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SYNTHETIC STUDIES OF 1-TETRA-*O*-BENZOYL-B-D-GLUCOPYRANOSYL-3-(2)-SUBSTITUTED HYDRAZINO-1, 3-BENZOTHIAZOLYL THIOCARBAMIDES

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ABSTRACT
A series of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-(2)-substituted hydrazino-1, 3-benzothiazolyl thiocarbamides **3a-g** have been synthesized by the interaction of Tetra-*O*-benzoyl-β-D-glucopyranosyl isothiocyanate **1** with various 2-hydrazino benzothiazoles **2a-g**.The identities of these new compounds have been established on the basis of chemical transformations and IR, ^{1}H NMR and Mass spectral studies.
Keywords: Synthetic studies, thiocarbamides, isothiocyanate, spectral analysis.

INTRODUCTION

The development of new and different antimicrobial agents has been a very important step and much of the research program efforts are directed toward to the design of new and available drugs, because of the unsatisfactory status of treatments of microorganisms, drug side effects, and the acquisition by the infecting organisms of resistance to the available drugs[1]. A review of the literature revealed that many effective antimicrobial agents have a heterocyclic system in their molecule[2]. Recent observation suggests that several analogues of benzimidazole ring system such as benzoxazole and benzothiazole derivatives also indicate potential activity with lower toxicity in the antimicrobial therapeutic approach in man[3-7]. Benzothiazole derivatives have attracted a great deal of interest due to their biological and commercial importance.

They have been found to have antiviral[8], antibacterial[9], antimicrobial[10], and fungicidal activities[11]. They are also useful as antiallergic[12], antidiabetic[13], antitumor[14], anti-inflammatory[15], anthelmintic[16], and anti HIV agents[17]. The present study is aiming at the synthesis of heterocyclic systems containing the substituted benzothiazole moiety that would be expected to have antimicrobial activity. The present study is aiming at the synthesis of heterocyclic systems containing the substituted benzothiazole moiety that would be expected to have antimicrobial activity. A series of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-(2)-substituted hydrazino-1, 3-benzothiazolyl thiocarbamides**3a-g**have been synthesized by the interaction of Tetra-*O*-benzoyl-β-D-glucopyranosylisothiocyanate**1** with various 2-hydrazino benzothiazoles **2a-g**.

The structures of the products were confirmed by the spectral (IR, ^{1}H NMR and Mass[18-25]) and elemental analysis (Table-1).

EXPERIMENTAL

Melting points of all synthesized compounds were determined using open capillary tube on Mac digital melting point apparatus and were uncorrected. The IR spectrum was recorded in KBr Disks on SHIMADZU IR affinity – 1 – FTIR spectrometer. The NMR spectrum was recorded in Brucker DRX – 300 instruments operating at 300 MHz using $CDCl_3$ solution with TMS as internal standard. The mass spectrum was recorded on a THERMO Finnigan LCO Advantage max ion trop Mass Spectrometer. Specific rotations were measured on Equip-Tronics EQ-801 Digital Polarimeter. Thin layer chromatography (TLC) was performed on silica gel G for TLC (Merck) and spots were visualized by iodine vapours.

General Methods

Tetra-*O*-benzoyl-β-D-glucopyranosylisothiocyanate (1)

Tetra-*O*-benzoyl-β-D-glucopyranosylisothiocyanate1 have been synthesized by the interaction of Tetra-*O*-benzoyl-β-D-glucopyranosyl bromide with lead thiocyanate.

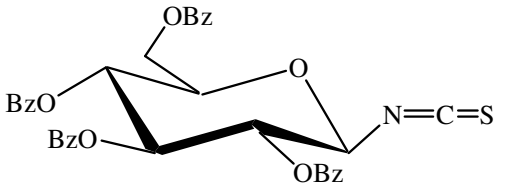
2-Hydrazinobenzothiazoles (2a-g)

Take 2mL of 80% hydrazine hydrate keep it in ice bath and add to it 2 mL of conc. HCl with constant stirring. Then add 5 mL ethylene glycol Keep the flask at room temperature for 5 min Then add 1 gm of 2-amino benzothiazole add reflux on oil bath for 2 hr.

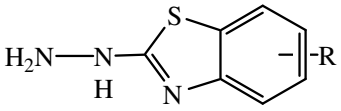
1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-(2)-substituted hydrazino-1, 3-benzothiazolyl thiocarbamides (3a-g)

Mixture of Tetra-*O*-benzoyl-β-D-glucopyranosylisothiocyanate**1** (0.002M, 1.7gm) and Hydrazinobenzothiazoles 2a (0.002M, 0.32gm) was reflux in benzene for about 3 hr. The benzene was distilled off and the sticky mass triturated several times with petroleum ether (60-80 ^{o}C) to afford solid. It was purified by ethanol-water.

Similarly, when the reaction was extended to other 2-Hydrazino benzothiazoles **2b-g** the corresponding benzothiazolylthiocarbamides **3b-g** has been synthesized.

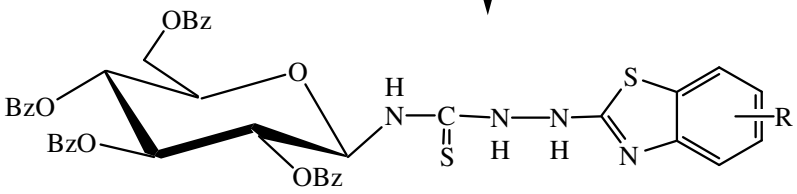


Tetra-*O*-benzoyl-β-D-glucopyranosyl isothiocyanate 1



2- Hydrazino benzothiazoles 2a-g

Reflux
Benzene
3 hr



1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-(2)-substituted hydrazino-1, 3-benzothiazolyl thiocarbamides 3a-g

Where, Bz = COC_6H_5

R = (a) H, (b) 4-Chloro, (c) 5-Chloro, (d) 6-Chloro, (e) 4-Methyl, (f) 5-Methyl, (g) 6-Methyl.

Scheme-1

Spectral analysis

3a: IR(KBr cm^{-1}):3338 (N-H), 3061 (Aromatic C-H), 2974 (Aliphatic C-H), 1741 (C=O),1531 (C=C), 1280 (C-N), 1176 (C-O), 850 (Characteristics of glucose), 771, 717 (mono sub. Ar-H); ^{1}H NMR ($CDCl_3$,ppm):δ 8.04-7.24 (20H, m, Aromatic protons), 5.90-4.84 (7H, m, glucopyranosyl protons), 1.2-1.18 (2H, s, hump N-H),1.16 (1H, s, N-H);Mass (m/z):802 (M^{+}),647, 619, 579, 355, 231, 105.

3d: IR(KBr cm^{-1}):3456 (N-H), 3061 (Aromatic C-H), 2954 (Aliphatic C-H), 1743 (C=O),1490 (C=C), 1286 (C-N), 1176 (C-O), 844 (Characteristics of glucose) 719 (mono sub. Ar-H); ^{1}H NMR

($CDCl_3$,ppm):δ 8.07-7.25 (23H, m, Aromatic protons), 5.92-4.43 (7H, m, glucopyranosyl protons), 0.87 (1H, s, N-H),1.25 (1H, s, N-H),2.31 (1H, s, N-H),1.93 (3H, s, CH_3);Mass (m/z):817 (M^+),723, 661, 619, 579, 557, 413, 397, 240, 133, 105.

3g: IR(KBr cm^{-1}):3444 (N-H), 3062 (Aromatic C-H), 2960 (Aliphatic C-H), 1734 (C=O),1527 (C=C), 1271 (C-N), 1176 (C-O), 852 (Characteristics of glucose) 771, 717 (mono sub. Ar-H); ^1H NMR ($CDCl_3$,ppm):δ 8.08-7.25 (23H, m, Aromatic protons), 5.92-4.38 (7H, m, glucopyranosyl protons), 0.87 (1H, s, N-H),1.25 (1H, s, N-H),2.36 (1H, s, N-H);Mass (m/z):837 (M^+),723, 661, 619, 579, 557, 497, 352, 185, 150, 105.

Table-1: Physical Data of compounds 3(a-g)

S. No.	Products	m.p. (°C)	Yield (%)	R_f Value	$[\alpha]_D^{31}$ (c, in $CHCl_3$]	Elemental Analysis % Found (Required)	
						N	S
1.	3a	172	74	0.92	+52.5 (0.96)	6.90 (6.98)	7.92 (7.98)
2.	3b	179	72.94	0.86	+122.2 (0.9)	6.82 (6.86)	7.82 (7.84)
3.	3c	174	82.21	0.87	+85.1 (0.94)	6.81 (6.86)	7.79 (7.84)
4.	3d	176	80.50	0.90	+52.6 (0.95)	6.78 (6.86)	7.77 (7.84)
5.	3e	187	75.96	0.79	+42.2 (0.96)	6.58 (6.69)	7.55 (7.65)
6.	3f	192	76.29	0.82	+84.1 (0.91)	6.55 (6.69)	7.57 (7.65)
7.	3g	184	78.97	0.86	+81.2 (0.97)	6.60 (6.69)	7.59 (7.65)

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