyclohex), 63.77 (CH_2-5′), 74.11 (CH-3′), 79.43 (CH-2′), 82.14 (CH-1′), 86.53 (CH-4′), 124.57 (CH-2), 126.88 (CH-4), 128.49 (CH-5), 129.44 (CH-6), 134.75 (C-3), 140.96 (C-1), 166.51 (CO). IR spectrum (CCl_4): 3389, 3347, 3347, 2930, 2858, 1669, 1608, 1589, 1507, 1480, 1472, 1463, 1451, 1435, 1406, 1389, 1373, 1361, 1351, 1320, 1308, 1257, 1218, 1152, 1113, 1092, 1082, 940, 838, 672 cm^{-1}.

3.2.8. 1β-[3-(Benzylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2h**)

According to the general procedure, a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.783 mmol, 495 mg), $Pd(OAc)_2$ (5 mol %, 0.039 mmol, 8.8 mg), Xantphos (10 mol %, 0.078 mmol, 45.3 mg), benzylamine (2.35 mmol, 3 equiv, 0.25 mL), K_3PO_4 (3.13 mmol, 4 equiv, 665 mg) and toluene (2.1 mL) was heated at 80 °C for 4 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:10 to provide the title compound as a white foam (377 mg, 79%). HRMS (FAB) for $C_{37}H_{64}NO_5Si_3$: [M+H] calculated, 686.4092; found, 686.4087. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.52, −0.14, 0.09 and 0.10 (4×s, 18H, CH_3Si), 0.77, 0.91 and 0.94 (3×s, 3×9H, $(CH_3)_3C$), 3.78 (d, 2H, $J_{5',4'}$=3.5, H-5′), 3.81 (dd, 1H, $J_{2',1'}$=8.3, $J_{2',3'}$=4.4, H-2′), 4.04 (td, 1H, $J_{4',5'}$=3.5, $J_{4',3'}$=1.4, H-4′), 4.11 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.4, H-3′), 4.61 (dd, 1H, J_{gem}=14.7, J_{vic}=5.5, CH_aH_bPh), 4.68 (dd, 1H, J_{gem}=14.7, J_{vic}=5.8, CH_aH_bPh), 6.36 (br dd, 1H, J_{vic}=5.8, 5.5, NH), 4.80 (d, 1H, $J_{1',2'}$=8.3, H-1′), 7.28–7.32 (m, 1H, H-*p*-Ph), 7.33–7.37 (m, 4H, H-*o*,*m*-Ph), 7.40 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.62 (dddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.2, $J_{6,1'}$=0.4, H-6), 7.71 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.8, H-2), 7.78 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.2, H-4). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.56, −5.53, −5.43, −5.34, −4.50 and −4.35 (CH_3Si), 17.89, 18.05 and 18.30 ($C(CH_3)_3$), 25.78, 25.88 and 25.94 ($(CH_3)_3C$), 44.12 (CH_2Ph), 63.76 (CH_2-5′), 74.12 (CH-3′), 79.48 (CH-2′), 82.07 (CH-1′), 86.55 (CH-4′), 124.74 (CH-2), 126.92 (CH-4), 127.60 (CH-*p*-Ph), 127.97 (CH-*o*-Ph), 128.51 (CH-5), 128.75 (CH-*m*-Ph), 129.99 (CH-6), 134.09 (C-3), 138.15 (C-*i*-Ph), 141.15 (C-1), 167.23 (CO). IR spectrum (CCl_4): 3459, 3357, 1673, 1607, 1588, 1538, 1511, 1500, 1481, 1472, 1463, 1456, 1436, 1406, 1389, 1361, 1307, 1288, 1253, 1218, 1153, 1112, 1097, 1080, 1030, 940, 923, 837, 698, 671 cm^{-1}.

3.2.9. 1β-[3-(Methylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2i**)

According to the general procedure a mixture of 3-bromophenyl-*C*-ribonucleoside **1** (0.549 mmol, 347 mg), $Pd(OAc)_2$ (5 mol %, 0.027 mmol, 6.2 mg), Xantphos (10 mol %, 0.055 mmol, 31.8 mg), $MeNH_2 \cdot HCl$ (1.65 mmol, 3 equiv, 112 mg), K_3PO_4 (2.20 mmol, 4 equiv, 466 mg) and toluene (1.4 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/AcOH 25:1 to 13:1 to provide the title compound as a white foam (295 mg, 88%). HRMS (ESI) for $C_{31}H_{60}NO_5Si_3$: [M+H] calculated, 610.3774; found, 610.3764. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.52, −0.14, 0.098, 0.102, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.79, 0.93 and 0.94 (3×s, 3×9H, $(CH_3)_3C$), 3.00 (d, 3H, J=4.9, CH_3N), 3.79 (dd, 1H, J_{gem}=10.9, $J_{5'b,4'}$=3.2, H-5′b), 3.81 (dd, 1H, J_{gem}=10.9, $J_{5'a,4'}$=3.7, H-5′a), 3.82 (dd, 1H, $J_{2',1'}$=8.3, $J_{2',3'}$=4.4, H-2′), 4.05 (ddd, 1H, $J_{4',5'}$=3.7, 3.2, $J_{4',3'}$=1.4, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.4, H-3′), 6.11 (br q, 1H, J=4.9, NH), 4.81 (d, 1H, $J_{1',2'}$=8.3, H-1′), 7.39 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.58 (dddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.2, $J_{6,1'}$=0.5, H-6), 7.70 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.8, H-2), 7.74 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.2, H-4). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.52, −5.45, −4.50, −4.49 and −4.34 (CH_3Si), 17.91, 18.06 and 18.33 ($C(CH_3)_3$), 25.79, 25.88 and 25.94 ($(CH_3)_3C$), 26.75 (CH_3N), 63.82 (CH_2-5′), 74.17 (CH-3′), 79.54 (CH-2′), 82.14 (CH-1′), 86.55 (CH-4′), 124.53 (CH-2), 126.85 (CH-4), 128.45 (CH-5), 129.89 (CH-6), 134.43 (C-3), 141.05 (C-1), 168.15 (CO). IR spectrum (CCl_4): 3476, 3363, 2955, 2897, 1677, 1608, 1588, 1519, 1485, 1472, 1463, 1436, 1419, 1389, 1362, 1308, 1288, 1256, 1219, 1154, 1113, 1095, 1080, 998, 940, 924, 813, 727, 691, 671 cm^{-1}.

3.2.10. 1β-{4-[(Pyrrolidine-1-yl)carbonyl]phenyl}-1-deoxy-2,3,5-tri-O-(tert-butyldimethylsilyl)-D-ribofuranose (**5a**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.223 mmol, 141 mg), $Pd(OAc)_2$ (5 mol %, 0.011 mmol, 2.5 mg), Xantphos (10 mol %, 0.022 mmol,

13 mg), pyrrolidine (0.669 mmol, 3 equiv, 0.06 mL), K_3PO_4 (0.893 mmol, 4 equiv, 189 mg) and toluene (0.5 mL) was heated at 80 °C for 3 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:16 to provide the title compound as a colourless light oil (128 mg, 88%). HRMS (FAB) for $C_{34}H_{64}NO_5Si_3$: [M+H] calculated, 650.4092; found, 650.4108. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.43, −0.12, 0.09, 0.12 and 0.13 (5×s, 18H, CH_3Si), 0.80, 0.935 and 0.941 (3×s, 3×9H, $(CH_3)_3C$), 1.84–1.86 and 1.92–1.98 (2×m, 2×2H, CH_2-pyr), 3.37 and 3.40 (2×dt, 2H, J_{gem}=10.8, J_{vic}=6.8, CH_2N-pyr), 3.64 (t, 2H, J_{vic}=7.0, CH_2N-pyr), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.9, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.9, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.6, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.9, 2.9, $J_{4',3'}$=2.0, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=2.0, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.6, H-1′), 7.47 (s, 4H, H-2,3,5,6). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.52, −5.43, −5.33, −4.50, −4.46 and −4.43 (CH_3Si), 17.94, 18.06 and 18.35 ($C(CH_3)_3$), 24.47 (CH_2-pyr), 25.84, 25.87 and 25.98 ($(CH_3)_3C$), 26.42 (CH_2-pyr), 46.18 and 49.56 (CH_2N-pyr), 63.58 (CH_2-5′), 73.78 (CH-3′), 79.64 (CH-2′), 82.64 (CH-1′), 85.98 (CH-4′), 126.63 (CH-2,6), 126.94 (CH-3,5), 136.49 (C-4), 142.68 (C-1), 169.73 (CO). IR spectrum (CCl_4): 2955, 2930, 2896, 2885, 2858, 1634, 1614, 1574, 1513, 1485, 1472, 1463, 1463, 1420, 1405, 1392, 1362, 1343, 1257, 1227, 1213, 1186, 1153, 1112, 1112, 1095, 1081, 1020, 939, 838, 681, 672, 637 cm^{-1}.

3.2.11. 1β-{4-[(Piperidine-1-yl)carbonyl]phenyl}-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5b**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.701 mmol, 443 mg), $Pd(OAc)_2$ (5 mol %, 0.035 mmol, 7.9 mg), Xantphos (10 mol %, 0.070 mmol, 40.6 mg), piperidine (2.10 mmol, 3 equiv, 0.21 mL), K_3PO_4 (2.80 mmol, 4 equiv, 595 mg) and toluene (1.8 mL) was heated at 80 °C for 3 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:8 to provide the title compound as a colourless light oil (431 mg, 92%). HRMS (FAB) for $C_{35}H_{66}NO_5Si_3$: [M+H] calculated, 664.4267; found, 664.4249. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.42, −0.11, 0.09, 0.11 and 0.13 (5×s, 18H, CH_3Si), 0.80, 0.936 and 0.939 (3×s, 3×9H, $(CH_3)_3C$), 1.44–1.52 and 1.64–1.70 (2×br m, 6H, CH_2-pip), 3.25–3.35 and 3.64–3.76 (2×br m, 2×2H, CH_2N-pip), 3.78 (dd, 1H, J_{gem}=10.9, $J_{5'b,4'}$=2.9, H-5′b), 3.82 (dd, 1H, J_{gem}=10.9, $J_{5'a,4'}$=3.9, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.6, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.9, 2.9, $J_{4',3'}$=2.1, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=2.1, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.6, H-1′), 7.32–7.35 (m, 2H, H-3,5), 7.45–7.49 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.53, −5.45, −5.38, −4.49, −4.46 and −4.43 (CH_3Si), 17.94, 18.06 and 18.34 ($C(CH_3)_3$), 24.61 and 25.62 (CH_2-pip), 25.82, 25.87 and 25.97 ($(CH_3)_3C$), 26.48 (CH_2-pip), 43.13 and 48.75 (CH_2N-pip), 63.56 (CH_2-5′), 73.75 (CH-3′), 79.67 (CH-2′), 82.66 (CH-1′), 85.95 (CH-4′), 126.61 (CH-3,5), 126.80 (CH-2,6), 135.76 (C-4), 142.23 (C-1), 170.34 (CO). IR spectrum (CCl_4): 2951, 2930, 2897, 2887, 2858, 1638, 1615, 1574, 1512, 1472, 1463, 1454, 1443, 1429, 1410, 1389, 1370, 1362, 1352, 1310, 1288, 1273, 1258, 1219, 1187, 1153, 1110, 1095, 1080, 1029, 1021, 1003, 939, 885, 852, 838, 696, 671, 635 cm^{-1}.

3.2.12. 1β-{4-[(Morpholine-4-yl)carbonyl]phenyl}-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5c**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.752 mmol, 475 mg), $Pd(OAc)_2$ (5 mol %, 0.038 mmol, 8.4 mg), Xantphos (10 mol %, 0.075 mmol, 43.5 mg), morpholine (2.26 mmol, 3 equiv, 0.19 mL), K_3PO_4 (3.00 mmol, 4 equiv, 638 mg) and toluene (1.9 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:16 to provide the title compound as a colourless light oil (448 mg, 89%). HRMS (FAB) for $C_{34}H_{64}NO_6Si_3$: [M+H] calculated, 666.4042; found, 666.4023. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.44, −0.11, 0.09, 0.12 and 0.13 (5×s, 18H, CH_3Si), 0.80, 0.937 and 0.940 (3×s, 3×9H, $(CH_3)_3C$), 3.33–3.49, 3.52–3.68 and 3.68–3.82 (3×br m, 8H, H-morph), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.9, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.8, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.7, $J_{2',3'}$=4.4, H-2′), 4.05 (ddd, 1H, $J_{4',5'}$=3.8, 2.9, $J_{4',3'}$=1.9, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.9, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.7, H-1′), 7.34–7.38 (m, 2H, H-3,5), 7.48–7.52 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.53, −5.45, −5.38, −4.50, −4.47 and −4.43 (CH_3Si), 17.93, 18.05 and 18.34 ($C(CH_3)_3$), 25.81, 25.86 and 25.97 ($(CH_3)_3C$), 42.64 and 48.26 (CH_2N-morph), 63.56 (CH_2-5′), 66.85 (CH_2O-morph), 73.79 (CH-3′), 79.68 (CH-2′), 82.54 (CH-1′), 86.07 (CH-4′), 126.93 (CH-3,5), 126.97 (CH-2,6), 134.50 (C-4), 142.94 (C-1), 170.46 (CO). IR spectrum (CCl_4): 3930, 2957, 2897, 2858, 1645, 1615, 1574, 1509, 1482, 1462, 1456, 1423, 1408, 1389, 1362, 1300, 1280, 1258, 1219, 1187, 1154, 1118, 1110, 1095, 1080, 1026, 1018, 1011, 939, 894, 839, 698, 671 cm^{-1}.

3.2.13. 1β-[4-(Dibutylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5d**)

According to the general procedure a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.627 mmol, 396 mg), $Pd(OAc)_2$ (5 mol %, 0.031 mmol, 7.0 mg), Xantphos (10 mol %, 0.063 mmol, 36.3 mg), dibutylamine (1.88 mmol, 3 equiv, 0.32 mL), K_3PO_4 (2.51 mmol, 4 equiv, 532 mg) and toluene (1.6 mL) was heated at 80 °C for 3 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:6 to provide the title compound as a colourless light oil (422 mg, 95%). HRMS (FAB) for $C_{38}H_{74}NO_5Si_3$: [M+H] calculated, 708.4875; found, 708.4865. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.44, −0.11, 0.090, 0.092, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.77–0.82 (br m, 3H, $CH_3CH_2CH_2CH_2N$), 0.81, 0.935 and 0.940 (3×s, 3×9H, $(CH_3)_3C$), 0.95–0.99 (br m, 3H, $CH_3CH_2CH_2CH_2N$), 1.07–1.15, 1.37–1.43, 1.43–1.50 and 1.60–1.68 (4×br m, 4×2H, $CH_3CH_2CH_2CH_2N$), 3.16 and 3.47 (2×br m, 2×2H, $CH_3CH_2CH_2CH_2N$), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.8, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.8, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.6, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.8, 2.8, $J_{4',3'}$=1.9, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.9, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.6, H-1′), 7.37–7.32 (m, 2H, H-3,5), 7.44–7.48 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.54, −5.45, −5.31, −4.48, −4.47 and −4.44 (CH_3Si), 13.75 and 13.94 ($CH_3CH_2CH_2CH_2N$), 17.91, 18.06 and 18.33 ($C(CH_3)_3$), 19.79 and 20.33 ($CH_3CH_2CH_2CH_2N$), 25.83, 25.87 and 25.97 ($(CH_3)_3C$), 29.65 and 30.91 ($CH_3CH_2CH_2CH_2N$), 44.51 and 48.82 ($CH_3CH_2CH_2CH_2N$), 63.58 (CH_2-5′), 73.81 (CH-3′), 79.59 (CH-2′), 82.63 (CH-1′), 85.98 (CH-4′), 126.21 (CH-3,5), 126.75 (CH-2,6), 136.68 (C-4), 141.80 (C-1), 171.62 (CO). IR spectrum (CCl_4): 2958, 2897, 1637, 1615, 1573, 1512, 1471, 1463, 1423, 1408, 1389, 1379, 1362, 1307, 1257, 1221, 1188, 1152, 1110, 1097, 1080, 1021, 939, 838, 700, 682, 672, 635 cm^{-1}.

3.2.14. 1β-[4-(Dimethylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5e**)

According to the general procedure a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.287 mmol, 181 mg), $Pd(OAc)_2$ (5 mol %, 0.014 mmol, 3.2 mg), Xantphos (10 mol %, 0.029 mmol, 16.6 mg), $Me_2NH\cdot HCl$ (0.573 mmol, 2 equiv, 46.8 mg), K_3PO_4 (1.15 mmol, 4 equiv, 243 mg) and toluene (0.8 mL) was heated at 80 °C for 4 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:16 to provide the title compound as a colourless light oil (155 mg, 87%). HRMS (FAB) for $C_{32}H_{62}NO_5Si_3$: [M+H] calculated, 624.3936; found, 624.3945. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.42, −0.11, 0.07, 0.09, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.80, 0.936 and 0.940 (3×s, 3×9H, $(CH_3)_3C$), 2.90–3.05 and 3.05–3.15 (2×br s, 2×3H, CH_3N), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.9, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.8, H-5′a), 3.86 (dd, 1H, $J_{2',1'}$=7.6, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.8, 2.9, $J_{4',3'}$=2.0, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4,

$J_{3',4'}$=2.0, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.6, H-1′), 7.35–7.39 (m, 2H, H-3,5), 7.46–7.49 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.52, −5.44, −5.34, −4.49, −4.46 and −4.43 (CH_3Si), 17.94, 18.06 and 18.35 ($C(CH_3)_3$), 25.83, 25.88 and 25.98 ($(CH_3)_3C$), 35.37 and 39.53 (CH_3N), 63.59 (CH_2-5′), 73.80 (CH-3′), 79.67 (CH-2′), 82.63 (CH-1′), 86.00 (CH-4′), 126.75 (CH-2,6), 126.92 (CH-3,5), 135.57 (C-4), 142.44 (C-1), 171.65 (CO). IR spectrum (CCl_4): 2956, 2897, 1644, 1616, 1574, 1513, 1489, 1472, 1463, 1406, 1390, 1390, 1362, 1308, 1258, 1217, 1187, 1153, 1111, 1095, 1077, 1020, 939, 838, 701, 671, 634 cm^{-1}.

3.2.15. 1β-[4-(Cyclopropylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5f**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.548 mmol, 346 mg), $Pd(OAc)_2$ (5 mol %, 0.027 mmol, 6.1 mg), Xantphos (10 mol %, 0.055 mmol, 31.7 mg), cyclopropylamine (1.64 mmol, 3 equiv, 0.11 mL), K_3PO_4 (2.19 mmol, 4 equiv, 465 mg) and toluene (1.5 mL) was heated at 80 °C for 3 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:12 to provide the title compound as a white foam (283 mg, 81%). HRMS (FAB) for $C_{33}H_{62}NO_5Si_3$: [M+H] calculated, 636.3936; found, 636.3951. ^{1}H NMR (500.0 MHz, $CDCl_3$): −0.49, −0.13, 0.089, 0.092, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.62–0.66 (m, 2H, CH_2-cycloprop), 0.80 (s, 9H, $(CH_3)_3C$), 0.85–0.87 (m, 2H, CH_2-cycloprop), 0.93 and 0.94 (2×s, 2×9H, $(CH_3)_3C$), 2.90 (ttd, 1H, J=7.0, 3.9, 2.9, CH-cycloprop), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.8, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.7, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.9, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.7, 2.8, $J_{4',3'}$=1.7, H-4′), 4.12 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.7, H-3′), 4.80 (d, 1H, $J_{1',2'}$=7.9, H-1′), 6.24 (br d, 1H, J=2.9, NH), 7.47–7.51 (m, 2H, H-2,6), 7.67–7.71 (m, 2H, H-3,5). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.54, −5.41, −5.38, −4.50, −4.48 and −4.41 (CH_3Si), 6.76 (CH_2-cycloprop), 17.89, 18.05 and 18.35 ($C(CH_3)_3$), 23.09 (CH-1-cycloprop), 25.82, 25.86 and 25.98 ($(CH_3)_3C$), 63.63 (CH_2-5′), 73.97 (CH-3′), 79.55 (CH-2′), 82.33 (CH-1′), 86.27 (CH-4′), 126.54 (CH-3,5), 126.96 (CH-2,6), 133.43 (C-4), 144.64 (C-1), 168.63 (CO). IR spectrum (CCl_4): 3457, 3346, 3094, 3011, 2956, 2897, 1678, 1662, 1615, 1575, 1533, 1520, 1487, 1472, 1463, 1424, 1407, 1389, 1362, 1308, 1288, 1258, 1214, 1186, 1153, 1112, 1095, 1081, 1019, 939, 838, 684, 671, 638 cm^{-1}.

3.2.16. 1β-[4-(Cyclohexylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5g**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.653 mmol, 413 mg), $Pd(OAc)_2$ (5 mol %, 0.033 mmol, 7.3 mg), Xantphos (10 mol %, 0.065 mmol, 37.8 mg), cyclohexylamine (1.96 mmol, 3 equiv, 0.22 mL), K_3PO_4 (2.61 mmol, 4 equiv, 555 mg) and toluene (1.6 mL) was heated at 80 °C for 2 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:8 to provide the title compound as a white foam (385 mg, 87%). HRMS (FAB) for $C_{36}H_{68}NO_5Si_3$: [M+H] calculated, 678.4405; found, 678.4419. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.46, −0.13, 0.090, 0.092, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.81, 0.936 and 0.943 (3×s, 3×9H, $(CH_3)_3C$), 1.18–1.22, 1.23–1.27, 1.39–1.48, 1.63–1.69, 1.73–1.79 and 2.02–2.08 (6×m, 10H, H-2,3,4,5,6-cyclohex), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.9, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.8, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.7, $J_{2',3'}$=4.4, H-2′), 3.98 (tdt, 1H, J=10.7, 8.1, 4.0, H-1-cyclohex), 4.04 (td, 1H, $J_{4',5'}$=3.8, 2.9, $J_{4',3'}$=1.8, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.8, H-3′), 4.81 (d, 1H, $J_{1',2'}$=7.7, H-1′), 5.94 (d, 1H, J=8.1, NH), 7.47–7.51 (m, 2H, H-2,6), 7.68–7.71 (m, 2H, H-3,5). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.52, −5.39, −5.31, −4.49, −4.46 and −4.41 (CH_3Si), 17.90, 18.06 and 18.36 ($C(CH_3)_3$), 24.93 and 25.59 (CH_2-3,4,5-cyclohex), 25.83, 25.87 and 25.99 ($(CH_3)_3C$), 33.27 (CH_2-2,6-cyclohex), 48.65 (CH-1-cyclohex), 63.61 (CH_2-5′), 73.88 (CH-3′), 79.54 (CH-2′), 82.45 (CH-1′), 86.12 (CH-4′), 126.52 (CH-3,5), 126.91 (CH-2,6), 134.15 (C-4), 144.36 (C-1), 166.36 (CO). IR spectrum (CCl_4): 3451, 3347, 2953, 2930, 2898, 2858, 1667, 1614, 1574, 1538, 1520, 1493, 1472, 1463, 1451, 1407, 1389, 1362, 1351, 1320, 1304, 1283, 1257, 1216, 1187, 1152, 1112, 1095, 1081, 1018, 939, 838, 699, 670, 638 cm^{-1}.

3.2.17. 1β-[4-(Benzylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5h**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.641 mmol, 405 mg), $Pd(OAc)_2$ (5 mol %, 0.032 mmol, 7.2 mg), Xantphos (10 mol %, 0.064 mmol, 37.1 mg), benzylamine (1.92 mmol, 3 equiv, 0.21 mL), K_3PO_4 (2.56 mmol, 4 equiv, 544 mg) and toluene (1.6 mL) was heated at 80 °C for 3 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/EA 100:2 to 100:10 to provide the title compound as a white foam (378 mg, 86%). HRMS (FAB) for $C_{37}H_{64}NO_5Si_3$: [M+H] calculated, 686.4092; found, 686.4097. ^{1}H NMR (600.1 MHz, $CDCl_3$): −0.48, −0.13, 0.09, 0.10, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.81 and 0.94 (2×s, 27H, $(CH_3)_3C$), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.8, H-5′b), 3.81 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.7, H-5′a), 3.86 (dd, 1H, $J_{2',1'}$=7.8, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.7, 2.8, $J_{4',3'}$=1.7, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.7, H-3′), 4.62 and 4.64 (2×dd, 2H, J_{gem}=14.6, J_{vic}=5.6, CH_2Ph), 4.81 (d, 1H, $J_{1',2'}$=7.8, H-1′), 6.50 (br t, 1H, J_{vic}=5.6, NH), 7.28–7.32 (m, 1H, H-*p*-Ph), 7.34–7.38 (m, 4H, H-*o*,*m*-Ph), 7.48–7.52 (m, 2H, H-2,6), 7.73–7.77 (m, 2H, H-3,5). ^{13}C NMR (150.9 MHz, $CDCl_3$): −5.56, −5.42, −5.38, −4.52, −4.50 and −4.43 (CH_3Si), 17.86, 18.02 and 18.32 ($C(CH_3)_3$), 25.80, 25.84 and 25.96 ($(CH_3)_3C$), 44.09 (CH_2Ph), 63.60 (CH_2-5′), 73.92 (CH-3′), 79.54 (CH-2′), 82.34 (CH-1′), 86.20 (CH-4′), 126.67 (CH-3,5), 126.98 (CH-2,6), 127.55 (CH-*p*-Ph), 127.97 (CH-*o*-Ph), 128.73 (CH-*m*-Ph), 133.40 (C-4), 138.19 (C-*i*-Ph), 144.70 (C-1), 167.11 (CO). IR spectrum (CCl_4): 3460, 3356, 3089, 3067, 3033, 2956, 2897, 2887, 1672, 1657, 1614, 1575, 1523, 1514, 1472, 1455, 1407, 1389, 1362, 1305, 1295, 1289, 1263, 1257, 1217, 1152, 1113, 1095, 1080, 1031, 1019, 939, 838, 698, 672, 638 cm^{-1}.

3.2.18. 1β-[4-(Methylcarbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5i**)

According to the general procedure, a mixture of 4-bromophenyl-*C*-ribonucleoside **4** (0.211 mmol, 133 mg), $Pd(OAc)_2$ (5 mol %, 0.011 mmol, 2.4 mg), Xantphos (10 mol %, 0.021 mmol, 12.2 mg), $MeNH_2 \cdot HCl$ (0.634 mmol, 3 equiv, 42.8 mg), K_3PO_4 (0.845 mmol, 4 equiv, 180 mg) and toluene (0.52 mL) was heated at 80 °C for 3.5 h. The crude product mixture was purified by flash column chromatography on silica gel in gradient hexane/AcOH 83:1 to 13:1 to provide the title compound as a white foam (208 mg, 81%). HRMS (ESI) for $C_{31}H_{60}NO_5$: [M+H] calculated, 610.3774; found, 610.3771. ^{1}H NMR (499.8 MHz, $CDCl_3$): −0.49, −0.13, 0.091, 0.094, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.80, 0.936 and 0.942 (3×s, 3×9H, $(CH_3)_3C$), 3.02 (d, 3H, J=4.9, CH_3N), 3.78 (dd, 1H, J_{gem}=10.9, $J_{5'b,4'}$=2.9, H-5′b), 3.82 (dd, 1H, J_{gem}=10.9, $J_{5'a,4'}$=3.8, H-5′a), 3.85 (dd, 1H, $J_{2',1'}$=7.8, $J_{2',3'}$=4.4, H-2′), 4.04 (ddd, 1H, $J_{4',5'}$=3.8, 2.9, $J_{4',3'}$=1.7, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.7, H-3′), 4.81 (d, 1H, $J_{1',2'}$=7.8, H-1′), 6.14 (br q, 1H, J=4.9, NH), 7.48–7.52 (m, 2H, H-2,6), 7.69–7.73 (m, 2H, H-3,5). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.52, −5.41, −5.40, −4.49, −4.46 and −4.40 (CH_3Si), 17.91, 18.06 and 18.36 ($C(CH_3)_3$), 25.83, 25.88 and 25.99 ($(CH_3)_3C$), 26.80 (CH_3N), 63.66 (CH_2-5′), 74.00 (CH-3′), 79.62 (CH-2′), 82.36 (CH-1′), 86.27 (CH-4′), 126.54 (CH-3,5), 127.02 (CH-2,6), 135.75 (C-4), 144.50 (C-1), 168.01 (CO). IR spectrum (CCl_4): 3477, 3357, 2956, 2897, 1675, 1662, 1615, 1574, 1547, 1530, 1500, 1472, 1463, 1414, 1407, 1389, 1362, 1303, 1290, 1274, 1257, 1216, 1187, 1152, 1113, 1095, 1080, 1066, 939, 838, 699, 671, 638 cm^{-1}.

3.3. General procedure for the preparation of unsubstituted benzamide nucleosides 2j and 5j using $AcONH_4$—method A

A flame-dried septum-sealed flask containing 3- or 4-bromophenyl-*C*-ribonucleoside **1** or **4** (1 mmol), $Pd(OAc)_2$ (10 mol %,

0.1 mmol), Xantphos (20 mol %, 0.2 mmol), $AcONH_4$ (3 mmol) ($AcONH_4$ was not dried prior to use) and K_3PO_4 (4 mmol) was evacuated and backfilled with $CO_{(g)}$. Then, toluene (2.5 mL) was added via syringe. The reaction mixture was stirred at room temperature for 5 min and then immersed into a preheated oil bath (80 °C) and vigorously stirred until the starting material had been completely consumed (TLC analysis, Hexanes/EA/AcOH 7:2:1). The reaction mixture was then allowed to cool to room temperature, diluted with Et_2O, filtered through a plug of Celite (eluting with Et_2O) and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel.

3.4. General procedure for the preparation of unsubstituted benzamide nucleosides 2j and 5j using NH_4Cl—method B

A flame-dried septum-sealed flask containing 3- or 4-bromophenyl-*C*-ribonucleoside **1** or **4** (1 mmol), $Pd(OAc)_2$ (5 mol %, 0.05 mmol), Xantphos (10 mol %, 0.1 mmol), NH_4Cl (3 mmol) (NH_4Cl was not dried prior to use) and K_3PO_4 (4 mmol) was evacuated and backfilled with $CO_{(g)}$. Then, toluene/DMSO 1:1 (3 mL) was added via syringe. The reaction mixture was stirred at room temperature for 5 min and then immersed into a preheated oil bath (80 °C) and vigorously stirred until the starting material had been completely consumed (TLC analysis, Hexanes/EA/AcOH 7:2:1). The reaction mixture was then allowed to cool to room temperature, diluted with Et_2O, filtered through a plug of Celite (eluting with Et_2O) and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel.

3.5. General procedure for the preparation of unsubstituted benzamide nucleosides 2j and 5j using $NH_{3(g)}$—method C

A flame-dried septum-sealed flask containing 3- or 4-bromophenyl-*C*-ribonucleoside **1** or **4** (1 mmol), $Pd(OAc)_2$ (10 mol %, 0.1 mmol), Xantphos (20 mol %, 0.2 mmol) was evacuated and backfilled with $CO_{(g)}/NH_{3(g)}$ mixture (approx. ratio 1:1). Then, toluene (2.5 mL) was added via syringe. The reaction mixture was stirred at room temperature for 5 min and then immersed into a preheated oil bath (80 °C) and vigorously stirred until the starting material had been completely consumed (TLC analysis, Hexanes/EA/AcOH 7:2:1). The reaction mixture was then allowed to cool to room temperature, diluted with Et_2O, filtered through a plug of Celite (eluting with Et_2O) and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel.

*3.5.1. 1β-[3-(Carbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**2j**)*

Prepared according to the general procedure, *method A*. Starting from **1** (104 mg, 0.165 mmol) using $Pd(OAc)_2$ (10 mol %, 0.016 mmol, 3.7 mg), Xantphos (20 mol %, 0.033 mmol, 19 mg), $AcONH_4$ (0.494 mmol, 3 equiv, 38 mg), K_3PO_4 (0.658 mmol, 4 equiv, 140 mg) and toluene (0.41 mL). Reaction time 15 h. Flash chromatography in gradient hexane/AcOH 25:1 to 25:2 gave **2j** as colourless light oil (74 mg, 76%). HRMS (FAB) for $C_{30}H_{57}NO_5Si_3Na$: [M+Na] calculated, 618.3436; found, 618.3438. ^1H NMR (499.8 MHz, $CDCl_3$): −0.51, −0.14, 0.10, 0.11, 0.12 and 0.14 (6×s, 6×3H, CH_3Si), 0.79, 0.94 and 0.95 (3×s, 3×9H, $(CH_3)_3C$), 3.79 (dd, 1H, J_{gem}=10.9, $J_{5'b,4'}$=3.3, H-5′b), 3.82 (dd, 1H, J_{gem}=10.9, $J_{5'a,4'}$=3.7, H-5′a), 3.84 (dd, 1H, $J_{2',1'}$=8.3, $J_{2',3'}$=4.4, H-2′), 4.05 (ddd, 1H, $J_{4',5'}$=3.7, 3.3, $J_{4',3'}$=1.5, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.5, H-3′), 4.82 (d, 1H, $J_{1',2'}$=8.3, H-1′), 5.80–6.00 and 6.00–6.20 (2×br s, 2H, NH_2), 7.41 (td, 1H, $J_{5,4}$=$J_{5,6}$=7.7, $J_{5,2}$=0.5, H-5), 7.64 (dt, 1H, $J_{6,5}$=7.7, $J_{6,2}$=$J_{6,4}$=1.7, H-6), 7.76–7.80 (m, 2H, H-2,4). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.56, −5.43, −5.44, −4.51, −4.49 and −4.34 (CH_3Si), 17.90, 18.05 and 18.33 ($C(CH_3)_3$), 25.78, 25.87 and 25.95 (($CH_3)_3C$), 63.81 (CH_2-5′), 74.18 (CH-3′), 79.56 (CH-2′), 82.05 (CH-1′), 86.61 (CH-4′), 125.44 (CH-2), 127.12 (CH-4), 128.46 (CH-5), 130.47 (CH-6), 133.07 (C-3), 141.21 (C-1), 169.29 (CO). IR spectrum (CCl_4): 3538, 3506, 3421, 3352, 3295, 3166, 2955, 2847, 1685, 1609, 1587, 1472, 1463, 1438, 1407, 1389, 1379, 1361, 1308, 1257, 1257, 1218, 1153, 1112, 1095, 1082, 940, 926, 838, 692, 672 cm^{-1}.

Prepared according to the general procedure, *method B*. Starting from **1** (337 mg, 0.5333 mmol) using $Pd(OAc)_2$ (5 mol %, 0.027 mmol, 6.0 mg), Xantphos (10 mol %, 0.053 mmol, 31.0 mg), NH_4Cl (1.60 mmol, 3 equiv, 95.6 mg), K_3PO_4 (2.13 mmol, 4 equiv 453 mg) and toluene/DMSO 1:1 mixture (1.9 mL). Reaction time 3.5 h. Flash chromatography in gradient hexane/AcOH 25:1 to 25:2 gave **2j** as colourless light oil (216 mg, 68%).

Prepared according to the general procedure, *method C*. Reaction mixture containing **1** (107 mg, 0.1693 mmol), $Pd(OAc)_2$ (10 mol %, 0.017 mmol, 3.8 mg), Xantphos (20 mol %, 0.034 mmol, 20 mg), K_3PO_4 (0.677 mmol, 4 equiv 144 mg) was evacuated and backfilled with $CO_{(g)}/NH_3$ (ca. 1:1 mixture). Then toluene (0.42 mL) was added and reaction mixture was stirred for 16 h. Flash chromatography in gradient hexane/AcOH 25:1 to 25:2 gave **2j** as colourless light oil (63 mg, 62%).

*3.5.2. 1β-[4-(Carbamoyl)phenyl]-1-deoxy-2,3,5-tri-O-(tert-butyldimethyl-silyl)-D-ribofuranose (**5j**)*

Prepared according to the general procedure, *method A*. Starting from **4** (126 mg, 0.200 mmol) using $Pd(OAc)_2$ (10 mol %, 0.02 mmol, 4.5 mg), Xantphos (20 mol %, 0.04 mmol, 23 mg), $AcONH_4$ (0.598 mmol, 46 mg, 3 equiv), K_3PO_4 (0.797 mmol, 169 mg, 4 equiv) and toluene (0.5 mL). Reaction time 16 h. Flash chromatography in gradient hexane/AcOH 25:1 to 25:2 gave **5j** as colourless light oil (72 mg, 60%). HRMS (ESI) for $C_{30}H_{58}NO_5Si_3$: [M+H] calculated, 596.3592; found, 596.3617. ^1H NMR (500.0 MHz, $CDCl_3$): −0.48, −0.13, 0.09, 0.10, 0.12 and 0.13 (6×s, 6×3H, CH_3Si), 0.81, 0.939 and 0.943 (3×s, 3×9H, $(CH_3)_3C$), 3.78 (dd, 1H, J_{gem}=11.0, $J_{5'b,4'}$=2.8, H-5′b), 3.82 (dd, 1H, J_{gem}=11.0, $J_{5'a,4'}$=3.7, H-5′a), 3.86 (dd, 1H, $J_{2',1'}$=7.9, $J_{2',3'}$=4.4, H-2′), 4.05 (ddd, 1H, $J_{4',5'}$=3.7, 2.8, $J_{4',3'}$=1.7, H-4′), 4.13 (dd, 1H, $J_{3',2'}$=4.4, $J_{3',4'}$=1.7, H-3′), 4.82 (d, 1H, $J_{1',2'}$=7.9, H-1′), 5.55–5.57 and 5.90–6.20 (2×br s, 2H, NH_2); 7.51–7.55 (m, 2H, H-2,6), 7.75–7.78 (m, 2H, H-3,5). ^{13}C NMR (125.7 MHz, $CDCl_3$): −5.52, −5.41, −4.49, −4.46 and −4.39 (CH_3Si), 17.91, 18.06 and 18.36 ($C(CH_3)_3$), 25.83, 25.88 and 25.99 (($CH_3)_3C$), 63.66 (CH_2-5′), 74.02 (CH-3′), 79.64 (CH-2′), 82.33 (CH-1′), 86.33 (CH-4′), 127.08 (CH-2,6), 127.11 (CH-3,5), 132.38 (C-4), 145.34 (C-1), 169.08 (CO). IR spectrum (CCl_4): 3539, 3507, 3422, 3352, 3291, 3212, 3163, 2956, 2897, 1615, 1604, 1583, 1575, 1513, 1472, 1463, 1416, 1407, 1388, 1377, 1361, 1304, 1257, 1215, 1187, 1153, 1113, 1095, 1080, 1018, 939, 838, 672, 642 cm^{-1}.

Prepared according to the general procedure, *method B*. Starting from **4** (312 mg, 0.494 mmol) using $Pd(OAc)_2$ (5 mol %, 0.025 mmol, 5.5 mg), Xantphos (10 mol %, 0.050 mmol, 27 mg), NH_4Cl (1.48 mmol, 79 mg, 3 equiv), K_3PO_4 (1.98 mmol, 419 mg, 4 equiv) and toluene/DMSO 1:1 mixture (1.8 mL). Reaction time 3.5 h. Flash chromatography in gradient hexane/AcOH 25:1 to 25:2 gave **5j** as colourless light oil (192 mg, 65%).

Prepared according to the general procedure, *method C*. Reaction mixture containing **4** (128 mg, 0.203 mmol), $Pd(OAc)_2$ (10 mol %, 0.020 mmol, 4.5 mg), Xantphos (20 mol %, 0.041 mmol, 23 mg), K_3PO_4 (0.810 mmol, 172 mg, 4 equiv) was evacuated and backfilled with $CO_{(g)}/NH_3$ (ca. 1:1 mixture). Then toluene (0.50 mL) was added and reaction mixture was stirred for 14 h. Flash chromatography in gradient hexane/AcOH 25:1 to 25:2 gave **5j** as colourless light oil (93 mg, 69%).

3.6. General procedure for the deprotection of TBDMS-group—method A

$Et_3N{\cdot}3HF$ (163 μL, 1.00 mmol, 10 equiv) was added to the solution of silylated compound **2a–i** or **5a–i** (0.10 mmol) in THF

(1.00 mL) and the resulting mixture was stirred at 40 °C for 2 days. After the reaction was complete (monitored by TLC eluted in $CHCl_3$/MeOH 8:2), solvent was removed under reduced pressure, the crude product was dissolved in water and solid K_2CO_3 was added until basic pH. Solvents were removed under reduced pressure and crude product purified by reversed-phase chromatography (H_2O/MeOH as a eluent) to obtain free *C*-ribonucleosides **3a**–**i** and **6a**–**i**.

3.7. General procedure for the deprotection of TBDMS-group—method B

Mixture of CF_3COOH/H_2O 9:1 (0.75 mL) was added to silylated compounds **2j** or **5j** (0.10 mmol) and resulting homogeneous mixture was stirred for 1.5 h at room temperature. After the reaction was complete (monitored by TLC, eluted in $CHCl_3$/MeOH 8:2) solvents were removed under reduced pressure and the crude product was co-evaporated with 3×50 mL of toluene. Subsequent purification by reversed-phase chromatography afforded desired nucleoside **3j** and **6j**.

*3.7.1. 1β-{3-[(Pyrrolidine-1-yl)carbonyl]phenyl}-1-deoxy-D-ribofuranose (**3a**)*

Compound **3a** was prepared from **2a** (306 mg, 0.471 mmol) according to general procedure, method A, in 86% yield, as a colourless oil, which after lyophylization furnished white powder, mp 115–118 °C. HRMS (FAB) for $C_{16}H_{22}NO_5$: [M+H] calculated, 308.1498; found, 308.1506. ^{1}H NMR (500.0 MHz, DMSO-d_6): 1.79 and 1.86 (2×p, 2×2H, J_{vic}=6.8, CH_2-pyr), 3.33–3.39 (m, 2H, CH_2N-pyr), 3.45 (t, 2H, J_{vic}=6.8, CH_2N-pyr), 3.52 and 3.57 (2×br ddd, 2H, J_{gem}=11.4, $J_{5',OH}$=5.5, $J_{5',4'}$=4.4, H-5′), 3.67 (br ddd, 1H, $J_{2',1'}$=7.3, $J_{2',OH}$=7.0, $J_{2',3'}$=5.1, H-2′), 4.82 (td, 1H, $J_{4',5'}$=4.4, $J_{4',3'}$=3.1, H-4′), 3.89 (br ddd, 1H, $J_{3',2'}$=5.1, $J_{3',OH}$=4.5, $J_{3',4'}$=3.1, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.3, H-1′), 4.87 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.97 (d, 1H, $J_{OH,3'}$=4.5, OH-3′), 5.04 (d, 1H, $J_{OH,2'}$=7.0, OH-2′), 7.38 (br dd, 1H, $J_{5,4}$=8.4, $J_{5,6}$=6.6, H-5), 7.41 (br dt, 1H, $J_{4,5}$=8.4, $J_{4,2}$=$J_{4,6}$=2.0, H-4), 7.46 (br dt, 1H, $J_{6,5}$=6.6, $J_{6,2}$=$J_{6,4}$=2.0, H-6), 7.52 (br t, 1H, $J_{2,4}$=$J_{2,6}$=2.0, H-2). ^{13}C NMR (125.7 MHz, DMSO-d_6): 24.19 and 26.23 (CH_2-pyr), 46.17 and 49.19 (CH_2N-pyr), 62.23 (CH_2-5′), 71.67 (CH-3′), 78.01 (CH-2′), 82.78 (CH-1′), 85.51 (CH-4′), 124.98 (CH-2), 126.19 (CH-4), 127.80 (CH-5), 128.13 (CH-6), 137.18 (C-3), 141.78 (C-1), 168.53 (CO). IR spectrum (KBr): 3386, 3252, 1632, 1613, 1600, 1578, 1491, 1465, 1458, 1451, 1422, 1342, 1337, 1313, 1220, 1170, 1128, 1090, 1059, 1050, 1043, 1000, 877, 806, 704 cm^{-1}. $[\alpha]_D^{20}$ −29.5 (*c* 3.69, MeOH). Anal. Calcd for $C_{16}H_{21}NO_5 \cdot 0.5H_2O$: C, 60.75; H, 7.01; N, 4.43. Found: C, 60.44; H, 6.87; N, 4.22.

*3.7.2. 1β-{3-[(Piperidine-1-yl)carbonyl]phenyl}-1-deoxy-D-ribofuranose (**3b**)*

Compound **3b** was prepared from **2b** (291 mg, 0.438 mmol) according to general procedure, method A, in 93% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (FAB) for $C_{17}H_{24}NO_5$: [M+H] calculated, 322.1654; found, 322.1650. ^{1}H NMR (500.0 MHz, DMSO-d_6): 1.41–1.49, 1.49–1.56 and 1.56–1.64 (3×br m, 3×2H, CH_2-pip), 3.21–3.31 (br m, 2H, CH_2N-pip), 3.52 (ddd, 1H, J_{gem}=11.7, $J_{5'b,OH}$=5.5, $J_{5'b,4'}$=4.7, H-5′b), 3.56 (ddd, 1H, J_{gem}=11.7, $J_{5'a,4'}$=5.5, $J_{5'a,4'}$=4.4, H-5′a), 3.53–3.58 (br m, 2H, CH_2N-pip), 3.67 (ddd, 1H, $J_{2',1'}$=7.3, $J_{2',OH}$=7.1, $J_{2',3'}$=5.4, H-2′), 3.82 (ddd, 1H, $J_{4',5'}$=4.7, 4.4, $J_{4',3'}$=3.1, H-4′), 3.82 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.7, $J_{3',4'}$=3.1, H-3′), 4.58 (d, 1H, $J_{1',2'}$=7.3, H-1′), 4.87 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.97 (d, 1H, $J_{OH,3'}$=4.7, OH-3′), 5.05 (d, 1H, $J_{OH,2'}$=7.1, OH-2′), 7.24 (dt, 1H, $J_{4,5}$=7.6, $J_{4,2}$=$J_{4,6}$=1.5, H-4), 7.37 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.5, H-2), 7.38 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.6, H-5), 7.44 (dt, 1H, $J_{6,5}$=7.6, $J_{6,2}$=$J_{6,4}$=1.5, H-6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 24.32, 25.52 and 26.25 (CH_2-pip), 42.54 and 48.27 (CH_2N-pip), 62.26 (CH_2-5′), 71.72 (CH-3′); 78.02 (CH-2′), 82.73 (CH-1′), 85.54 (CH-4′), 124.52 (CH-2), 125.81 (CH-4), 127.38 (CH-6), 128.34 (CH-5), 136.45 (C-3), 141.98 (C-1), 169.19 (CO). IR spectrum (KBr): 3435, 1696, 1632, 1620, 1602, 1584, 1492, 1445, 1432, 1369, 1350, 1287, 1210, 1114, 1075, 1052, 1028, 887, 853, 806, 701 cm^{-1}. $[\alpha]_D^{20}$ −24.1 (*c* 4.10, MeOH). Anal. Calcd for $C_{17}H_{23}NO_5 \cdot 0.5H_2O$: C, 61.80; H, 7.32; N, 4.24. Found: C, 61.59; H, 7.25; N, 4.14.

*3.7.3. 1β-{3-[(Morpholine-4-yl)carbonyl]phenyl}-1-deoxy-D-ribofuranose (**3c**)*

Compound **3c** was prepared from **2c** (180 mg, 0.270 mmol) according to general procedure, method A, in 89% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (FAB) for $C_{16}H_{22}NO_6$: [M+H] calculated, 324.1447; found, 324.1443. ^{1}H NMR (500.0 MHz, DMSO-d_6): 3.31–3.35 (br m, 2H, CH_2N-morph); 3.53 and 3.56 (2×ddd, 2H, J_{gem}=11.8, $J_{5',OH}$=5.5, $J_{5',4'}$=4.6, H-5′), 3.56–3.66 (br m, 6H, CH_2N-morph and CH_2O-morph), 3.67 (ddd, 1H, $J_{2',1'}$=7.3, $J_{2',OH}$=7.1, $J_{2',3'}$=5.4, H-2′), 3.83 (dt, 1H, $J_{4',5'}$=4.6, $J_{4',3'}$=2.8, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.8, $J_{3',4'}$=2.8, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.3, H-1′), 4.87 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.97 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.05 (d, 1H, $J_{OH,2'}$=7.1, OH-2′), 7.30 (dt, 1H, $J_{4,5}$=7.5, $J_{4,2}$=$J_{4,6}$=1.5, H-4), 7.38 (dd, 1H, $J_{5,6}$=7.7, $J_{5,4}$=7.5, H-5), 7.42 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.5, H-2), 7.47 (dt, 1H, $J_{6,5}$=7.7, $J_{6,2}$=$J_{6,4}$=1.5, H-6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 42.26 and 47.95 (CH_2N-morph), 62.22 (CH_2-5′), 66.33 (CH_2O-morph), 71.69 (CH-3′), 78.03 (CH-2′), 82.69 (CH-1′), 85.53 (CH-4′), 124.96 (CH-2), 126.24 (CH-4), 127.71 (CH-6), 128.39 (CH-5), 135.45 (C-3), 142.09 (C-1), 169.40 (CO). IR spectrum (KBr): 3435, 1631, 1620, 1601, 1585, 1491, 1463, 1433, 1389, 1389, 1362, 1338, 1303, 1279, 1264, 1210, 1113, 1068, 1054, 889 cm^{-1}. $[\alpha]_D^{20}$ −29.5 (*c* 3.86, MeOH). Anal. Calcd for $C_{16}H_{21}NO_6 \cdot 0.5H_2O$: C, 57.82; H, 6.67; N, 4.21. Found: C, 58.21; H, 6.68; N, 3.90.

*3.7.4. 1β-[3-(Dibutylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3d**)*

Compound **3d** was prepared from **2d** (258 mg, 0.364 mmol) according to general procedure, method A, in 83% yield, as a colourless oil, which after lyophylization furnished a colourless oil. HRMS (ESI) for $C_{20}H_{32}NO_5$: [M+H] calculated, 366.2275; found, 366.2264. ^{1}H NMR (499.8 MHz, DMSO-d_6): 0.71 and 0.93 (2×bt, 2×3H, J_{vic}=7.1, $CH_3CH_2CH_2CH_2N$), 1.02–1.10, 1.28–1.38, 1.39–1.44 and 1.50–1.60 (4×br m, 4×2H, $CH_3CH_2CH_2CH_2N$), 3.10 and 3.38 (2×br t, 2×2H, J_{vic}=7.6, $CH_3CH_2CH_2CH_2N$), 3.55 (ddd, 1H, J_{gem}=11.7, $J_{5'b,OH}$=5.5, $J_{5'b,4'}$=4.5, H-5′b), 3.55 (ddd, 1H, J_{gem}=11.7, $J_{5'a,4'}$=5.5, $J_{5'a,4'}$=4.5, H-5′a), 3.65 (td, 1H, $J_{2',1'}$=$J_{2',OH}$=7.2, $J_{2',3'}$=5.4, H-2′), 3.82 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.4, H-4′), 3.88 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.9, $J_{3',4'}$=3.4, H-3′), 4.58 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.95 (d, 1H, $J_{OH,3'}$=4.9, OH-3′), 5.00 (d, 1H, $J_{OH,2'}$=7.2, OH-2′), 7.19 (ddd, 1H, $J_{4,5}$=7.5, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4), 7.32 (tt, 1H, $J_{2,4}$=$J_{2,6}$=1.8, $J_{2,5}$=$J_{2,1'}$=0.5, H-2), 7.40 (ddd, 1H, $J_{5,6}$=7.7, $J_{5,4}$=7.5, $J_{5,2}$=0.5, H-5), 7.43 (dddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.3, $J_{6,1'}$=0.6, H-6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 13.62 and 14.07 ($CH_3CH_2CH_2CH_2N$), 19.42, 19.95, 29.47 and 30.44 ($CH_3CH_2CH_2CH_2N$), 43.95 and 48.23 ($CH_3CH_2CH_2CH_2N$), 62.21 (CH_2-5′), 71.62 (CH-3′), 78.01 (CH-2′), 82.73 (CH-1′), 85.44 (CH-4′), 123.89 (CH-2), 125.34 (CH-4), 126.87 (CH-6), 128.25 (CH-5), 137.27 (C-3), 141.86 (C-1), 170.62 (CO). IR spectrum (KBr): 3351, 1633, 1614, 1603, 1585, 1493, 1428, 1378, 1310, 1204, 1166, 1111, 1076, 1054, 806, 706 cm^{-1}. $[\alpha]_D^{20}$ −34.4 (*c* 3.37, MeOH). Anal. Calcd for $C_{20}H_{31}NO_5 \cdot 0.5H_2O$: C, 64.45; H, 8.61; N, 3.74. Found: C, 64.52; H, 8.42; N, 3.76.

*3.7.5. 1β-[3-(Dimethylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3e**)*

Compound **3e** was prepared from **2e** (134 mg, 0.215 mmol) according to general procedure, method A, in 95% yield, as a colourless oil, which after lyophylization furnished white powder, mp 122–125 °C. HRMS (FAB) for $C_{14}H_{20}NO_5$: [M+H] calculated, 282.1341; found, 282.1338. ^{1}H NMR (500.0 MHz, DMSO-d_6): 2.80–2.92 and 2.92–3.04 (2×br s, 2×3H, CH_3N), 3.53 and 3.56 (2×ddd,

2H, J_{gem}=11.7, $J_{5',OH}$=5.5, $J_{5',4'}$=4.4, H-5′), 3.68 (ddd, 1H, $J_{2',1'}$=7.2, $J_{2',OH}$=7.1, $J_{2',3'}$=5.4, H-2′), 3.82 (td, 1H, $J_{4',5'}$=4.4, $J_{4',3'}$=3.4, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.8, $J_{3',4'}$=3.4, H-3′), 4.58 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.93 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.01 (d, 1H, $J_{OH,2'}$=7.1, OH-2′), 6.60 (ddd, 1H, $J_{4,5}$=7.6, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4), 7.38 (dd, 1H, $J_{5,6}$=7.7, $J_{5,4}$=7.6, H-5), 7.41 (tt, 1H, $J_{2,4}$=$J_{2,6}$=1.8, $J_{2,5}$=$J_{2,1'}$=0.6, H-2), 7.45 (dddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.3, $J_{6,1'}$=0.7, H-6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 34.90 and 39.16 (CH_3N), 62.18 (CH_2-5′), 71.61 (CH-3′), 77.91 (CH-2′), 82.73 (CH-1′), 85.47 (CH-4′), 124.77 (CH-2), 126.00 (CH-4), 127.33 (CH-6), 128.11 (CH-5), 136.38 (C-3), 141.81 (C-1), 170.35 (CO). IR spectrum (KBr): 3362, 3252, 3175, 1623, 1606, 1585, 1512, 1479, 1432, 1412, 1400, 1310, 1222, 1164, 1131, 1098, 1081, 1053, 1046, 804, 700 cm^{-1}. $[\alpha]_D^{20}$ −28.4 (*c* 3.49, MeOH). Anal. Calcd for $C_{14}H_{19}NO_5\cdot 0.4H_2O$: C, 58.28; H, 6.92; N, 4.85. Found: C, 58.58; H, 6.69; N, 4.48.

3.7.6. 1β-[3-(Cyclopropylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3f**)

Compound **3f** was prepared from **2f** (283 mg, 0.445 mmol) according to general procedure, method A, in 89% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{15}H_{20}NO_5$: [M+H] calculated, 294.1336; found, 294.1336. ^{1}H NMR (500.0 MHz, DMSO-d_6): 0.54–0.58 and 0.66–0.72 (2×m, 2×2H, CH_2-cycloprop), 2.83 (tq 1H, *J*=7.3, 4.0, CH-cycloprop), 3.54 (ddd, 1H, J_{gem}=11.7, $J_{5'b,OH}$=5.6, $J_{5'b,4'}$=4.8, H-5′b), 3.58 (ddd, 1H, J_{gem}=11.7, $J_{5'a,OH}$=5.6, $J_{5'a,4'}$=4.5, H-5′a), 3.70 (ddd, 1H, $J_{2',1'}$=7.1, $J_{2',OH}$=6.8, $J_{2',3'}$=5.4, H-2′), 3.82 (ddd, 1H, $J_{4',5'}$=4.8, 4.5, $J_{4',3'}$=3.7, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.8, $J_{3',4'}$=3.7, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.1, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.93 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.01 (d, 1H, $J_{OH,2'}$=6.8, OH-2′), 7.39 (dd, 1H, $J_{5,4}$=7.8, $J_{5,6}$=7.6, H-5), 7.54 (dddd, 1H, $J_{6,5}$=7.6, $J_{6,2}$=1.7, $J_{6,4}$=1.3, $J_{6,1'}$=0.6, H-6), 7.68 (ddd, 1H, $J_{4,5}$=7.8, $J_{4,2}$=1.7, $J_{4,6}$=1.3, H-4), 7.78 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.7, H-2), 8.41 (d, 1H, *J*=4.0, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 5.98 and 6.00 (CH_2-cycloprop), 23.28 (CH-cycloprop), 62.23 (CH_2-5′), 71.58 (CH-3′), 77.73 (CH-2′), 83.04 (CH-1′), 85.42 (CH-4′), 125.42 (CH-2), 126.30 (CH-4), 128.16 (CH-5), 129.11 (CH-6), 134.52 (C-3), 141.71 (C-1), 167.91 (CO). IR spectrum (KBr): 3351, 3092, 3012, 1642, 1607, 1584, 1534, 1482, 1424, 1303, 1205, 1113, 1072, 1050, 813, 698 cm^{-1}. $[\alpha]_D^{20}$ −29.1 (*c* 2.51, MeOH). Anal. Calcd for $C_{15}H_{19}NO_5\cdot 0.5H_2O$: C, 59.59; H, 6.67; N, 4.63. Found: C, 59.81; H, 6.41; N, 4.29.

3.7.7. 1β-[3-(Cyclohexylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3g**)

Compound **3g** was prepared from **2g** (190 mg, 0.280 mmol) according to general procedure, method A, in 91% yield, as a colourless oil, which after crystallization from EA furnished white cotton-like crystals, mp 189–191 °C. HRMS (ESI) for $C_{18}H_{24}NO_5$: [M−H] calculated, 334.1649; found, 334.1650. ^{1}H NMR (500.0 MHz, DMSO-d_6): 1.07–1.17, 1.23–1.37, 1.57–1.64, 1.57–1.64 and 1.77–1.85 (5×m, 10H, H-2,3,4,5,6-cyclohex), 3.54 (ddd, 1H, J_{gem}=11.7, $J_{5'b,OH}$=5.6, $J_{5'b,4'}$=4.8, H-5′b), 3.58 (ddd, 1H, J_{gem}=11.7, $J_{5'a,4'}$=5.6, $J_{5'a,4'}$=4.5, H-5′a), 3.71 (ddd, 1H, $J_{2',1'}$=7.0, $J_{2',OH}$=6.8, $J_{2',3'}$=5.4, H-2′), 3.71–3.79 (br m, 1H, H-1-cyclohex), 3.83 (ddd, 1H, $J_{4',5'}$=4.8, 4.5, $J_{4',3'}$=3.7, H-4′), 3.90 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.9, $J_{3',4'}$=3.7, H-3′), 4.60 (d, 1H, $J_{1',2'}$=7.0, H-1′), 4.81 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.91 (d, 1H, $J_{OH,3'}$=4.9, OH-3′), 5.00 (d, 1H, $J_{OH,2'}$=6.8, OH-2′), 7.39 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.54 (dt, 1H, $J_{6,5}$=7.7, $J_{6,2}$=$J_{6,4}$=1.5, H-6), 7.71 (dt, 1H, $J_{4,5}$=7.7, $J_{4,2}$=$J_{4,6}$=1.5, H-4), 7.79 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.5, H-2), 8.15 (d, 1H, *J*=8.0, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 25.16, 25.47 and 32.63 (CH_2-2,3,4,5,6-cyclohex), 48.50 (CH-1-cyclohex), 62.17 (CH_2-5′), 71.51 (CH-3′), 77.67 (CH-2′), 83.07 (CH-1′), 85.34 (CH-4′), 125.56 (CH-2), 126.36 (CH-4), 128.04 (CH-5), 128.86 (CH-6), 134.94 (C-3), 141.62 (C-1), 165.66 (CO). IR spectrum (KBr): 3410, 3299, 3264, 2935, 2854, 1635, 1612, 1587, 1542, 1486, 1449, 1441, 1369, 1350, 1337, 1320, 1301, 1210, 1129, 1087, 1063, 815, 703, 695 cm^{-1}. $[\alpha]_D^{20}$ −11.0 (*c* 3.18, MeOH). Anal. Calcd for $C_{18}H_{25}NO_5\cdot 1H_2O$: C, 61.17; H, 7.70; N, 3.96. Found: C, 61.27; H, 7.47; N, 3.96.

3.7.8. 1β-[3-(Benzylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3h**)

Compound **3h** was prepared from **2h** (210 mg, 0.306 mmol) according to general procedure, method A, in 88% yield, as a colourless oil, which after crystallization from EA furnished white crystals, mp 135–137 °C. HRMS (ESI) for $C_{19}H_{20}NO_5$: [M−H] calculated, 342.1336; found, 342.1340. ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.55 and 3.58 (2×ddd, 2×1H, J_{gem}=11.6, $J_{5',OH}$=5.6, $J_{5',4'}$=4.6, H-5′), 3.71 (ddd, 1H, $J_{2',1'}$=7.1, $J_{2',OH}$=6.9, $J_{2',3'}$=5.4, H-2′), 3.83 (td, 1H, $J_{4',5'}$=4.6, $J_{4',3'}$=3.7, H-4′), 3.90 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.9, $J_{3',4'}$=3.7, H-3′), 4.48 (d, 2H, *J*=6.0, CH_2Ph), 4.61 (d, 1H, $J_{1',2'}$=7.1, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.94 (d, 1H, $J_{OH,3'}$=4.9, OH-3′), 5.03 (d, 1H, $J_{OH,2'}$=6.9, OH-2′), 7.22–7.26 (m, 1H, H-*p*-Ph), 7.30–7.35 (m, 4H, H-*o*,*m*-Ph), 7.43 (dd, 1H, $J_{5,4}$=7.7, $J_{5,6}$=7.6, H-5), 7.58 (dddd, 1H, $J_{6,5}$=7.6, $J_{6,2}$=1.8, $J_{6,4}$=1.3, $J_{6,1'}$=0.5, H-6), 7.79 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4), 7.87 (tt, 1H, $J_{2,4}$=$J_{2,6}$=1.8, $J_{2,5}$=$J_{2,1'}$=0.6, H-2), 9.04 (t, 1H, *J*=6.0, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 42.81 (CH_2Ph), 62.24 (CH_2-5′), 71.61 (CH-3′), 77.75 (CH-2′), 82.99 (CH-1′), 85.45 (CH-4′), 125.49 (CH-2), 126.37 (CH-4), 126.95 (CH-*p*-Ph), 127.40 (CH-*o*-Ph), 128.28 (CH-5), 128.51 (CH-*m*-Ph), 129.33 (CH-6), 134.39 (C-3), 139.94 (C-*i*-Ph), 141.85 (C-1), 166.51 (CO). IR spectrum (KBr): 3420, 3370, 3292, 1642, 1620, 1607, 1581, 1542, 1496, 1480, 1451, 1430, 1317, 1317, 1292, 1219, 1170, 1126, 1079, 1054, 1028, 812, 704, 693 cm^{-1}. $[\alpha]_D^{20}$ −21.9 (*c* 3.19, MeOH). Anal. Calcd for $C_{19}H_{21}NO_5$: C, 66.46; H, 6.16; N, 4.08. Found: C, 66.13; H, 6.19; N, 3.93.

3.7.9. 1β-[3-(Methylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3i**)

Compound **3i** was prepared from **2i** (250 mg, 0.410 mmol) according to general procedure, method A, in 93% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{13}H_{18}NO_5$: [M+H] calculated, 268.1179; found, 268.1185. ^{1}H NMR (600.1 MHz, DMSO-d_6): 2.78 (d, 3H, *J*=4.6, CH_3), 3.54 (ddd, 1H, J_{gem}=11.6, $J_{5'b,OH}$=5.6, $J_{5'b,4'}$=4.7, H-5′b), 3.58 (ddd, 1H, J_{gem}=11.6, $J_{5'a,4'}$=5.6, $J_{5'a,4'}$=4.5, H-5′a), 3.69 (ddd, 1H, $J_{2',1'}$=7.2, $J_{2',OH}$=6.9, $J_{2',3'}$=5.4, H-2′), 3.83 (ddd, 1H, $J_{4',5'}$=4.7, 4.5, $J_{4',3'}$=3.5, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.5, $J_{3',4'}$=3.7, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.94 (d, 1H, $J_{OH,3'}$=4.9, OH-3′), 5.03 (d, 1H, $J_{OH,2'}$=6.9, OH-2′), 7.41 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7, H-5), 7.54 (dddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.3, $J_{6,1'}$=0.6, H-6), 7.70 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4), 7.80 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.8, H-2), 8.41 (q, 1H, *J*=4.6, NH). ^{13}C NMR (150.9 MHz, DMSO-d_6): 26.48 (CH_3), 62.25 (CH_2-5′), 71.62 (CH-3′), 77.76 (CH-2′), 82.99 (CH-1′), 85.45 (CH-4′), 125.25 (CH-2), 126.16 (CH-4), 128.22 (CH-5), 129.09 (CH-6), 134.62 (C-3), 141.78 (C-1), 166.97 (CO). IR spectrum (KBr): 3412, 1639, 1607, 1585, 1549, 1484, 1434, 1412, 1325, 1307, 1307, 1217, 1158, 1115, 1075, 1052, 814, 698 cm^{-1}. $[\alpha]_D^{20}$ −10.9 (*c* 2.87, MeOH). Anal. Calcd for $C_{13}H_{17}NO_5\cdot 0.5H_2O$: C, 56.51; H, 6.57; N, 5.07. Found: C, 56.48; H, 6.64; N, 4.85.

3.7.10. 1β-[3-(Carbamoyl)phenyl]-1-deoxy-D-ribofuranose (**3j**)

Compound **3j** was prepared from **2j** (350 mg, 0.587 mmol) according to general procedure, method B, in 90% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{12}H_{16}NO_5$: [M+H] calculated, 254.1023; found, 254.1024. ^{1}H NMR (600.1 MHz, DMSO-d_6): 3.54 (ddd, 1H, J_{gem}=12.0, $J_{5'b,OH}$=5.7, $J_{5'b,4'}$=4.7, H-5′b), 3.58 (ddd, 1H, J_{gem}=12.0, $J_{5'a,4'}$=5.7, $J_{5'a,4'}$=4.5, H-5′a), 3.70 (ddd, 1H, $J_{2',1'}$=7.2, $J_{2',OH}$=6.9, $J_{2',3'}$=5.4, H-2′), 3.82 (ddd, 1H, $J_{4',5'}$=4.7, 4.5, $J_{4',3'}$=3.5, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.8, $J_{3',4'}$=3.5, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.7, OH-5′), 4.94 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.03 (d, 1H, $J_{OH,2'}$=6.9, OH-2′), 7.36 (br s, 1H, NH_aH_b), 7.40 (t, 1H, $J_{5,4}$=$J_{5,6}$=7.7,

H-5), 7.55 (dddd, 1H, $J_{6,5}$=7.7, $J_{6,2}$=1.8, $J_{6,4}$=1.3, $J_{6,1'}$=0.5, H-6), 7.76 (ddd, 1H, $J_{4,5}$=7.7, $J_{4,2}$=1.8, $J_{4,6}$=1.3, H-4), 7.85 (t, 1H, $J_{2,4}$=$J_{2,6}$=1.8, H-2), 7.95 (br s, 1H, NH_aH_b). ^{13}C NMR (150.9 MHz, DMSO-d_6): 62.25 (CH_2-5′), 71.63 (CH-3′), 77.78 (CH-2′), 83.01 (CH-1′), 85.47 (CH-4′), 125.68 (CH-2), 126.61 (CH-4), 128.16 (CH-5), 129.37 (CH-6), 134.32 (C-3), 141.71 (C-1), 168.21 (CO). IR spectrum (KBr): 3419, 1659, 1617, 1607, 1582, 1486, 1397, 1217, 1114, 1072, 1050, 695 cm^{-1}. $[\alpha]_D^{20}$ −25.6 (*c* 1.29, MeOH). Anal. Calcd for $C_{21}H_{15}NO_5 \cdot 0.5H_2O$: C, 54.96; H, 6.15; N, 5.34. Found: C, 54.56; H, 6.15; N, 5.12.

3.7.11. 1β-{4-[(Pyrrolidine-1-yl)carbonyl]phenyl}-1-deoxy-D-ribofuranose (**6a**)

Compound **6a** was prepared from **5a** (258 mg, 0.397 mmol) according to general procedure, method A, in 90% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{16}H_{20}NO_5$: [M−H] calculated, 306.1336; found, 306.1342. ^{1}H NMR (500.0 MHz, DMSO-d_6): 1.76–1.83 and 1.83–1.90 (2×m, 2×2H, CH_2-pyr), 3.38 (t, 2H, *J*=6.6, CH_2N-pyr), 3.45 (t, 2H, *J*=6.9, CH_2N-pyr), 3.53 and 3.56 (2×br dd, 2×1H, J_{gem}=12.1, $J_{5',4'}$=4.5, H-5′), 3.68 (dd, 1H, $J_{2',1'}$=7.2, $J_{2',3'}$=5.3, H-2′), 3.83 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.4, H-4′), 3.90 (dd, 1H, $J_{3',2'}$=5.3, $J_{3',4'}$=3.4, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.83 (br s, 1H, OH-5′), 5.01 (br s, 1H, OH-3′), 5.05 (br s, 1H, OH-2′), 7.42–7.46 (m, 2H, H-2,6), 7.46–7.48 (m, 2H, H-3,5). ^{13}C NMR (125.7 MHz, DMSO-d_6): 24.11 and 26.17 (CH_2-pyr), 46.10 and 49.13 (CH_2N-pyr), 62.23 (CH_2-5′), 71.70 (CH-3′), 77.89 (CH-2′), 82.75 (CH-1′), 85.46 (CH-4′), 126.07 (CH-2,6), 127.02 (CH-3,5), 136.33 (C-4), 143.30 (C-1), 168.35 (CO). IR spectrum (KBr): 3341, 2969, 2925, 2877, 1606, 1565, 1516, 1482, 1462, 1444, 1444, 1405, 1340, 1304, 1253, 1230, 1209, 1184, 1113, 1113, 1078, 1054, 1018, 835, 687, 635 cm^{-1}. $[\alpha]_D^{20}$ −37.9 (*c* 2.82, MeOH). Anal. Calcd for $C_{16}H_{21}NO_5 \cdot 4/5H_2O$: C, 59.73; H, 7.08; N, 4.35. Found: C, 59.58; H, 6.91; N, 4.26.

3.7.12. 1β-{4-[(Piperidine-1-yl)carbonyl]phenyl}-1-deoxy-D-ribofuranose (**6b**)

Compound **6b** was prepared from **5b** (308 mg, 0.464 mmol) according to general procedure, method A, in 91% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{17}H_{24}NO_5$: [M+H] calculated, 322.1649; found, 322.1650. ^{1}H NMR (500.0 MHz, DMSO-d_6): 1.41–1.49, 1.49–1.57 and 1.58–1.64 (3×br m, 3×2H, CH_2-pip), 3.21–3.31 (br m, 2H, CH_2N-pip), 3.50–3.60 (br m, 4H, H-5′ and CH_2N-pip), 3.69 (dd, 1H, $J_{2',1'}$=7.3, $J_{2',3'}$=5.3, H-2′), 3.83 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.4, H-4′), 3.90 (dd, 1H, $J_{3',2'}$=5.3, $J_{3',4'}$=3.4, H-3′), 4.58 (d, 1H, $J_{1',2'}$=7.3, H-1′), 4.83 (br s, 1H, OH-5′), 4.98 (br s, 1H, OH-3′), 5.04 (br s, 1H, OH-2′), 7.29–7.33 (m, 2H, H-3,5), 7.43–7.46 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 24.25 and 25.74 (CH_2-pip), 42.49 and 48.25 (CH_2N-pip), 62.24 (CH_2-5′), 71.72 (CH-3′), 77.88 (CH-2′), 82.72 (CH-1′), 85.48 (CH-4′), 126.31 (CH-2,6), 126.60 (CH-3,5), 135.63 (C-4), 142.84 (C-1), 169.07 (CO). IR spectrum (KBr): 3393, 2935, 2858, 1632, 1606, 1569, 1515, 1470, 1445, 1407, 1368, 1352, 1288, 1277, 1113, 1113, 1075, 1055, 1027, 1018, 1004, 888, 854, 835, 684, 634 cm^{-1}. $[\alpha]_D^{20}$ −28.2 (*c* 2.87, MeOH). Anal. Calcd for $C_{17}H_{23}NO_5 \cdot 2/3H_2O$: C, 61.14; H, 7.36; N, 4.19. Found: C, 61.14; H, 7.26; N, 4.07.

3.7.13. 1β-{4-[(Morpholine-4-yl)carbonyl]phenyl}-1-deoxy-D-ribofuranose (**6c**)

Compound **6c** was prepared from **5c** (317 mg, 0.480 mmol) according to general procedure, method A, in 87% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{16}H_{22}NO_6$: [M+H] calculated, 324.1442; found, 324.1442. ^{1}H NMR (500.0 MHz, DMSO-d_6): 3.33–3.37 (br m, 2H, CH_2N-morph), 3.50–3.70 (br m, 6H, CH_2N+CH_2O-morph), 3.53 and 3.56 (2×ddd, 2×1H, J_{gem}=11.7, $J_{5',OH}$=5.5, $J_{5',4'}$=4.5, H-5′), 3.69 (ddd, 1H, $J_{2',1'}$=7.3, $J_{2',OH}$=7.1, $J_{2',3'}$=5.3, H-2′), 3.83 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.4, H-4′), 3.90 (dddd, 1H, $J_{3',2'}$=5.3, $J_{3',OH}$=4.8, $J_{3',4'}$=3.4, $J_{3',1'}$=0.4, H-3′), 4.59 (d, 1H, $J_{1',2'}$=7.3, H-1′), 4.82 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.94 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.01 (d, 1H, $J_{OH,2'}$=7.1, OH-2′), 7.35–7.38 (m, 2H, H-3,5), 7.44–7.48 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 42.50 and 48.00 (CH_2N-morph), 62.21 (CH_2-5′), 66.28 (CH_2O-morph), 71.70 (CH-3′), 77.89 (CH-2′), 82.67 (CH-1′), 85.47 (CH-4′), 126.31 (CH-2,6), 127.02 (CH-3,5), 134.63 (C-4), 143.26 (C-1), 169.27 (CO). IR spectrum (KBr): 3401, 2965, 2921, 2858, 1611, 1569, 1513, 1463, 1436, 1436, 1407, 1389, 1362, 1331, 1302, 1280, 1260, 1221, 1184, 1158, 1114, 1114, 1066, 1052, 1026, 1012, 895, 834, 688, 634 cm^{-1}. $[\alpha]_D^{20}$ −27.5 (*c* 4.21, MeOH). Anal. Calcd for $C_{16}H_{21}NO_6 \cdot 0.5H_2O$: C, 57.82; H, 6.67; N, 4.21. Found: C, 57.67; H, 6.57; N, 4.17.

3.7.14. 1β-[4-(Dibutylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6d**)

Compound **6d** was prepared from **5d** (220 mg, 0.310 mmol) according to general procedure, method A, in 87% yield, as a colourless oil, which after lyophylization furnished colourless oil. HRMS (FAB) for $C_{20}H_{32}NO_5$: [M+H] calculated, 366.2280; found, 366.2275. ^{1}H NMR (500.0 MHz, DMSO-d_6): 0.71 and 0.92 (2×br t, 2×3H, J_{vic}=7.1, $CH_3CH_2CH_2CH_2N$), 1.00–1.10, 1.26–1.38, 1.38–1.49 and 1.49–1.60 (4×br m, 4×2H, $CH_3CH_2CH_2CH_2N$), 3.14 and 3.38 (2×br t, 2×2H, J_{vic}=7.1, $CH_3CH_2CH_2CH_2N$), 3.53 (ddd, 1H, J_{gem}=11.7, $J_{5'b,OH}$=5.5, $J_{5'b,4'}$=4.9, H-5′b), 3.56 (ddd, 1H, J_{gem}=11.7, $J_{5'a,4'}$=5.5, $J_{5'a,4'}$=4.4, H-5′a), 3.67 (td, 1H, $J_{2',1'}$=$J_{2',OH}$=7.1, $J_{2',3'}$=5.4, H-2′), 3.82 (ddd, 1H, $J_{4',5'}$=4.9, 4.4, $J_{4',3'}$=3.2, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.8, $J_{3',4'}$=3.2, H-3′), 4.58 (d, 1H, $J_{1',2'}$=7.1, H-1′), 4.85 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.98 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.04 (d, 1H, $J_{OH,2'}$=7.1, OH-2′), 7.17–7.31 (m, 2H, H-3,5), 7.38–7.48 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 13.70 and 14.11 ($CH_3CH_2CH_2CH_2N$), 19.45, 19.98, 29.50 and 30.47 ($CH_3CH_2CH_2CH_2N$), 43.92 and 48.26 ($CH_3CH_2CH_2CH_2N$), 62.24 (CH_2-5′), 71.66 (CH-3′), 77.97 (CH-2′), 82.86 (CH-1′), 85.45 (CH-4′), 126.27 and 126.31 (CH-2,3,5,6), 136.51 (C-4), 142.50 (C-1), 170.59 (CO). IR spectrum (KBr): 3399, 3334, 3278, 1619, 1568, 1512, 1430, 1405, 1381, 1307, 1210, 1179, 1119, 1103, 1080, 1047, 1018, 839, 682, 638 cm^{-1}. $[\alpha]_D^{20}$ −34.2 (*c* 3.30, MeOH). Anal. Calcd for $C_{20}H_{31}NO_5$: C, 65.73; H, 8.55; N, 3.83. Found: C, 65.35; H, 8.39; N, 3.72.

3.7.15. 1β-[4-(Dimethylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6e**)

Compound **6e** was prepared from **5e** (150 mg, 0.240 mmol) according to general procedure, method A, in 95% yield, as a colourless oil, which after lyophylization furnished white powder, mp 103–105 °C. HRMS (ESI) for $C_{14}H_{18}NO_5$: [M−H] calculated, 280.1179; found, 280.1190. ^{1}H NMR (500.0 MHz, DMSO-d_6): 2.90 and 2.97 (2×br s, 2×3H, CH_3N), 3.53 and 3.56 (2×ddd, 2H, J_{gem}=11.7, $J_{5',OH}$=5.6, $J_{5',4'}$=4.5, H-5′), 3.65–3.71 (br m, 1H, H-2′), 3.83 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.3, H-4′), 3.87–3.92 (br m, 1H, H-3′), 4.58 (d, 1H, $J_{1',2'}$=7.3, H-1′), 4.86 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.99 (br s, 1H, OH-3′), 5.06 (br d, 1H, $J_{OH,2'}$=7.1, OH-2′), 7.33–7.37 (m, 2H, H-3,5), 7.42–7.46 (m, 2H, H-2,6). ^{13}C NMR (125.7 MHz, DMSO-d_6): 35.02 and 39.29 (CH_3N), 62.27 (CH_2-5′), 71.76 (CH-3′), 77.97 (CH-2′), 82.77 (CH-1′), 85.51 (CH-4′), 126.25 (CH-2,6), 127.01 (CH-3,5), 135.62 (C-4), 142.99 (C-1), 170.36 (CO). IR spectrum (KBr): 3333, 1613, 1569, 1520, 1493, 1407, 1401, 1305, 1265, 1218, 1182, 1114, 1087, 1053, 1018, 836, 693 cm^{-1}. $[\alpha]_D^{20}$ −26.1 (*c* 3.10, MeOH). Anal. Calcd for $C_{14}H_{19}NO_5 \cdot 0.5H_2O$: C, 57.92; H, 6.94; N, 4.82. Found: C, 58.20; H, 6.85; N, 4.57.

3.7.16. 1β-[4-(Cyclopropylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6f**)

Compound **6f** was prepared from **5f** (140 mg, 0.220 mmol) according to general procedure, method A, in 89% yield, as a colourless oil, which after lyophylization furnished white powder, mp 94–98 °C. HRMS (ESI) for $C_{15}H_{18}NO_5$: [M−H] calculated, 292.1179; found, 292.1191. ^{1}H NMR (500.0 MHz, DMSO-d_6): 0.54–0.58 and

0.63–0.74 (2×m, 2×2H, CH_2-cycloprop), 2.83 (tq 1H, J=7.2, 4.2, CH-cycloprop), 3.53 and 3.57 (2×ddd, 2×1H, J_{gem}=11.7, $J_{5',OH}$=5.4, $J_{5',4'}$=4.6, H-5′), 3.66 (ddd, 1H, $J_{2',1'}$=7.1, $J_{2',OH}$=7.0, $J_{2',3'}$=5.4, H-2′), 3.82 (td, 1H, $J_{4',5'}$=4.6, $J_{4',3'}$=3.6, H-4′), 3.88 (bddd, 1H, $J_{3',2'}$=5.4, $J_{3',OH}$=4.7, $J_{3',4'}$=3.6, H-3′), 4.60 (d, 1H, $J_{1',2'}$=7.1, H-1′), 4.82 (t, 1H, $J_{OH,5'}$=5.4, OH-5′), 4.93 (d, 1H, $J_{OH,3'}$=4.7, OH-3′), 5.01 (d, 1H, $J_{OH,2'}$=7.1, OH-2′), 7.43–7.46 (m, 2H, H-2,6), 7.74–7.78 (m, 2H, H-3,5), 8.42 (d, 1H, J=4.2, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 5.95 and 5.96 (CH_2-cycloprop), 23.20 (CH-cycloprop), 62.14 (CH_2-5′), 71.57 (CH-3′), 77.91 (CH-2′), 82.77 (CH-1′), 85.41 (CH-4′), 125.99 (CH-2,6), 127.08 (CH-3,5), 133.58 (C-4), 144.80 (C-1), 167.54 (CO). IR spectrum (KBr): 3452, 3355, 3316, 3262, 3093, 3007, 1733, 1649, 1633, 1614, 1571, 1538, 1525, 1504, 1493, 1422, 1320, 1306, 1277, 1211, 1189, 1120, 1080, 1047, 1016, 841, 809, 697, 640 cm^{-1}. $[\alpha]_D^{20}$ −24.8 (*c* 3.49, MeOH). Anal. Calcd for $C_{15}H_{19}NO_5 \cdot 2/3H_2O$: C, 59.06; H, 6.71; N, 4.59. Found: C, 59.01; H, 6.71; N, 4.31.

3.7.17. 1β-[4-(Cyclohexylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6g**)

Compound **6g** was prepared from **5g** (120 mg, 0.177 mmol) according to general procedure, method A, in 85% yield, as a colourless oil, which after crystalisation from EA furnished white needles, mp 191–195 °C. HRMS (ESI) for $C_{18}H_{24}NO_5$: [M−H] calculated, 334.1649; found, 334.1657. ^{1}H NMR (500.0 MHz, DMSO-d_6): 1.06–1.17, 1.22–1.36, 1.57–1.64, 1.68–1.76 and 1.78–1.85 (5×m, 10H, H-2,3,4,5,6-cyclohex), 3.54 and 3.57 (2×ddd, 2×1H, J_{gem}=11.7, $J_{5',OH}$=5.6, $J_{5',4'}$=4.5, H-5′), 3.66 (ddd, 1H, $J_{2',1'}$=7.0, $J_{2',OH}$=6.9, $J_{2',3'}$=5.3, H-2′), 3.70–3.79 (br m, 1H, H-1-cyclohex), 3.83 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.7, H-4′), 3.88 (ddd, 1H, $J_{3',2'}$=5.3, $J_{3',OH}$=4.9, $J_{3',4'}$=3.7, H-3′), 4.60 (d, 1H, $J_{1',2'}$=7.0, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.94 (d, 1H, $J_{OH,3'}$=4.9, OH-3′), 5.01 (d, 1H, $J_{OH,2'}$=6.9, OH-2′), 7.43–7.47 (m, 2H, H-2,6), 7.77–7.80 (m, 2H, H-3,5), 8.13 (d, 1H, J=8.0, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 25.17, 25.48 and 32.65 (CH_2-2,3,4,5,6-cyclohex), 48.49 (CH-1-cyclohex), 62.16 (CH_2-5′), 71.56 (CH-3′), 77.95 (CH-2′), 82.85 (CH-1′), 85.39 (CH-4′), 125.93 (CH-2,6), 127.21 (CH-3,5), 134.06 (C-4), 144.67 (C-1), 165.45 (CO). IR spectrum (KBr): 3436, 3366, 3314, 3272, 2935, 2854, 1615, 1575, 1540, 1508, 1449, 1438, 1415, 1348, 1335, 1314, 1307, 1283, 1224, 1188, 1131, 1082, 1074, 1050, 1019, 839, 693, 635 cm^{-1}. $[\alpha]_D^{20}$ −27.1 (*c* 3.51, MeOH). Anal. Calcd for $C_{18}H_{25}NO_5 \cdot 1H_2O$: C, 61.17; H, 7.70; N, 3.96. Found: C, 61.15; H, 7.60; N, 3.94.

3.7.18. 1β-[4-(Benzylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6h**)

Compound **6h** was prepared from **5h** (334 mg, 0.487 mmol) according to general procedure, method A, in 93% yield, as a colourless oil, which after lyophylization furnished white powder, mp 151–153 °C. HRMS (ESI) for $C_{19}H_{20}NO_5$: [M−H] calculated, 342.1336; found, 342.1343. ^{1}H NMR (500.0 MHz, DMSO-d_6): 3.54 (ddd, 1H, J_{gem}=11.6, $J_{5'b,OH}$=5.5, $J_{5'b,4'}$=4.6, H-5′b), 3.58 (ddd, 1H, J_{gem}=11.6, $J_{5'a,OH}$=5.5, $J_{5'a,4'}$=4.4, H-5′a), 3.67 (ddd, 1H, $J_{2',1'}$=7.2, $J_{2',OH}$=7.0, $J_{2',3'}$=5.3, H-2′), 3.83 (ddd, 1H, $J_{4',5'}$=4.6, 4.4, $J_{4',3'}$=3.5, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.3, $J_{3',OH}$=4.9, $J_{3',4'}$=3.5, H-3′), 4.48 (d, 2H, J=6.2, CH_2Ph), 4.61 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.86 (t, 1H, $J_{OH,5'}$=5.5, OH-5′), 4.98 (d, 1H, $J_{OH,3'}$=4.9, OH-3′), 5.06 (d, 1H, $J_{OH,2'}$=7.0, OH-2′), 7.21–7.27 (m, 1H, H-*p*-Ph), 7.29–7.35 (m, 2H, H-*o*-Ph), 7.29–7.35 (m, 2H, H-*m*-Ph), 7.46–7.50 (m, 2H, H-2,6), 7.84–7.88 (m, 2H, H-3,5), 9.03 (t, 1H, J=6.2, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 42.77 (CH_2Ph), 62.19 (CH_2-5′), 71.64 (CH-3′), 78.02 (CH-2′), 82.76 (CH-1′), 85.49 (CH-4′), 126.18 (CH-2,6), 126.96 (CH-*p*-Ph), 127.24 (CH-3,5), 127.38 (CH-*o*-Ph), 128.53 (CH-*m*-Ph), 133.49 (C-4), 139.98 (C-*i*-Ph), 145.09 (C-1), 166.31 (CO). IR spectrum (KBr): 3440, 3324, 3264, 3088, 3063, 3030, 2938, 2885, 1637, 1614, 1587, 1571, 1535, 1505, 1496, 1454, 1422, 1318, 1309, 1286, 1210, 1120, 1080, 1046, 1041, 1030, 1017, 841, 699, 641, 616 cm^{-1}. $[\alpha]_D^{20}$ −29.0 (*c* 2.79, MeOH). Anal. Calcd for $C_{19}H_{21}NO_5 \cdot 0.2H_2O$: C, 65.77; H, 6.22; N, 4.04. Found: C, 65.69; H, 6.09; N, 3.95.

3.7.19. 1β-[4-(Methylcarbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6i**)

Compound **6i** was prepared from **5i** (205 mg, 0.336 mmol) according to general procedure, method A, in 90% yield, as a colourless oil, which after lyophylization furnished white hygroscopic powder. HRMS (ESI) for $C_{13}H_{18}NO_5$: [M+H] calculated, 268.1179; found, 268.1181. ^{1}H NMR (499.8 MHz, DMSO-d_6): 2.78 (d, 3H, J=4.6, CH_3), 3.54 and 3.57 (2×ddd, 2H, J_{gem}=11.7, $J_{5',OH}$=5.4, $J_{5',4'}$=4.5, H-5′), 3.67 (dd, 1H, $J_{2',1'}$=7.2, $J_{2',3'}$=5.3, H-2′), 3.83 (td, 1H, $J_{4',5'}$=4.5, $J_{4',3'}$=3.4, H-4′), 3.89 (dd, 1H, $J_{3',2'}$=5.3, $J_{3',4'}$=3.4, H-3′), 4.60 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.83 (t, 1H, $J_{OH,5'}$=5.4, OH-5′), 4.96 (br s, 1H, OH-3′), 5.03 (br s, 1H, OH-2′), 7.44–7.48 (m, 2H, H-2,6), 7.76–7.80 (m, 2H, H-3,5), 8.38 (q, 1H, J=4.6, NH). ^{13}C NMR (125.7 MHz, DMSO-d_6): 26.42 (CH_3), 62.18 (CH_2-5′), 71.63 (CH-3′), 77.91 (CH-2′), 82.75 (CH-1′), 85.47 (CH-4′), 126.12 (CH-2,6), 126.98 (CH-3,5), 133.69 (C-4), 144.73 (C-1), 166.70 (CO). IR spectrum (KBr): 3415, 3362, 3270, 3195, 1671, 1643, 1619, 1572, 1564, 1545, 1508, 1469, 1412, 1404, 1328, 1316, 1305, 1217, 1186, 1161, 1129, 1108, 1074, 1052, 1035, 1016, 836, 706 cm^{-1}. $[\alpha]_D^{20}$ −26.4 (*c* 3.46, MeOH). Anal. Calcd for $C_{13}H_{17}NO_5 \cdot 1H_2O$: C, 54.73; H, 6.71; N, 4.91. Found: C, 54.61; H, 6.52; N, 4.73.

3.7.20. 1β-[4-(Carbamoyl)phenyl]-1-deoxy-D-ribofuranose (**6j**)

Compound **6j** was prepared from **5j** (332 mg, 0.557 mmol) according to general procedure, method B, in 90% yield, as a colourless oil, which after lyophylization furnished white powder, mp 166–172 °C. HRMS (ESI) for $C_{12}H_{16}NO_5$: [M+H] calculated, 254.1023; found, 254.1023. ^{1}H NMR (499.8 MHz, DMSO-d_6): 3.54 (ddd, 1H, J_{gem}=11.6, $J_{5'b,OH}$=5.6, $J_{5'b,4'}$=4.6, H-5′b), 3.57 (ddd, 1H, J_{gem}=11.6, $J_{5'a,4'}$=5.6, $J_{5'a,4'}$=4.4, H-5′a), 3.67 (ddd, 1H, $J_{2',1'}$=7.2, $J_{2',OH}$=7.0, $J_{2',3'}$=5.3, H-2′), 3.83 (ddd, 1H, $J_{4',5'}$=4.6, 4.4, $J_{4',3'}$=3.4, H-4′), 3.89 (ddd, 1H, $J_{3',2'}$=5.3, $J_{3',OH}$=4.8, $J_{3',4'}$=3.4, H-3′), 4.61 (d, 1H, $J_{1',2'}$=7.2, H-1′), 4.82 (t, 1H, $J_{OH,5'}$=5.6, OH-5′), 4.93 (d, 1H, $J_{OH,3'}$=4.8, OH-3′), 5.01 (d, 1H, $J_{OH,2'}$=7.0, OH-2′), 7.31 (br s, 1H, NH_aH_b), 7.45 (m, 2H, H-2,6), 7.82 (m, 2H, H-3,5), 7.92 (br s, 1H, NH_aH_b). ^{13}C NMR (125.7 MHz, DMSO-d_6): 62.17 (CH_2-5′), 71.63 (CH-3′), 77.94 (CH-2′), 82.75 (CH-1′), 85.45 (CH-4′), 126.03 (CH-2,6), 127.41 (CH-3,5), 133.43 (C-4), 144.97 (C-1), 167.98 (CO). IR spectrum (KBr): 3442, 3349, 3288, 3238, 3199, 1649, 1614, 1599, 1568, 1513, 1418, 1394, 1309, 1211, 1189, 1131, 1100, 1080, 1052, 1016, 841, 830, 633 cm^{-1}. $[\alpha]_D^{20}$ −31.1 (*c* 2.80, MeOH). Anal. Calcd for $C_{12}H_{15}NO_5 \cdot 0.2H_2O$: C, 56.11; H, 6.04; N, 5.45. Found: C, 56.18; H, 6.01; N, 5.34.

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Supplementary data

Copies of the ^{1}H and ^{13}C NMR spectra of all new compounds. Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.tet.2009.04.006.

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