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SYNTHESIS OF 1-(3-ARYLIMINO-4-ARYL-5-IMINO-1,2,4-THIADIAZOLIDINE)-3-TETRA-*O*-BENZOYL-β-D-GLUCOSYL CARBAMIDES

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ABSTRACT

Synthesis of 1-(3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine)-3-tetra-*O*-benzoyl-β-D-glucosyl carbamides were realized by successful oxidation of aryl thiocarbamides into 3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidines (IIa-c), (so called Hector's base) by using hydrogen peroxide in ethanolic medium, which on reacting with tetra-*O*-benzoyl-β-D-glucosyl isocyanate (I) produces a new series of 1-(3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine)-3-tetra-*O*-benzoyl-β-D-glucosyl carbamides (IIIa-c). The newly synthesized compounds have been characterized by usual chemical transformations and IR spectral analysis.The polarimetric study of the compounds has been carries out.

Keywords: Aryl thiocarbamides, Glucosyl isocyanate, 1,2,4-thiadiazolidines.

INTRODUCTION

The Hector's base are known to be formed by oxidation of monoaryl thiocarbamides with the help of numerous oxidizing agents[1].The drug having Hector's base nucleus enhances pharamaceutical, medicinal, agricultural and industrial application[2]. These drugs have shown a diverse range of physiological activities[3], plant growth promoting activity[4], antismog, herbicidal, antibactrial[5], amoebicidal, antidiabetic agent[6]. The noteworthy and diversified applicability of sugar derivatives have increase our interest to investigate derivatives of 1-(3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine)-3-tetra-*O*-benzoyl-β-D-glucosyl carbamides (IIIa-c). The compounds have characterized by IR spectral analysis. The polarimetric study has been carried out.

EXPEMENTAL

All the chemicals were research grade. All the melting points recorded were found uncorrected. The structures of newly synthesized compounds were confirmed on the basis of elemental and spectral analysis.IR spectra were recorded in KBr disks on SHIMADZU IR affinity-1 FTIR spectrometer. Optical rotation $[\alpha]^{31}_{D}$ were measured on the Equip –Tronic EQ-801 Digital polarimeter at 31°C in $CHCl_3$. Thin layer chromatography was visualized by iodine vapors.

The compounds described in this paper were first time synthesized by multistep reaction protocol.The reagents required for the given synthesis are obtained as-

(1) Synthesis of tetra-*O*-benzoyl-β-D- glucosyl isocyanate (I)

Tetra-*O*-benzoyl-β-D- glucosyl isocyanate (I)[7] is prepared by the interaction of tetra-*O*-benzoyl-α-D-glucosyl bromide and lead cyanate in xylene medium.

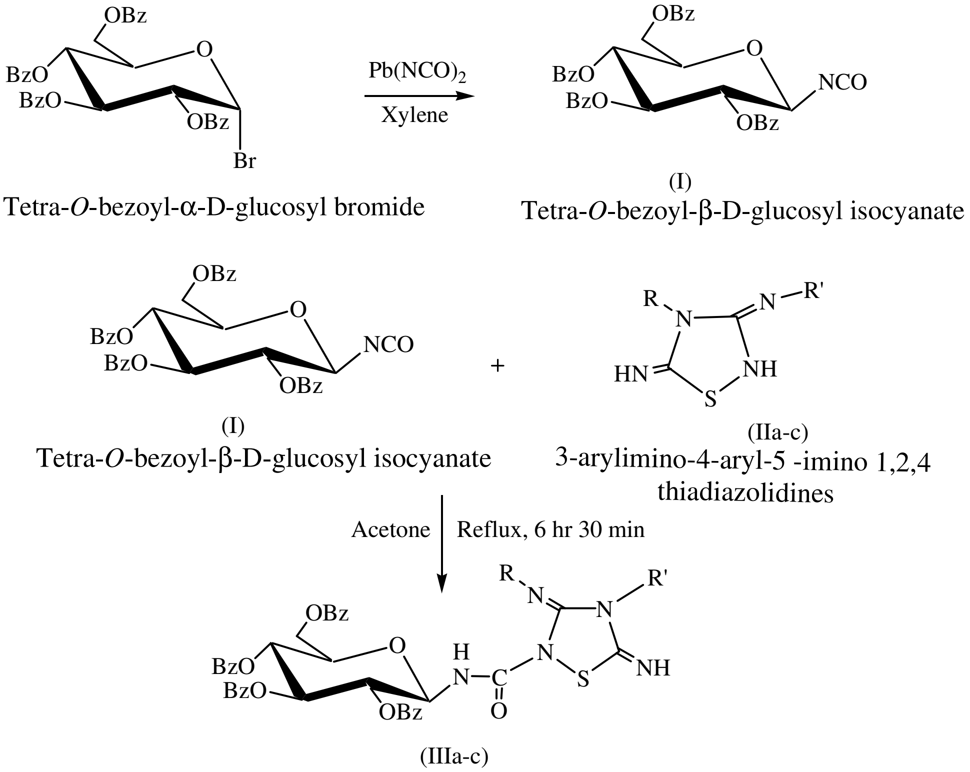
(2) Syntheis of 3-phenylimino-4-phenyl-5-imino-1,2,4-thiadiazolidine (IIa)

To an ethanolic suspension of phenyl thicarbamide (10g in 30ml) acidified with conc. Hydrochloric acid (10ml) was added gradually with proper stirring hydrogen peroxide (20ml of 20 volume strength) reaction was exothermic and thiocarbamide went into solution and a clear solution was obtained. After complete addition of hydrogen peroxide reaction mixture was allowed to stand for 15 min. Suphur was liberated. The liberated sulphur was removed by filtration. The clear filtrate on treatment with dil. Ammonium hydroxide produces 3-phenylimino-4-phenyl-5-imino-1,2,4-thiadiazolidine (IIa) white solid crystallised from ethanol.

(3) Synthesis 1-(3-phenylimino-4-phenyl-5-imino-1,2,4-thiadiazolidine)-3-tetra-*O*- benzoyl-β-D-glucosyl carbamide (IIIa)

The mixture of tetra-*O*-benzoyl-β-D-glucosyl isocyanate (I) (0.6 g, 0.001M) and 3-phenylimino-4-phenyl-5-imino-1,2,4-thiadiazolidine (IIa) (0.2 g, 0.001 M) was refluxed in acetone (30 ml) medium for 6½ hr. The reaction was monitorised by TLC. After the completion of reaction the solvent was removed by distillation and the sticky mass obtained as residue was triturated several times with petroleum ether, a white product separate out.

The structures of all the products synthesized were established on the basis of usual chemical transformations and IR spectral analysis[8-11].



Tetra-*O*-bezoyl-α-D-glucosyl bromide

(I)
Tetra-*O*-bezoyl-β-D-glucosyl isocyanate

(I)
Tetra-*O*-bezoyl-β-D-glucosyl isocyanate

(IIa-c)
3-arylimino-4-aryl-5 -imino 1,2,4 thiadiazolidines

(IIIa-c)
1-(3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine) -3-tetra-*O*-benzoyl-β-D-glucosyl carbamides

Where, Bz = COC_6H_5
R = R` = (a) Phenyl, (b) *o*-Cl-phenyl, (c) *o*-totyl

Scheme - 1

RESULT AND DISCUSSION

The mixture of tetra-*O*-benzoyl-β-D-glucosyl isocyanate (I) and 3-phenylimino-4-phenyl-5-imino-1,2,4-thiadiazolidine (IIa) was refluxed in acetone (30 ml) medium for 6½ hr. The reaction was monitorised by TLC. After the completion of reaction the solvent gets distilled off to obtained a sticky residue. This residue on tituration with petroleum ether afforded a white solid.

When the interaction of tetra-*O*-benzoyl-β-D- glucosyl isocyanate (I) was extended to other 3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine (IIb-c) the related 1-(3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine)-3-tetra-*O*-benzoyl-β-D-glucosyl carbamides (IIIb-c) were obtained.

The IR spectral analysis and elemental analysis (Table-1) clearly indicated the product and assign their structure.

Spectral data

IIIa: IR (KBr, cm^{-1}): 3321.42 (N-H), 3062.96 (Ar. C-H), 1730.15 (C=O), 1666.50 (C=N), 1311.59 (C-N), 1249.87 (C-O), 852.54 (C-S), 935.48 (β-D-glucosyl ring deformation), 707.80 (Monosubstituted benzene).

IIIb: IR (KBr, cm^{-1}): 3377.36 (N-H), 3062.96 (Ar. C-H), 1730.15 (C=O), 1531.48 (C=N), 1312.52 (C-N), 1253.73 (C-O), 883.40 (C-S), 937.40 (β-D- glucosyl ring deformation), 754.17 (Disubstituted benzene).

IIIc: IR (KBr, cm^{-1}): 3321.42 (N-H), 3062.96 (Ar. C-H), 1730.15 (C=O), 1537.27 (C=N), 1313.52 (C-N), 1253.73 (C-O), 854.47 (C-S), 935.48 (β-D- glucosyl ring deformation), 756.10 (Disubstituted benzene).

Table-1: Physical Characterization of Synthesis 1-(3-arylimino-4-aryl-5-imino-1,2,4-thiadiazolidine)-3-tetra-*O*-benzoyl-β-D-glucosyl carbamides (IIIa-c)

S. No.	Product (IIIa-c)	Yield (%)	R_f value (3:7, Acetone:Pet ether)	m.p. (°C)	$[\alpha]^{31}_D$	Elemental analysis Found (Required)	
						N	S
1.	IIIa	66.56	0.24	97-99	-70	7.67 (7.87)	2.95 (3.59)
2.	IIIb	68.74	0.79	102-104	-200	7.47 (7.30)	3.00 (3.48)
3.	IIIc	66.39	0.64	90-93	-110	7.00 (7.03)	3.11 (3.334)

C and H analysis were found satisfactory in all cases.

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