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SYNTHESIS OF SOME NEW -1, 2, 4-DITHIAZOLIDINE HYDROCHLORIDES

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ABSTRACT

Several 3-hepta-O-benzoyl-β-D-lactosyl-4-phenyl-5-arylimino-1, 2, 4-dithiazolidine hydrochlorides have been prepared by the interaction of 1-hepta-O-benzoyl-β-D-lactosyl-S-chloro isothiocarbamoyl chloride and 1-phenyl 3-aryl thiocarbamides . The structure of these new N-lactosylated -1,2,4-dithiazolidine hydrochlorides have been established on the basis of usual chemical transformations and IR , NMR, and Mass spectral analyses.

Key word: 1-Hepta-O-benzoyl-β-D-lactosyl-S-chloro isothiocarbamoyl chloride, 1-3-Diaryl thiocarbamides, 1,2,4-Dithiazolidine hydrochlorides, Synthesis.

INTRODUCTION

Very few compounds containing thioamido group and having lactosyl substituent on nitrogen have been reported and tested for their biological activity[1-3]. Chemistry of N-phenyl-S-chloro isothiocarbamoyl chloride with special utility in the synthesis of nitrogen and sulphur containing five and six membered heterocyclic compounds have been exhaustively investigated by number of chemists[4-6]. In view of our interest in the synthesis of newer types of 1,2,4-dithiazolidines; herein a simple method for the synthesis of 1,2,4-dithiazolidines has been reported.

EXPERIMENTAL

Melting points were taken in open capillary tubes and are uncorrected. Specific rotations were measured on Equip-Tronics Digital Polarimeter at 28^0C 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.

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Synthesis of 1-hepta-O-benzoyl-β-D-lactosyl-S-chloro isothiocarbamoyl chloride (1)

Chlorine gas (generated from 8 g $KMnO_4$ and 50 mL conc. HCl) was passed through a chloroform solution of 1-hepta–O-benzoyl-β-D-lactosyl isothiocyanate (0.005M, 5.5 g) maintaining the temperature below 10^0C. Then the solvent was distilled off and the resulting syrupy mass was triturated several times with petroleum ether (60-80^0C) to afford **(1)**.

Preparation of 1- phenyl 3-aryl- thiocarbamide (2a-g)

1-Phenyl-3-aryl thiocarbamides **(2a-g)** were prepared by the interaction of phenyl isothiocyanate and appropriate aryl amines in benzene medium.

Synthesis of 3-hepta-O-benzoyl-β-D-lactosylimino-4-phenyl-5-arylimino-1,2,4-dithiazolidine hydrochlorides 3(a-g)

A mixture of 1-hepta-O-benzoyl-β-D-lactosyl-S-chloro isothiocarbamoyl chloride **(1)** (0.005 M) and 1-phenyl-3-aryl thiocarbamides **(2a-g)** (0.005 M) in chloroform was refluxed for 3 h. Then the solvent was distilled off and the resulting syrupy mass was triturated several times with petroleum ether (60-80^0C) to afford a pale yellow solid **(3a-g)** (Table 1). The products were purified by chloroform – petroleum ether.

RESULTS AND DISCUSSION

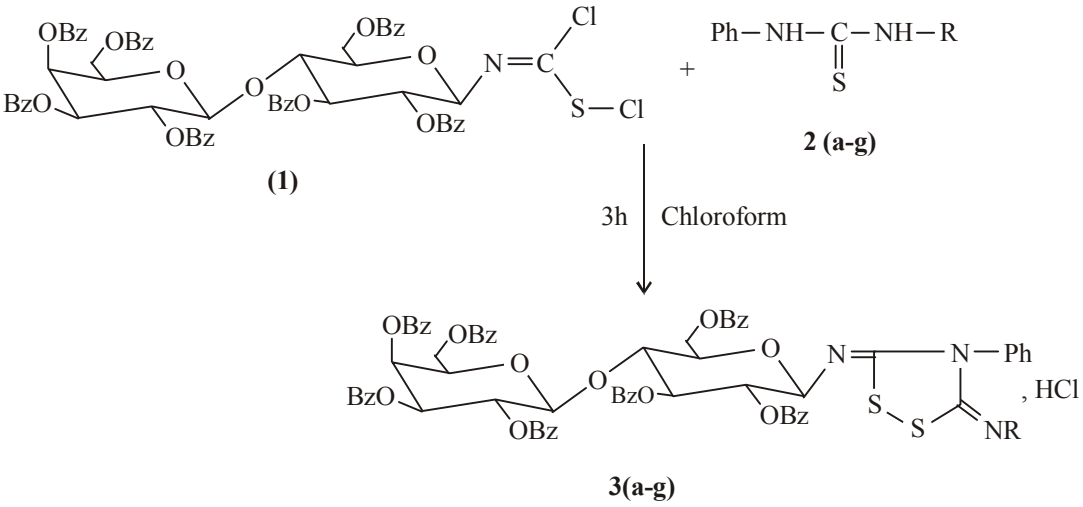
The condensation of 1-hepta-O-benzoyl-β-D-lactosyl-S-chloro isothiocarbamoyl chloride **(1)** and 1-phenyl 3-aryl thiocarbamides **(2a-g)** in $CHCl_3$ was carried out for 3 h. After condensation, the solvent was distilled off and the resulting syrupy mass was triturated several times with petroleum ether (60-80^0C) to afford a pale yellow solid of 3-hepta-O-benzoyl-β-D-lactosyl-4-phenyl-5arylimino-1,2,4-dithiazolidine hydrochlorides **(3a-g) (**Table 1**)**. The products were purified by chloroform-petroleum ether. The structure of the products were confirmed by spectral analysis (IR[7], NMR[8] and Mass[9]). The specific rotation of the products were also recorded[10]. All the compounds have been screened for both; antibacterial and antifungal activity.

Table 1: Characterization data of the synthesized compounds

Product	M.P. (^{o}C)	Yield (%)	$[\alpha]^D_{28}$ (c, $CHCl_3$)
3a	155	83	$+80^0$ (0.156)
3b	146	87	$+108^0$ (0.157)

Cont...

Product	M.P. (^oC)	Yield (%)	$[\alpha]^D_{28}$ (c, $CHCl_3$)
3c	151	90	$+120^0$ (0.155)
3d	144	89	$+95^0$ (0.156)
3e	150	91	$+148^0$ (0.157)
3f	148	85	$+134^0$ (0.158)
3g	142	88	$+110^0$ (0.156)
Satisfactory C, H, N and S analysis were obtained in all cases			



Scheme 1

Where , R = (a) phenyl, (b) p - tolyl, (c) o – tolyl, (d) m- tolyl, (e) p - Cl-phenyl, (f) o - Cl-phenyl and (g) m- Cl -phenyl.

Spectral data

3a. IR(KBr): 3065.4 cm^{-1} (Ar-H stretching),1728.2 cm^{-1} (C=O), 1602.1 cm^{-1} (C=N), 1269.5 cm^{-1} (C-O), 850.5 cm^{-1} (lactosyl C-H deformation) ,765.8 cm^{-1} (C-S) and 708.9 cm^{-1} (C-H aromatic); ^{1}H NMR (ppm) : δ 7.12-7.07 (10H, m, Ar-H) 7.14-5.73 (10H, m, lactosyl protons), 4.57-4.21(4H, d, -OCH_2) and 5.91-5.73 (35H, m,7-COC_6H_5); Mass (m/z) : 1411 (M^+), 1412, 1337, 1052, 579, 391, 335 and 105.

3b. IR(KBr): 3064.5 cm^{-1} (Ar-H stretching),1728.8 cm^{-1} (C=O), 1601.7 cm^{-1} (C=N), 1269.8 cm^{-1} (C-O), 854.4 cm^{-1} (lactosyl C-H deformation), 756.3 cm^{-1} (C-S) and 709.2 cm^{-1}

(C-H aromatic); ^{1}H NMR (ppm) : δ 7.12-7.07 (9H, m, Ar-H) 7.14-5.73 (10H, m, lactosyl protons), 4.57-4.21 (4H,d,-OCH_2), 5.91-5.73 (35H, m, 7-COC_6H_5) and 4.57-4.21 (3H, S, -CH_3); Mass (m/z):1424 (M^+), 1425, 1351, 1052, 579, 391, 335 and 105.

3f. IR (KBr): 3066 cm^{-1} (Ar-H stretching),1728.6 cm^{-1} (C=O), 1601.1 cm^{-1} (C=N), 1270.3 cm^{-1} (C-O), 854.4 cm^{-1} (lactosyl C-H deformation), 763.8 cm^{-1} (C-S) and 709.7cm^{-1} (C-H aromatic); ^{1}H NMR (ppm) : δ 7.12-7.07 (9H,m,Ar-H) 7.14-5.73 (10H,m, lactosyl protons), 4.57-4.21 (4H,d,-OCH_2) and 5.91-5.73(35H, m, 7-COC_6H_5) ; Mass (m/z): 1447 (M^+), 1448, 1372, 1052, 579, 391, 335 and 105.

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