Carbohydrate Research, 153 (1986) 318–324
Elsevier Science Publishers B.V., Amsterdam – Printed in The Netherlands

Note

Synthesis of glycosylaminothiazoles*

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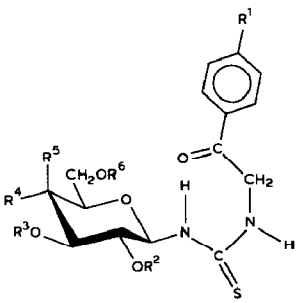
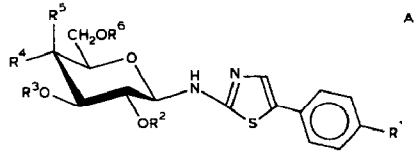
(Received December 10th, 1985; accepted for publication, January 23rd, 1986)

The reaction of glycosyl isothiocyanates with aliphatic α-aminoketone hydrochlorides yields glycosylthiourea derivatives[2,3] which give glycosylimidazolinethiones (**15**) by cyclodehydration. We have described[4] the preparation of *N*-phenacyl-*N'*-(2,3,4,6-tetra-*O*-acetyl-β-D-glycopyranosyl)thioureas (**1–3** and **5–6**) and the synthesis of 1,3-dihydro-5-methyl-1-(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl)-2*H*-imidazole-2-thione (**15**, D-*gluco*, R = Me) by reaction of 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl isothiocyanate with α-aminoacetone hydrochloride. We now report the preparation of new phenacylglycosylthioureas (**4** and **7**) and the synthesis of glycosylaminothiazoles (**8–14**).

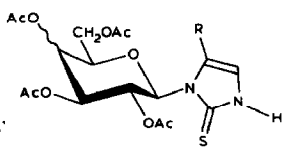
Following a procedure similar to that described[4], *N*-(*p*-bromophenacyl)-*N'*-



1–7



8–14, 16–18



15

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	16	17	18
R^1	H	Me	OMe	Br	H	Me	Br	H	Me	OMe	Br	H	Me	Br	Me	Br	Br
R^2, R^3, R^6	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	Ac	H	H	H
R^4	OAc	OAc	OAc	OAc	H	H	H	OAc	OAc	OAc	OAc	H	H	H	OH	OH	H
R^5	H	H	H	H	OAc	OAc	OAc	H	H	H	H	OAc	OAc	OAc	H	H	OH

*Thiourea Derivatives of Carbohydrates, Part VII. For Part VI, see ref. 1.

0008-6215/86/$ 03.50

TABLE I

^{1}H-N.M.R. DATA (δ SCALE, J IN Hz) FOR **4** AND **7** IN $CDCl_3$ AT 90 MHz

Compound	*Glycosylamino group*								*Heterocycle*		
	H-1'	*H-2'*	*H-3'*	*H-4'*	*H-5'*	*H-6',6''*	*OAc*	*NH*	*N'H*	*CH_2*	*Ar*
4	5.76t $J_{1',2'}$ 9 $J_{1',N'H}$ 9	5.20t[a] $J_{2',3'}$ 9	5.44t[a] $J_{3',4'}$ 9	5.15t[a] $J_{4',5'}$ 9	4.00m[a]	4.40–3.80m	2.06s (6 H) 2.04s (3 H) 2.02s (3 H)	7.50t J_{NH,CH_2} 3.7	7.42d	5.06d J_{gem} 19.5[b]	8.00–7.40m
7	5.70m	←5.60–5.20m→				←4.22s→	2.18s (3 H) 2.08s (3 H) 2.06s (3 H) 2.01s (3 H)	7.46t	7.12d $J_{1',N'H}$ 8	5.04d J_{gem} 19[b]	7.98–7.55m

[a]Obtained by extrapolation to zero concentration of $Eu(fod)_3$. [b]Obtained in the presence of $Eu(fod)_3$.

TABLE II

^{1}H-N.M.R. DATA (δ SCALE J IN Hz) FOR **8–14**

Compound	*Glycosylamino group*											
	H-1'	*H-2'*	*H-3'*	*H-4'*	*H-5'*	*H-6'*	*H-6''*	*OAc*	*NH*	*H-4*	*Ar*	*CH$_3$*
8[a]	5.10d[c] $J_{1',2'}$ 9[d]	5.25t[c] $J_{2',3'}$ 9[d]	5.07t[c] $J_{3',4'}$ 9[d]	5.05t[c]	3.90m	4.38dd $J_{6',6''}$ 12	4.12dd $J_{5',6''}$ 2.9	2.01s (3 H) 2.03s (3 H) 2.06s (6 H)	7.00–5.70	7.45s[c]	7.60–7.20m	—
9[a]	5.15d[c] $J_{1',2'}$ 9[d]	5.32t[c] $J_{2',3'}$ 9[d]	5.12t[c] $J_{3',4'}$ 9[d]	5.05t[c]	3.88m	4.36dd $J_{5',6'}$ 5 $J_{6',6''}$ 12	4.12dd	2.03s (3 H) 2.06s (9 H)	—	7.25s[c]	7.40–7.00m	2.35s
10[a]	5.15d[c] $J_{1',2'}$ 9[d]	5.20t[c] $J_{2',3'}$ 9[d]	5.03t[c] $J_{3',4'}$ 9[d]	5.05t[c]	4.00–3.75m	4.38dd $J_{5',6'}$ 5 $J_{6',6''}$ 12	4.12dd $J_{5',6''}$ 3	2.03s (3 H) 2.06s (9 H)	6.25s	7.25s	7.45–6.80m	3.82s
11[a]	5.12[c] $J_{1',2'}$ 9[d]	5.30t[c] $J_{2',3'}$ 9[d]	5.10t[c] $J_{3',4'}$ 9[d]	5.05t[c]	3.90m	4.38dd $J_{5',6'}$ 5 $J_{6',6''}$ 12	4.12dd $J_{5',6''}$ 3	2.06s (3 H) 2.08s (9 H)	—	7.40s	7.60–7.20m	—
12[b]	5.10t $J_{1',2'}$ 9.5 $J_{1',NH}$ 9.5	5.26dd $J_{2',3'}$ 8.7	5.18dd $J_{3',4'}$ 2.9	5.47d	← 4.25–4.05m →			2.02s (3 H) 2.04s (3 H) 2.07s (3 H) 2.17s (3 H)	6.10d	7.38s	7.45–7.20m	—
13[b]	5.08d $J_{1',2'}$ 10	5.26dd $J_{2',3'}$ 8.5	5.18dd $J_{3',4'}$ 3	5.48dd $J_{4',5'}$ 0.9	← 4.19–4.06m →			2.02s (3 H) 2.04s (3 H) 2.07s (3 H) 2.17s (3 H)	6.05s	7.34s	7.23m	2.35s
14[b]	5.07d $J_{1',2'}$ 8.3	5.26dd $J_{2',3'}$ 10	5.16dd $J_{3',4'}$ 2.9	5.48d	← 4.20–4.05m →			2.02s (3 H) 2.04s (3 H) 2.08s (3 H) 2.17s (3 H)	6.17s	7.37s	7.49–7.25m	—

[a]$CDCl_3$ at 90 MHz. [b]$CDCl_3$ at 200 MHz. [c]Obtained by extrapolation to zero concentration of $Eu(fod)_3$. [d]Obtained in the presence of $Eu(fod)_3$.

(2,3,4,6-tetra-*O*-acetyl-β-D-gluco(galacto)pyranosylthiourea (**4** or **7**) was prepared by reaction of 2,3,4,6-tetra-*O*-acetyl-β-D-gluco(galacto)pyranosyl isothiocyanate[5,6] with *p*-bromophenacylamine hydrochloride. The structures of **4** and **7** were demonstrated by elemental analyses and u.v., i.r., and ^{1}H-n.m.r. (Table I) data. A strong i.r. absorption at 1685–1695 cm^{-1} was assigned to the Ar–C=O group. The ^{1}H-n.m.r. spectrum of **4** could be interpreted completely on the basis of the shifts induced by the incremental addition of Eu(fod)$_3$. These assignments (Table I) were confirmed by double resonance experiments. The values of $J_{1',2'}$, $J_{2',3'}$, $J_{3',4'}$, and $J_{4',5'}$ were in the range (8–10 Hz) for antiperiplanar protons and were indicative of a major $^4C_1$(D) conformation in solution.

The cyclodehydration reaction with acetic anhydride and phosphoric acid of **1–7** afforded the 5-aryl-2-[2,3,4,6-tetra-*O*-acetyl-β-D-gluco(galacto)pyranosylamino]thiazoles **8–14**.

The analytical and u.v., i.r., and ^{1}H-n.m.r. (Table II) data for **8–14** and the mass spectrum of **11** were consistent with the structures assigned. Compounds **8–14** had λ_{max} at 300 nm, characteristic of arylsubstituted aminothiazoles[7], and no i.r. band for Ar–C=O. The δ values of H-4 of these products were similar to those for simple arylaminothiazoles[8]. The mass spectrum of **11** showed two peaks at *m/z* 256 and 254 (64%) assigned to 2-amino-5-(*p*-bromophenyl)thiazole, and significant peaks at *m/z* 214, 212, 201, 199, 196, 194, 182, and 180 characteristic of simple thiazoles[9].

Conventional base-catalysed deacetylation of **9**, **11**, and **14** produced the corresponding 5-aryl-2-[β-D-gluco(galacto)pyranosylamino]thiazoles **16–18**, the structures of which were in agreement with the i.r. data and elemental analyses.

The isomeric structures of 5-aryl-1,3-dihydro-1-(2,3,4,6-tetra-*O*-acetyl-β-D-glycopyranosyl)-2*H*-imidazole-2-thiones (**15**, R = aryl) for **8–14** were ruled out by several data. Thus, the characteristic λ_{max} of imidazoline-2-thiones[4,10,11] at ~260 nm was absent; the δ value expected[4] for H-4 of **15** was ~6.4 whereas H-4 in **8–14** resonated at δ 7.4; and **16–18** had no i.r. band (~1290 cm^{-1}) for the C=S group[4].

The $^3J_{H,H}$ values for **8–14** (Table II) showed that the $^4C_1$(D) conformation preponderated in solutions in chloroform.

EXPERIMENTAL

General methods. — Melting points are uncorrected. Optical rotations were measured at 5461 and 5895 Å, using a 1-cm cell. I.r. spectra were recorded for KBr discs. ^{1}H-N.m.r. spectra were obtained at 90 (continuous wave) and 200 MHz (F.t.) Assignments were confirmed by double resonance experiments and H/D exchange. Overlapping signals were separated from each other by incremental additions of Eu(fod)$_3$. The e.i. mass spectrum was obtained at 70 eV, with an ion-source temperature of 200°. T.l.c. was performed on silica gel HF_{254} (Merck) with ether–hexane (6:1) and detection by u.v. light, iodine vapour, or charring with sulphuric acid. Flash chromatography was conducted on Silica Gel 60 (Merck, 230 mesh).

N-(p-*Bromophenacyl*)-N'-[*2,3,4,6-tetra*-O-*acetyl*-β-D-*gluco(galacto)pyranosyl*]-*thioureas* (**4** *and* **7**). — A solution of *p*-bromophenacylamine hydrochloride (1.29 mmol) in water was neutralised with sodium hydrogencarbonate (1.29 mmol) and added to a solution of 2,3,4,6-tetra-*O*-acetyl-β-D-gluco(galacto)pyranosyl isothiocyanate (1.29 mmol) in acetone (12 mL) under nitrogen. The resulting solution was kept at room temperature for *t* h. The solvent was evaporated under diminished pressure, and the residue was washed with water and crystallised from ethanol. The following compounds were prepared in this manner.

N-(*p*-Bromophenacyl)-*N'*-(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl)-thiourea (**4**; 0.7 g, 90%; *t* 1 h), m.p. 177–178°, $[\alpha]_D^{20}$ −4.5° (*c* 0.9, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 253 nm (ε_{mM} 26.0); ν_{max} 3300 and 3280 (NH), 1750 (CO ester), 1685 (CO ketone), 1585 and 1540 (C=C aromatic), 1225 (C–O–C and C=S), and 820 cm^{-1} (CH aromatic). The ^{1}H-n.m.r. data are given in Table I.

Anal. Calc. for $C_{23}H_{27}BrN_2O_{10}S$: C, 45.78; H, 4.51; N, 4.64; S, 5.31; Br, 13.24. Found: C, 45.89; H, 4.74; N, 4.36; S, 5.37; Br, 13.08.

N-(*p*-Bromophenacyl)-*N'*-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyl)-thiourea (**7**; 0.60 g, 78%; *t* 4 h), m.p. 113–114°, $[\alpha]_D^{21}$ +8° (*c* 0.7, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 253 nm (ε_{mM} 28.0); ν_{max} 3340 and 3290 (NH), 1750 (CO ester), 1695 (CO ketone), 1585 and 1550 (C=C aromatic), 1230 (C–O–C and C=S), and 820 cm^{-1} (CH aromatic). The ^{1}H-n.m.r. data are given in Table I.

Anal. Calc. for $C_{23}H_{27}BrN_2O_{10}S$: C, 45.78; H, 4.51; N. 4.64; S. 5.31; Br, 13.24. Found: C, 45.47; H, 4.70; N, 4.90; S, 5.20; Br, 13.50.

5-Aryl-2-[*2,3,4,6-tetra*-O-*acetyl*-β-D-*gluco(galacto)pyranosylamino*]*thiazoles* (**8–14**). — To a solution of *N*-phenacyl-*N'*-[2,3,4,6-tetra-*O*-acetyl-β-D-gluco-(galacto)pyranosyl]thiourea (**1–7**, 0.68 mmol) in acetic anhydride (6.8 mL) was added phosphoric acid (0.34 mL). The mixture was stirred at room temperature for *t* h. The reactions were monitored by t.l.c. The resulting solution was poured into ice–water (80 mL), the solid products were collected, and solutions in chloroform (40 mL) were washed with saturated aqueous sodium hydrogencarbonate (3 × 15 mL) and then water, dried ($MgSO_4$), and concentrated. Solutions of the residues in aqueous 96% ethanol were treated with Amberlist IR-45 (HO^-) resin (8 mL), filtered, and concentrated, and the resulting syrup was purified as indicated. The following compounds were prepared in this manner.

5-Phenyl-2-(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosylamino)thiazole (**8**; 0.24 g, 70%; *t* 7 h), m.p. 122–124° (from ethanol), $[\alpha]_D^{21}$ −11°, $[\alpha]_{546}^{21}$ −17° (*c* 1, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 302 nm (ε_{mM} 25.8); ν_{max} 3380 (NH), 1740 (CO), 1550 and 1490 (C=C aromatic, NH), 1225 (C–O–C), 750 and 680 cm^{-1} (CH aromatic). The ^{1}H-n.m.r. data are given in Table II.

Anal. Calc. for $C_{23}H_{26}N_2O_9S$: C, 54.53; H, 5.17; N, 5.53; S, 6.33. Found: C, 54.66; H, 5.40; N, 5.42; S, 5.98.

2-(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosylamino)-5-(*p*-tolyl)thiazole (**9**; 0.26 g, 73%; *t* 23 h), m.p. 134° (from ethanol), $[\alpha]_D^{21}$ −14°, $[\alpha]_{546}^{21}$ −20° (*c* 1, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 302 nm (ε_{mM} 21.2); ν_{max} 3370 (NH), 1735 (CO), 1545 and 1525

(C=C aromatic, NH), 1220 (C–O–C), and 805 cm^{-1} (CH aromatic). The ^1H-n.m.r. data are given in Table II.

Anal. Calc. for $C_{24}H_{28}N_2O_9S$: C, 55.37; H, 5.42; N, 5.38; S, 6.16. Found: C, 55.48; H, 5.64; N, 5.50; S, 6.36.

5-(*p*-Methoxyphenyl) - 2 - (2,3,4,6 - tetra - *O* - acetyl - β - D -glucopyranosyl - amino)thiazole (**10**; 0.25 g, 70%; *t* 28 h), m.p. 139–141° (from ethanol), $[\alpha]_D^{21}$ −19°, $[\alpha]_{546}^{21}$ −22° (*c* 1, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 303 nm (ε_{mM} 21.1); ν_{max} 3480, 3340 and 3180 (NH), 1740 (CO), 1570, 1515, 1450 (C=C aromatic, NH), 1230 (C–O–C), and 815 cm^{-1} (CH aromatic). The ^1H-n.m.r. data are given in Table II.

Anal. Calc. for $C_{24}H_{28}N_2O_{10}S$: C, 53.72; H, 5.26; N, 5.22; S, 5.97. Found: C, 53.61; H, 5.29; N, 5.25; S, 6.15.

5-(*p*-Bromophenyl)-2-(2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosylamino)-thiazole (**11**; 0.27 g, 68%; *t* 17 h), m.p. 145–147° (from ethanol), $[\alpha]_D^{21}$ −17°, $[\alpha]_{546}^{21}$ −27° (*c* 1, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 310 nm (ε_{mM} 20.5); ν_{max} 3460 and 3140 (NH), 1740 (CO), 1545, 1520 and 1445 (C=C aromatic and NH), 1240 (C–O–C), and 810 cm^{-1} (CH aromatic). The ^1H-n.m.r. data are given in Table II. Mass spectrum: *m/z* 586 and 584 (1%, M^+), 544 and 542 (1), 527 and 525 (1), 526 and 524 (1), 331 (7), 256 and 254 (64), 214 and 212 (21), 201 and 199 (14).

Anal. Calc. for $C_{23}H_{25}BrN_2O_9S$: C, 47.18; H, 4.30; N, 4.78; S, 5.48; Br, 13.65. Found: C, 46.48; H, 4.36; N, 4.82; S, 5.78; Br, 13.39.

5-Phenyl-2-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosylamino)thiazole (**12**; 0.1 g, 29%; *t* 5 h), isolated as an amorphous solid by column chromatography (6:1 ether–hexane), had $[\alpha]_D^{21}$ +8° (*c* 0.6, chloroform); $\lambda_{max}^{CH_2Cl_2}$ 300 nm (ε_{mM} 17.8); ν_{max} 3300 (NH), 1740 (CO), 1525 and 1500 (C=C aromatic, NH), 1220 (C–O–C), 750 and 685 (CH aromatic). The ^1H-n.m.r. data are given in Table II.

Anal. Calc. for $C_{23}H_{26}N_2O_9S$: C, 54.53; H, 5.17; N, 5.53; S, 6.33. Found: C, 54.24; H, 5.29; N, 5.53; S, 6.66.

2-(2,3,4,6-Tetra-*O*-acetyl-β-D-galactopyranosylamino)-5-(*p*-tolyl)thiazole (**13**; 0.13 g, 35%; *t* 24 h), isolated as an amorphous solid by column chromatography (6:1 ether–hexane), had $\lambda_{max}^{CH_2Cl_2}$ 300 nm (ε_{mM} 21.9); ν_{max} 3300 (NH), 1745 (CO), 1525 and 1505 (C=C aromatic and NH), 1220 (C–O–C), and 805 cm^{-1} (CH aromatic). The ^1H-n.m.r. data are given in Table II.

Anal. Calc. for $C_{24}H_{28}N_2O_9S$: C, 55.30; H, 5.42; N, 5.38; S, 6.23. Found: C, 54.90; H, 5.24; N, 5.15; S, 6.90.

5-(*p*-Bromophenyl)-2-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosylamino)-thiazole (**14**; 0.2 g, 49%; *t* 5 h), isolated as an amorphous solid by column chromatography (6:1 ether–hexane), had $[\alpha]_D^{20}$ +14° (*c* 0.6, dichloromethane); $\lambda_{max}^{CH_2Cl_2}$ 305 nm (ε_{mM} 18.4); ν_{max} 3300 (NH), 1745 (CO), 1520 and 1500 (C=C aromatic and NH), 1220 (C–O–C), and 815 cm^{-1} (CH aromatic). The ^1H-n.m.r. data are given in Table II.

Anal. Calc. for $C_{23}H_{25}BrN_2O_9S$: C, 47.18; H, 4.30; N, 4.78; S, 5.48; Br, 13.64. Found: C, 47.30; H, 4.31; N, 4.67; S, 5.78; Br, 13.62.

5-Aryl-2-[β-D-gluco(galacto)pyranosylamino]thiazoles (**16–18**). — A solution

of 5-aryl-2-[2,3,4,6-tetra-*O*-acetyl-β-D-gluco(galacto)pyranosylamino]thiazole (**9**, **11**, or **14**, 0.25 mmol) in methanol (3.5 mL) and methanolic 0.5% sodium methoxide (0.3 mL) was stirred for *t* min at room temperature. The reactions were monitored by t.l.c. The solutions were treated with Amberlist IR-120 (H^+) resin (5 mL), filtered, and concentrated, and the residue was crystallised from ethanol. The following compounds were prepared in this manner.

2-(β-D-Glucopyranosylamino)-5-(*p*-tolyl)thiazole (**16**; 0.066 g, 75%; *t* 15 min), m.p. 209–210°; ν_{max} 3225, 3260 (OH, NH), 1560 and 1530 (C=C aromatic, NH), and 805 cm^{-1} (CH aromatic).

Anal. Calc. for $C_{16}H_{20}N_2O_5S$: C, 54.53; H, 5.72; N, 7.95. Found: C, 54.27; H, 6.25; N, 7.94.

5-(*p*-Bromophenyl)-2-(β-D-glucopyranosylamino)thiazole (**17**; 0.080 g, 77%; *t* 10 min), m.p. 219–220°; ν_{max} 3225, 3260 (OH, NH), 1560 and 1530 (C=C aromatic, NH), and 805 cm^{-1} (CH aromatic).

Anal. Calc. for $C_{15}H_{17}BrN_2O_5S$: C, 43.17; H, 4.11; N, 6.71; S, 7.68; Br, 19.15. Found: C, 43.38; H, 4.38; N, 6.33; S, 7.87; Br, 19.32.

5-(*p*-Bromophenyl)-2-(β-D-galactopyranosylamino)thiazole (**18**; 0.073 g, 70%; *t* 10 min), m.p. 178–179°; ν_{max} 3340, 3230 (OH, NH), 1560, 1530 and 1500 (C=C aromatic, NH), and 815 cm^{-1} (CH aromatic).

Anal. Calc. for $C_{15}H_{17}BrN_2O_5S$: C, 43.17; H, 4.11; N, 6.71; S, 7.68; Br, 19.15. Found: C, 42.88; H, 4.14; N, 6.42; S, 8.01; Br, 18.77.

ACKNOWLEDGMENTS

We thank Dr. Cert Ventulá for the mass spectrum, and the Ministry of Education and Science and the Ministry of Health of Spain for the awards of scholarships (to M.C.O.M.).

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