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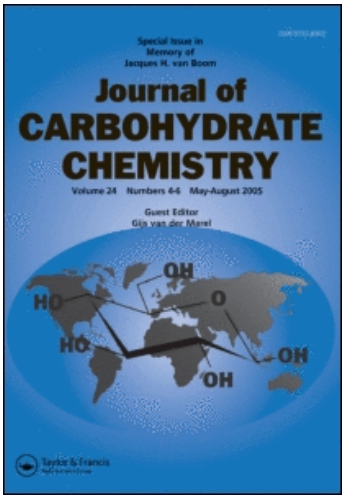

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Glycosylation of 4-Aryl-1-thioxo[1,2,4]-triazolo[4,3-a]quinazolin-5(4H)-one

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ABSTRACT

Reaction of 4-aryl-1-thioxo [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-ones (**2a,b**) with acetylated glycosyl bromides **3a–c** under alkaline conditions afforded the corresponding *S*-glycoside derivatives **4**, **5** and *N*-glycoside derivatives **6**, **7**. Oxidation of *S*-glycosyl derivatives **4**, **5** with *m*-chloroperbenzoic acid yielded the corresponding sulphones **8**, **9**, whereas the *N*-glycosyl derivatives **6**, **7** yielded 1-oxo derivatives **10**, **11**. However their *O*-deacetylation with sodium methoxide in methanol caused cleavage of the *S*-glycosyl residue and gave N^2-glycosylated analogues **12**, **13**, **14** and **15**.

Key Words: 4-Aryl-1-thioxo[1,2,4]triazolo[4,3-a]quinazolin-5(4H)-one; *O*-acetylated glycosyl bromides; S- and N-glycosides; Synthesis and spectral studies.

INTRODUCTION

The 4(3H)-quinazolinone ring system represents an important class of heterocyclic compounds in medicinal chemistry having anticonvulsant,[1,2] antihypertensive,[3,4] antidiabetic,[5] and anti-tumor activity.[6–8] On the other hand, heterocycles containing the 1,2,4-triazole nucleus are considered to be very interesting heterocyclic ring

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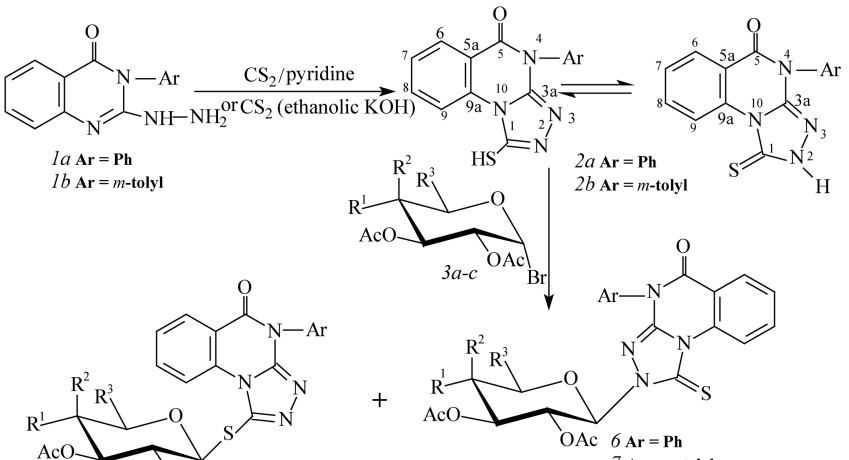
systems because of their therapeutic importance. For example, derivatives of 1,2,4-triazole have been found recently to have significant antiseptic[9] and analgesic properties.[10] Prompted by the aforesaid biological and pharmaceutical activities, and in continuation of our previous work aimed at introducing carbohydrate moieties into biologically versatile heterocyclic rings to improve their pharmacological activity,[11–16] we report here the synthesis of a new series of compounds in which a 4-aryl-1-thioxo [1,2,4] triazolo [4,3-a] quinazolin-5(4H)-one ring is used as a carrier for the glycosidic moiety.

RESULTS AND DISCUSSION

4-Aryl-1-thioxo [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-ones (**2a,b**) were prepared by the reaction of 3-aryl-2-thioxo-4 (3H)-quinazolinones[17] with the hydrazine hydrate in ethanol as the reaction medium.[18,19] The resulting 3-aryl-2-hydrazino-4(3H)-quinazolinones (**1a,b**) were cyclized with CS_2 in the presence of KOH in C_2H_5OH to obtain the desired triazoloquinazolinone derivatives **2a,b** and hydrazino derivatives **1a,b** also underwent ring closure with CS_2 in the presence of pyridine as a base.

Condensation of **2a,b** with 2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl bromide (**3a**), 2,3,4,6-tetra-*O*-acetyl-α-D-galactopyranosyl bromide (**3b**) or 2,3,4-tri-*O*-acetyl-α-D-xylopyranosyl bromide (**3c**) in acetone and in the presence of K_2CO_3 each afforded two products that were separated by silica gel chromatography (3:1, petroleum ether:Acetone) (Scheme 1).



Compound No.	R^1	R^2	R^3
3a-7a	OAc	H	CH_2OAc
3b-7b	H	OAc	CH_2OAc
3c-7c	OAc	H	H

Scheme 1. Reaction of compounds **2a,b** with *O*-acetylated glycosyl bromides.

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Several instrumental techniques were used to elucidate the structures of the *S*-glycosides **4**, **5** and *N*-glycosides **6**, **7** (Experimental). The IR spectra generally indicate the absence of the υ_{NH} of compounds **2a,b** but show the characteristic frequency of the newly formed functional group(s) (Experimental). The β-configurations of **4**, **5**, **6** and **7** were supported by their NMR data (Experimental). The chemical shifts and coupling constants of anomeric protons (H-1′) in **4**, **5**, **6** and **7** were in accordance with the reported value for β-glucosides, β-galactosides and β-xylosides.[20] Large coupling constants of the anomeric proton in *S*- and *N*-glycoside derivatives corresponds to the diaxial orientation of H-1′ and H-2′ protons, indicating the β-configuration and the $^4C_1$ conformation for this anomer (Experimental).

The ^{1}H NMR spectra provided several pieces of evidence for assigning the structures of the *S*- and *N*-glycosides derivatives. The ^{1}H NMR spectra gave an interesting peak corresponding to H-9 at δ 9.94–9.98 ppm in the case of the *N*-glycoside derivatives **6** and **7**. This downfield chemical shift is attributed to the strong interaction between the thione group (C = S) and H-9. This peak was used as an indicator for the *N*-glycoside derivatives, whereas the chemical shift for H-9 in the *S*-glycoside derivatives **4** and **5** occurs at δ 8.22–8.26 ppm. The anomeric proton (H-1′) of *N*-glycoside derivatives **6** and **7** in the ^{1}H NMR spectra was observed downfield to the peak assigned to the anomeric proton of *S*-glycoside derivatives **4** and **5** (Experimental). This suggested that magnetic anisotropy effects associated with the thione group (C = S) might be responsible for the deshielding observed for the anomeric proton.[21,22] The ^{13}C NMR spectra showed a signal at δ 164.8–165.2 ppm due to the thione group (C = S) that is associated with *N*-glycoside derivatives **6** and **7**, while the *S*-glycoside derivatives **4** and **5** gave the chemical shift at δ 144.2–144.6 ppm for the same carbon C-1 (C-S). In the ^{13}C NMR spectra of **6** and **7** the position of the C-1′ carbon atom is shifted downfield by 4.7 ppm in comparison with the C-1′ (anomeric carbon atom) signal of **4** and **5** (Experimental).

The mass spectra of **4**, **5**, **6** and **7** do not give reliable evidence to discriminate between the *S*- and *N*-glycoside derivatives. The FAB mass spectra of **4b** and **6b** showed the characteristic dissociation of poly-*O*-acetylated *N*- and *S*-glycosides[13,23] glycosidic bond fragments with *m*/*z* 294 [B + 1] (the aglycone) and 331 (the carbohydrate moiety). Possible fragmentation patterns of **4b** and **6b** are presented in Scheme 2.

The regiochemistry of this glycosylation was established by oxidation. Oxidation of the thioglycosides **4a,b** and **5a,c** with *m*-chloroperbenzoic acid in dichloromethane at room temperature[24] yielded the sulphone derivatives **8a,b** and **9a,b** respectively (Scheme 3). Oxidation of the *N*-glycosides **6a,b** and **7a,c** under the same conditions gave the corresponding *N*-glycoside of 1-oxo derivatives **10a,b** and **11a,b**, respectively (Scheme 3).

The analytical and spectral data (IR, ^{1}H NMR, ^{13}C NMR and mass spectra) were consistent with the assigned structures (Experimental). The analytical data for **9a** revealed a molecular formula $C_{30}H_{30}N_4O_{12}S$. The mass spectrum of **11b** was compatible with the molecular formula $C_{27}H_{26}N_4O_9$. The IR spectra of **8a,b** and **9a,b** contained absorption bands at 1210–1195 cm^{-1} and 1380–1375 cm^{-1} attributed to sulphonyl group (SO_2).

The IR and ^{13}C NMR spectra of **10a,b** and **11a,b** showed the absence of the signal due to thione (C = S) observed in **6a,b** and **7a,b** (Experimental). The β-configuration and $^4C_1$ conformation for these compounds were confirmed from ^{1}H NMR data. A comparison of the ^{1}H NMR parameters of sulphone derivative **9a** with those of the *S*-glycoside

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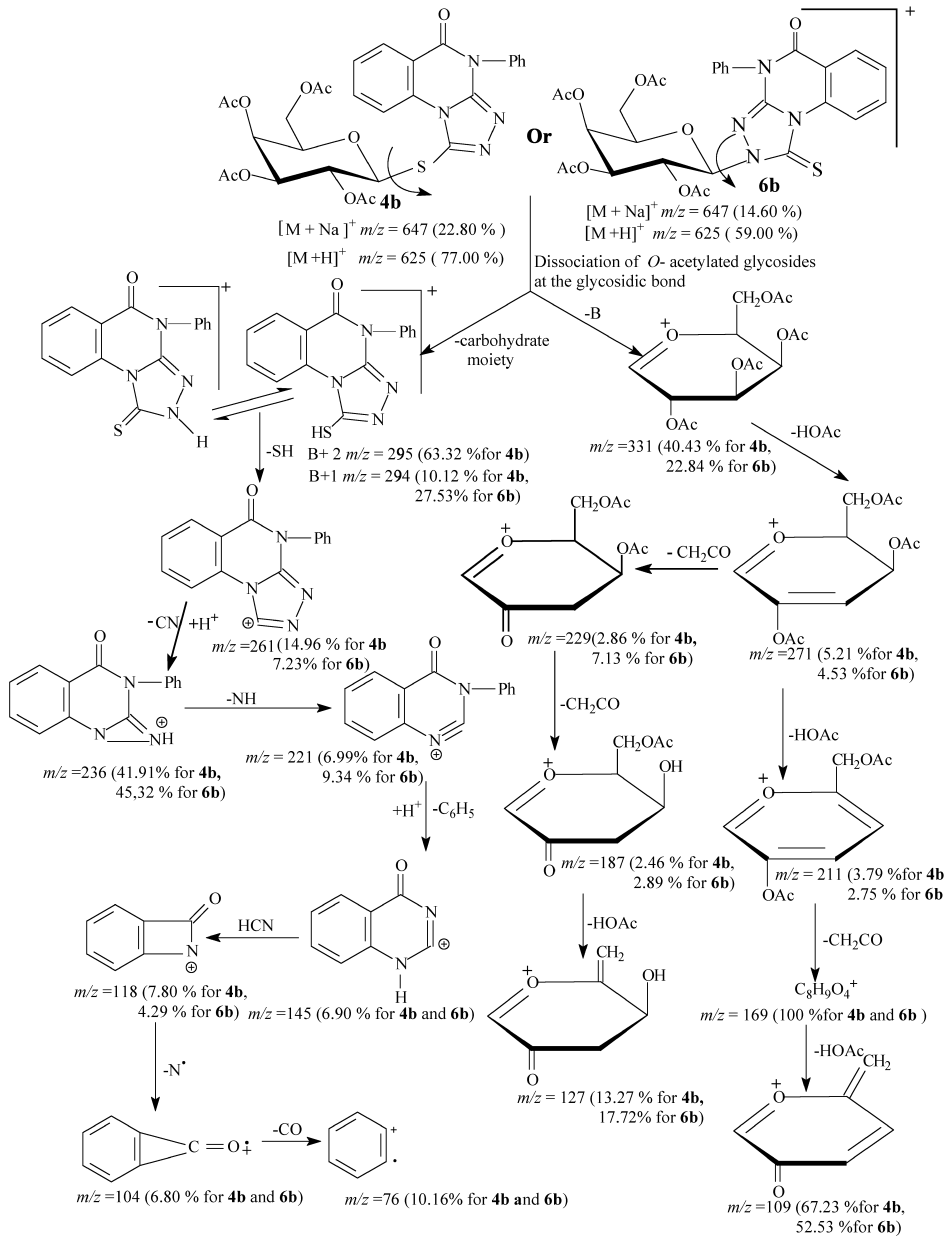

Scheme 2. Proposed mass spectral cleavage pathway of compounds **4b** and **6b**.

derivative **5a**, shows that the chemical shift for the signal of H-1′ (anomeric proton) appears 0.39 ppm lower field than that of **5a** (Experimental). The chemical shift of the anomeric proton (H-1′) and anomeric carbon atom (C-1′) of the carbohydrate moiety for **10a** resonated at higher field than *N*-glycoside[22] **6a** in ^{1}H and ^{13}C NMR spectra, respectively (Experimental).

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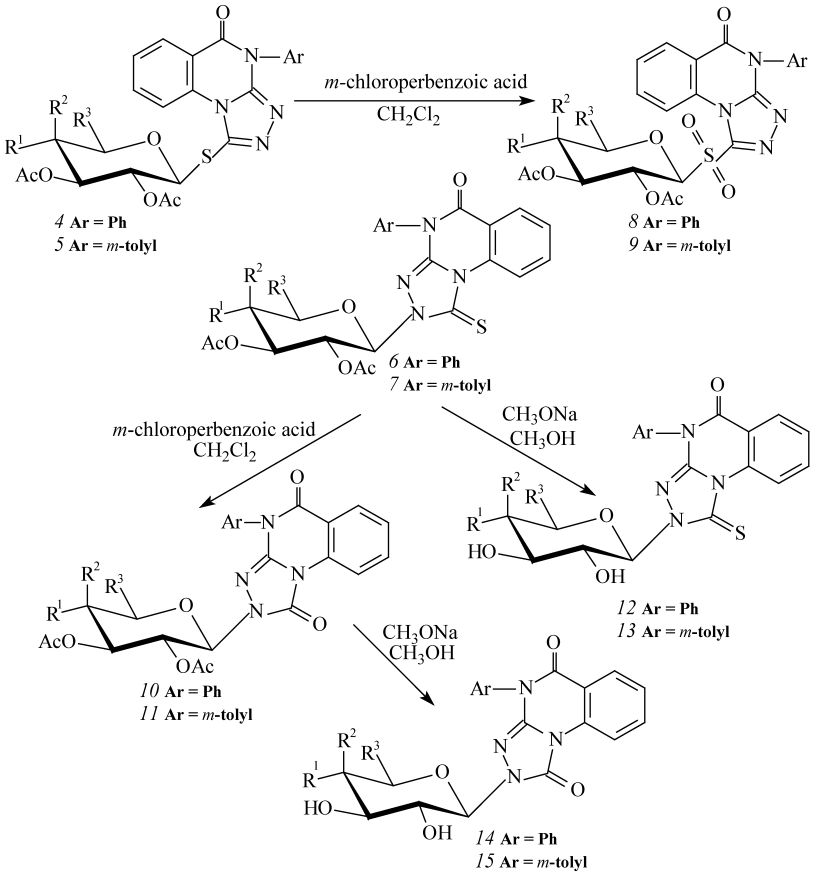

Compound No.	R^1	R^2	R^3
8a-15a	OAc	H	CH_2OAc
8b, 10b, 12b	H	OAc	CH_2OAc
9b, 11b, 13b, 15b	OAc	H	H

Scheme 3. Oxidation of compounds **4–7** and deacetylation of compounds **10–13**.

Attempts to deprotect *S*-glycoside derivatives **4**, **5**, **8** and **9** were carried out with sodium methoxide in methanol resulting in its decomposition. Deprotection of nucleoside derivatives **6a,b**, **7a,c**, **10a** and **11a,b** under the same conditions gave the expected deacetylated nucleosides **12a,b**, **13a,b**, **14a** and **15a,b**, respectively (Scheme 3). The structures of new compounds were established for the reaction products based on their analytical and spectral data (Experimental). The IR spectra of deacetylated nucleosides are characterized by the absence of $\upsilon_{C=O}$ ester stretching vibration at 1755–1743 cm^{-1} region and the presence of two strong bands at 1694–1672 and 3447–3435 cm^{-1} region due to $\upsilon_{C=O}$ and υ_{OH} groups, respectively.

The β-configuration in the sugar moiety was assigned on the basis of the coupling constant values of the anomeric proton, easily measurable in ^1H NMR spectra of the deacetylated nucleosides (Experimental).

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EXPERIMENTAL

Melting points were determined with a Buchi SMP-20 apparatus and are uncorrected. IR spectra were recorded on a Unicam SP 1200 spectrophotometer using KBr discs. ^{1}H and ^{13}C NMR spectra were recorded on a Varian CFT-20 operating 300 MHz for ^{1}H and 100 MHz for ^{13}C measurements. Tetramethylsilane (TMS) was used as internal standard and chemical shifts are expressed in δ-ppm values. Low-resolution mass spectra were recorded on micromass spectrometer model 7070 F at energy 70 eV and inlet temperature 90°C. FAB (LSIMS) spectra were recorded on a VG-Autospec instrument. Analytical TLC was performed on EM silica gel 60 F_{254} sheet (0.2 mm) with benzene/acetone (5:2, v/v) and in 2-propanol/benzene/25% ammonia (10:5:1, v/v/v) as the developing eluents A and B. The spots were detected with UV lamp model UV GL-58. $[\alpha]_D$ were measured on a polarimeter SR-6 at 25°C in $CHCl_3$. Elemental analyses were obtained from the laboratory service of microanalysis, Tanta University and from microanalysis center Cairo University, Cairo, Egypt.

4-Phenyl-1-thioxo [1,2,4] triazolo [4,3-a] quinazolin-5(4H)-one (2a). This compound was obtained in 82% as previously described,[25] mp 320–322°C (Lit.[25] 310–315°C, without any spectral data), additional data, R_f = 0.45(solvent system A), R_f = 0.72 (solvent system B); IR: 3434 (NH), 1211 (C = S), 1695 (C = O quinazolinone) cm^{-1}, ^{1}H NMR (DMSO-d_6): δ_H 10.29 (s, 1H, NH disappeared with D_2O), 7.50–8.2(m, 9H, Ar-H), ^{13}C NMR (DMSO-d_6): δ_c 148.7 (C-3a), 163.8(C-5), 116.4(C-5a), 131.2(C-6), 129.9(C-7), 137.8(C-8), 119.7(C-9), 138.2(C-9a), 137.2(C-1 of Ph), 129.8(2C, C-2,6 of Ph), 128.6(2C, C-3,5 of Ph), 129.3(C-4 of Ph), 165.2 (C = S); MS (EI): *m/z* (%) 295(25.49, M + 1), 294(100, M^+), 261(14.92), 236(40.67), 221(7.22), 145(6.89), 118(8.43), 104(6.77).

1-Thioxo-4-(*m*-tolyl)[1,2,4] triazolo[4,3-a]quinazolin-5(4H)-one (2b). This compound was obtained in 77% as previously reported[25]), mp 310–312°C (Lit.[25] 308–311°C, without any spectral data), additional data, R_f = 0.48 (solvent system A), R_f = 0.75(solvent system B); IR: 3495 (NH), 1231(C = S), 1698 (C = O quinazolinone) cm^{-1}; ^{1}H NMR (DMSO-d_6): δ_H 11.12 (s, 1H, NH disappeared with D_2O), 7.54–8.35 (m, 8H, Ar-H), 2.51(s, 3H, CH_3, J = 7.2 Hz); ^{13}C NMR (DMSO-d_6): δ_c 149.7(C-3a), 162.1(C-5), 118.0(C-5a), 129.7(C-6), 131.3(C-7), 135.5(C-8), 125.4(C-9), 138.8(C-9a), 137.9(C-1 of Ph), 129.6(C-2 of Ph), 137.5(C-3 of Ph), 128.7(C-4 of Ph), 128.2(C-5 of Ph), 123.4(C-6 of Ph), 29.6(*m*-CH_3), 165.2 (C = S); MS (EI): *m/z* (%) 309(100, M^+ + 1), 308 (83.65, M^+), 275 (42.55), 247(82.43), 232 (12.34), 162(25.32), 134(10.52), 118(15.0).

General procedure for the preparation of acetylated *S*-glycosides 4a–c, 5a–c and acetylated *N*-glycosides 6a–c, 7a–c. To a suspension of anhydrous K_2CO_3 (4.6 g, 33.3 mmol) in 50 mL of dry acetone at room temperature was added 4-aryl-1-thioxo [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-ones (**2a,b**) (5.6 mmol) and then (11.2 mmol) of *O*-acetylated glycosyl bromides **3a–c** and the reaction mixture was stirred for 10 h. The precipitate (KBr) was filtered off, and the filtrate was concentrated to dryness. The residue was dissolved in a 3:1 mixture of petroleum ether and acetone and transferred to a 50 × 2-cm column of silica gel (L 100/250 μ). The product was eluted with a

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mixture of petroleum ether and acetone, and the fractions containing *N*-glycosides **6** and **7** were collected. The fractions were monitored by TLC in system A. The subsequent fractions contained *S*-glycosides **4** and **5**. The following title compounds were prepared as just described.

4-Phenyl-1-(2′,3′,4′,6′-tetra-*O*-acetyl-*β*-D-glucopyranosylthio)[1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (4a). This compound was obtained in 58% yield, mp 215–217°C (from aqueous ethanol), R_f = 0.46 (solvent in system A), R_f = 0.78 (solvent in system B), $[\alpha]_D^{25}$ + 52 (*c* 1.0, $CHCl_3$); IR: 1746(C = O ester), 1686(C = O quinazolinone), 1628 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.41–8.22 (m, 9H, Ar-H), 5.82(d, 1H, *β* H-1′, $J_{1'2'}$ = 9.2 Hz), 5.08(t, 1H, H-2′, $J_{2',3'}$ = 9.2 Hz), 5.36(t, 1H, H-3′, $J_{3',4'}$ = 9.0 Hz), 5.13(t, 1H, H-4′, J4′,5′ = 9.2 Hz), 3.95(m, 1H, H-5′), 4.25(dd, 1H, H-6′, $J_{6',6''}$ = 12.4 Hz, $J_{6'5'}$ = 3.3 Hz), 4.15(dd, 1H, H-6″, $J_{6'',5'}$ = 2.6 Hz) 2.09, 2.05, 2.01, 1.98(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 150.1(C-3a), 160.3(C-5), 118.4(C-5a), 129.9(C-6), 127.4(C-7), 135.2(C-8), 116.8(C-9), 134.2(C-9a), 138.7(C-1 of Ph), 129.4(2C, C-2,6 of Ph), 128.9(2C, C-3,5 of Ph), 129.8(C-4 of Ph), 144.6 (C-S), 83.7(*β* C-1′), 71.2(C-2′), 72.3(C-3′), 68.2(C-4′), 75.2(C-5′), 63.1(C-6′), 20.4, 20.5 (2CO*CH*$_3$), 20.3(2CO*CH*$_3$), 170.2, 169.6, 169.2, 168.6 (4 *CO*CH_3).

Anal. Calcd for $C_{29}H_{27}N_4O_{10}S$ (623.61): C, 55.85; H, 4.36; N, 8.98; S, 5.14. Found: C, 55.63; H, 4.05; N, 8.67; S, 4.94.

4-Phenyl-1-(2′,3′,4′,6′-tetra-*O*-acetyl-*β*-D-galactopyranosylthio)[1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (4b). This compound was obtained in 53% yield, mp 219–221°C (from ethanol), R_f = 0.47(solvent in system A), R_f = 0.78 (solvent in system B), $[\alpha]_D^{25}$ + 37 (*c* 1.0, $CHCl_3$); IR: 1748(C = O ester), 1685(C = O quinazolinone), 1631 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.44–8.26 (m, 9H, Ar-H), 5.86(d, 1H, *β*H-1′, $J_{1'2'}$ = 10.2 Hz), 5.28(t, 1H, H-2′, $J_{2',3'}$ = 10.2 Hz), 5.21(t, 1H, H-3′, $J_{3',4'}$ = 3.4 Hz), 5.47(t, 1H, H-4′, $J_{4',5'}$ = 3.2 Hz), 4.19–4.09(m, 3H, H-5′, H-6′, H-6″), 2.12, 2.02, 2.00, 1.98(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 150.5 (C-3a), 160.2(C-5), 118.4(C-5a), 129.7(C-6), 126.9(C-7), 134.7(C-8), 117.2(C-9), 134.0(C-9a), 138.6(C-1 of Ph), 129.2(2C, C-2,6 of Ph), 128.8(2C, C-3,5 of Ph), 129.9(C-4 of Ph), 144.5 (C-S), 82.8(*β* C-1′), 67.5(C-2′), 72.2(C-3′), 67.3(C-4′), 75.0(C-5′), 63.4(C-6′), 20.7(2CO*CH*$_3$), 20.6 (2CO*CH*$_3$). 170.5, 169.6, 169.3, 168.6 (4 *CO*CH_3); MS (FAB): *m*/*z* (%) 647 (22.8, $[M + Na]^+$, 625 (77.0, $[M + H]^+$, 295 (63.32, B + 2), 294 (10.12, B + 1), 331(40.43, sugar moiety), 169 (100, $C_8H_9O_4^+$).

Anal. Calcd for $C_{29}H_{27}N_4O_{10}S$ (623.61): C, 55.85; H, 4.36; N, 8.98; S, 5.14. Found: C, 56.07; H, 4.08; N, 8.68; S, 5.32.

4-Phenyl-1-(2′,3′,4′-tri-*O*-acetyl-*β*-D-xylopyranosylthio)[1,2,4] triazolo[4,3-a] quinazolin-5 (4H)-one (4c). This compound was obtained in 50% yield, mp 210–212°C (from ethanol), R_f = 0.44(solvent in system A), R_f = 0.77 (solvent in system B), $[\alpha]_D^{25}$ + 64 (*c* 1.0, $CHCl_3$); IR: 1745(C = O ester), 1688(C = O quinazolinone), 1627 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.39–8.25 (m, 9H, Ar-H), 5.84(d, 1H, *β* H-1′, $J_{1'2'}$ = 7.8 Hz), 4.98(t, 1H, H-2′, $J_{2',3'}$ = 7.5 Hz), 5.27(t, 1H, H-3′, $J_{3',4'}$ = 7.5 Hz), 4.84(m, 1H, H-4′, $J_{4',5'}$ = 4.6 Hz), 4.05 (dd, 1H, H-5′, $J_{5',5''}$ = 11.9 Hz), 3.73 (dd, 1H, H-5″, $J_{4',5''}$ = 7.4 Hz), 2.04, 2.00, 1.96 (each s, each 3H, 3Ac); MS (FAB): *m*/*z* (%)

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574 (14.45, $[M + Na]^+$, 552 (48.37, $[M^+H]^+$, 294 (17.93, B + 1), 259 (38.37, sugar moiety), 221 (7.45), 145 (13.29), 118 (5.17), 76(14.34), 60 (100, AcOH).

Anal. Calcd for $C_{26}H_{23}N_4O_8S$ (551.55): C, 56.62; H, 4.20; N, 10.16; S, 5.81. Found: C, 56.89; H, 3.92; N, 9.84; S, 5.99.

1-(2′,3′,4′,6′-Tetra-*O*-acetyl-*β*-D-glucopyranosylthio)-4-(*m*-tolyl) [1,2,4]triazolo [4,3-a] quinazolin-5 (4H)-one (5a). This compound was obtained in 62% yield, mp 207–209°C (from EtOAc)), R_f = 0.48 (solvent in system A), R_f = 0.81 (solvent in system B), $[\alpha]_D^{25}$ + 78 (*c* 1.0, $CHCl_3$); IR: 1752(C = O ester), 1684(C = O quinazolinone), 1625 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.38–8.25 (m, 8H, Ar-H), 2.47 (s, 3H, *m*-CH_3, J = 7.1 Hz), 5.80(d, 1H, *β* H-1′, $J_{1'2'}$ = 9.2 Hz), 5.05(t, 1H, H-2′, $J_{2',3'}$ = 9.5 Hz), 5.33(t, 1H, H-3′, $J_{3',4'}$ = 10.4 Hz), 5.10(t, 1H, H-4′, J4′,5′ = 9.0 Hz), 3.91(m, 1H, H-5′), 4.21(dd, 1H, H-6′, $J_{6',6''}$ = 12.7 Hz, $J_{6'5'}$ = 5.5 Hz), 4.13(dd, 1H, H-6″, $J_{6',6''}$ = 12.4 Hz, $J_{6'',5'}$ = 2.5 Hz), 2.03, 2.01, 1.98, 1.95(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 149.7 (C-3a), 160.5(C-5), 117.9(C-5a), 129.6(C-6), 127.6(C-7), 135.0(C-8), 117.1(C-9), 135.2(C-9a), 136.4(C-1 of Ph), 129.6(C-2 of Ph), 138.5(C-3 of Ph), 129.3(C-4 of Ph), 128.6(C-5 of Ph), 126.1(C-6 of Ph), 21.3 (*m*-CH_3), 144.2 (C-S), 82.4(*β* C-1′), 69.8(C-2′), 73.2(C-3′), 67.5(C-4′), 74.4(C-5′), 61.9(C-6′), 20.6, 20.5, 20.3, 20.1 (4CO*CH*$_3$), 170.2, 169.3, 169.0, 168.2 (4 *CO*CH_3); MS (FAB): *m/z* (%) 661(24.25, ($[M + Na]^+$), 639 (39.41, $[M^+H]^+$), 331 (43.21, sugar moiety), 308 (100, B + 1), 275(21.13), 235 (4.45), 229 (5.33), 187 (3.85), 145 (6.75), 118 (5.17), 109 (68.54), 76(14.34).

Anal. Calcd for $C_{30}H_{29}N_4O_{10}S$ (637.64): C, 56.51; H, 4.58; N, 8.79; S, 5.03. Found: C, 55.68; H, 4.34; N, 9.00; S, 5.21.

1-(2′,3′,4′,6′-Tetra-*O*-acetyl-*β*-D-galactopyranosylthio)-4-(*m*-tolyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (5b). This compound was obtained in 60% yield, mp 202–204°C (from EtOAc), R_f = 0.48(solvent in system A), R_f = 0.84 (solvent in system B), $[\alpha]_D^{25}$ + 42 (*c* 1.0, $CHCl_3$); IR: 1744(C = O ester), 1681(C = O quinazolinone), 1623 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.35–8.23 (m, 8H, Ar-H), 2.45(s, 3H, CH_3) 5.82(d, 1H, *β* H-1′, $J_{1'2'}$ = 10.0 Hz), 5.24(t, 1H, H-2′, $J_{2',3'}$ = 10.2 Hz), 5.19(t, 1H, H-3′, $J_{3',4'}$ = 3.4 Hz), 5.45(t, 1H, H-4′, J4′,5′ = 3.2 Hz), 4.18–4.03(m, 3H, H-5′, H-6′, H-6″), 2.10, 1.99, 1.98, 1.96(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 149.8 (C-3a), 160.3(C-5), 118.2(C-5a), 129.2(C-6), 127.5(C-7), 135.2(C-8), 116.3(C-9), 135.3(C-9a), 135.9(C-1 of Ph), 129.4(C-2 of Ph), 138.3(C-3 of Ph), 129.4(C-4 of Ph), 128.7(C-5 of Ph), 126.3(C-6 of Ph), 21.6 (*m*-CH_3), 144.3 (C-S), 81.9(*β* C-1′), 67.9(C-2′), 71.3(C-3′), 66.3(C-4′), 74.2(C-5′), 61.8(C-6′), 20.4(2CO*CH*$_3$), 20.2(2CO*CH*$_3$), 169.7, 169.5, 169.3, 168.4 (4, *CO*CH_3).

Anal. Calcd for $C_{30}H_{29}N_4O_{10}S$ (637.64): C, 56.51; H, 4.58; N, 8.79; S, 5.03. Found: C, 56.78; H, 4.90; N, 8.46; S, 5.18.

1-(2′,3′,4′-Tri-*O*-acetyl-*β*-D-xylopyranosylthio)-4-(*m*-tolyl)[1,2,4] triazolo[4,3-a] quinazolin-5 (4H)-one (5c). This compound was obtained in 59% yield, mp 232–234°C (from EtOH), R_f = 0.46 (solvent in system A), R_f = 0.80 (solvent in system B), $[\alpha]_D^{25}$ + 37 (*c* 1.0, $CHCl_3$); IR: 1743(C = O ester), 1682(C = O quinazolinone), 1624 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.35–8.24 (m, 8H, Ar-H), 2.41 (s, 3H, *m*-CH_3), 5.80(d, 1H, *β* H-1′, $J_{1'2'}$ = 7.6 Hz), 4.96(t, 1H, H-2′, $J_{2',3'}$ = 7.5 Hz), 5.24(t, 1H, H-3′, $J_{3',4'}$ = 7.4 Hz), 4.82(m, 1H, H-4′, $J_{4',5'}$ = 4.5 Hz), 4.03(dd, 1H, H-5′, $J_{5',5''}$ = 11.9 Hz), 3.66 (dd, 1H, H-5″, $J_{4',5''}$ = 7.6 Hz), 2.03, 2.02, 1.93 (each s, each 3H, 3Ac).

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Anal. Calcd for $C_{27}H_{25}N_4O_8S$ (565.57): C, 57.34; H, 4.46; N, 9.91; S, 5.67. Found: C, 57.80; H, 4.77; N, 9.62; S, 5.78.

4-Phenyl-N^2-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-glucopyranosyl)-1-thioxo-[1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (6a). This compound was obtained in 32% yield, mp 193–195°C (from aqueous ethanol), R_f = 0.54 (solvent in system A), R_f = 0.84 (solvent in system B), $[\alpha]_D^{25}$ + 38 (*c* 0.5, $CHCl_3$); IR: 1747(C = O ester), 1692(C = O quinazolinone), 1625 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.52–9.97 (m, 9H, Ar-H), 6.42(d, 1H, β H-1′, $J_{1'2'}$ = 9.6 Hz), 5.53(t, 1H, H-2′, $J_{2',3'}$ = 9.6 Hz), 5.44(t, 1H, H-3′, $J_{3',4'}$ = 9.6 Hz), 5.21(t, 1H, H-4′, J4′,5′ = 9.6 Hz), 3.92(m, 1H, H-5′), 4.37(dd, 1H, H-6′, $J_{6',6''}$ = 12.6 Hz, $J_{6'5'}$ = 3.8 Hz), 4.17(dd, 1H, H-6″, $J_{6'',5'}$ = 2.6 Hz) 2.12, 2.08, 2.05, 1.96(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 147.4 (C-3a), 160.2(C-5), 117.8(C-5a), 129.5(C-6), 127.4(C-7), 134.6(C-8), 117.2(C-9), 136.4(C-9a), 138.5(C-1 of Ph), 128.8(2C, C-2,6 of Ph), 129.0(2C, C-3,5 of Ph), 129.7(C-4 of Ph), 164.8 (C = S), 87.4(β C-1′), 71.5(C-2′), 73.8(C-3′), 69.4(C-4′), 73.9(C-5′), 62.3(C-6′), 20.7, 20.4(2CO*CH*$_3$), 20.3(2CO*CH*$_3$), 170.5, 169.8, 169.2, 168.5(4 *CO*CH_3).

Anal. Calcd for $C_{29}H_{27}N_4O_{10}S$ (623.61): C, 55.85; H, 4.36; N, 8.98; S, 5.14. Found: C, 56.09; H, 4.62; N, 9.19; S, 4.97.

4-Phenyl-N^2-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-galactopyranosyl)-1-thioxo [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (6b). This compound was obtained in 24% yield, mp 202–204°C (from ethanol), R_f = 0.54 solvent in system A), R_f = 0.83 (solvent in system B), $[\alpha]_D^{25}$ + 73 (*c* 0.5, $CHCl_3$); IR: 1745(C = O ester), 1690 (C = O quinazolinone), 1628 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.52–9.96 (m, 9H, Ar-H), 6.38(d, 1H, β H-1′, $J_{1'2'}$ = 9.6 Hz), 5.49(t, 1H, H-2′, $J_{2',3'}$ = 9.6 Hz), 5.19(t, 1H, H-3′, $J_{3',4'}$ = 4.0 Hz), 5.49(t, 1H, H-4′, $J_{4',5'}$ = 3.7 Hz), 4.35–4.17(m, 3H, H-5′, H-6′, H-6″), 2.23, 2.06, 2.00, 1.98(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 146.8 (C-3a), 160.4(C-5), 118.0(C-5a), 129.8(C-6), 127.4(C-7), 135.0(C-8), 117.1(C-9), 136.1(C-9a), 138.3C-1 of Ph), 126.5(2C, C-2,6 of Ph), 128.5(2C, C-3,5 of Ph), 129.7(C-4 of Ph), 164.8 (C = S), 86.4(β C-1′), 70.5 (C-2′), 73.0(C-3′), 68.4(C-4′), 74.4(C-5′), 62.3 (C-6′), 20.6(2CO*CH*$_3$), 20.5 (2CO*CH*$_3$). 170.4, 169.9, 169.0, 168.7 (4 *CO*CH_3); MS (FAB): *m/z* (%) 647 (14.6, $[M + Na]^+$, 625 (59.0, $[M^+H]^+$, 294 (27.53, B + 1), 331(22.84, sugar moiety), 169 (100, $C_8H_9O_4^+$).

Anal. Calcd for $C_{29}H_{27}N_4O_{10}S$ (623.61): C, 55.85; H, 4.36; N, 8.98; S, 5.14. Found: C, 55.67; H, 4.15; N, 8.69; S, 5.03.

4-Phenyl-N^2-(2′,3′,4′-tri-*O*-acetyl-β-D-xylopyranosyl)-1-thioxo[1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (6c). This compound was obtained in 37% yield, mp 192–194°C (from methanol), R_f = 0.52 (solvent in system A), R_f = 0.83 (solvent in system B), $[\alpha]_D^{25}$ + 33 (*c* 0.5, $CHCl_3$); IR: 1744(C = O ester), 1692(C = O quinazolinone), 1623 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.42–9.95 (m, 9H, Ar-H), 6.43(d, 1H, β H-1′, $J_{1'2'}$ = 9.4 Hz), 5.03(t, 1H, H-2′, $J_{2',3'}$ = 9.4 Hz), 5.38(t, 1H, H-3′, $J_{3',4'}$ = 9.2 Hz), 4.86(m, 1H, H-4′, $J_{4',5'}$ = 4.3 Hz), 4.07 (dd, 1H, H-5′, $J_{5',5}$ = 12.2 Hz), 3.78 (dd, 1H, H-5″, $J_{4',5''}$ = 8.1 Hz), 2.07, 2.04, 1.98 (each s, each 3H, 3Ac).

Anal. Calcd for $C_{26}H_{23}N_4O_8S$ (551.55): C, 56.62; H, 4.20; N, 10.16; S, 5.81. Found: C, 56.35; H, 4.52; N, 9.93; S, 5.96.

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N^2-(2′,3′,4′,6′-Tetra-*O*-acetyl-*β*-D-glucopyranosyl)-1-thioxo-4-(*m*-tolyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (7a). This compound was obtained in 32% yield, mp 189–191°C (from aqueous ethanol), R_f = 0.56 (solvent in system A), R_f = 0.85 (solvent in system B), $[\alpha]_D^{25}$ + 68 (*c* 0.5, $CHCl_3$); IR: 1755(C = O ester), 1694(C = O quinazolinone), 1631 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.60–9.98 (m, 8H, Ar-H), 2.45 (s, 3H, *m*-CH_3, J = 7.1 Hz), 6.42(d, 1H, *β* H-1′, $J_{1'2'}$ = 9.7 Hz), 5.52(t, 1H, H-2′, $J_{2',3'}$ = 9.7 Hz), 5.42(t, 1H, H-3′, $J_{3',4'}$ = 9.7 Hz), 5.20(t, 1H, H-4′, J4′,5′ = 9.7Hz), 3.91(m, 1H, H-5′), 4.35(dd, 1H, H-6′, $J_{6',6''}$ = 12.3 Hz, $J_{6'5'}$ = 3.6 Hz), 4.16(dd, 1H, H-6″, $J_{6'',5'}$ = 2.2 Hz) 2.10, 2.06, 2.02, 1.96(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 147.2 (C-3a), 160.0(C-5), 117.2(C-5a), 129.1(C-6), 127.8(C-7), 135.2(C-8), 117.3(C-9), 133.7(C-9a), 137.5(C-1 of Ph), 129.4(C-2of Ph), 138.3(C-3 of Ph), 129.7(C-4 of Ph), 128.1(C-5 of Ph), 126.4(C-6 of Ph), 21.5 (*m*-CH_3), 165.2 (C = S), 87.6(*β* C-1′), 73.5(C-2′), 72.4(C-3′), 70.2(C-4′), 73.8(C-5′), 61.7(C-6′), 20.5, 20.4, 20.3, 20.0 (4CO*CH*$_3$), 170.4, 169.9, 169.6, 169.4 (4 *CO*CH_3); MS (FAB): *m/z* (%) 661(18.35, $[M + Na]^+$, 639 (59.0, $[M^+H]^+$, 308(17.32, B + 1), 331(31.62, sugar moiety), 271 (6.21), 229(4.23), 109(100).

Anal. Calcd for $C_{30}H_{29}N_4O_{10}S$ (637.64): C, 56.51; H, 4.58; N, 8.79; S, 5.03. Found: C, 56.27; H, 4.28; N, 9.06; S, 5.21.

N^2-(2′,3′,4′,6′-Tetra-*O*-acetyl-*β*-D-galactopyranosyl)-1-thioxo-4-(*m*-tolyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (7b). This compound was obtained in 29% yield, mp 202–204°C (from ethanol), R_f = 0.55(solvent in system A), R_f = 0.86 (solvent in system B), $[\alpha]_D^{25}$ + 58 (*c* 0.5, $CHCl_3$); IR: 1743(C = O ester), 1693(C = O quinazolinone), 1624 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.58–9.94 (m, 8H, Ar-H), 2.43 (s, 3H, *m*-CH_3, J = 7.1 Hz), 6.40(d, 1H, *β* H-1′, $J_{1'2'}$ = 10.3Hz), 5.47(t, 1H, H-2′, $J_{2',3'}$ = 10.3 Hz), 5.22(t, 1H, H-3′, $J_{3',4'}$ = 3.8 Hz), 5.52(t, 1H, H-4′, $J_{4',5'}$ = 3.9 Hz), 4.33-4.19(m, 3H, H-5′, H-6′, H-6″), 2.28, 2.02, 2.01, 2.00(each s, each 3H, 4Ac).

Anal. Calcd for $C_{30}H_{29}N_4O_{10}S$ (637.64): C, 56.51; H, 4.58; N, 8.79; S, 5.03. Found: C, 56.24; H, 4.93; N, 8.46; S, 4.92.

N^2-(2′,3′,4′-Tri-*O*-acetyl-*β*-D-xylopyranosyl)-1-thioxo-4-(*m*-tolyl) [1,2,4]triazolo [4,3-a] quinazolin-5 (4H)-one (7c). This compound was obtained in 28% yield, mp 213–215°C (from EtOAc)), R_f = 0.56 (solvent in system A), R_f = 0.87 (solvent in system B), $[\alpha]_D^{25}$ + 94 (*c* 0.5, $CHCl_3$); IR: 1748(C = O ester), 1691(C = O quinazolinone), 1629 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.58–9.96 (m, 8H, Ar-H), 2.41 (s, 3H, *m*-CH_3, J = 7.2 Hz), 6.42 (d, 1H, *β* H-1′, $J_{1'2'}$ = 7.2 Hz), 5.05(t, 1H, H-2′, $J_{2',3'}$ = 7.2 Hz), 5.41(t, 1H, H-3′, $J_{3',4'}$ = 7.2 Hz), 4.88(m, 1H, H-4′, $J_{4',5'}$ = 4.5 Hz), 4.09 (dd, 1H, H-5′, $J_{5',5''}$ = 11.5 Hz), 3.83 (dd, 1H, H-5″, $J_{4',5''}$ = 8.4 Hz), 2.09, 2.07, 2.03 (each s, each 3H, 3Ac).

Anal. Calcd for $C_{27}H_{25}N_4O_8S$ (565.57): C, 57.34; H, 4.46; N, 9.91; S, 5.76. Found: C, 57.17; H, 4.74; N, 9.69; S, 5.57.

Oxidation of *S*-glycosides 4a,b, 5a,c and *N*-glycosides 6a,b, 7a,b with *m*-chloroperbenzoic acid. General procedure. A solution of substrate (50 mmol) and *m*-chloroperbenzoic acid (0.270 g, 1.56 mmol) in dichloromethane (20 mL) was stirred at room temperature for 5 h. The solution was filtered and washed with saturated sodium bicarbonate (25 mL) followed by water (25 mL). The organic layer was dried under anhydrous Na_2SO_4, filtered and concentrated to dryness under reduced pressure.

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The residue was crystallized from methanol to yield sulphone derivatives **8a,b, 9a,b** and 1-oxo-derivatives **10a,b, 11a,b**, respectively.

4-Phenyl-1-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-glucopyranosulfonyl)[1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (8a). This compound was obtained in 74% yield, mp 189–192°C, R_f = 0.37(solvent in system A), R_f = 0.68 (solvent in system B), $[\alpha]_D^{25}$ + 36 (*c* 1.0, $CHCl_3$); IR: 1744(C = O ester), 1689(C = O quinazolinone), 1632 (C = N) , 1375 and 1200 cm^{-1} (SO_2); ^1H NMR ($CDCl_3$): δ_H 7.32–8.14 (m, 9H, Ar-H), 6.24(d, 1H, β H-1′, $J_{1'2'}$ = 9.7 Hz), 5.51–5.27(m, 3H, H-2′, H-3′, H-4′), 3.47 (m, 1H, H-5′), 4.46(m, 2H, H-6′, H-6″) 2.15–2.00 (4 CH_3); ^{13}C NMR ($CDCl_3$): δ_c 150.4(C-3a), 161.6(C-5), 118.7(C-5a), 130.2(C-6), 127.0(C-7), 135.6(C-8), 117.3(C-9), 135.4(C-9a), 138.6(C-1 of Ph), 129.5(2C, C-2,6 of Ph), 130.4(2C, C-3,5 of Ph), 129.6(C-4 of Ph), 146.2 (C-S), 83.6(β C-1′), 71.4(C-2′), 72.5(C-3′), 68.5(C-4′), 75.7 (C-5′), 63.4(C-6′), 20.6–20.2 (4CO*CH*$_3$), 170.6–169.2 (4 *CO*CH_3).

Anal. Calcd for $C_{29}H_{27}N_4O_{12}S$ (655.61): C, 53.13; H, 4.15; N, 8.55; S, 4.89. Found: C, 52.98; H, 3.82; N, 8.29; S, 4.71.

4-Phenyl-1-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-galactopyranosylsulfonyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (8b). This compound was obtained in 69 % yield, mp 233–235°C, R_f = 0.36 (solvent in system A), R_f = 0.68 (solvent in system B), $[\alpha]_D^{25}$ + 29 (*c* 1.0, $CHCl_3$); IR: 1746(C = O ester), 1685(C = O quinazolinone), 1629 (C=N), 1365 and 1198 cm^{-1} (SO_2).

Anal. Calcd for $C_{29}H_{27}N_4O_{12}S$ (655.61): C, 53.13; H, 4.15; N, 8.55; S, 4.89. Found: C, 52.90; H(C = N), 1380 and 1195 cm^{-1} (SO_2)., 4.47; N, 8.83; S, 5.10.

1-(2′,3′,4′,6′-Tetra-*O*-acetyl-β-D-glucopyranosylsulfonyl)-4-(*m*-tolyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (9a). This compound was obtained in 72% yield, mp 207–209°C, R_f = 0.36 (solvent in system A), R_f = 0.70(solvent in system B), $[\alpha]_D^{25}$ + 59 (*c* 1.0, $CHCl_3$); IR: 1748(C = O ester), 1692(C = O quinazolinone), 1628 (C = N), 1377 and 1210 cm^{-1} (SO_2); ^1H NMR ($CDCl_3$): δ_H 7.26–8.32 (m, 8H, Ar-H), 2.46 (s, 3H, *m*-CH_3, J = 7.0 Hz), 6.29(d, 1H, β H-1′, $J_{1'2'}$ = 9.7 Hz), 5.55–5.35(m, 3H, H-2′, H-3′, H-4′), 3.98–4.54(m, 3H, H-5′, H-6′, H-6″), 2.16–2.03 (4 CH_3); MS (FAB): *m*/*z* (%) 693(12.37, $[M + Na]^+$, 671 (53.62, $[M^+H]^+$, 308(8.45, B + 1), 331(100, sugar moiety).

Anal. Calcd for $C_{30}H_{29}N_4O_{12}S$ (669.63): C, 53.81; H, 4.37; N, 8.37; S, 4.79. Found: C, 53.67; H, 4.47; N, 8.06; S, 4.59.

1-(2′,3′,4′-Tri-*O*-acetyl-β-D-xylopyranosylsulfonyl)-4-(*m*-tolyl)[1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (9b). This compound was obtained in 74% yield, mp 268–270°C, R_f=0.35(solvent in system A), R_f=0.72 (solvent in system B), $[\alpha]_D^{25}$ + 22 (*c* 1.0, $CHCl_3$); IR: 1746(C=O ester), 1692(C=O quinazolinone), 1627(C=N), 1378 and 1198 cm^{-1} (SO_2).

Anal. Calcd for $C_{27}H_{25}N_4O_{10}S$ (597.57): C, 54.27; H, 4.22; N, 9.38; S, 5.37. Found: C, 54.04; H, 4.56; N, 9.11; S, 5.19.

1-Oxo-4-phenyl-N^2-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-glucopyranosyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (10a). This compound was obtained in 76% yield, mp 223–225°C, R_f = 0.38 (solvent in system A), R_f = 0.71 (solvent in system B), $[\alpha]_D^{25}$ + 48 (*c* 0.5, $CHCl_3$); IR: 1749(C = O ester), 1690(C = O quinazolinone),

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1670 (C = O), 1625 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.37–9.32 (m, 9H, Ar-H), 5.92(d, 1H, β H-1′, $J_{1'2'}$ = 10.2 Hz), 5.61(t, 1H, H-2′, $J_{2',3'}$ = 10.2 Hz), 5.56(t, 1H, H-3′, $J_{3',4'}$ = 10.0 Hz), 5.26(t, 1H, H-4′, J4′,5′ = 10.2 Hz), 3.95(m, 1H, H-5′), 4.41(dd, 1H, H-6′, $J_{6',6''}$ = 12.3 Hz, $J_{6'5'}$ = 3.9 Hz), 4.28(dd, 1H, H-6″, $J_{6'',5'}$ = 2.8 Hz) 2.16, 2.10, 2.08, 2.00(each s, each 3H, 4Ac); ^{13}C NMR ($CDCl_3$): δ_c 147.8 (C-3a), 161.0(C-5), 117.5(C-5a), 129.8(C-6), 127.7(C-7), 134.8(C-8), 117.6(C-9), 135.7(C-9a), 138.3(C-1 of Ph), 128.6(2C, C-2,6 of Ph), 128.7(2C, C-3,5 of Ph), 129.5(C-4 of Ph), 153.3 (C = O), 84.3(β C-1′), 73.7 (C-2′), 73.2(C-3′), 70.8(C-4′), 69.3(C-5′), 62.8(C-6′), 21.2, 20.7 (2CO*CH*$_3$), 20.5(2CO*CH*$_3$), 171.3, 170.5, 169.7 169.0 (4 *CO*CH_3).

Anal. Calcd for $C_{29}H_{27}N_4O_{11}$ (607.55): C, 57.33; H, 4.48; N, 9.22. Found: C, 57.09; H, 4.83; N, 8.91.

1-Oxo-4-phenyl-N^2-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-galactopyranosyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (10b). This compound was obtained in 64% yield, mp 254–256°C R_f = 0.36 solvent in system A), R_f = 0.72 (solvent in system B), $[\alpha]_D^{25}$ + 39 (*c* 0.5, $CHCl_3$); IR: 1746(C = O ester), 1690(C = O quinazolinone), 1675 (C = O), 1624 cm^{-1} (C = N); ^{13}C NMR ($CDCl_3$): δ_c 147.4 (C-3a), 160.8(C-5), 118.1(C-5a), 129.5(C-6), 128.2(C-7), 134.5(C-8), 117.5(C-9), 135.6(C-9a), 137.7(C-1 of Ph), 128.6(2C, C-2,6 of Ph), 128.7(2C, C-3,5 of Ph), 129.5(C-4 of Ph), 152.7 (C = O), 85.7(β C-1′), 72.5(C-2′), 73.0(C-3′), 72.4(C-4′), 68.6(C-5′), 62.4(C-6′), 21.3–2.00 (4CO*CH*$_3$), 170.6–168.7 (4 *CO*CH_3).

Anal. Calcd for $C_{29}H_{27}N_4O_{11}$ (607.55): C, 57.33; H, 4.48; N, 9.22. Found: C, 57.08; H, 4.22; N, 8.93.

1-Oxo-N^2-(2′,3′,4′,6′-tetra-*O*-acetyl-β-D-glucopyranosyl)-4-(*m*-tolyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (11a). This compound was obtained in 79% yield, mp 258–261°C, R_f = 0.38 (solvent in system A), R_f = 0.73 (solvent in system B), $[\alpha]_D^{25}$ + 85 (*c* 0.5, $CHCl_3$); IR: 1752(C = O ester), 1690(C = O quinazolinone), 1680 (C = O), 1629 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.48–9.62 (m, 8H, Ar-H), 2.48 (s, 3H, *m*-CH_3, J = 7.0 Hz), 5.90(d, 1H, β H-1′, $J_{1'2'}$ = 8.4 Hz), 5.58(t, 1H, H-2′, $J_{2',3'}$ = 8.4 Hz), 5.48(t, 1H, H-3′, $J_{3',4'}$ = 8.6 Hz), 5.31(t, 1H, H-4′, J4′,5′ = 8.4Hz), 3.94(m, 1H, H-5′), 4.42(dd, 1H, H-6′, $J_{6',6''}$ = 12.6 Hz, $J_{6'5'}$ = 3.8 Hz), 4.21(dd, 1H, H-6″, $J_{6'',5'}$ = 2.5 Hz) 2.21, 2.08, 2.06, 2.02(each s, each 3H, 4Ac).

Anal. Calcd for $C_{30}H_{29}N_4O_{11}$ (621.58): C, 57.97; H, 4.70; N, 9.01. Found: C, 57.71; H, 4. 39; N, 8.70

1-Oxo-N^2-(2′,3′,4′-Tri-*O*-acetyl-β-D-xylopyranosyl)-4-(*m*-tolyl) [1,2,4] triazolo [4,3-a] quinazolin-5 (4H)-one (11b). This compound was obtained in 75% yield, mp 297–300°C, R_f = 0.35(solvent in system A), R_f = 0.74 (solvent in system B), $[\alpha]_D^{25}$ + 115 (*c* 0.5, $CHCl_3$); IR: 1753(C = O ester), 1689(C = O quinazolinone), 1675 (C = O), 1625 cm^{-1} (C = N); ^1H NMR ($CDCl_3$): δ_H 7.58–9.96 (m, 8H, Ar-H), 2.45 (s, 3H, *m*-CH_3, J = 7.0 Hz), 5.89(d, 1H, β H-1′, $J_{1'2'}$ = 9.3 Hz), 5.18(t, 1H, H-2′, $J_{2',3'}$ = 9.3 Hz), 5.46(t, 1H, H-3′, $J_{3',4'}$ = 9.0 Hz), 4.83(m, 1H, H-4′, $J_{4',5'}$ = 4.8 Hz), 4.17 (dd, 1H, H-5′, $J_{5',5''}$ = 12.2 Hz), 3.90 (dd, 1H, H-5″, $J_{4',5''}$ = 9.0 Hz), 2.10, 2.08, 2.06 (each s, each 3H, 3Ac), ^{13}C NMR ($CDCl_3$): δ_c 147.6 (C-3a), 161.5 (C-5), 117.4(C-5a), 128.6(C-6), 127.6(C-7), 135.4(C-8), 117.8(C-9), 134.0(C-9a), 137.7(C-1 of Ph), 128.8(C-2of Ph), 138.4(C-3 of Ph), 129.6(C-4 of Ph), 128.0(C-5 of Ph), 126.7(C-6 of

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Ph), 21.7 (*m*-CH_3), 154.3(C = O), 84.8(β C-1′), 73.5(C-2′), 72.7(C-3′), 70.4(C-4′), 62.2(C-6′), 21.0, 20.7, 20.5 (3CO*CH*$_3$), 171.0, 170.3, 169.4 (3 *CO*CH_3); MS (FAB): *m*/*z* (%) 573 (26.42, $[M + Na]^+$, 551 (100, $[M^+H]^+$, 292(11.12, B + 1), 259(53.15, sugar moiety), 235 (5.32), 145(12.15), 118(4.85).

Anal. Calcd for $C_{27}H_{25}N_4O_9$ (549.51): C, 59.01; H, 4.59; N, 10.20. Found: C, 58.72; H, 4.27; N, 10.48.

General deacetylation method. The acetylated product (3.5 mmol) was dissolved in 25 mL of anhydrous methanol, then 1.5 mL of a 1 N NaOMe methanolic solution were added. The reaction mixture was kept at room temperature for the appropriate time (20–30 min.). The process was controlled by TLC (system B) until total deacylation of starting material. The reaction mixture was neutralized with ion-exchange resin Dowex-50 (H^+ form) to pH 6–7. The solution was filtered, the filtrate was concentrated to dryness under reduced pressure and the residue was recrystallized from absolute ethanol. The following products were prepared in this manner.

***N*^{2}-(β-D-Glucopyranosyl)-4-phenyl-1-thioxo[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (12a).** This compound was obtained in 83% yield, mp 274–275°C, R_f = 0.52(solvent in system B), $[\alpha]_D^{25}$ + 18 (*c* 0.8, $CHCl_3$); IR: 3443(OH), 1687(C = O quinazolinone), 1628 cm^{-1} (C = N); ^{1}H NMR (DMSO-d_6): δ_H 7.48–9.24 (m, 9H, Ar-H), 5.41(d, 1H, β H-1′, $J_{1'2'}$ = 9.3 Hz), 3.27–4.45(m, 6H, H-2′, H-3′, H-4′, H-5′, H-6′, H-6″), 4.60 (brs, 1H, D_2O exch., OH), 4.80–5.60 (m, 3H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 146.2 (C-3a), 160.4(C-5), 117.4(C-5a), 128.8(C-6), 127.6(C-7), 134.3(C-8), 117.3(C-9), 136.4(C-9a), 138.0(C-1 of Ph), 128.5(2C, C-2,6 of Ph), 129.2(2C, C-3,5 of Ph), 129.2(C-4 of Ph), 163.9 (C = S), 84.8(β C-1′), 74.6(C-2′), 78.2(C-3′), 72.8(C-4′), 79.1(C-5′), 61.8(C-6′),); MS (FAB): *m*/*z* (%) 479(100, $[M + Na]^+$.

Anal. Calcd for $C_{21}H_{20}N_4O_6S$ (456.47): C, 55.26; H, 4.42; N, 12.27; S, 7.02. Found: C, 54.97; H, 4.09; N, 11.99; S, 6.78.

***N*^{2}-(β-D-Galactopyranosyl)-4-phenyl-1-thioxo[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (12b).** This compound was obtained in 74% yield, mp 289–291°C, R_f = 0.51(solvent in system B), $[\alpha]_D^{25}$ + 26 (*c* 0.8, $CHCl_3$); IR: 3440(OH), 1689(C = O quinazolinone), 1631 cm^{-1} (C = N); ^{1}H NMR (DMSO-d_6): δ_H 7.47–9.28 (m, 9H, Ar-H), 5.42(d, 1H, β H-1′, $J_{1'2'}$ = 10.3 Hz), 3.25–4.42(m, 6H, H-2′, H-3′, H-4′, H-5′, H-6′, H-6″), 4.58 (brs, 1H, D_2O exch., OH), 4.76–5.58 (m, 3H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 146.2 (C-3a), 160.5(C-5), 117.4(C-5a), 129.0(C-6), 127.6(C-7), 134.5(C-8), 117.2(C-9), 136.4(C-9a), 138.2(C-1 of Ph), 128.6(2C, C-2,6 of Ph), 129.2(2C, C-3,5 of Ph), 129.2(C-4 of Ph), 164.1 (C = S), 84.3 (β C-1′), 73.8(C-2′), 78.4(C-3′), 71.6(C-4′), 79.6(C-5′), 62.9(C-6′),); MS (FAB): *m*/*z* (%) 457(100, $[M + H]^+$.

Anal. Calcd for $C_{21}H_{20}N_4O_6S$ (456.47): C, 55.26; H, 4.42; N, 12.27; S, 7.02. Found: C, 54.93; H, 4.22; N, 11.93; S, 7.24.

***N*^{2}-(β-D-Glucopyranosyl)-1-thioxo-4-(*m*-tolyl)[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (13a).** This compound was obtained in 89% yield, mp 229–230°C, (solvent, R_f=0.55 (solvent in system B), $[\alpha]_D^{25}$ + 27 (*c* 0.8, $CHCl_3$); IR: 3439(OH), 1694(C=O quinazolinone), 1631 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6): δ_H 7.52–9.27 (m, 8H, Ar-H), 2.47 (s, 3H, *m*-CH_3, J=7.2 Hz), 5.40(d, 1H, β H-1′, $J_{1'2'}$=10.5 Hz), 3.35–4.43

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(m, 6H, H-2′, H-3′, H-4′, H-5′, H-6′, H-6″), 4.40 (brs, 1H, D_2O exch., OH), 5.00–5.60 (m, 3H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 147.5 (C-3a), 160.4(C-5), 117.3(C-5a), 128.6(C-6), 127.6(C-7), 135.6(C-8), 117.5(C-9), 134.0(C-9a), 137.4(C-1 of Ph), 129.7(C-2of Ph), 138.7(C-3of Ph), 129.9(C-4 of Ph), 128.2(C-5 of Ph), 126.7(C-6 of Ph), 21.8(*m*-CH_3), 164.8 (C = S), 84.3(β C-1′), 74.6(C-2′), 78.5(C-3′), 72.4(C-4′), 79.4(C-5′), 62.3 (C-6; MS (FAB): *m/z* (%) 493(100, $[M + Na]^+$.

Anal. Calcd for $C_{22}H_{22}N_4O_6S$ (470.49): C, 56.16; H, 4.71; N, 11.91; S, 6.81. Found: C, 56.42; H, 4.94; N, 11.65; S, 6.58.

N^2-(β-D-Xylopyranosyl)-1-thioxo-4-(*m*-tolyl)[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (13b). This compound was obtained in 80% yield, mp 315–316°C, R_f = 0.54 (solvent in system B), $[\alpha]_D^{25}$ + 37 (*c* 0.8, $CHCl_3$); IR: 4435(OH), 1687(C = O quinazolinone), 1629 cm^{-1} (C = N); ^1H NMR (DMSO-d_6): δ_H 7.53–9.24 (m, 8H, Ar-H), 2.47 (s, 3H, *m*-CH_3, J = 7.0 Hz), 5.45(d, 1H, β H-1′, $J_{1'2'}$ = 8.2 Hz), 3.18–4.52(m, 5H, H-2′, H-3′, H-4′, H-5′, H-5″), 4.47 (brs, 1H, D_2O exch., OH), 5.00–5.68 (m, 2H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 147.4 (C-3a), 160.6(C-5), 117.8(C-5a), 129.5(C-6), 128.0(C-7), 135.2(C-8), 117.3(C-9), 133.7(C-9a), 137.5(C-1 of Ph), 129.4(C-2of Ph), 138.5(C-3 of Ph), 129.8(C-4 of Ph), 128.5(C-5 of Ph), 126.4(C-6 of Ph), 21.8 (*m*-CH_3), 164.3 (C = S), 84.8(β C-1′), 74.5(C-2′), 78.4(C-3′), 72.5(C-4′), 65.3(C-5′); MS (FAB): *m/z* (%) 463 (54.30, $[M + Na]^+$, 441 (100, $[M^+H]^+$.

Anal. Calcd for $C_{21}H_{20}N_4O_5S$ (440.47): C, 57.26; H, 4.58; N, 12.72; S, 7.28. Found: C, 56.94; H, 4.86; N, 12.41; S, 7.45.

N^2-(β-D-Glucopyranosyl)-1-oxo-4-phenyl[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (14a). This compound was obtained in 68% yield, mp 313–315°C, R_f=0.54 (solvent in system B), $[\alpha]_D^{25}$ + 19 (*c* 0.8, $CHCl_3$); IR: 3445(OH), 1685(C=O quinazolinone), 1672 (C = O), 1628 cm^{-1} (C = N); ^1H NMR (DMSO-d_6): δ_H 7.42–9.10 (m, 9H, Ar-H), 4.73(d, 1H, β H-1′, $J_{1'2'}$ = 8.7 Hz), 3.37–4.49 (m, 6H, H-2′, H-3′, H-4′, H-5′, H-6′, H-6″), 4.64 (brs, 1H, D_2O exch., OH), 4.91–5.67 (m, 3H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 146.6 (C-3a), 160.7(C-5), 117.9(C-5a), 129.2(C-6), 127.8(C-7), 134.7(C-8), 117.5 (C-9), 136.8(C-9a), 138.4(C-1 of Ph), 128.6(2C, C-2,6 of Ph), 129.7(2C, C-3,5 of Ph), 129.6(C-4 of Ph), 153.3 (C = O), 81.4 (β C-1′), 74.2(C-2′), 78.8(C-3′), 73.6(C-4′), 79.5(C-5′), 62.4(C-6′),); MS (FAB): *m/z* (%) 463(100, $[M + Na]^+$.

Anal. Calcd for $C_{21}H_{20}N_4O_7$ (440.41): C, 57.27; H, 4.58; N, 12.72. Found: C, 57.54; H, 4.87; N, 12.37.

N^2-(β-D-Glucopyranosyl)-1-oxo-4-(*m*-tolyl)[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (15a). This compound was obtained in 72% yield, mp 328–330°C, (solvent, R_f = 0.57 (solvent in system B), $[\alpha]_D^{25}$ + 39 (*c* 0.8, $CHCl_3$); IR: 3446(OH), 1694(C = O quinazolinone), 1675 (C = O), 1631 cm^{-1} (C = N); ^1H NMR (DMSO-d_6): δ_H 7.47–9.12 (m, 8H, Ar-H), 2.43 (s, 3H, *m*-CH_3, J = 7.0 Hz), 4.80(d, 1H, β H-1′, $J_{1'2'}$ = 9.4 Hz), 3.40–4.54 (m, 6H, H-2′, H-3′, H-4′, H-5′, H-6′, H-6″), 4.47 (brs, 1H, D_2O exch., OH), 5.25–5.63 (m, 3H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 147.7 (C-3a), 161.0(C-5), 117.5(C-5a), 128.3(C-6), 127.6(C-7), 135.6(C-8), 117.5(C-9), 134.0(C-9a), 137.4(C-1 of Ph), 130.1(C-2of Ph), 138.4(C-3of Ph), 129.4(C-4 of Ph), 128.6(C-5 of Ph),

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126.8(C-6 of Ph), 21.7(*m*-CH_3), 153.8 (C = O), 82.5(β C-1′), 73.2(C-2′), 77.9(C-3′), 73.6(C-4′), 79.7(C-5′), 62.8 (C-6; MS (FAB): *m/z* (%) 477(100, $[M + Na]^+$.

Anal. Calcd for $C_{22}H_{22}N_4O_7$ (454.43): C, 58.15; H, 4.88; N, 12.33. Found: C, 57.84; H, 5.08; N, 12.62.

N^2-(β-D-Xylopyranosyl)-1-oxo-4-(*m*-tolyl)[1,2,4]triazolo[4,3-a] quinazolin-5 (4H)-one (15b). This compound was obtained in 80% yield, mp 355–356°C, R_f = 0.57 (solvent in system B), $[\alpha]_D^{25}$ + 50 (*c* 0.8, $CHCl_3$); IR: 3447(OH), 1689(C=O quinazolinone), 1675 (C = O), 1627 cm^{-1} (C=N); ^1H NMR (DMSO-d_6): δ_H 7.48–9.15 (m, 8H, Ar-H), 2.44 (s, 3H, *m*-CH_3, J = 7.0 Hz), 4.82(d, 1H, β H-1′, $J_{1'2'}$ = 7.9 Hz), 3.28–4.50(m, 5H, H-2′, H-3′, H-4′, H-5′, H-5″), 4.46 (brs, 1H, D_2O exch., OH), 5.18–5.65 (m, 2H, D_2O exch., OH); ^{13}C NMR (DMSO-d_6): δ_c 147.7 (C-3a), 161.3(C-5), 117.6(C-5a), 128.5(C-6), 127.3(C-7), 135.7(C-8), 117.5(C-9), 134.2(C-9a), 137.6(C-1 of Ph), 130.0(C-2 of Ph), 138.5(C-3 of Ph), 129.5(C-4 of Ph), 128.7(C-5 of Ph), 126.9(C-6 of Ph), 21.7 (*m*-CH_3), 153.7 (C = O), 82.4(β C-1′), 73.3(C-2′),77.8(C-3′), 73.0(C-4′), 64.6(C-5′); MS (FAB): *m/z* (%) 425 (100, $[M^+H]^+$.

Anal. Calcd for $C_{21}H_{20}N_4O_6$ (424.41): C, 59.43; H, 4.75; N, 13.20. Found: C, 59.67; H, 4.94; N, 12.87.

REFERENCES

1. Johne, S. Quinazoline, derivatives pharmaceutical research. Pharmazie **1981**, *26* (9), 583–596.
2. Mannschreck, A.; Koller, H.; Stuhler, G.; Davies, M.A.; Traber, J. The enantiomers of methaqualone and their unequal anticonvulsive activity. Eur. J. Med. Chem. **1984**, *19*, 381–384.
3. Hess, H.J.; Cronin, T.H.; Scriabine, A.J. Antihypertensive 2-amino-4(3H)-quinazolinones. J. Med. Chem. **1968**, *11*, 130–136.
4. Hussain, M.A.; Chiu, A.T.; Price, W.A.; Timmermans, P.B.; Shefter, E. Antihypertensive activity of 2[(2-hydroxyphenyl)-amino]-4(3H)-quinazolinone. Pharm. Res. **1988**, *5*, 242–244.
5. Malamas, M.S.; Millen, J. Quinazoline acetic acids and related analogues as aldose reductase inhibitors. J. Med. Chem. **1991**, *34*, 1492–1503.
6. Baker, A.J. Preparation of 4-anilinoquinazolines as anticancer agent. Eur. Patentbl. **1995**, *635*, 498 [Chem. Abstr. **1995**, *122*, 214099].
7. Baek, D.J.; Park, Y.K.; Heo, H.I.; Lee, M.; Yang, Z.; Choi, M. Synthesis of 5-substituted quinazolinone derivatives and their inhibitors activity in vitro. Bioorg. Med. Chem. Lett. **1998**, *8*, 3287–3290.
8. Webber, S.E.; Bleckman, T.M.; Attard, J.; Deal, J.G.; Kathardekar, V.; Welsh, K.M.; Webber, S.; Janson, C. Design of thymidylate synthetase inhibitors using protein crystal structures: the synthesis and biological evaluation of a novel class of 5-substituted quinazolinones. J. Med. Chem. **1993**, *36*, 733–746.
9. Dobosz, M.; Rekas, J.; Pachuta, A. Synthesis of thiocyanates derived from 1,2,4-triazole. Acta Pol. Pharm. **1989**, *46*, 10–13 [Chem. Abstr. **1990**, *112*, 216797].
10. Drelkorn, B.A.; Thibault, T.D. Triazolo[4,3-a]quinoxolines for control of rice. US Patent **1977**, 48322 [Chem. Abstr. **1977**, *86*, 166387].

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11. Abdel-Megeed, M.F.; Saleh, M.A.; Aly, Y.A.; Abdo, I.M. Synthesis, conformational and configurational studies of some new acetylated glycosides of 2-thio-3-aryl-4(3H)-quinazolinones, their thiono and 3,1-benzothiazine2,4-dithione. Nucleosides, Nucleotides Nucleic Acids **1995**, *14*, 1985–1996.
12. Saleh, M.A. Synthesis of some new 3-substituted thio-11H-1,2,4-triazolo[4,3-c] pyrimido[5,4-b]indoles. Sulfur Lett. **2000**, *23* (6), 265–273.
13. Saleh, M.A.; Abbas, Y.A.; Abdel-Hai, F.E.; Youssef, S.A. Synthesis and antiviral evaluation of N-glycosides derived from 6-amino-3-aryl-2-methyl-4(3H)-quinazolinones. Nucleosides Nucleotides Nucleic Acids **2001**, *20* (10 & 11), 1891–1902.
14. Saleh, M.A.; Abdel-Megeed, M.F.; Abdo, M.A.; Shokr, A.M. Synthesis and antiviral evaluation of some new glycosyl thioureas containing a quinazolinone nucleus. Nucleosides Nucleotides Nucleic Acids **2002**, *21* (1), 93–106.
15. Saleh, M.A. Reaction of glycosyl isothiocyanates with 3-indolylaminomethyl-ketone hydrochloride. Nucleosides Nucleotides Nucleic Acids **2002**, *21* (4 & 5), 401–409.
16. Saleh, M.A. A convenient synthesis of novel nucleosides of 2-thioxo-5H-3,4-dihydropyrimido[5,4-b]indol-4-one. Sulfur Lett. **2002**, *25* (6), 235–245.
17. Abdel-Megeed, M.F.; Aly, Y.A.; Saleh, M.A.; Abdo, I.M.; El-Hiti, G.A.; Smith, K. Novel one pot procedure for the preparation of 3-substituted 2-thioxo-4(3H)-quinazolinones. Sulfur Lett. **1995**, *19*, 129–140.
18. Badawy, M.A.; Abdel-Hady, S.A.L.; Ibrahim, Y.A.; Kadry, A.K. Chemistry of quinazolines: reinvestigation of the action of hydrazine on thioxo derivatives. J. Heterocycl. Chem. **1985**, *22*, 1535, 1536.
19. Kottke, K.; Kohmstedt, H. On the hydrazinolysis of 3-aryl-4-oxo-2-thioxo-1,2,3,4-tetrahydroquinazolines and their derivatives. Part 7: synthesis of compounds with aminoguanidine structure. Pharmazie **1982**, *37* (9), 635–637.
20. Wang, L.; Lin, T.S.; Sartorelli, A.C. Conformational and configurational studies on *N*-(substituted phenyl) pyranosylamine derivatives by high resolution nuclear magnetic resonance spectroscopy. J. Org. Chem. **1983**, *48*, 2891–2897.
21. Long, R.A.; Townsend, L.B. Characterization certain sulphur containing nucleosides by ^{1}H nuclear magnetic resonance spectroscopy using the magnetic anistropy effect of thione group. Chem. Commun. **1970**, *13*, 1087–1089.
22. Gracz, H.S.; Sochacka, E.; Malkiewicz, A.; Kuo, K.; Charles, W.; Agris, P.F. Chemistry and structure of the modified uridines in the anticodon wobble position of transfer RNA are determined by thiolation. J. Am. Chem. Soc. **1987**, *109*, 7171–7177.
23. Salekh, M.A.; Krosavina, L.S.; Vigodorchik, M.M.; Turchin, K.F.; Sapukova, A.A.; Anisimova, O.S.; Suvorov, N.N. *N*-Glucosylation of aminoindolines and the bisglucosides of 5-aminoindole. J. Org. Chem. (USSR) (Eng. Trans.) **1989**, *25*, 2619–2628 [Chem. Abstr. **1990**, *112*, 235704].
24. Buchanan, J.G.; McCaig, A.E.; Wightman, R.H. The synthesis of 4-alkylsulphonyl-5-amino and 5-amino-4-phosph-imidazole nucleosides as potential inhibitors of purine biosynthesis. J. Chem. Soc., Perkin Trans. 1 **1990**, *4*, 955–963.
25. Kottke, K.; Kuhmstedt, H. Synthesis of compounds with an aminoguanidin structure. Part 2. 4-Aryl-5-oxo-1-thioxo-1,2,4,5-tetrahydro-*s*-triazolo[4,3-a] quinazoline and derivatives. Pharmazie **1978**, *33*, 124, 125.

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