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SYNTHESIS AND SPECTRAL STUDIES OF *N*-GLUCOPYRANOSYL SUBSTITUTED-1, 3, 4-THIADIAZOLIDINES

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ABSTRACT
A series of 2-Tetra-*O*-benzoyl-β-D-glucopyranosyl-4-phenyl-5-arylimino-1, 3, 4-thiadiazolidines have been synthesized by the interaction of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-phenyl amino thiocarbamide with various aryl isocyanodichloride. The synthesized compounds were structurally confirmed by analytical and IR, ^{1}H NMR and Mass spectral analysis.
Keywords: 1, 3, 4-thiadiazolidines, aryl isocyanodichloride, Spectral studies, thiocarbamide

INTRODUCTION

Heterocyclic compounds are found to exhibit anti-inflammatory, anti-parasitic, anti-tubercular, antidiabetic activity[1-3]. In recent years, there has been increasing interest in the synthesis of heterocyclic compounds by cyclization of appropriate linear compounds. Organosulfur compounds play an important role in modern organic synthesis. Recently in our laboratory there are various reports on sugar heterocyclic possessing antimicrobial and antifungal activities [4-6].

In view of applications of these compounds in various fields, we report the synthesis of a series of 2-Tetra-*O*-benzoyl-β-D-glucopyranosyl-4-phenyl-5-arylimino-1, 3, 4-thiadiazolidines **(3a-h)** have been synthesized by the interaction of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-phenyl amino thiocarbamide **(1a-h)** with various aryl isocyanodichloride **2**.The structures of the products were confirmed by the spectral (IR, ^{1}H NMR and Mass[8-15]) and elemental analysis (Table-1).

EXPERIMENTAL

Melting points of all synthesized compounds were determined using open capillary tube on Mac digital melting point apparatus and were uncorrected. The IR spectrum was recorded in KBr Disks on SHIMADZU IR affinity – 1 – FTIR spectrometer. The NMR spectrum was recorded in Brucker DRX – 300 instruments operating at 300 MHz using $CDCl_3$ solution with TMS as internal standard. The mass spectrum was recorded on a THERMO Finnigan LCO Advantage max ion trop Mass Spectrometer. Specific rotations were measured on Equip-Tronics EQ-801 Digital Polarimeter. Thin layer chromatography (TLC) was performed on silica gel G for TLC (Merck) and spots were visualized by iodine vapours.

General Methods

Synthesis of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-phenyl amino thiocarbamide (1)

It was prepared by refluxing Tetra-*O*-benzoyl-β-D-glucopyranosylisothiocyanate and phenyl hydrazine in benzene for 1 hr.

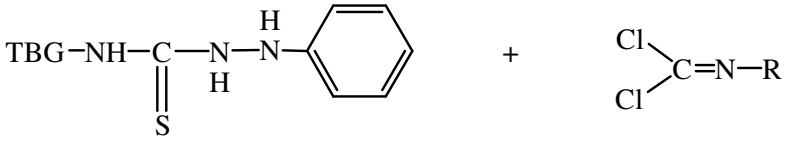
Preparation of aryl isocyanodichlorides (2 a-h)[7]

It were prepared by passing excess amount of gaseous chlorine into the solutions of aryl isothiocyanates in chloroform, aryl isocyanodichlorides were obtained as pale yellow oil.

2-Tetra-*O*-benzoyl-β-D-glucopyranosyl-4-phenyl-5-arylimino-1, 3, 4-thiadiazolidines (3a-h)

Mixture of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-phenyl amino thiocarbamides (**1)** (0.002M, 1.49gm) and phenyl isocyanodichloride **(2a)** (0.002M, 0.268gm) was reflux in benzene for about 3 hr. The benzene was distilled off and the sticky mass triturated several times with petroleum ether (60-80 ^{o}C) to afford solid **(3a)**. It was purified by ethanol-water.

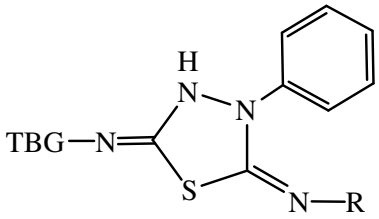
Similarly, when the reaction of 1-Tetra-*O*-benzoyl-β-D-glucopyranosyl-3-phenyl amino thiocarbamide (**1)** was extended to other aryl isocyanodichlorides **(2b-h)** the corresponding 1, 3, 4-thiadiazolidines **(3b-h)** has been synthesized.



1-Tetra-*O*-benzoyl-β-D-glucopyranosyl 3-phenyl amino thiocarbamide **(1)**

Aryl isocyanodichlorides **(2a-h)**

Reflux
Chloroform
3 hr



2-Tetra-*O*-benzoyl-β-D-glucopyranosyl-4-phenyl-5-arylimino-1, 3, 4-thiadiazolidines **(3a-h)**

Where,

Bz = COC_6H_5

R = a) Phenyl, b) *o*-tolyl, c) *m*-tolyl, d) *p*-tolyl, e) *o*-Cl phenyl, f) *m*-Cl phenyl, g) *p*-Cl phenyl, h) *p*-methoxy.

Scheme-1

Spectral analysis

3a: IR(KBr cm^{-1}): 3310(N-H),3061 (Aromatic C-H), 2972 (Aliphatic C-H), 1735 (C=O),1600 (C=N), 1313 (C-O), 1269 (C-N), 852 (Characteristics of glucose), 1176 (C – S); ^{1}H NMR ($CDCl_3$,pm): δ 8.18-7.13 (30H, m, Aromatic protons), 6.57-4.25 (7H, m, glucopyranosyl protons), 4.35 (1H, s, N-H); Mass (m/z):619, 579, 457, 335,105.

3d: IR(KBr cm^{-1}): 3315(N-H),3059 (Aromatic C-H), 2954 (Aliphatic C-H), 1730 (C=O),1651 (C=N), 1600 (C=C), 1315 (C-O),1273(C-N), 852 (Characteristics of glucose), 1176 (C-S).; ^{1}H NMR

($CDCl_3$,ppm):δ 8.18-7.05 (29H, m, Aromatic protons), 6.87-4.25 (7H, m, glucopyranosyl protons), 4.52 (1H, s, N-H),2.32 (3H, s, CH_3); Mass (m/z):860 (M^+), 579, 335,108.

3h: IR(KBr cm^{-1}): 3061 (Aromatic C-H), 2960 (Aliphatic C-H), 1735 (C=O),1620 (C=N), 1600 (C=C), 1313 (C-N), 1278 (C-O), 1070 (Characteristics of glucose); ^1H NMR ($CDCl_3$,ppm):δ 8.18-6.98 (29H, m, Aromatic protons), 6.87-4.40 (7H, m, glucopyranosyl protons), 3.77 (1H, s, N-H),4.48 (3H, s, O-CH_3);Mass (m/z):877 (M^++1), 876 (M^+), 579, 457, 105.

Table-1: Physical Data of compounds 3(a-h)

S. No.	Products	m.p. (°C)	Yield (%)	R_f Value	Elemental Analysis % Found (Required)		$[\alpha]_D^{31}$ (c, in $CHCl_3$]
					N	S	
1.	3a	179	74	0.86	6.59 (6.61)	3.75 (3.78)	+81.2° (0.97 in $CHCl_3$)
2.	3b	185	72.94	0.92	6.48 (6.50)	3.65 (3.71)	+84.1° (0.91 in $CHCl_3$)
3.	3c	174	85.21	0.87	6.47 (6.50)	3.68 (3.71)	+42.2° (0.96 in $CHCl_3$)
4.	3d	178	70.50	0.79	6.48 (6.50)	3.69 (3.71)	+52.6° (0.95 in $CHCl_3$)
5.	3e	190	75.25	0.85	6.28 (6.35)	3.60 (3.63)	+85.1° (0.94 in $CHCl_3$)
6.	3f	181	76.27	0.92	6.29 (6.35)	3.61 (3.63)	+122.2° (0.9 in $CHCl_3$)
7.	3g	171	80.21	0.81	6.32 (6.35)	3.59 (3.63)	+52.50° (0.96 in $CHCl_3$)
8.	3h	196	76.57	0.78	6.36 (6.39)	3.62 (3.65)	+76.2° (0.94 in $CHCl_3$)

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REFERENCES

1. P.C.Tome Joao, Maria G.Neves, A. Tome, A.S.Gavaleiro Jose, Ana F.Mendonca, I.N.Pegado, R. Duarte and M.L.Valdeira, *Bioorg. Med. Chem.*, **14**, 3878 (2005).
2. M. Shiozaki, H. Doi, D. S. Tanaka, T.Shimozato and S. Kurakata, *Bioorg. Med. Chem.*, **14**, 3011(2006).
3. S.Hout, N. Azas, A. Darque, M. Robin, C. Di Giorgio, M.Gasquet, J.Galy and Timon-Savid, *P. Parasitology,* **129**, 525 (2004).
4. A. V. Yadgire and S. P. Deshmukh, *Indian J. Heterocyclic Chem.*, **22**, 67 (2012).
5. P. T. Agrawal and S. P.Deshmukh, *J. Indian Chem. Soc*., **87**, 1259 (2010).
6. K. M. Heda and S. P. Deshmukh, *Rasayan J. Chem.*, **5 (1)**, 24 (2012).
7. G. M. Dyson. And T. Harimgton, *J. Chem. Soc.*, **2**, 191 (1940).

8. R. M. Silverstein, G. C. Bassler and T. C. Morill, Spectrometric Identification of Organic Compound, 5th Edn. , Sons, Inc, New York, (a) p. 127, (b) p.100 (2001).
9. D.H.Williams and I. Fleming, Spectroscopic Methods in Organic Chemistry, IV, Tata McGraw Hill, p. 42 (1991).
10. S. Cao, F. D. Tropper, R. Roy, *ChemInform*, **26 (42)**, 17 (1995).
11. Z. Dai, F. Qu, C. C. Wu and W. Li, *J. Chem. Research (S)*, 106 (2001).
12. R. Varma, S. Y. Kulkarni, C. I. Jose and V. S. Pansare, *Carbohydr. Res*., **25**, 133 (1984).
13. A. Vargas-Berenguel, F. Ortega-Caballero, F. Santoyo Gonzalez, J. J. Garcia Lopez, J. Gimenez-Martine, L. Garcia-Fuentes and E. Ortiz-Salmeron, *Chem. Eur. J.*, **8(4)**, 822 (2002).
14. J. Isac-García, F. G. Calvo-Flores, F. Hernández-Mateo, F. Santoyo-González,*Eur. J. Org. Chem*., **2**, 383 (2001).
15. H. H. A. M. Hassan and A. H. F. El-Husseiney, *Polish J. Chem.*, **75**, 803 (2001).

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