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SYNTHESIS OF 1-HEPTA-O-BENZOYL-β-D-LACTOSYL-5-SUBSTITUTED-2-S-BENZYL-2-ISOTHIOBIURETS

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ABSTRACT

Certain1-hepta-O-benzoyl-β-D-lactosyl-5-substituted-2-S-benzyl-2-isothiobiurets have been synthesized for the first time by the interaction of 1-hepta-O-benzoyl-β-D-lactosyl-2-*S*-benzyl isothiocarbamide with various isocyanates. The structures of these new 2-isothiobiurets have been established on the basis of usual chemical transformations and IR, NMR, and Mass spectral analysis. The polarimetric study of title compounds has been carried out.

Key words: Iosothiocarbamide, Isocyantes, Isothiobiurets.

INTRODUCTION

In recent years the chemistry of thiobiurets and related compounds has attracted increasing attention. Physiological and potential chemotherapeutic[1] properties of numerous derivatives have been studied, and possible technical applications, particularly in the field of plastic and resins are embedded in an intensive patent literature. Carbohydrate compounds also shows antibacterial and antifungal activity[2,3].

Isothiobiurets and its alkylated derivatives act as antipyretics when administered subcutaneously (to rabbits). Lethal doses cause decreased blood pressure, lung edema and general collapse[4].

In our laboratory, we have prepared several S-hepta-O-acetyl-lactosyl-1-aryl-5-phenyl-2-isothiobiurets and tested for their biological activity[5,6]. So in view of our interest in the synthesis of new ever type of N-lactosylated isothiobiurets, here we have reported the simple method for the synthesis of isothiobiurets having lactosyl substituent by the interaction of hepta-O-benzoyl-β-D-lactosyl-2-*S*-benzyl isothiocarbamide with various isocyanates.

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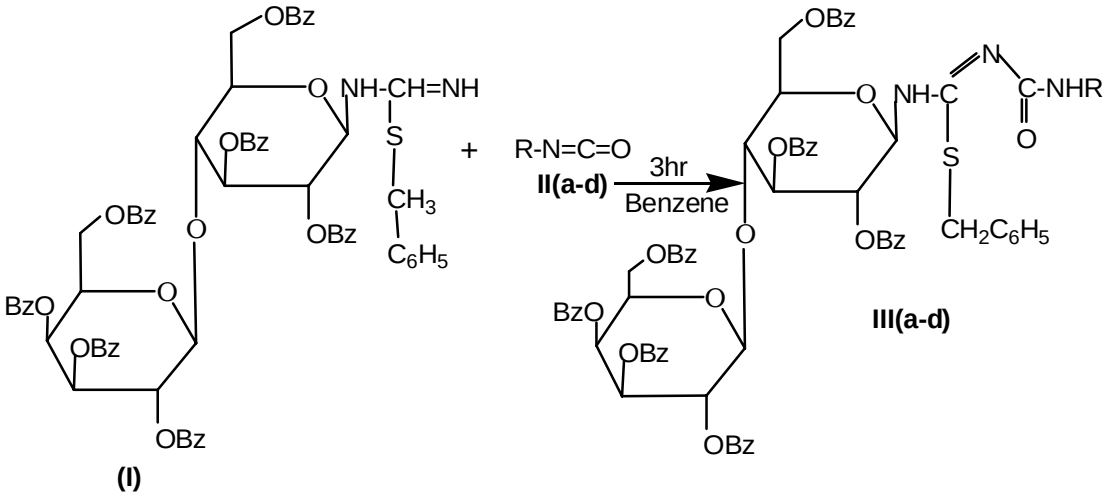
RESULTS AND DISCUSSION

Condensation of 1-hepta-O-benzoyl-β-D-lactosyl-2-*S*-benzyl isothiocarbamide **(I)** has been carried out with 1-hepta-O-acetyl-β-D-lactosyl isocyanate **(IIa-d)** in benzene medium for 3 hr., which gives 1-hepta-O-benzoyl β- D-lactosyl-5- substituted-2-S-benzyl-2-monothiobiurtes **(IIIa-d)**. The structure of the products were confirmed by spectral analysis (IR[7], NMR[8]and Mass[9]). The specific rotations of the products were also recorded[10].



1-Hepta-O-benzoyl-β-D-lactosyl -2-S-1-hepta-O-benzoyl-β-D-lactosyl-5-benzyl isothiocarbamide substituted-2-S-benzyl-2-monothiobiurtes

Scheme 1

Where, R = Where,

R = (a) Hepta-O-acetyl-β-D-lactosyl,

(b) Hepta-O-acetyl-β-D-maltosyl,

(c) Tetra-O-acetyl-β-D-glucosyl,

(d) Tetra-O-acetyl-β-D-galactosyl.

EXPERIMENTAL

Melting points were taken in open capillary tubes and are uncorrected. Specific rotations were measured on Equip-Tronics Digital Polarimeter at 28°C in $CHCl_3$. IR spectra were recorded on Perkin-Elmer spectrum RXI FTIR spectrophotometer (4000-450 cm^{-1}).

^{1}H NMR were recorded in $CDCl_3$ on Bruker DRX-300 spectrometer operating at 300 MHz. The Mass spectra were recorded on Jeol-SX-102(FAB) instrument.

Preparation of 1-hepta-O-benzoyl-β-D-lactosyl-2-S-benzyl isothiocarbamide (I)

The required 1-hepta-O-benzoyl-β-D-lactosyl-S-benzyl-isodithiocarbamide was prepared by already known method. Details of a typical preparation are as follows:

To an ethanolic suspension of thiocarbamide (0.005 M, 6 g in 30 mL) was added benzyl chloride (0.005 M, 3.4 g) and the reaction mixture was refluxed for 90 min. Afterwards, it was cooled and rendered basic with dilute ice cold ammonium hydroxide a sticky residue was obtained which on standing for 1 or 2 hr. solidified (5 g). It was filtered, washed with petroleum ether.

Preparation of sugar isocyanate

To a suspension of hepta-O-acetyl-α-D-lactosyl-bromide (0.03 M, 21 g) in sodium dried xylene (80 mL) was added lead cyanate (0.03 M, 9 g). The reaction mixture was refluxed gently for 3 hr. with frequent shaking. This solution was then cooled and the liberated lead bromide was removed by filtration. The xylene filtrate was then treated with petroleum ether (60-80^{o}C) with stirring, a pale yellow solid. The products were purified by chloroform – petroleum ether.

Synthesis of 1-hepta-O-benzoyl-β-D-lactosyl-substituted-2-S-benzyl-2-isothio-biurets (IIIa-d)

To a benzene solution of 1-hepta-O-benzoyl-β-D-lactosyl-2-S-benzyl isothiocarbamide **(I)** (0.005 M, 3.6 g in 40 mL) was added benzene solution of 1-hepta-O-acetyl-β-D-lactosyl isocyanate (IIa-d) (0.005 M, 1.9 g, 20 mL) and reaction mixture was refluxed over boiling water bath for 3 hr. After heating solvent benzene was distilled off and the sticky mass obtained as residue was triturated several times with petroleum ether to afford a solid **(IIIa-d)** Table 1. The products were purified by chloroform – petroleum ether.

Spectral data

IIIa. IR (KBr): 3065.4 cm^{-1} (Ar-H stretching), 1729 cm^{-1} (C=O), 1600 cm^{-1} (C=N), 850.5 cm^{-1} (lactosyl C-H deformation), 708.9 cm^{-1} (C-H aromatic); ^{1}H NMR (ppm) : δ 7.12-7.07 (10H, m, Ar-H), 8.08-7.89 (2H, S, N-H) 7.14-5.73 (20H, m, lactosylprotons), 4.57-4.21 (5H, d, -OCH_2), 5.91-5.73 (35H, m, 7-COC_6H_5); 4.57-4.21 (21H, S, 7-$COCH_3$) Mass (m/z): 1879 (M^+), 1880, 1052, 579, 391, 335, 105.

Table 1: Synthesis of 1-hepta-*O*-benzoyl-β-D-lactosyl-5-Substituted-2-*S*-benzyl-2–isothiobiurets (IIIa-d)

Reactants- (I) 1-hepta-O-benzoyl-β-D-lactosyl-2-*S*-benzyl isothiocarbamide **(I)**

(II) Sugar isocyanate **(IIa-d)**

Product	Melting point (^oC)	Yield (%)	Analysis found (requires)		$[\alpha]^D_{28}$ (c, 0.156, $CHCl_3$)
			N (%)	S (%)	
IIIa	160^{o}C	80	2.21 (2.23)	1.62 (1.70)	+ 113^0
IIIb	110-112^{o}C	82	2.19 (2.23)	1.52 (1.70)	+ 95^0
IIIc	120-122^{o}C	86	2.51 (2.63)	1.89 (2.00)	+ 98^0
IIId	115^{o}C	85	2.41 (2.63)	1.89 (2.00)	+ 138^0

IIIc. IR (KBr): 3065.4 cm^{-1} (Ar-H stretching), 1747 cm^{-1} (C=O), 1600 cm^{-1} (C=N), 850.5 cm^{-1} (lactosyl C-H deformation), 708.9 cm^{-1} (C-H aromatic); ^{1}H NMR (ppm) : δ7.12-7.07 (10H, m, Ar-H), 8.08-7.89 (2H, S, N-H) 7.14-5.73 (20H, m, lactosylprotons), 4.57-4.21 (5H, d, $-OCH_2$), 5.91-5.73 (35H, m, 7-COC_6H_5); 4.57-4.21 (12H, S, 4-$COCH_3$) Mass (m/z): 1594 (M^+), 1595, 1053, 579, 391, 335, 105.

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