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RESEARCH ARTICLE

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The approach to the synthesis of novel amino-*C*-glycosides

Abstract Stereoselective 1,3-dipolar cycloadditions of *exo*-glycals **1** to nitrones **2**, **5** and **8** were investigated under the catalysis of Lewis acid or in a refluxing benzene or toluene solution, and afforded the corresponding cycloadducts of ketosyl spiro-isoxazolidines. The reductive cleavage of the N-O bond in the isoxazolidine ring and debenzylation by the catalytic hydrogenation [$Pd(OH)_2$/C] were approached using the glucose-type cycloadducts **6b** and **6e** to alkyl-*C*-glycoside derivatives **12**, providing a new access to a novel alkyl-*C*-glycoside containing an amino group on the side alkyl chain.

Keywords exo-glycal, *C*-glycoside, 1,3-dipolar cycloaddition, ketosyl spiro-isoxazolidine

1 Introduction

C-Glycosides are one of the important carbohydrate mimetics and have gained considerable attention in recent years because of their attractive biological activities as pharmaceutical targets and resistance to enzymatic degradation in vivo. Consequently, a large number of *C*-glycosides have been synthesized for investigating their bioactivity and for discovering new drugs, and various synthetic methodologies have been developed for instance, by anionic, cationic, free radical and rearrangement reactions, cycloaddition reaction, metalmediated reactions [1–18], as well as the cyclization reactions of open chain(s) in a monosaccharide as the exclusive approach to the *C*-saccharides [19].

exo-Glycal is a very important synthetic precursor, and has been widely used in the synthesis of *C*-glycosidic derivatives [20–27]. We have also developed convenient method for stereospecifically synthesizing α-ketosyl disaccharides by the stereoselective glycosiylation of *exo*-glycal [28–30]. As the continuation of applying *exo*-glycals to the synthesis of *C*-glycoside derivatives, we wish to report herein the approaches to the synthesis of novel aminoalkyl-*C*-glycoside derivatives via the key step of 1,3-dipolar cycloaddition (scheme 1, 2 and 3) using *exo*-glycals as the precursor.

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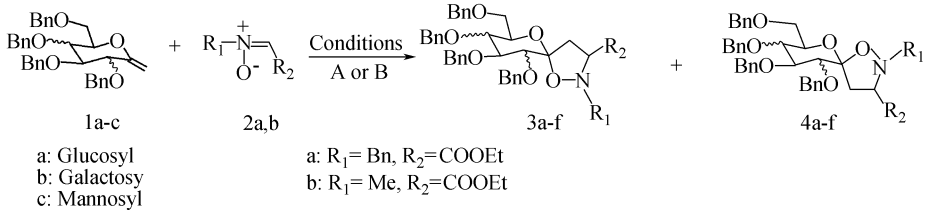
2 Results and discussion

The *exo*-glycals (**1**) were synthesized by the methylenation of their corresponding sugar lactones with Tebbe's reagent [31]. The nitrones (**2**), (**5**) and (**8**) were readily prepared by the reaction of the corresponding alkyl hydroxylamines and the aldehydes, respectively.

In order to examine the reaction condition, the 1,3-cycloaddition was firstly performed with *exo*-glycals (**1a–c**) and the nitrones (**2a–b**) in different conditions and provided the corresponding cycloadducts of spiro- isoxazolidine in mixtures of α,β-isomers (**3**) and (**4**) [32]. As shown in Table 1, under the catalysis of Lewis acid, $BF_3{\cdot}Et_2O$ (called condition **A**), the cycloaddition reactions could be regiospecifically carried out in low temperature and afforded a mixture of the corresponding diastereoselective ketosyl spiro-isooxazolidines **3** and **4** with the α-isomer (**3**) as the predominant. It should be mentioned that other Lewis acids, such as $ZnCl_2$, $ZnBr_2$, $AlCl_3$, $BF_3{\cdot}OEt_2$, $Yb(OTf)_3$, TsOH, Cp_2TiCl_2 and Et_2AlCl were found to be not effective for the reaction. Moreover, in the absence of the Lewis acid catalyst the reaction could not occur at low temperature, but in higher temperature (in a refluxing benzene solution, so-called condition **B**) the reaction could proceed efficiently with a considerable decrease of the α-stereoselectivity, and in some cases the β-selectivity was slightly prefered.

On the basis of the reaction of **1** and **2**, the cycloaddition reactions of **1a–c** and the carbohydrate nitrone (**5**) were investigated [33]. It was found that the reaction of **1a** and **5** did not go well at low temperature in the presence of Lewis acid (condition **A**) or in refluxing benzene (condition **B**).



Scheme 1 Cycloaddition of *exo*-glycals **1** and nitrones **2**

Reagents and reaction conditions: (A) $BF_3 \cdot OEt_2$ (1.25 eq), CH_2Cl_2, −78℃, 4 h; (B) benzene, reflux, 24 h

Table 1 1,3-Dipolar cycloadditions of the **1a–c** and nitrones **2a–b**

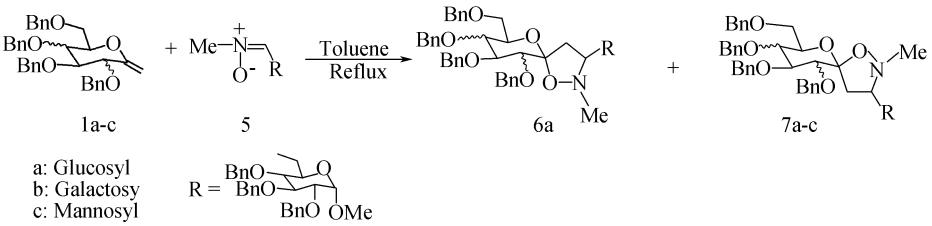
Entry	1	2	Conditions	3 (α-anomer)(%)		4 (β-anomer)(%)		α : β
1	**1a**	**2a**	A	**a**	71.9	**a**	6.4	11.3 : 1.0
2	**1b**	**2a**	A	**b**	72.6	**b**	4.9	14.8 : 1.0
3	**1c**	**2a**	A	**c**	58.0	**c**	10.8	5.4 : 1.0
4	**1a**	**2b**	A	**d**	70.1	**d**	7.7	9.1 : 1.0
5	**1b**	**2b**	A	**e**	68.6	**e**	7.8	8.8 : 1.0
6	**1c**	**2b**	A	**f**	53.7	**f**	28.6	1.9 : 1.0
7	**1a**	**2a**	B	**a**	43.4	**a**	52.3	1.0 : 1.2
8	**1b**	**2a**	B	**b**	31.0	**b**	31.5	1.0 : 1.0
9	**1c**	**2a**	B	**c**	23.4	**c**	42.1	1.0 : 1.8
10	**1a**	**2b**	B	**d**	41.0	**d**	41.4	1.0 : 1.0
11	**1b**	**2b**	B	**e**	37.5	**e**	41.3	1.0 : 1.1
12	**1c**	**2b**	B	**f**	12.3	**f**	38.2	1.0 : 3.1

Conditions: (A) *exo*-glycals (**1**) (0.2 mmol), nitrone (**2**) (0.25 mmol), BF3·OEt_2 (1.25 eq), CH_2Cl_2, −78℃, 4 h
(B) *exo*-glycals (**1**) (0.2 mmol), nitrone (**2**) (0.25 mmol), benzene, reflux, 24 h

But when the reaction was performed in refluxing toluene, it provided the corresponding cycloadducts, spiro- isoxazolidine disaccharides **6a** and **7a** (Scheme 2). However, in the cases of the galactose and mannose derivatives **1b** and **1c** only the O-β-anomeric isomers **7b** (53.2%) and **7c** (50.7%) were obtained in the reaction, respectively.

Under similar condition, the cycloaddition reactions of **1a,b** and **8a-d** were studied. After refluxing the toluene solution for certain times, the reaction provided the corresponding cycloadducts, ketosyl spiro-isoxazolidine **9** and **10** (scheme 3), respectively, and the results are listed in Table 2. Interestingly, the isomerization of *exo*-glucal (**1a**) to the corresponding glucal (**11**) was observed in the 1,3-dipolar cycloaddition of **1a** and **8**.

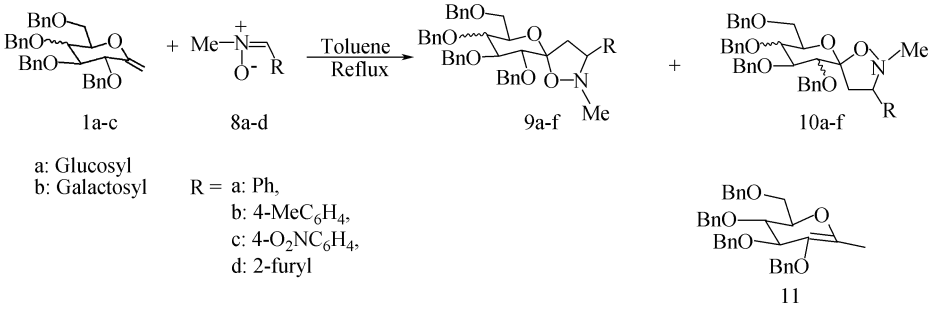
As shown in scheme 2 and Table 2, compared with that of **1** and **2** (in scheme 1 and Table 1) the cycloadditions of the *exo*-glycals (**1**) with nitrones (5) and (**8**) were not so efficient. The reactivity was slightly related to the property of the substituent on nitrones (5) and (**8**), that is, the electron-withdrawing group was beneficial to the reaction. It should be mentioned that Lewis acids, such as $ZnCl_2$, $ZnBr_2$, $AlCl_3$, $BF_3 \cdot OEt_2$, $Yb(OTf)_3$ were not effective to the reaction, even though it has been reported that some Lewis acids could efficiently promote the 1,3-dipolar cycloadditions of some nitrones and electron-rich alkenes such as enolic compounds in a stereoselective way [34-37]. On the other hand, the isomerization of *exo*-glucal (**1a**) to the corresponding glucal (**11**) may probably be one of the reasons that resulted in the low yields of the cycloaddition reactions due to the much low reactivity of (**11**) to the nitrone (**8**).



	6[a]	7[a]
a	17.4	52.1
b	-	53.2[b]
c	-	50.7[b]

[a]: Isolated yields; [b]: No α -isomer was obtained.

Scheme 2 Cycloaddition of *exo*-glycals **1** and nitrones **5**



Scheme 3 Cycloaddition of *exo*-glycals (**1**) and nitrones (**8**)

Table 2 1,3-dipolar cycloaddition of *exo*-glycals (**1**) with nitrones (**8**)

Entry	*Exo*-glycal (**1**)	Nitrone (**8**)	Time (h)	**9**[a]		**10**[a]
1	**1a**	**a** (R = Ph)	100	**a**	41.9 b	
2	**1a**	**b** (R = 4-$CH_3C_6H_4$)	100	**b**	28.9	34.6
3	**1a**	**c** (R = 4-O_2N C_6H_4)	40	**c**	52.4	43.7
4	**1a**	**d** (R = 2-Furyl)	80	**d**	39.1	43.0
5	**1b**	**b** (R = 4-$CH_3C_6H_4$)	80	**e**	79.6 [b]	
6	**1b**	**d** (R = 2-Furyl)	80	**f**	75.8 [b]	

[a]: Isolated yields
[b]: The α,β-isomers **9** and **10** could not be separated by silica gel column chromatography

The structures of cycloadducts **3**, **4**, **6**, **7**, **9** and **10** were determined by the analyses of their spectral data of ^{1}H-NMR, ^{13}C-NMR, 2D (H-H, C-H) COSY and NOESY experiments. It has been reported that for the *C*-glycosides [38], *C*-ketosides [28] and *C*-spiro-ketosides [30], their ^{13}C-NMR spectral signals of α-anomeric carbons appeared in the higher field than those of the β-anomeric carbons. Accordingly, the anomeric configurations of the compounds were established with the comparison of the chemical shifts of the anomeric carbons in the corresponding α- and β-anomers.

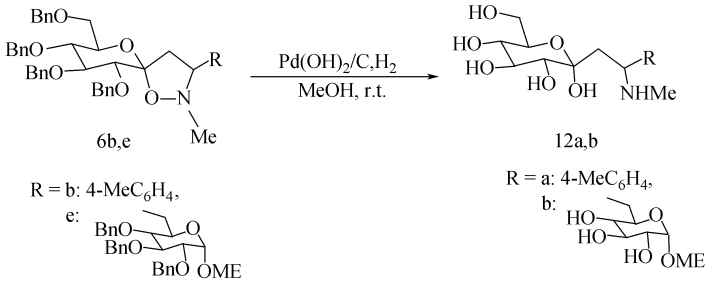
The reductive cleavage of the N-O bond of the cycloadducts, ketosyl spiro-isooxazolidines was approached by the palladium catalytic hydrogenation with **6b** and **6e** where the sugar moiety is glucose as shown in scheme 4. Under the catalysis of $Pd(OH)_2/C$ (20%) the cleavage of the O-N bond and debenzylation of the ketosyl spiro-isooxazolidines **6b** and **6e** were finished by one-step hydrogenation reaction and afforded the novel alkyl-*C*-glycoside derivatives having an amino group on the side chain (**12a**) and (**12b**) in yields of 45.6% and 51.6%, respectively. The structures of the products (**12**) were identified by the analysis of NMR and MS spectra.

In summary, we have described a stereoselective 1,3-dipolar cycloaddition of *exo*-glycal and nitrones, which provided a new kind of spiro-isoxazolidinic ketosides in a mixture of α,β-anomeric isomers. The reductive cleavage of the O–N bond of the cycloadducts, glucose type products (**6b**) and (**6b**), was approached by $Pd(OH)_2/C$ catalytic hydrogenation, affording the corresponding novel aminoalkyl-*C*-glycosides (**12a**) and (**12b**), respectively, which is a new access to alkyl-*C*-glycoside derivatives. A further investigation of applying the ketosyl spiro-isoxazolidines to the synthesis of novel *C*-glycoside derivatives is under way in this laboratory.

3 Experimental

3.1 General methods

Melting points were measured on a micro melting point apparatus and were uncorrected. ^{1}H-NMR, ^{13}C-NMR and COSY spectra were measured on a FT-NMR Bruker AVANCE



Scheme 4 Catalytic hydrogenation of the ketosyl spiro-isooxazolidines **6a** and **6e**

400 (400 MHz) NMR spectrometer using tetramethylsilane (Me_4Si) as an internal standard. Mass spectra (MS) were carried out on a VG-7070E mass spectrometer with FAB (fast atomic bombardment) using 3-nitrobenzyl alcohol (NBA) as the matrix. Thin-layer chromatography (TLC) was performed on precoated plates (Qingdao GF_{254}) with detection by UV light or with phosphomolybdic acid in $EtOH/H_2O$ followed by heating. Column chromatography was performed using SiO_2 (Qingdao 300~400 mesh).

3.2 General procedure for the 1,3-dipolar cycloaddition of **1** and **2** [32]

Under the catalysis of Lewis acid in low temperature (condition A): $BF_3{\cdot}Et_2O$ (32 μL, 0.25 mmol, 1.25 equiv.) was added to a solution of **1a** (107 mg, 0.2 mmol), **2**a (52 mg, 0.25 mmol) and Molecular sieves 4A (MS 4A, 300 mg) in 4 mL of CH_2Cl_2 under argon atmosphere at −78℃. The solution was stirred at the same temperature for 4 h, then the reaction temperature was gradually increased up to 0℃, then, triethylamine (0.1 mol) was added to quench the reaction. The solvent was removed under reduced pressure, and the residue was applied on silica gel column chromatography using AcOEt:Hexane (1:5) as the eluent to produce the corresponding cycloadducts **3** (107.6 mg, 71.9%) and **4** (9.5 mg, 6.4%).

In refluxing benzene without Lewis acid (condition B): A solution of **1a** (107 mg, 0.2 mmol) and **2** (52 mg, 0.25 mmol) in 5 mL of benzene was refluxed under argon for 24 h. The reaction was monitored by TLC (AcOEt : Hexane = 1 : 2). After completion, the solvent was removed under reduced pressure. The residue was submitted to silica gel column chromatography using AcOEt:Hexane (1 : 5) as the eluent to produce the corresponding cycloadducts **3** (64.5 mg, 43.4%) and **4** (77.8 mg, 52.3%).

Under the same conditions of (A**)** and (B), the 1,3-dipolar cycloadditions of **1b**–**c** with **2a**, and **1a–c** with **2b** were carried out and afforded the corresponding products of **3** and **4**. The results are listed in Table 1.

3.3 General procedure for the 1,3-dipolar cycloaddition of **1** and **8** [33]

A solution of **1a** (322.0 mg, 0.60 mmol) and **8b** (134.3 mg, 0.90 mmol) in 15 mL of dry toluene was refluxed under N_2 atmosphere for 100 h. The reaction was monitored by TLC (AcOEt:Petrolum ether = 1:4). After the reaction completed, the solvent was removed under reduced pressure, and the residue was applied on silica gel column chromatography using AcOEt:Petrolum ether (1:6) as the eluent to afford the corresponding cycloadducts **9b** (118.9 mg, 28.9%), **10b** (143.4 mg, 34.6%), and the isomerized by-product **11** (glucal) (72.6 mg, 22.5%).

With the same procedure, cycloadditions of **1** with **8** were carried out and produced the corresponding products **9** and **10**. The results are shown in Table 2.

3.4 The hydrogenation of the cycloadduct **6** by the catalysis of [$Pd(OH)_2$/C]

The cycloadduct **6b** (137.2 mg, 0.2 mmol) was dissolved in 5 mL of methanol. Twenty milligrams of $Pd(OH)_2$/C (20 wt %)was added to the solution , and the mixture was stirred vigorously under H_2 atmosphere at room temperature for 12 h. After the reaction completed, the catalyst was removed by filtration through a Celite pad and the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography (AcOEt:MeOH=1:1) to produce the corresponding debenzylated product **12a** (29.8 mg, 45.6%).

Following the same procedure, the catalytic hydrogenation of cycloadducts **6e** was carried out and gave the product of **12b** in the yield of 51.6%.

3.5 The spectral data of some new compounds

9b: Colorless syrup; ^{1}H-NMR ($CDCl_3$) δ: 2.34 (t, 1H, J = 11 Hz, 2-H), 2.36 (s, 3H, CH_3), 2.55 (dd, 1H, J = 5 Hz, J = 8 Hz, 2-H), 2.62 (s, 3H, NCH_3), 3.60 (d, 1H, J = 10 Hz, 4-H), 3.76 (t, 1H, J = 10 Hz, 6-H), 3.83–3.91 (m, 3H, 1-H, 8-H, 8-H), 4.03 (t, 1H, J = 10 Hz, 5-H), 4.08–4.11 (m, 1H, 7-H), 4.56 (d, 1H, J = 12 Hz, CH_2Ph), 4.59 (d, 1H, J = 11 Hz, CH_2Ph), 4.64 (d, 1H, J = 12 Hz, CH_2Ph), 4.70 (d, 1H, J = 12 Hz, CH_2Ph), 4.78–4.82 (m, 2H, CH_2Ph), 4.98 (s, 2H, CH_2Ph), 5.06 (d, 1H, J=11 Hz, CH_2Ph), 7.10–7.20 (m, 4H, CH_3-ArH), 7.32–7.41 (m, 20H, ArH); ^{13}C-NMR ($CDCl_3$) δ: 21.54 (CH_3), 43.45 (2-C), 49.45 (NCH_3), 68.78 (8-C), 71.62 (7-C), 72.58 (1-C), 73.83 (CH_2Ph), 75.36 (CH_2Ph), 75.83 (CH_2Ph), 75.93 (CH_2Ph), 78.60 (6-C), 80.76 (4-C), 84.46 (5-C), 104.94 (3-C), and aromatic carbons; FAB-MS (m/z): 687 $(M+H)^+$.

10b: Colorless syrup; ^{1}H-NMR ($CDCl_3$)δ: 2.35 (s, 3H, CH_3), 2.39 (dd, 1H, J = 11 Hz, J = 7 Hz, 2-H), 2.49 (dd, 1H, J = 11 Hz, J = 7 Hz, 2-H), 2.79 (s, 3H, NCH_3), 3.58 (d, 1H, J = 10 Hz, 4-H), 3.73–3.78 (m, 2H, 6-H, 8-H), 3.85 (dd, 1H, J = 7 Hz, J = 3 Hz, 8-H), 4.03 (t, 1H, J = 10 Hz, 5-H), 4.05 (d, 1H, J = 8 Hz, 7-H), 4.09–4.13 (m, 1H, 1-H), 4.54 (d, 1H, J = 12 Hz, CH_2Ph), 4.57 (d, 1H, J = 11 Hz, CH_2Ph), 4.61 (d, 1H, J = 12 Hz, CH_2Ph), 4.68 (d, 1H, J = 10 Hz, CH_2Ph), 4.87 (d, 1H, J = 12 Hz, CH_2Ph), 4.91 (d, 1H, J = 11 Hz, CH_2Ph), 4.97–5.03 (m, 2H, CH_2Ph), 7.11–7.23 (m, 4H, CH_3-ArH), 7.30–7.42 (m, 20H, ArH); ^{13}C-NMR ($CDCl_3$) δ: 21.54 (CH_3), 38.21 (2-C), 47.11 (NCH_3), 69.04 (8-C), 71.15 (7-C), 72.58 (1-C), 73.85 (CH_2Ph), 75.42 (CH_2Ph), 75.77 (CH_2Ph), 76.05 (CH_2Ph), 78.61 (6-C), 79.26 (4-C), 84.58 (5-C), 106.18 (3-C), and aromatic carbons; FAB-MS (m/z): 687 $(M+H)^+$.

9c: Colorless syrup; ^{1}H-NMR ($CDCl_3$) δ: 2.28 (t, 1H, J = 11 Hz), 2.53 (dd, 1H, J = 5 Hz, J = 8 Hz), 2.63 (s, 3H, NCH_3), 3.58 (d, 1H, J = 10 Hz), 3.65 (t, 1H, J = 9 Hz),

3.78–3.84 (m, 3H), 4.01 (t, 1H, J = 10 Hz), 4.07 (d, 1H, J = 10 Hz), 4.57 (d, 1H, J = 12 Hz), 4.61 (d, 1H, J = 11 Hz), 4.66 (d, 1H, J = 12 Hz), 4.73 (d, 1H, J = 12 Hz), 4.88 (d, 1H, J = 11 Hz), 4.95 (s, 2H), 5.08 (d, 1H, J = 11 Hz), 7.20–7.40 (m, 20H), 7.52 (d, 2H, J = 9 Hz), 8.17 (d, 2H, J = 8 Hz); ^{13}C-NMR ($CDCl_3$) δ: 43.84, 49.09, 68.83, 72.52, 73.06, 73.82, 75.35, 75.88, 75.95, 78.62, 80.38, 84.44, 105.26, and aromatic carbons; FAB-MS (m/z): 718 $(M+H)^+$.

10c: Colorless syrup; ^{1}H-NMR ($CDCl_3$)δ: 2.36–2.48 (m, 2H), 2.83 (s, 3H, NCH_3), 3.55 (d, 1H, J = 10 Hz), 3.70–3.75 (m, 2H), 3.81 (dd, 1H, J = 7 Hz, J = 3 Hz), 4.02 (t, 1H, J = 10 Hz), 4.09 (d, 1H, J = 9 Hz), 4.18 (t, 1H, J = 8 Hz), 4.54–4.61 (m, 3H), 4.67 (d, 1H, J = 13 Hz), 4.87 (d, 1H, J = 10 Hz), 4.91–5.00 (m, 3H), 7.19–7.37 (m, 20H), 7.54 (d, 2H, J = 8 Hz), 8.14 (d, 2H, J = 8 Hz); ^{13}C-NMR ($CDCl_3$) δ: 37.86, 47.35, 69.05, 70.02, 72.00, 73.85, 75.43, 75.93, 76.08, 78.56, 79.08, 84.52, 107.02, and aromatic carbons; FAB-MS (m/z): 718 $(M+H)^+$.

9d: Colorless syrup; ^{1}H-NMR ($CDCl_3$) δ: 2.31 (t, 1H, J = 12 Hz, 2-H), 2.45 (dd, 1H, J = 4 Hz, J = 9 Hz, 2-H), 2.75 (s, 3H, NCH_3), 3.62 (d, 1H, J = 9 Hz, 4-H), 3.70 (t, 1H, J = 10 Hz, 6-H), 3.76 (d, 1H, J = 9 Hz, 8-H), 3.82 (dd, 1H, J = 9 Hz, J = 4 Hz, 8-H), 3.89–3.93 (m, 1H, 1-H), 4.02 (t, 1H, J = 9 Hz, 5-H), 4.07–4.09 (m, 1H, 7-H), 4.57 (d, 1H, J = 12 Hz, CH_2Ph), 4.63 (d, 1H, J = 11 Hz, CH_2Ph), 4.73 (d, 1H, J = 12 Hz, CH_2Ph), 4.87–4.91 (m, 2H, CH_2Ph), 4.93 (d, 1H, J = 12 Hz, CH_2Ph), 4.96 (d, 1H, J=12 Hz, CH_2Ph), 5.09 (d, 1H, J = 11 Hz, CH_2Ph), 6.24 (d, 1H, J = 8 Hz, furyl), 6.31–6.39 (m, 1H, furyl), 7.21–7.43 (m, 21H, ArH); ^{13}C-NMR ($CDCl_3$) δ: 43.93 (2-C), 45.08 (NCH_3), 68.79 (8-C), 72.81 (7-C), 73.02 (1-C), 73.86 (CH_2Ph), 75.35 (CH_2Ph), 75.90 (CH_2Ph), 75.98 (CH_2Ph), 78.67 (6-C), 80.63 (4-C), 84.33 (5-C), 105.99 (3-C), and aromatic carbons; FAB-MS (m/z): 663 $(M+H)^+$.

10d: Colorless syrup; ^{1}H-NMR ($CDCl_3$)δ: 2.36 (dd, 1H, J = 11 Hz, J = 7 Hz, 2-H), 2.51 (dd, 1H, J = 12 Hz, J = 7 Hz, 2-H), 2.88 (s, 3H, NCH_3), 3.60 (d, 1H, J = 10 Hz, 4-H), 3.76 (t, 1H, J = 10 Hz, 6-H), 3.78–3.87 (m, 2H, 8-H, 8-H), 3.94 (t, 1H, J = 10 Hz, 5-H), 4.06–4.10 (m, 1H, 1-H), 4.01 (d, 1H, J = 8 Hz, 7-H), 4.54–4.63 (m, 3H, CH_2Ph), 4.67 (d, 1H, J = 11 Hz, CH_2Ph), 4.87 (d, 1H, J = 11 Hz, CH_2Ph), 4.90 (d, 1H, J = 12 Hz, CH_2Ph), 4.96 (d, 1H, J = 12 Hz, CH_2Ph), 5.06 (d, 1H, J = 11 Hz, CH_2Ph), 6.21 (d, 1H, J = 7 Hz, furyl), 6.29~6.38 (m, 1H, furyl), 7.20–7.42 (m, 21H, ArH); ^{13}C-NMR ($CDCl_3$) δ: 43.01 (2-H), 48.88 (NCH_3), 69.09 (8-C), 70.93 (7-C), 71.80 (1-C), 73.95 (CH_2Ph), 75.69 (CH_2Ph), 75.79 (CH_2Ph), 75.98 (CH_2Ph), 78.57 (6-C), 79.04 (4-C), 84.38 (5-C), 108.04 (3-C), and aromatic carbons; FAB-MS (m/z): 663 $(M+H)^+$.

12a: Colorless amorphous solid; ^{1}H-NMR ($CDCl_3$) δ: 2.08–2.13 (m, 2H), 2.25 (s, br., 1H, OH), 2.33 (s, 3H, CH_3), 2.36 (s, br., 1H, NH), 2.76 (s, 3H, NCH_3), 3.54–3.61 (m 4H), 3.76–3.93 (m, 3H), 7.19–7.34 (m, 4H); ^{13}C-NMR ($CDCl_3$) δ: 23.39, 36.06, 41.82, 49.39, 60.86, 71.68, 72.19, 75.23, 78.82, 97.60, 128.12, 128.19, 128.88, 128.93, 136.36, 138.25; FAB-MS (m/z): 328 $(M+H)^+$.

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