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Synthesis of Heteroaromatic *N*-β-Glycosides of *N*-Acetylglucosamine under the Conditions of Phase Transfer Catalysis: I. Glucosaminides of 2-Oxobenzazoles

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Abstract—Glycosylation of benzothiazolone-2, 5-methylbenzoxazolone-2, and benzothiazolone-2 with the full acetate of α-*D*-glucosaminyl chloride in the phase transfer systems investigated (solid–organic solvent and aqueous alkali–organic solvent) regioselectively leads to the corresponding *N*-β-*D*-glucosaminides, which is proved by ^{1}H NMR spectroscopy and X-ray analysis.

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Key words: amide–imide tautomers, crown ethers, N-β-glycosylation, phase transfer catalysis, quaternary ammonium salts, regioselectivity, X-ray analysis

INTRODUCTION

We have established [1] that, under phase transfer conditions (solid–organic solvent), thiol–thione pairs of mercapto derivatives of 1,3,4-oxadiazole and 1,2,4-triazole are easily glucosaminated in the presence of crown ethers with 2-acetamido-3,4,6-tri-*O*-acetyl-2-deoxy-α-*D*-glucopyranosyl chloride (**I**) to form a mixture of the corresponding *S*- and *N*-β-glucosaminides.[2] At the same time, 5-sulfonylbenzoxazol–2-thiol, benzothiazole-2-thiol, and 3-ethylquinazoline-2-thiol regioselectively form only *S*-β-glucosaminides under similar conditions [2].

There is no literature information on the selectivity of PTC glycosylation of heterocyclic compounds with *N*-acetylglucosamine derivatives that can undergo lactim–lactam (amide–imidol) tautomeric transformations [3]. It is only known that some glycosyl donors (neutral carbohydrates) selectively glycosylate derivatives of (*2H*) phthalazinone-1 at nitrogen or oxygen atoms and the selectivity of process depends on its conditions and the nature of the substituent in position 4 of phthalazine cycle [4, 5]. For example, the glycosylation of 4-chloro- and 4-phenyl-(*2H*)-phthalazinone-1 silver salts with acetobromoglucose leads to the corresponding *O*-β-glucosides, while (*2H*)-phthalazinone-1 does not enter the reaction discussed. In the case of similar sodium (potassium) derivatives, only 4-phenyl-(*2H*)-phthalazinone-1 reacts in aqueous acetone is possible to give *N*-β-glucoside; however, *O*-β-glucoside is not formed at all [4, 5].

We continued the search for new effective and selective phase transfer glycosylation reactions [1, 2, 6–8] of heterocyclic compounds and tried the glucosaminylation with α-chloride (**I**) of some amide–imidol tautomers [3] [namely, benzoxazolone-2 (**II**), 5-methylbenzoxazolone-2 (**III**), and benzothiazolone-2 (**IV**)] (scheme) in various phase transfer systems (25°C) in the presence of CE catalysts and TEBAC.

RESULTS AND DISCUSSION

The stoichiometric glucosamination of azoles (**II**)–(**IV**) according our method A [1, 2, 7, 8] was carried out in dry acetonitrile in the presence of anhydrous potassium carbonate and catalytic amounts of 15C5, B18C6, or DB18C6. A mixture of excess glycosyl acceptor was stirred with α-chloride (**I**) when the reaction was carried out in biphasic system aqueous alkali–chloroform (method B according to [9]); potassium hydroxide

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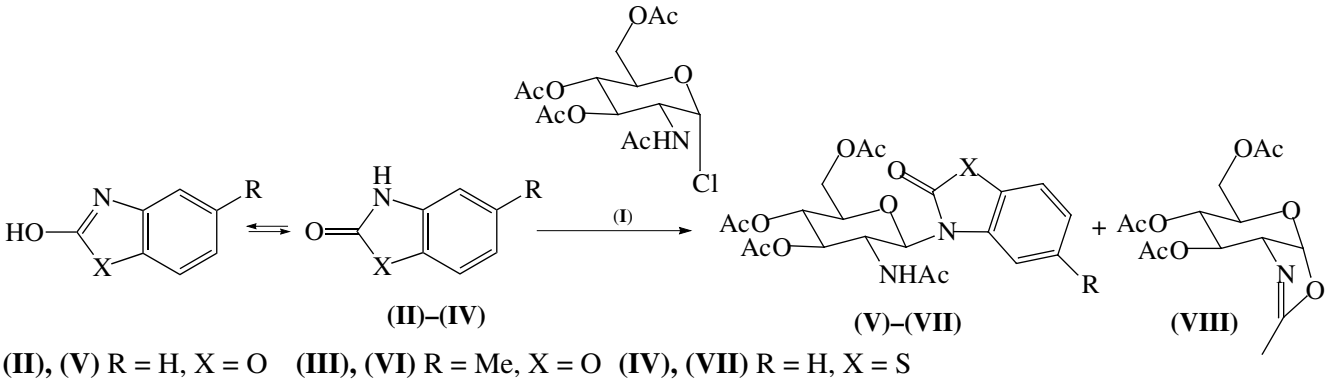
[2] Abbreviations: CE, crown ether; 15C5, 15-crown-5; 18C6, 18-crown-6; DB18C6, dibenzo-18-crown-6; PTC, phase transfer catalysis; and TEBAC, triethylbenzylammonium chloride.



Scheme of synthesis of 2-oxobenzazole *N*-β-glucosaminides (**V**)–(**VII**).

served as a base and OEBAC or 15C5, as a phase transfer catalyst. The method C consisted in the glycosylation of the azoles in the presence of sodium hydride suspension in mineral oil, the process being carried out as in method A (see the Experimental section).

The composition of reaction mixture was monitored by TLC, while determining the time of full conversion of glycosyl donor (**I**). The products (**V**)–(**VII**) were isolated in pure state by column chromatography and investigated with the help of ^{1}H NMR spectroscopy and X-ray analysis. The conditions of the phase transfer reactions and the yields of target products are listed in Table 1.

We found that, under the conditions described above, the glycosylation reactions by the methods A–C regioselectively proceed and exclusively lead to *N*-β-glycosaminides of 2-oxobenzazoles (**V**)–(**VII**). Significant amounts of 2-methyl-(3,4,6-tri-*O*-acetyl-1,2-dideoxy-α-*D*-glucopyrano)-[1,2-*d*]-2-oxozoline (**VIII**), a known [1, 7] side product, are also formed. We identified (**VIII**) with the help of independent sample by TLC only in the reaction mixtures, because it is unstable and decomposes

Table 1. The conditions of obtaining *N*-β-glucosaminides (**V**)–(**VII**)*

Glycoside	Base/method	Azole–α-chloride (**I**)–base molar ratio	Catalyst, mol %	Reaction time, h	Yield, %
(**V**)	K_2CO_3	1 : 1 : 1	–	10	31
(**V**)	K_2CO_3	1 : 1 : 4.5	–	6.5	38
(**V**)	K_2CO_3/A	1 : 1 : 1	15C5, 20	4.5	61
(**V**)	K_2CO_3/A	1 : 1 : 1	B18C6, 20	9	48
(**V**)	K_2CO_3/A	1 : 1 : 1	DB18C6, 20	9	43
(**V**)	K_2CO_3/A	1 : 1 : 4.5	15C5, 20	4	64
(**V**)**	KOH/B	2 : 1 : 1.85	TEBAC, 80	5.5	46
(**V**)**	KOH/B	2 : 1 : 1.85	15C5, 80	7	43
(**V**)	NaH/C	1 : 1 : 1.1	15C5, 20	7.5	55
(**VI**)	K_2CO_3	1 : 1 : 1	–	11	28
(**VI**)	K_2CO_3	1 : 1 : 4.5	–	7.5	33
(**VI**)	K_2CO_3/A	1 : 1 : 1	15C5, 20	4.5	58
(**VI**)	K_2CO_3/A	1 : 1 : 4.5	15C5, 20	4	62
(**VI**)	NaH/C	1 : 1 : 1.1	15C5, 20	7	57
(**VI**)**	KOH/B	2 : 1 : 1.85	TEBAC, 80	5	45
(**VI**)**	KOH/B	2 : 1 : 1.85	15C5, 80	7	46
(**VII**)	K_2CO_3/A	1 : 1	15C5, 20	3	52
(**VII**)	K_2CO_3/A	1 : 4.5	15C5, 20	2	62

* Unless otherwise stated, solvent is acetonitrile; the reaction time corresponds to the full conversion of chloride (**I**) (TLC monitoring).
** Solvent is $CHCl_3$.

Table 2. Proton chemical shifts in ^{1}H NMR spectra of compounds (**V**)–(**VII**)

Group or atom	(**V**)	(**VI**)	(**VII**)
H1 ($J_{1,2}$)	5.66 d (9.3)	5.65 d (9.9)	5.87 d (9.9)
H2 ($J_{2,3}$)	4.58 ddd (9.6)	4.55 ddd (9.6)	4.69 ddd (9.5)
H3 ($J_{3,4}$)	5.37 dd (9.9)	5.37 dd (9.8)	5.35 dd (9.3)
H4 ($J_{4,5}$)	5.28 dd (9.5)	5.23 dd (8.7)	5.26 dd (9.0)
H5, H6	4.18 m	4.16 m	4.18 m
NHAc	1.63 s	1.64 s	1.63 s
OAc	1.94 s, 2.02 s 2.03 s	1.94 s, 2.02 s 2.04 s	1.92 s, 2.02 s 2.04 s
NH ($J_{NH,2}$)	8.09 d (9.3)	8.07 d (9.0)	8.05 d (9.0)
Alk	–	2.38 s	–
CH_{arom}	7.20 m, 7.35 d, 7.59 d	6.97 d, 7.21 d 7.41 s	7.21 t, 7.37 t 7.62 d, 7.73 d

under the conditions of column chromatography. No corresponding products of *O*-alkylation were found.

It was established (Table 1) that the use of 15C5 in the reaction by method A provides a minimum time of reaction (2–4.5 h) and the best yields (52–64%) of *N*-glycosides (**V**)–(**VII**). Under the experimentally found optimum conditions at stoichiometric ratio of reagents, the conversion time of the substrate and yields of products (**V**)–(**VII**) are insignificantly changed even at increased (by 4.5 times) amount of potassium carbonate. The substitution of B18C6 or DB18C6 for 15C5 twofold reduces the glycosylation speed and by 20% the yield of glycoside (**V**) (Table 1). In the absence of CE, the yields of target products appreciably decrease and the reaction times increase.

The use of excess azole, aqueous potassium hydroxide and almost stoichiometric quantities of OEBAC (80 mol. % relative to α-chloride) (method B, Table 1) results (in comparison with method A) in a decrease in yields of (**V**) and (**VI**) by 20%. The substitution of 15C5 for OEBAC does not affect the yields of target products (**V**) and (**VI**); however, the conversion time of α-chloride (**I**) increases, which agrees with the known ideas o about the catalytic properties of quaternary ammonium salts and CEs in such phase transfer systems [10–13].

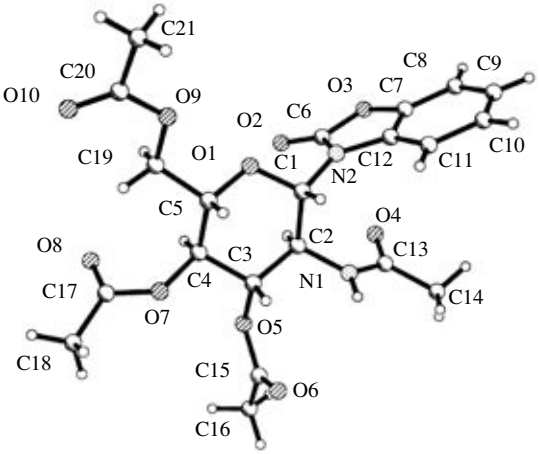
Glycosides (**V**) and (**VI**) were obtained by method C in the yields similar to those achieved by method A; however, the reaction times were much longer (Table 1).

The structures of compounds synthesized were proved by the methods of ^{1}H NMR spectroscopy and X-ray analysis. The chemical shifts and spin–spin coupling constants of anomeric protons in the ^{1}H NMR spectra of the isolated individual compounds (**V**)–(**VII**) (δ 5.65–5.87 ppm, $J_{1,2}$ 9.3–9.9 Hz), as well as of other skeletal protons (Table 2) close to those we earlier described for *N*-glycosides [1] allow the presumption that these are *N*-β-*D*-glucosaminides rather than the corresponding *O*-glycosides. Nevertheless, this conclusion undoubtedly needs an unequivocal proof.

To this end, we carried out an X-ray study of (**V**) (the figure and Tables 3–5), which, as expected, turned out to be an *N*-β-*D*-glucosaminide.

The coordinates of atoms, bond lengths, and torsion angles in full acetate (**V**) are given in Tables 3–5. In a crystal of compound (**V**), the six-membered pyranose cycle is in conformation $^4C_1$ (chair, the folding parameters: $S = 1.14$, $\Theta = 4.9°$, $\Psi = 9.7°$ [14]). All the substituents in the cycle are in equatorial conformation. Note the difference in lengths of bonds C_{sp3}–O in the carbohydrate residue (O1–C5 1.431 (6) Å and O1–C1 1.399 (5) Å), which is a consequence of the anomeric effect; this is described for related compounds (cf., e.g., [15, 16]).

Thus, we established that the glucosaminylation of azoles (**II**)–(**IV**) under the conditions of the investigated phase transfer processes, the most effective and convenient of which is method A, regioselectively leads to the corresponding *N*-β-glycosides. We have compared for the first time the data of X-ray analysis and ^{1}H NMR spectroscopy of glycosylation reaction by the example of (**V**) and found that, in our opinion, NMR allows the use of chemical shifts and spin coupling constants of protons to reveal and unequivocally identify the products of *N*-β-glycosylation of such type.



Molecular structure of *N*-β-glucosaminide (**V**)

EXPERIMENTAL

Melting points were determined on a PTP-1 device, and optical rotation at 20–22°C, on a Polamat-A polarimeter at λ 546 nm. ^{1}H NMR spectra were obtained on a Varian VXR-300 (300 MHz) instrument for solutions in DMSO-d_6 with tetramethylsilane as internal standard. TLC was carried out on precoated plates Sorbfil-AFB-UV (Sorbpolymer, Russia). Substance spots were

detected by spraying the plates with 2% sulfuric acid solution in *n*-butanol followed by heating at 200–300°C. We used 10 : 1 benzene–ethanol solvent system. Kieselgel 60 (0.063–0.200 mm, Merck) was used for the separation of substances by column chromatography. The results of elemental analysis of the compounds synthesized corresponded to the calculated values.

Benzoxazol-2-one, 5-methylbenzoxazol-2-one, and benzothiazol-2-one were obtained by the procedure [18]. The reference compound (**VIII**) was synthesized by the Lemieux method [19]. TEBAC (99%) was from Aldrich; crown ethers 15C5 (98%), B18C6 (98%), and DB18C6 (98%) were from the Bogatsky Physicochemical Institute of the National Academy of Sciences of Ukraine).

Acetonitrile was boiled with P_2O_5, fractioned, boiled over freshly calcined potash, distilled, and the distillate was fractioned using a Vigreaux column. Dry K_2CO_3 was obtained by its heating for 5 h at 340–360°C.

X-Ray analysis. Crystals of (**V**) are rhombic, $C_{21}H_{24}N_2O_{10}$, at –109°C $a = 6.305(1)$, $b = 13.778(4)$, $c = 25.804(5)$ Å, $V = 2241.7(9)$ Å^3, $M_r = 464.42$, $Z = 4$, spatial group $P2_12_12_1$; $d_{calc} = 1.376$ g/cm^3; $\mu(MoK_\alpha) = 0.111$ mm^{-1}; F (000) = 976. Parameters of elementary cell and intensity of 2294 independent reflections were measured on an automatic four-circle Siemens P3/PC X-ray diffractometer (MoK_α, graphite monochromator, $\theta/2\theta$ scanning, $2\theta_{max} = 50°$). The structure was computed by a direct method on a SHELXTL set of programs [17]. Positions of hydrogen atoms were revealed from differential synthesis of electronic density and refined using a rider model with $U_{iso} = nU_{equiv}$ of no hydrogen atom connected with the given hydrogen (n = 1.5 for methyl groups and n = 1.2 for other hydrogen atoms). The structure was refined according to squares of structural amplitudes, by a full matrix method of the least squares in anisotropic approximation for nonhydrogen atoms (up to $wR_2 = 0.108$) using 2219 reflections ($R_1 = 0.046$ from 1295 reflections with $F > 4\sigma(F)$, S = 0.931). Final coordinates of atoms are given in Table 3, lengths of bonds, in Table 4, and valent angles, in Table 5.

Table 3. Coordinates ($\times 10^4$, Å) and (in parentheses) equivalent isotropic thermal parameters ($\times 10^3$, Å^2 of atoms in a crystal of (**V**)

Atom	*x*	*y*	*z*	*U*(eq)
N1	3215 (7)	11942 (3)	1754 (2)	26 (1)
N2	6110 (6)	11927 (3)	851 (2)	19 (1)
O1	5645 (5)	10281 (2)	812 (1)	25 (1)
O2	9243 (6)	11402 (3)	1243 (2)	44 (1)
O3	8943 (6)	12892 (3)	868 (2)	37 (1)
O4	6032 (6)	12928 (3)	1909 (1)	38 (1)
O5	3529 (5)	10117 (2)	2315 (1)	26 (1)
O6	–9 (7)	10299 (3)	2398 (2)	52 (1)
O7	3121 (6)	8494 (2)	1663 (1)	29 (1)
O8	5991 (9)	7560 (3)	1804 (2)	58 (1)
O9	5405 (7)	8744 (2)	124 (1)	34 (1)
O10	5657 (7)	7144 (3)	–10 (2)	42 (1)
C1	4746 (8)	11137 (3)	1003 (2)	22 (1)
C2	4447 (9)	11118 (3)	1591 (2)	22 (1)
C3	3364 (9)	10186 (4)	1765 (2)	25 (1)
C4	4419 (9)	9306 (3)	1524 (2)	22 (1)
C5	4472 (9)	9425 (3)	939 (2)	27 (1)
C6	8169 (8)	12000 (4)	1019 (2)	30 (1)
C7	7337 (9)	13370 (4)	628 (2)	27 (1)
C8	7372 (12)	14293 (4)	419 (2)	43 (2)
C9	5540 (11)	14621 (4)	207 (2)	43 (2)
C10	3729 (11)	14074 (4)	194 (2)	42 (2)
C11	3674 (9)	13128 (4)	405 (2)	27 (1)
C12	5537 (8)	12792 (3)	615 (2)	22 (1)
C13	4120 (10)	12806 (4)	1891 (2)	33 (1)
C14	2592 (11)	13595 (4)	2000 (3)	52 (2)
C15	1691 (10)	10207 (4)	2594 (2)	33 (1)
C16	2142 (11)	10168 (4)	3160 (2)	40 (2)
C17	4092 (13)	7660 (4)	1823 (2)	39 (2)
C18	2537 (13)	6963 (4)	2035 (2)	56 (2)
C19	5543 (10)	8596 (3)	670 (2)	31 (1)
C20	5454 (8)	7942 (4)	–179 (2)	29 (1)
C21	5292 (13)	8200 (4)	–733 (2)	53 (2)

Method A. The corresponding azole (**II**)–(**IV**) (1.37 mmol), finely ground anhydrous K_2CO_3 (0.85 g, 6.17 mmol), and 15C5 (0.054 ml, 0.274 mmol) were added to a solution of chloride (**I**) [20] (0.5 g, 1.37 mmol) in dry acetonitrile (30 ml); and the mixture was stirred at room temperature until the full conversion of chloride (**I**) (TLC). Solids were filtered off; the filtrate was evaporated to dryness at a reduced pressure; and the products (**V**)–(**VII**) were isolated by a column chromatography.

Method B. A mixture of chloride (**I**) (1.25 g, 3.42 mmol), azole (**II**) or (**III**) (6.84 mmol), TEBAC (0.62 g, 2.73 mmol), chloroform (12.5 ml), and 1.25 N KOH (5 ml, water solution) were stirred at room temperature until the full conversion of chloride (**I**) (TLC). The organic layer was separated; washed with 1 N KOH (5 ml) and water (2 × 10 ml); dried with anhydrous Na_2SO_4; filtered; and evaporated at a reduced pressure. Products (**V**) and (**VI**) were isolated by a column chromatography.

Method C. Sodium hydride (0.06 g, 1.64 mmol of 60% suspension in mineral oil) was added to a solution of azole (**II**) or (**III**) (1.37 mmol) in dry acetonitrile (30 ml). The mixture was stirred for 1 h at room temperature, and the obtained suspension of sodium salts

Table 4. Bond lengths (Å) in crystal of (**V**)

N1–C13	1.367 (7)	N1–C2	1.438 (6)
N2–C6	1.373 (6)	N2–C12	1.386 (6)
N2–C1	1.441 (6)	O1–C1	1.399 (5)
O1–C5	1.431 (6)	O2–C6	1.211 (6)
O3–C7	1.357 (6)	O3–C6	1.379 (6)
O4–C13	1.218 (7)	O5–C15	1.371 (6)
O5–C3	1.425 (6)	O6–C15	1.193 (7)
O7–C17	1.366 (7)	O7–C4	1.432 (6)
O8–C17	1.206 (8)	O9–C20	1.353 (6)
O9–C19	1.427 (6)	O10–C20	1.191 (6)
C1–C2	1.529 (7)	C2–C3	1.522 (7)
C3–C4	1.517 (7)	C4–C5	1.518 (7)
C5–C19	1.498 (7)	C7–C8	1.382 (8)
C7–C12	1.388 (7)	C8–C9	1.356 (9)
C9–C10	1.368 (9)	C10–C11	1.413 (8)
C11–C12	1.374 (7)	C13–C14	1.480 (8)
C15–C16	1.487 (8)	C17–C18	1.477 (9)
C20–C21	1.476 (8)		

Table 5. Valent angles in crystal of (**V**)

C13–N1–C2	122.5 (5)	C6–N2–C12	108.8 (4)
C6–N2–C1	122.2 (4)	C12–N2–C1	127.8 (4)
C1–O1–C5	113.9 (3)	C7–O3–C6	107.4 (4)
C15–O5–C3	117.1 (4)	C17–O7–C4	118.5 (5)
C20–O9–C19	116.9 (4)	O1–C1–N2	107.4 (4)
C20–O9–C19	116.9 (4)	O1–C1–N2	107.4 (4)
N1–C2–C3	109.7 (4)	N1–C2–C1	110.0 (4)
C3–C2–C1	111.3 (4)	O5–C3–C4	108.8 (4)
O5–C3–C2	108.5 (4)	C4–C3–C2	110.9 (4)
O7–C4–C3	105.7 (4)	O7–C4–C5	110.3 (4)
C3–C4–C5	109.4 (4)	O1–C5–C19	106.8 (4)
O1–C5–C4	109.1 (4)	C19–C5–C4	112.9 (4)
O2–C6–N2	129.0 (5)	O2–C6–O3	122.9 (5)
N2–C6–O3	108.0 (5)	O3–C7–C8	127.9 (5)
O3–C7–C12	110.0 (4)	C8–C7–C12	122.1 (5)
C9–C8–C7	116.8 (6)	C8–C9–C10	122.5 (5)
C9–C10–C11	121.3 (6)	C12–C11–C10	116.2 (5)
C11–C12–N2	133.3 (5)	C11–C12–C7	121.1 (4)
N2–C12–C7	105.6 (4)	O4–C13–N1	122.9 (5)
O4–C13–C14	122.4 (6)	N1–C13–C14	114.7 (6)
O6–C15–O5	123.1 (5)	O6–C15–C16	126.4 (5)
O5–C15–C16	110.6 (5)	O8–C17–O7	121.9 (6)
O8–C17–C18	126.9 (6)	O7–C17–C18	111.1 (7)
O9–C19–C5	108.8 (4)	O10–C20–O9	123.1 (5)
O10–C20–C21	125.7 (5)	O9–C20–C21	111.2 (4)

was treated with chloride (**I**) (0.5 g, 1.37 mmol) and 15C5 (0.054 ml, 0.274 mmol), and stirred at room temperature until the full conversion of the glycosyl donor (TLC). Solids were filtered off, the filtrate was evaporated to dryness at a reduced pressure, and products (**V**) and (**VI**) were separated b a column chromatography.

3-*N*-(2-Acetamido-3,4,6-tri-*O*-acetyl-2-deoxy-β-*D*-glucopyranosyl)-benzoxazol-2-one (V); mp 187–189°C, $[\alpha]_{546}$ –71° (*c* 1.0, chloroform).

3-*N*-(2-Acetamido-3,4,6-tri-*O*-acetyl-2-deoxy-β-*D*-glucopyranosyl)-5-methylbenzoxazol-2-one (VI); mp 178–180°C, $[\alpha]_{546}$ –75° (*c* 1.0, chloroform).

3-*N*-(2-Acetamido-3,4,6-tri-*O*-acetyl-2-deoxy-β-*D*-glucopyranosyl)benzothiazol-2-one (VII); mp 179–181°C, $[\alpha]_{546}$ –81° (*c* 1.0, chloroform).

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