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Preliminary communication

Saccharide oxadiazoles

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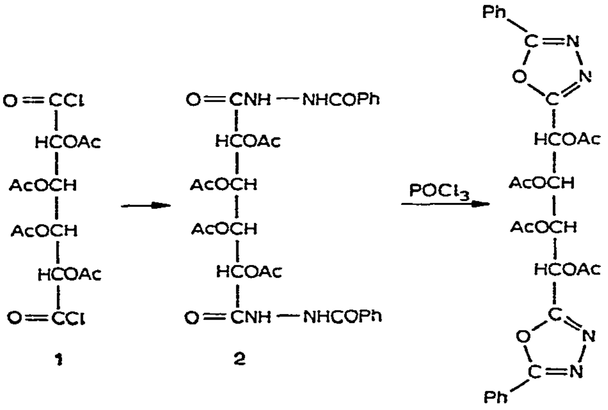
In a previous paper[1], we reported the oxidative cyclization of acetates of aroylhydrazones of aldehydo sugars to a new type of saccharide heterocycle, namely, 5-aryl-2-(polyacetoxyalkyl)-1,3,4-oxadiazoles. Because of the increasing biological and industrial uses of 1,3,4-oxadiazole derivatives[2], we have been interested in the preparation of new types of saccharide 1,3,4-oxadiazoles, and now describe the synthesis of the saccharide di-1,3,4-oxadiazole **3** by the dehydrative cyclization[3,4] of the newly prepared galactaric acid bis(benzoylhydrazide) derivative (**2**).

Treatment of a solution of 2,3,4,5-tetra-*O*-acetylgalactaroyl dichloride[5] (**1**) (1 g) in anhydrous benzene (150 ml) with benzoylhydrazine (0.8 g) in the same solvent (30 ml) at room temperature immediately gave a precipitate of 2,3,4,5-tetra-*O*-acetylgalactaric acid bis(benzoylhydrazide) (**2**) (1.1 g) as crystals, m.p. 275° (dec.). Elemental analysis gave values corresponding to those calculated for $C_{28}H_{30}N_4O_{12}$, and the i.r. spectrum showed four carbonyl bands, at 1755, 1730 (OAc), 1695, and 1670 cm^{-1} (CONH), in addition to NH absorption at 3350 cm^{-1}. The n.m.r. spectrum of **2** showed signals for four *O*-acetyl groups, at δ 2.15 (2 OAc) and 2.18 (2 OAc), and two phenyl groups (between δ 7.4 and 8.1 p.p.m.). Attempted preparation of **2** by direct condensation of 2,3,4,5-



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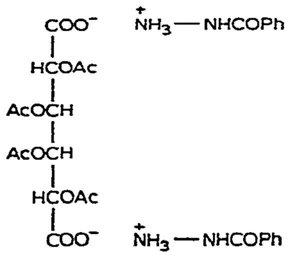
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tetra-*O*-acetylgalactaric acid with benzoylhydrazine failed, probably owing to formation of the unstable salt 4, which would decompose during crystallization to a sugar derivative (unidentified) and 1,2-dibenzoylhydrazine.

Dry 2 (0.5 g) was heated with phosphoryl chloride (10 ml) until complete dissolution occurred (10–15 min), and the mixture was cooled, and diluted with a cold, saturated solution of sodium hydrogen carbonate (200 ml). Extraction with chloroform and evaporation of the extract afforded 1,2,3,4-tetra-*O*-acetyl-1,4-bis(5-phenyl-1,3,4-oxadiazol-2-yl)-*galacto*-tetritol (3) (0.26 g), m.p. 217°; elemental analysis gave values corresponding to those calculated for $C_{28}H_{26}N_4O_{10}$ (two molecules of water per molecule less than for the starting dihydrazide 2), and 3 showed carbonyl ester absorption only at 1755 cm^{-1} (OAc), no amide or NH bands, and bands at 1385 and 980 cm^{-1} associated with the oxadiazole rings[6,7]. Both the dihydrazide 2 and the di-oxadiazole 3 were optically inactive.

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