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SYNTHESIS OF NEW 1-HEPTA-*O*-BENZOYL-β-D-LACTOSYL-3-ARYL THIOCARBAMIDES

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ABSTRACT

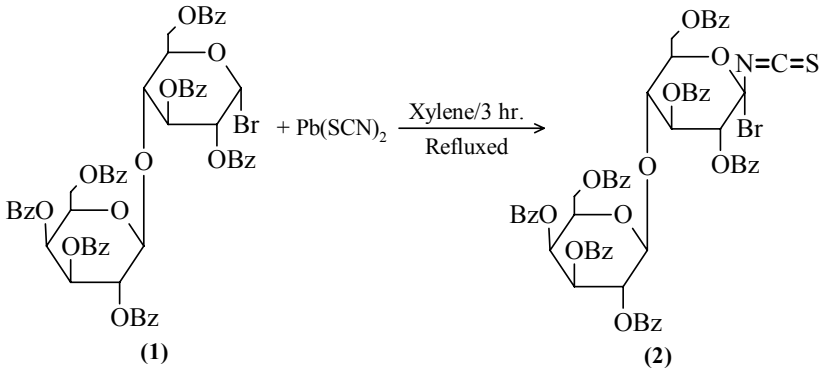
A series of new 1-hepta-*O*-benzoyl-β-D-lactosyl-3-aryl thiocarbamides have been synthesized by the interaction of hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate with aryl amines. The identities of these new *N*-lactosides have been established on the basis of usual chemical transformations and IR, NMR and Mass spectral studies.

Key words: 1-hepta-O-benzoyl-β-D-lactosyl-3-aryl thiocarbamides, Aryl Amine, N-lactoside, IR, NMR, Mass.

INTRODUCTION

Thiocarbamides and their derivatives show strong antimicrobial activity and are also versatile reagent in organic synthesis[1]. Although they have been known from long ago to be biologically active[2-4]. Their varied biological features are still of great scientific interest. Some derivatives of these possess antituberculosis, anticancer, antitumor, antipyretic activities[5,6].

In view of applications of thiocarbamides and its derivatives in medicinal chemistry and in many other ways, we herein report the synthesis of several 1-hepta-*O*-benzoyl-β-D-lactosyl-3-aryl thiocarbamides **(4a-g)** by the condensation of hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate **(2)** with aryl amines **(3a-g)**. The required lactosyl isothiocyanate was prepared by the reaction of hepta-*O*-benzoyl-α-D-lactosyl bromide **(1)** with lead thiocyanate[7] (Scheme 1).



(1) (2)

Scheme 1

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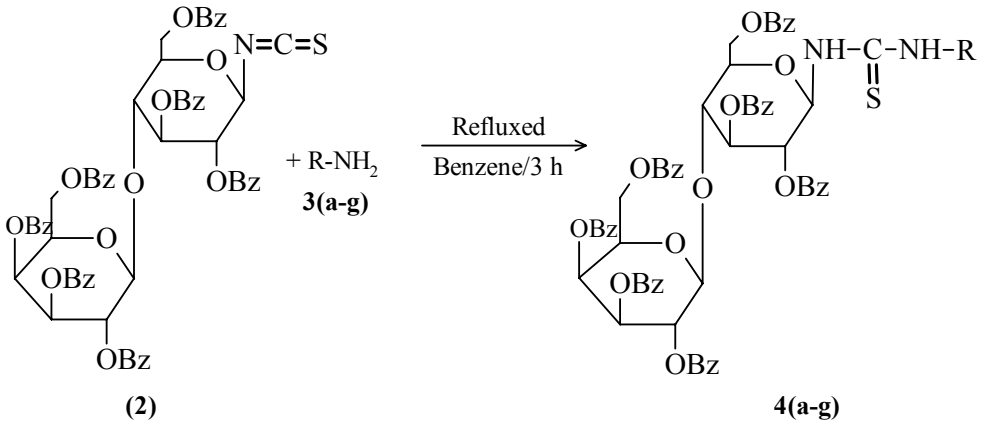

(2) **4(a-g)**

Scheme 2

Where,

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

EXPERIMENTAL

IR spectra were recorded on Perkin-Elmer spectrum RXI FTIR spectrophotometer (4000-450 cm^{-1})[8]. ^1H NMR was recorded in $CDCl_3$ on Bruker DRX-300 spectrometer operating at 300 MHz[9]. The mass spectra were recorded on Jeol-SX-102 (FAB) instrument[10]. Specific rotations were measured on Equip-Tronics Digital Polarimeter at 28°C in $CHCl_3$[11].

Synthesis of 1-hepta-O-benzoyl-β-D-lactosyl-3-aryl thiocarbamides (4 a-g)

(Scheme 2) (Table 1)

A mixture of hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate **(**2) (0.005, 5.5 g in 35 mL) and (0.005 M, 0.46 g) of aryl amines (3a-g) in 30 mL of benzene was refluxed for 3 h while monitoring by TLC. After completion of the reaction, the solvent was triturated with petroleum ether (60-80°C) to afford a white solid (4 a-g). The products were purified from acetone- petroleum ether.

4a. m.p. 128-132°C; yield 76%, $[\alpha]^{28}_D$ +190^0 (c, 1.1 in $CHCl_3$); IR (KBr): 3458 cm^{-1} (NH), 3066 cm^{-1} (Ar-H), 1729 cm^{-1} (C=O), 1271 cm^{-1} (C-N),1176 cm^{-1} (C-O), 1096 cm^{-1} (C=S), 1068,909 cm^{-1} (characteristic of lactose), 708 cm^{-1} (monosubtituted benzene); ^1H NMR (ppm) : δ 8.05-7.18 (40H, m, aromatic protons), 5.91-3.79 (16 H, m, 14 lactosyl protons, 2-NH protons); Mass (m/z): 1204 (M^+), 1145 (M-CH_3COOH), 1100 (M-CH_3COOH CH_2CO), 1052 (HBL^+), 579 (TBG^+), 391 (TBG^+ -$C_{12}H_{12}O_2$), 335 (TBG-$C_{14}H_{12}O_4$), 105 ($C_6H_5CO^+$); Anal. Calcd. for $C_{68}H_{56}O_{17}N_2S$: C, 67.77; H, 4.65; N, 2.32; S, 2.65%; Found: C, 67.70; H, 4.59; N, 2.30; S, 2.60%.

4b. m.p. 130-135°C; yield 79%, $[\alpha]^{28}_D$ + 250^0 (c, 1.11 in $CHCl_3$); IR (KBr): 3446 cm^{-1} (NH), 3068 cm^{-1} (Ar-H), 1728 cm^{-1} (C=O), 1271v (C-N), 1176 cm^{-1} (C-O), 1097 cm^{-1} (C=S), 1026, 909 cm^{-1} (characteristic of lactose), 710 cm^{-1} (monosubtituted benzene); ^1H NMR (ppm) : δ 8.04-7.17 (39H, m, aromatic protons), 5.92-3.79 (16H, m, 14 lactosyl protons, 2 NH protons), 2.28 (3H, s, -CH_3); Mass (m/z): 1218 (M^+ + 1), 1159 (M-CH_3COOH), 1052 (HBL^+), 579 (TBG^+), 391 (TBG^+-$C_{12}H_{12}O_2$), 335 (TBG-$C_{14}H_{12}O_4$), 105 ($C_6H_5CO^+$); Anal.calcd for $C_{69}H_{58}O_{17}N_2S$: C, 67.98; H, 4.76; N, 2.29; S, 2.56%; Found: C, 67.88; H, 4.69; N, 2.28; S, 2.56%.

4e. m.p. 145-150°C; yield 88%, $[\alpha]^{28}_{D}$ +170^0 (c, 1.11 in $CHCl_3$); IR (KBr): 3444 cm^{-1} (NH), 2949 cm^{-1} (Ar-H), 1728 cm^{-1} (C=O), 1272 cm^{-1} (C-N), 1176 cm^{-1} (C-O), 1097 cm^{-1} (C=S), 1026, 907 cm^{-1} (characteristic of lactose), 710 cm^{-1} (monosubtituted benzene); ^{1}H NMR (ppm) : δ 8.05-7.18 (39H, m, aromatic protons), 5.93-3.77 (16H, m, 14 lactosyl protons, 2 NH protons); Mass (m/z) : 1238 (M^+), 1178 (M-CH_3COOH), 1052 (HBL^+), 579 (TBG^+), 391 (TBG^+ -$C_{12}H_{12}O_2$), 335 (TBG-$C_{14}H_{12}O_4$), 105 ($C_6H_5CO^+$); Anal. calcd for $C_{68}H_{55}O_{17}N_2SCl$: C, 65.85; H, 4.43; N, 2.25; S, 2.58%; Found: C, 65.80; H, 4.40; N, 2.23; S, 2.54%.

Table 1: 1-hepta-*O*-benzoyl-β-D-lactosyl -3-aryl thiocarbamides (4 a-g)

Reactant: (a)1-hepta-O-benzoyl-β-D-lactosyl-isothiocyanate (0.005 M, 5.5 g) (2) (b) Aryl amines (3a-g)

Product	Melting point °C	% Yield	Analysis found (requires)		$[\alpha]^{28}_{D}$ (c,0.15)
			N (%)	S (%)	
4a	128-132	76	2.30 (2.32)	2.60 (2.65)	+190^0
4b	130-135	79	2.28 (2.29)	2.56 (2.56)	+250^0
4c	155-160	80	2.30 (2.29)	2.51 (2.56)	+140^0
4d	145	76	2.27 (2.29)	2.53 (2.56)	+180^0
4e	145-150	88	2.23 (2.25)	2.54 (2.58)	+170^0
4f	130	87	2.21 (2.25)	2.59 (2.58)	+140^0
4g	148	76	2.24 (2.25)	2.56 (2.58)	+170^0

RESULTS AND DISCUSSION

1-hepta-*O*-benzoyl-β-D-lactosyl-3-aryl thiocarbamides (4 a-g) were prepared by the condensation of 1-hepta-*O*-benzoyl-β-D-lactosyl isothiocyanate 2 with aryl amines (3a-g) in benzene medium for 3 h. Then, the solvent was distilled off and sticky residue obtained was triturated with petroleum ether (60-80°C) to afford a white solid (4a-g). The structure of the products were confirmed on the basis of IR[8], NMR[9] and Mass[10] spectral analysis. The specific rotation of the products were also recorded[11].

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REFERENCES

1. C. H. Cao, C. J. Zhou, H. Y. Gao and Y. T. Liu, J. Chin. Chem. Soc., **48**, 207 (2001).
2. M. Lacova, J. Chovancova, O. Hyblova and S. Varkonda, Chem. Pap., **44**, 131 (1990).
3. I. Chnlak, V. Sntorins and V. Sederka, Chem. Pap., **49**, 133 (1990).
4. T. Papenfnws, Ger. Offen. De., **3**, 528 (1987).
5. M. S. Shingare and D. B. Ingale, J. Ind. Chem. Soc., **53**, 1036 (1976).
6. B. Dash and M. Patra, Indian J. Chem., **19B**, 894 (1980).

7. J. C. Bailer, H. J. Emeleus, R. Nyholm and A. F. Trotman, Coprehensive Inorganic Chemistry, **2**, Pergamon Press, New York (1973) p. 141.

8. R. M. Silverstein, G. C. Bassler and T. C. Morril, Spectrometric Identification of Organic Compounds, 5th Ed., John Wiley and Sons, INC, New York (2003) p. 108-123.

9. N. B. Colthup, L. H. Daly and S. E. Wiberley, Introduction to Infrared and Raman Spectroscopy, Academic Press, New York (2003) p. 279.

10. D. H. Williams and I. Flemming, Spectroscopic Methods in Organic Chemistry, 4th Ed., Tata McGraw-Hill Publication, New Delhi (2003) p. 40, 41, 47, 53.

11. Weissberger and Arnold, Physical Methods of Organic Chemistry, Part II, 2nd Ed., Interscience Publisher, INC, New York (1949).