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Synthesis and Characterization of Some Novel Sugar Containing -2, 4-Isodithiobiurets

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Abstract

A series of Novel 1-aryl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiurets were synthesized by the interaction of 1-aryl-2-*S*-benzyl isothiocarbamides and hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate in benzene medium. The identities of these newly synthesized *N*-lactosides have been established on the basis of usual chemical transformations and IR, ^{1}H NMR and Mass spectral analysis. The polarimetric study of all compounds was carried out. The study of S-and N-glycosides is important in carbohydrate chemistry. Sugar isothiocyanate is good precursors and versatile intermediate for synthesis of S- and N-glycosides. Carbohydrates play an important role in the number of biological events and play an important role in their synthetic strategy as well. Similarly the amino sugars are an important class of glycosidase inhibitors and are arousing great interest as potential therapeutic agents.

***Keywords:** Lactosyl isothiocyanate; 1-aryl-2-S-benzyl isothiocarbamides; isodithiobiurets*

Introduction

Aryl/alkyl isothiocarbamides, due to their basic nature are found to interact with isothiocyante to form corresponding isodithiobiurests. Several non-glycosidic isodithiobiurests are known for their anticonvulsant and hypnotic activities, Glycobiology [1] has gained much attention because the oligosaccharide part and other glycoconjugates are responsible for their function in various biological processes viz. cell growth. Regulation, immunological responses, inflammation and bacterial and viral infections [2-4]. Literature survey reveals that synthesis of amino, diamino derivatives which exhibit biological and pharmaceutical activities such antimalerial effect [5,6]. Glycosyl thiourea has been widely used as important intermediate in the synthesis of nucleoside anologs [7-9]. Thiobiurets, imidazoles and thiazolines also shows anti-inflammatory, antitumor, hypnotic activities [10,11]. In recent years, steadily increasing research effort has centered on the production of glycosyl biurets because these compounds have been shown to possess many different biological activities. Some carbohydrate base urea exhibit relevant biological properties such as the antibiotic SF-1993, CV-1. Nitroso urea have shown to be alpha-glycosidase inhibitors, possesses antitumor activity. In the last years the intensive use of antibiotic has lead to an increase of the emergence of resistant bacteria [12]. There is a growing need for new class of antibacterial compounds having different mechanism of action compared to existing drugs.

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Materials and Methods

Experimental

Melting points of all synthesized compounds were determined using open capillary tube on Mac digital melting point apparatus and were uncorrected. The reagent grade chemicals were obtained from commercial sources and purified by either distillation or recrystallization before use. IR spectra were recorded in solid phase KBr disks on SHIMADZU IR affinity-1 FTIR spectrometer and 1H NMR spectra in CDCl3 on Bruker DRX-300 of NMR spectrometer 300 MHz. The Mass spectra were recorded on Waters UPLC-TQD Mass Spectrometer. Optical rotations were measured on Equip-Tronics EQ 800 Digital Polarimeter in $CHCl_3$. Purity of synthesized compounds has been checked by thin layer chromatography. It was performed on E. Merck pre-coated silica gel plates.

General procedure

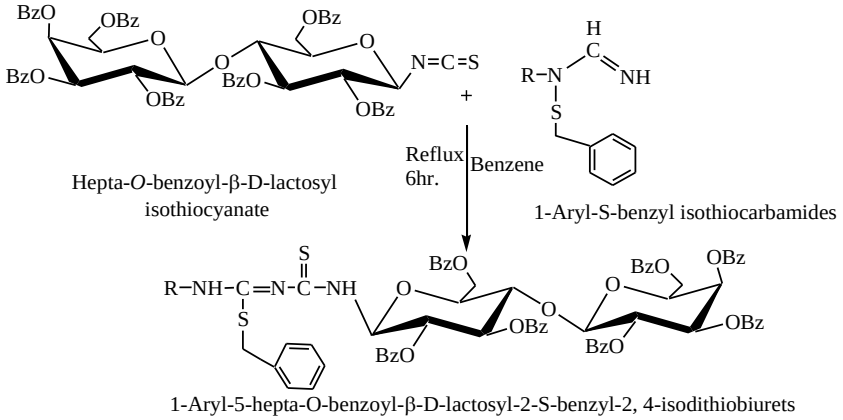

FIG. 1. **1-aryl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiurets**

Where, OBz=Benzoyl

R=(a) Phenyl (b) *o*-Cl-phenyl (c) *m*-Cl-phenyl (d) *p*-Cl-phenyl (e) *o*-tolyl (f) *p*-tolyl (FIG. 1)

Preparation of 1-phenyl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiurete

A benzene solution of 1-phenyl-2-*S*-benzyl isothiocarbamide (0.003 M, 0.72 g in 10 mL) was added to a benzene solution of hepta-*O*-benzoyl-1-β-D-lactosyl isothiocyanate (0.003 M, 3.3 g in 10 mL). The reaction mixture was refluxed for 6 hr and monitored by TLC afterwards, solvent was removed under reduced pressure to obtain sticky residue. This was triturated with petroleum ether (60 – 80°C) to afforded a pale yellow solid (88.99%). The crude product was 1.21 g crystallized by ethanol water m.p. 130°C, Anal. Calcd. For $C_{76}H_{63}O_{17}N_3S_2$, Required: C, 67.35; H, 4.65; N, 3.10; S, 4.73: Found C, 67.33; H, 4.62; N, 3.08; S, 4.75% III (a-f) (Scheme-III).

Results and Discussion

Herein, we report the synthesis of various 1-phenyl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzoyl-2, 4-isodithiobiurets III (a-f) have been synthesized by the interaction of hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate (I) with several 1-aryl-2-*S*-benzyl isothiocarbamides II (a-f) in benzene medium. All products were crystallized from ethanol before recording the physical data (Table 1). The purity of compounds was checked by TLC. The spectral analysis [13-15] IR, 1H NMR and Mass spectra of the product were observed. Optical rotation of the product was also recorded III (a-f) (Scheme-II).

Table 1. **Physical characterization of 1-aryl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzoyl-2, 4-isodithiobiurets (IIIa-f) (Scheme III). Reactants: i) Hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate, ii) 1-aryl-S-benzyl isothiocarbamids**

Sr. No	1-aryl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiurete	Comp.	Yield %	m. p. °C	Elemental analysis Found (Required)		R_f Petroleum ether: EtoAc,7: 3)
					N	S	
1	-Phenyl-	IIIa	68%	130°C	3.08(3.10)	4.75 (4.73)	0.60
2	-o-Cl-Phenyl	IIIb	85%	125°C	3.06(3.02)	4.68 (4.61)	0.65
3	-m-Cl-Phenyl	IIIc	80%	118°C	2.98(3.02)	4.58 (4.61)	0.59
4	-p-Cl-Phenyl	IIId	75%	113°C	3.04(3.02)	4.64 (4.61)	0.65
5	-o-tolyl	IIIe	82%	133°C	3.10(3.07)	4.72 (4.68)	0.50
6	-p-tolyl	IIIf	78%	128°C	3.05(3.07)	4.62 (4.68)	0.70

Spectral data

IIIa) 1-phenyl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiuret: m.p.: 130°C; Yield: 68.00%; IR (KBr, cm-1): υ, 3458 (N-H stretch), 3062.9 (Ar-H stretch), 2976.1 (Ali C-H stretch) 1730.1 (C=O), 1600.9 (C=N), 1379.1 (C-N), 1376.5 (C-O), 1026.4 and 908.4 (characteristic of lactose), 752.2 (C-S); 1H NMR ($CDCl_3$, ppm): δ8.04- 7.18 ppm (m, Ar-H), 6.70 -6.60 (2H, m, NH), δ 6.22 – 3.69 (m, lactosyl protons); Mass (m/z): (M^+) -1353, (M^+-H) -1352, (HBL^+)-1053, (HBL^+-C_7H_5O)-948, (HBL^+-C_7H_5O)-948, (HBL^+-$C_5H_5O_2$) - 931, (TBG)-579, (TBG-C_6H_5)- 474, ($C_6H_5O_2$)-109, C_7H_7- 91,. (Anal. Calcd. For $C_{76}H_{63}O_{17}N_3S_2$, Required: C, 67.35; H, 4.65; N, 3.10; S, 4.73: Found C, 67.33; H, 4.62; N, 3.08; S, 4.75%

IIIb) 1-*o*-Cl-phenyl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiuret: m.p.: 125°C; Yield: 85.00%;

IR (KBr, cm-1): υ, 3352 (N-H stretch), 3062.9 (Ar-H stretch), 2978.3 (Ali C-H stretch) 1730.1 (C=O), 1600.9 (C=N), 1176.5 (C-N), 1379.1 (C-O), 1070.4 and 910.4 (characteristic of lactose), 752.2 (C-S); 1H NMR ($CDCl_3$, ppm): δ8.32- 7.22 ppm (m, Ar-H), 6.70 -6.60 (2H, m, NH), δ 6.23 – 3.90 (m, lactosyl protons); Mass (m/z): (M^+) -1387, (M^+-H) -1386, (HBL^+)-1053, (HBL^+-C_7H_5O)-948, (HBL^+-C_7H_5O)-948, (HBL^+-$C_5H_5O_2$) - 931, (TBG)-579, (TBG-C_6H_5)- 474, ($C_6H_5O_2$)-109, C_7H_7- 91,. (Anal. Calcd. For $C_{76}H_{62}O_{17}N_3S_2Cl$, Required: C, 65.75; H, 6.79; N, 3.02; S, 4.61: Found C, 65.70; H, 6.82; N, 3.06; S, 4.68%

IIIc) 1- *m*-Cl -phenyl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiuret: m.p.: 118°C; Yield: 80.00%; (Anal. Calcd. For $C_{76}H_{62}O_{17}N_3S_2Cl$, Required: C, 65.75; H, 6.79; N, 3.02; S, 4.61: Found C, 65.68; H, 6.75; N, 2.98; S, 4.58%)

IIId) 1- *p*-Cl -phenyl -5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiuret: m.p.:

113°C; Yield: 75.00%; (Anal. Calcd. For $C_{76}H_{62}O_{17}N_3S_2Cl$, Required: C, 65.75; H, 6.79; N, 3.02; S, 4.61: Found C, 65.72; H, 6.73; N, 3.04; S, 4.64%)

IIIe) 1- *o*-tolyl -5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiuret: m.p.:

128-133°C; Yield: 82%; IR (KBr, cm-1): υ, 3462 (N-H stretch), 3062.9 (Ar-H stretch), 2978.0 (Ali C-H stretch) 1730.1 (C=O), 1600.9 (C=N), 1379.1 (C-N), 1176.5 (C-O), 1070.4 and 908.4 (characteristic of lactose), 785 (C-S); 1H NMR ($CDCl_3$, ppm): δ8.21- 7.02 ppm (m, Ar-H), δ 6.19 (2H, m, NH), δ 6.19 –3.68 (m, lactosyl protons), δ2.23-1.25 (s CH_3); Mass (m/z): (M^+)-1367, (M^+-CH_3)-1352, (M^+-C_7H_8ON)-1245, (HBL^+)-1053, (HBL-C_5H_8)-984, (HBL-$C_{14}H_{15}O_7$)-757, (TBG)-579, (TBG-$C_7H_8O_2$)-455, (TBG-$C_5H_{10}O_6$)-413, ($C_{12}H_{22}O_{11}$)-342, (TBG-$C_{13}H_{22}O_8$)-273,($C_8H_4N_3S_2$)-206, ($C_7H_5N_2S$)-149. (Anal Calcd. For $C_{77}H_{65}O_{17}N_3S_2$, Required: C, 67.59; H, 4.75; N, 3.07; S, 4.68: Found C, 67.50; H, 4.72; N, 3.10; S, 4.72%)

IIIf) 1-*p*-tolyl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-*S*-benzyl-2, 4-isodithiobiuret: m.p.:

128°C; Yield: 78.00%; (Anal. Calcd. For $C_{77}H_{65}O_{17}N_3S_2$, Required: C, 67.59; H, 4.75; N, 3.07; S, 4.68: Found C, 67.62; H, 4.78; N, 3.05; S, 4.62%)

Conclusion

In this research work, the characterizations of newly synthesized products were established on the basis of IR, ^{1}H NMR, and Mass spectral studies. Various 1-aryl-5-hepta-*O*-benzoyl-β-D-lactosyl-2-S-benzoyl-2, 4-isodithiobiurets were synthesized and yield of product ranged from 64%-85%.

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