



Microwave Assisted Synthesis of Some New N-lactosylated Thiocarbamates.

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Abstract

“Non-conventional” synthetic method has shown broad applications as a very efficient way to accelerate the course of many organic reactions, producing high yields, higher selectivity and lower quantities of side products consequently easier work-up and purification of the products. In view of these application of microwave a series of new 1-tetra- O - benzoyl -β-D- Glucosyl -3-aryl thiocarbamates have been synthesized by the interaction of tetra – O -benzoyl - β-D-glucosyl isothiocyanate with various alcohols by using microwave. The identities of these new N-lactosides have been established on the basis of usual chemical transformations and IR, NMR and Mass spectral studies.

Key words: Glucosyl isothiocyanate, Phenols and thiocarbamates.

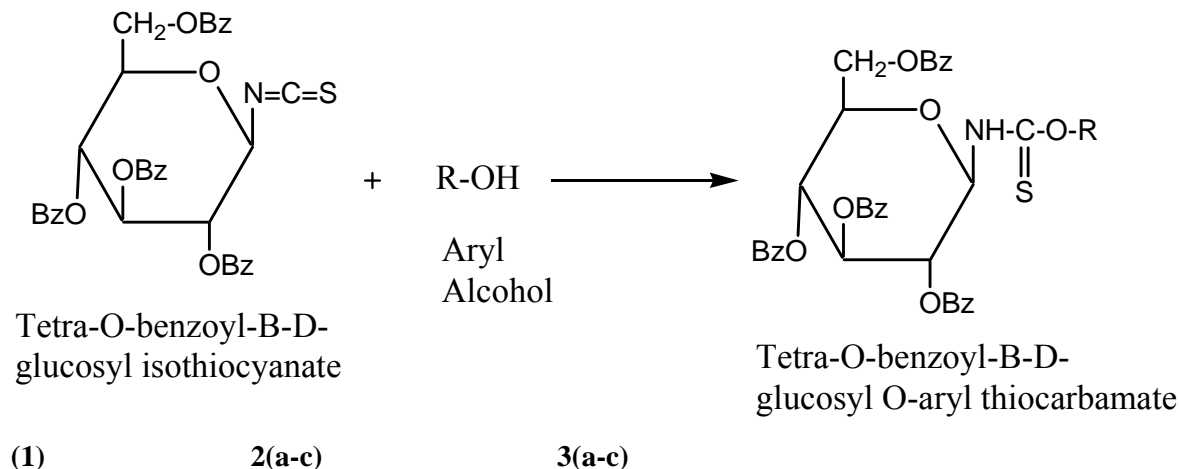
Introduction:

In recent years, the electromagnetic energy in the range of microwaves have gained special attention as regards the most various fields of utilization such as the alimentary (domestic ovens), analytical (small ovens devoted to the mineralization), and that one of bio-medical applications¹. In the field of organic synthetic chemistry, a certain delay has been suffered either in the base research for the clear improvements which can lead to higher yields of cleaner products, minor energy consumption, and environmental compatibility This delay can, however be rapidly reduced by use of electromagnetic energy caused by microwaves. Thus microwave energy can be used and is been used as an activating agent in chemistry for the synthesis of a large variety of compound. Numerous organic reaction assisted by microwave heating have been explained in various article and book^{2,3}

Thiocarbamates and their derivatives show strong antimicrobial activity and are also versatile reagent in organic synthesis⁴. Although they have been known from long ago to be biologically active^{5,6,7}, their varied biological features are still of great scientific interest. Some derivatives of these possess antituberculosis, anticancer, antitumor, antipyretic activities^{8,9}.

In view of applications of thiocarbamates and its derivatives in medicinal chemistry and in many other ways, we herein report the synthesis of several 1-tetra- O -benzoyl-β-D- Glucosyl-O-aryl thiocarbamates (**3a-c**) by the condensation of tetra-O –benzoyl - β-D-Glucosyl isothiocyanate **1** with various alcohols

(2a-c) (Scheme 1). The required lactosyl isothiocyanate was prepared by the reaction of tetra- *O* – benzoyl - α -D-glucosyl bromide with lead thiocyanate⁷.



Scheme-I

Where,

R= a)Phenol b) Resorcinol c) Crysol

Result and Discussion

1-tetra- *O* -benzoyl- β -D- glucosyl-*O*-aryl thiocarbamates (**3a-c**) were prepared by the condensation of 1-tetra-*O* –benzoyl - β -D-glucosyl isothiocyanate **1** with various alcohols (**2a-c**) in microwave for 25min. Then the reaction mixture was pour into ice cold water , with vigorous stirring; a white granular solid was separated out, crystallized from aqueous ethnaol (**3a-c**). The structure of the products were confirmed on the basis of IR¹⁰, NMR¹¹ and Mass¹² spectral analysis. The specific rotation of the products were also recorded¹³.

Experimental:

Specific rotations were measured on Equip-Tronics Digital Polarimeter at 28⁰ C in CHCl₃. IR spectra were recorded on Perkin-Elmer spectrum RXI FTIR spectrophotometer (4000-450 cm⁻¹). ¹H NMR was recorded in CDCl₃ on Bruker DRX-300 spectrometer operating at 300 MHz. The mass spectra were recorded on Jeol-SX-102(FAB) instrument.

Synthesis of tetra-*O*-benzoyl- β -D-glucosyl –*O*-aryl thiocarbamates (**4a-c**):

A mixture of tetra -*O*-benzoyl- β -D- glucosyl isothiocyanate **1** (0.005M,3.5g)in 30 ml of alcohols(**2a-c**) and the reaction mixture was kept in microwave for 5 min . Then the reaction mixture was pour into ice cold water , with vigorous stirring; a white granular solid was separated out, crystallized from aqueous ethnaol when 1-tetra-*O*-benzoyl- β -D-glucosyl –*O*-aryl thiocarbamates (**3a-c**) separate out .



4a. m.p. 95⁰C; yield 72.87%, $[\alpha]_D^{28} +133^0$ (c,1.11 in CHCl₃); IR(KBr): 3373 cm⁻¹ (N-H),1726 cm⁻¹ (C=O), 1284cm⁻¹ (C-N), 1180cm⁻¹ (C-O), ; ¹H NMR (ppm) : δ7.99-7.18 (25H, m, aromatic protons), 5.93-3.76 (7H,m, Glucosyl protons, 1 NH protons); , 2.99-1.20 MASS(m/z):731(M⁺), 579(TBG⁺), ,105(C₆H₅CO⁺); Anal.calcd for C₄₁H₃₃O₁₀NS: C,67.57; H,4.09; N,1.79; S,4.60% ; Found: C,67.30; H,4.51; N,1.91; S,4.73%.

4a. m.p. 145-150⁰C; yield 80%, $[\alpha]_D^{28} +155^0$ (c,1.11 in CHCl₃); IR(KBr): 3362 cm⁻¹ (N-H),1720 cm⁻¹ (C=O), 1289cm⁻¹ (C-N), 1279cm⁻¹ (C-O), ; ¹H NMR (ppm) : δ7.99-7.18 (22H, m, aromatic protons), 5.93-3.76 (7H,m, Glucosyl protons,), 1 NH protons); , 2.99-1.20 . MASS(m/z):731(M⁺), 579(TBG⁺), ,105(C₆H₅CO⁺); Anal.calcd for C₄₁H₃₄O₁₁NS: C,67.03; H,4.51; N,1.81; S,4.37% ; Found: C,67.56; H,4.09; N,1.69; S,4.13%.

Table -1 Tetra-*O*-benzoyl-β-D-glucosyl -*O*-aryl thiocarbamates(3a-c)

Reactants: a) Tetra-*O*-benzoyl-β-D-glucosyl-isothiocyanate(0.005M)(1)
b) Different alcohols (2a-c)

Product	Melting point °C	% yield	Analysis found (requires)		$[\alpha]_D^{28}$ (c,0.15)
			N(%)	S(%)	
3a	95	72.87	1.39 (1.91)	4.13(4.37)	+133 ⁰
3b	145-150	80	1.69 (1.81)	4.37(4.60)	+155 ⁰
3c	143-145	70	1.57 (1.69)	4.02(4.13)	+188 ⁰

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