

## NOTE

## Studies of Debenzylation, Debenzylation and Oxidative Cyclization of Certain N-Glucosylated-2,4-isodithiobiuret with Thiocarbamide and Elemental Sulphur

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Certain 1-aryl-2-S-benzyl-5-tetra-O-acetyl glucopyranosyl-2,4-isodithiobiurets, debenzylated with the help of thiocarbamide and elemental sulphur in presence of benzylamine and pyridine, respectively, have been investigated.

Elemental sulphur has been known to be an oxidising, dehydrogenating, aromatizing and cyclization reagent<sup>1</sup>. It is also used as sulphurizing and dealkylating agent<sup>2,3</sup>. Debenzylation of certain S-benzyl-2,4-isodithiobiurets was carried out by elemental sulphur<sup>4</sup>, which was subsequently cyclized into related 1,2,4-dithiazolidines. Debenzylation of S-benzyl-1-aryl isothiocarbamide and 1,5-diaryl-2-S-benzyl-2,4-isodithiobiurets was carried out with the help of thiocarbamide<sup>4,5</sup>. The present communication describes the debenzylation, debenzylation and oxidative cyclization of certain 1-aryl-2-S-benzyl-5-tetra-O-acetyl- $\beta$ -D-glucopyranosyl-2,4-isodithiobiurets, with the help of thiocarbamide and elemental sulphur in presence of benzyl amine and pyridine respectively. The former produces 1-aryl-2-benzylimino-5-tetra-O-acetyl- $\beta$ -D-glucopyranosyl amidino thiocarbamide and latter produce 3-phenyl-5-tetra-O-acetyl- $\beta$ -D-glucopyranosyl imidino 1,2,4-dithiazolidine respectively.

All chemicals used were of pure analytical grade as required. 1-Aryl thiocarbamide<sup>4</sup>, S-benzyl-1-aryl-isothiocarbamide<sup>4</sup>, tetra-O-acetyl- $\beta$ -D-glucopyranosyl isothiocyanate<sup>4</sup>, 1-aryl-2-S-benzyl-5-tetra-O-acetyl- $\beta$ -D-glucopyranosyl-2,4-isodithiobiurets were prepared as described<sup>4</sup> where aryl R = phenyl, *p*-Cl-phenyl or *o*-Cl-phenyl.

### (A) Debenzylation with thiocarbamide

(i) *1-Phenyl-2-benzylimino-5-tetra-O-acetyl- $\beta$ -D-glucopyranosyl amidinothiocarbamide* (1a): To a suspension of 1-phenyl-2-S-benzyl-5-tetra-O-acetyl- $\beta$ -D-glucopyranosyl-2,4-isodithiobiurets (29 g) in 20 mL ethanol, thiocarbamide (0.4 g) and benzyl amine (1 mL) were added. The reaction mixture was refluxed for 6 h on water bath. Hydrogen sulphide was found to be eliminated during the heating. After heating, resultant ethanolic solution was treated with excess of

aqueous hydrochloric acid (1:1) to remove the traces of unreacted benzylamine. The resultant semisolid isolated after water washing with aqueous acid was separated and triturated several times with petroleum ether when a white granular product was isolated (47%); m.p. 71°C; (Found: C, 55.92; H, 4.92; N, 9.18; S, 5.18; required: C, 56.67; H, 5.53; N, 9.12; S, 5.21). The positive Molish test confirm the presence of acetylated glucosyl group. On boiling with alkali it did not produce smell of mercaptans indicating the absence of mercapto benzyl group. Soluble in ethanol, methanol, acetone, benzene and insoluble in petroleum ether.

Similarly 1b and 1c were synthesised. (Table-1).

TABLE-1  
PHYSICAL DATA OF COMPOUNDS (1a-c)\*

Compound No.	Aryl	m.p. °C	Yield %
1a	Phenyl	71	47
1b	<i>p</i> -Cl-phenyl	60	53
1c	<i>o</i> -Cl-phenyl	72	62

\*All compounds gave satisfactory C, H, N and S analyses.

### (B) Debenzylation with elemental sulphur

*3-Phenylimino-5-tetra-O-acetyl-β-D-glucopyranosylimino-1,2,4-dithiazolidine* (2b): A reaction mixture of 1-phenyl-2-S-benzyl-5-tetra-O-acetyl-β-D-glucopyranosyl-2,4-isodithiobiurets (29 g), ethanol (20 mL), elemental sulphur (19 g) and pyridine (1 mL) was refluxed over a boiling water bath for 6 h, when hydrogen sulphide was found to be eliminated. The ethanolic clear solution was cooled and suspended elemental sulphur was removed by filtration. The ethanolic clear solution was poured into 100 mL cold water, then pale yellow oily liquid isolated. The oily liquid was triturated with petroleum ether to remove traces of benzaldehyde. After trituration with petroleum ether granular solid was isolated (73%) m.p. 86°C; (Found: C, 47.92; H, 4.28; N, 7.43; S, 11.78; required: C, 48.88; H, 4.81; N, 7.77; S, 11.85).

The product gives Molish test to confirm the presence of acetylated glucosyl group. On boiling with alkali it did not give a smell of mercaptans indicating the absence of mercapto benzyl group. Soluble in ethanol, methanol, acetone, benzene and insoluble in petroleum ether.

Similarly 2b-c were synthesised. (Table-2).

TABLE-2  
PHYSICAL DATA OF COMPOUNDS (2a-c)\*

Compound No.	Aryl	m.p. (°C)	Yield (%)
1.	Phenyl	86	73
2.	<i>p</i> -Cl-phenyl	69	82
3.	<i>o</i> -Cl-phenyl	55	72

\*All compounds gave satisfactory C, H, N and S, Cl analyses.

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