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"Synthesis, Characterization and Spectral Interpretation of 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide"

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ABSTRACT

Serial of the "1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide"was prepared by the interaction of the of Tetra-O-Benzoyl-β-D-glucosyl isothiocyanate and Phenyl thiocarbamide in Benzene Medium. The Reaction was refluxed for 3-hours in benzene medium and the benzene is evaporated and then the product is recrystalised by the pertroleum ether (60-80^{0}C). The identities of these new compounds have been established on the basis of usual chemical transformations and IR, 1HNMR and Mass spectral studies.

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.

Key words: Glucosyl isothiocyanate, Phenyl thiocarbamide, 2,4 dithiocarbamide etc.

INTRODUCTION

Isothiocyanates are important intermediates belonging to the family of compounds known as heterocumulenes. Isothiocyanates are versatile synthetic intermediates in organic chemistry due to their availability and their tendency to undergo nucleophilic addition and cycloadditions[1-3].Literature survey reveals that synthesis of amino, diamino derivatives which exhibit biological and pharmaceutical activities such antimalerial effect[4,5]. Acylureas and biurets are noncyclic compounds which upon replacement of one or both oxygen with sulphur results into the formation of thiobiurets and dithiobiurets respectively. Isodithiobiuret derivatives[6-7] shows potent biological activities such as anticonvulsant, hypnotic, analgesic, antifungal, insecticidal activity. Glycosyl thiourea has been widely

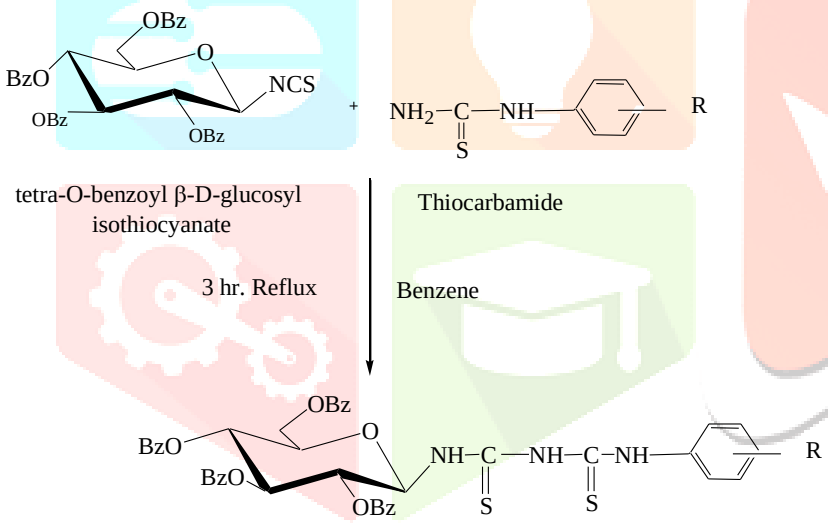
used as important intermediate in the synthesis of nucleoside anologs Nitroso urea have shown to be alpha-glycosidase inhibitors, possesses antitumor activity. In the last years the intensive use of antibiotic has lead to an increase of the emergence of resistant bacteria.[8]. There is a growing need for new class of antibacterial compounds having different mechanism of action compared to existing drugs.

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



1-tetra-O-benzoyl β-D-Glucosyl-5-aryl 2,4 dithiocarbamide

Figure 1: 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide

Where, OBz = Benzoyl

R = a) Phenyl b) *o*-Cl- phenyl c) *m*-Cl- phenyl d) *p*-Cl- phenyl

Preparation of 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide

A benzene solution of Tetra-O-Benzoyl-β-D-glucosyl thiocyanate (0.002 M, 1.358g in 10 mL) was added to a benzene solution of Phenyl thiocarbamide (0.002M, 0.304 g in 10 mL). The reaction mixture was refluxed for 3 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 (80%). The crude product was 1.21 g crystallized by ethanol water m.p. 150°C, Anal. Calcd. For $C_{42}H_{35}O_9N_3S_2$, Required: C, 60.64; H, 4.21; N, 5.05; S, 7.70: Found C, 60.62; H, 4.25; N, 5.01; S,7.68 %

III (a-d) (Scheme-III).

RESULTS AND DISCUSSION

Herein, we report the synthesis of various 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide **III (a-d)** have been synthesized by the interaction of Tetra-O-Benzoyl-β-D-glucosyl thiocyanate **(I)** with several aryl thiocarbamide **II(a-d)** 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[9,11] IR, 1H NMR and Mass spectra of the product were observed. Optical rotation of the product was also recorded **III (a-d) (Scheme-II).**

Table 1: **Physical characterization of 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide III (a-d) (Scheme III).**

Reactants: i) 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide, ii) aryl thiocarbamide

Sr. No	1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide	Comp.	Yield %	m. p. °C	Elemental analysis Found (Required)		R_f Petroleum ether:EtoAc,7: 3)
					N	S	
1	-Phenyl-	IIIa	80%	150°C	5.01(5.05)	7.68 (7.70)	0.58
2	-o-Cl-Phenyl	IIIb	85%	142°C	4.80(4.85)	7.33 (7.39)	0.63
3	-m-Cl-Phenyl	IIIc	80%	162°C	4.90(4.85)	7.30 (7.39)	0.55
4	-p-Cl-Phenyl	IIId	75%	158°C	4.84(4.85)	7.45 (7.39)	0.68

Spectral Data

IIIa) 1-tetra-O-Benzoyl-β-D-glucosyl-5-phenyl 2,4 dithiocarbamide: m.p.: 150°C; Yield: 80.00%;

IR (KBr, cm-1): υ, 3450 (N-H stretch), 3050 (Ar-H stretch), 2963 (Ali C-H stretch) 1720 (C=O), 1655 (C=N), 1255 (C=S),1222 (C-O), 1376.5 (C-O), 1090 (characteristic of glusose), 700 (C-S); **1H NMR ($CDCl_3$, ppm):** δ8.9- 7.18 ppm (m, Ar-H), 6.60 -6.40 (2H, m, NH),

δ 6.22 – 3.69 (m, glucosyl protons); **Mass (m/z):** (M^+) -830, (M-C_6H_6N)-738, (TBG)-579, (TBG-C_6H_5)-474, ($C_6H_5O_2$)-109, C_7H_7- 91,. (Anal. Calcd. For $C_{42}H_{35}O_9N_3S_2$, Required: C, 60.64; H, 4.21; N, 5.05; S, 7.70: Found C, 60.62; H, 4.25; N, 5.01; S,7.68 %

IIIb) 1-tetra-O-Benzoyl-β-D-glucosyl-5-o-Cl-phenyl 2,4 dithiocarbamide: m.p.: 142°C; Yield: 85.00%;

IR (KBr, cm-1): υ, 3458 (N-H stretch), 3050 (Ar-H stretch), 2968 (Ali C-H stretch) 1720 (C=O), 1650 (C=N), 1255 (C=S),1220 (C-O), 1376.5 (C-O), 1090 (characteristic of glusose), 700 (C-S); **1H NMR ($CDCl_3$, ppm):** δ8.10- 7.18 ppm (m, Ar-H), 6.70 -6.60 (2H, m, NH),

δ 6.22 – 3.69 (m, glucosyl protons); **Mass (m/z):** (M^+) -865, (M-C_6H_6N)-773, (TBG)-579, (TBG-C_6H_5)-474, ($C_6H_5O_2$)-109, C_7H_7- 91,. (Anal. Calcd. For $C_{42}H_{34}O_9N_3S_2Cl$, Required: C, 58.26; H, 3.93; N,4.85; S, 7.39: Found C, 58.30; H, 4.02; N, 4.80; S,7.33 %

IIIc) 1-tetra-O-Benzoyl-β-D-glucosyl-5-m-Cl-phenyl 2,4 dithiocarbamide: m.p. : 162ºC; Yield : 80.00%; (Anal. Calcd. For $C_{42}H_{34}O_9N_3S_2Cl$, Required: C, 58.26; H, 3.93; N, 4.85; S, 7.39: Found C, 58.32; H, 4.01; N, 4.90; S, 7.30 %)

IIId) 1-tetra-O-Benzoyl-β-D-glucosyl-5-p-Cl-phenyl 2,4 dithiocarbamide: m.p. : 158ºC; Yield : 75.00%; (Anal. Calcd. For $C_{42}H_{34}O_9N_3S_2Cl$, Required: C, 58.26; H, 3.93; N, 4.85; S, 7.39: Found C, 58.32; H, 4.01; N, 4.84; S, 7.45 %)

CONCLUSION:

In this research work, the characterizations of newly synthesized products were established on the basis of IR, ^{1}H NMR, & Mass spectral studies. Various 1-tetra-O-Benzoyl-β-D-glucosyl-5-aryl 2,4 dithiocarbamide were synthesized and yield of product ranged from 64-85%.

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