



EASY AND SELECTIVE ANOMERIC SYNTHESIS OF 1, 2, 3, 4, 6-PENTA-O-ACETYL- α -D (+) – GLUCOPYRANOSE

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ABSTRACT

An easy and economical acetylation method has been developed for the selective anomeric synthesis of 1, 2, 3, 4, 6-penta-O-acetyl- α -D (+)-glucopyranose. The molecular structure has been confirmed by melting point, optical rotation, FTIR, ^1H NMR and elemental analysis techniques.

KEY WORDS: 1, 2, 3, 4, 6-penta-O-acetyl- α -D (+)-glucopyranose, acetylation, anomeric

INTRODUCTION

Penta-O-acetyl- α -D-glucopyranose (α -D-glucose pentaacetate) has a strong functional aspect of insulintropic action by stimulating proinsulin biosynthesis (Malaisse et al., 1997). It has also been used as a rich carbon source for the growth of microbes and as a standard for the determination of acetyl content in many microbial culture media (Osman et al., 1986). The literature survey revealed that hitherto, chemically the acetylation synthetic methods of sugars have reported the use of acetic anhydride and a catalyst like perchloric acid, sulphuric acid, pyridine, hydrobromic acid, sodium acetate or zinc chloride (Wolfram and Thompson, 1963; Furniss et al., 1989; Pasto et al., 1992; Ault, 1998; Nicholas and Smith 1948; Barczai, 1950; Fritz and Schenk 1959; Hudson and Dale 1915; Redemann 1942; Conant 1928; Whitman and Schwenk 1946; Mohammed

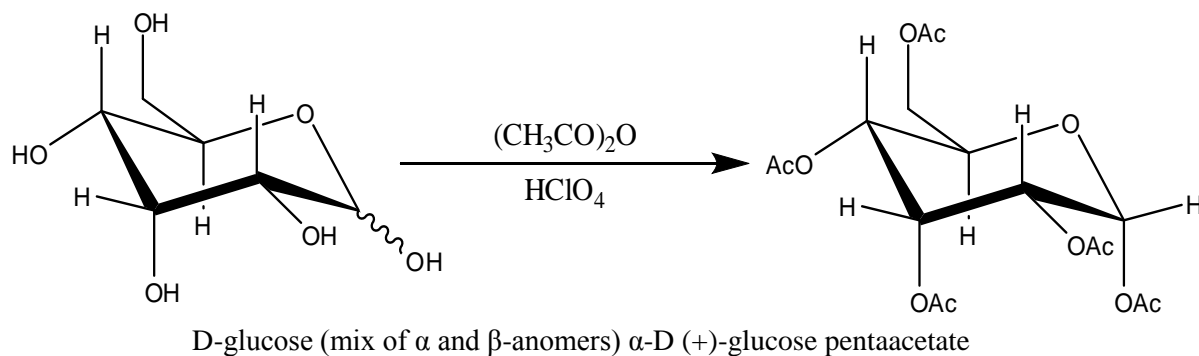
and Jwad 2011). In this short communication a new modified, easy and economical synthetic method has been described.

MATERIAL AND METHODS

The melting point was recorded using a Sonar melting point apparatus in an open capillary tube and was uncorrected. Optical rotation was determined at 25 $^{\circ}$ C on a Rudolph 21 CFR 11. The FTIR spectral study was performed on Shimadzu FTIR 8400 by KBr disc method. ^1H NMR spectrum was recorded on Jeol FX 300Q FTNMR spectrophotometer and the elemental analysis was carried out on Exeter Analytical Lnc Model CE-440 CHN Analyzer.

Synthetic procedure

In a typical and optimized synthesis of penta-O-acetyl- α -D-glucopyranose, 3.0g of



Scheme 1. Selective anomeric acetylation of D-glucose

D-glucose was taken in a conical flask containing 10 ml of acetic anhydride and 0.7 ml of perchloric acid of 70% was added drop wise with the help of a pipette with constant swirling till the glucose dissolved and the temperature maintained not to exceed above 35 °C. The mixture kept for half an hour at laboratory temperature and then poured in a beaker containing ice water. The product was formed after vigorous stirring, filtered off and washed with sufficient cold water. The obtained solid was dried and recrystallised from hot ethanol (Scheme 1).

Structural characterization of 1, 2, 3, 4, 6-penta-O-acetyl- α -D (+)-glucopyranose

White amorphous solid, yield: 90%; m.p. 110 °C; optical rotation: $[\alpha]_D^{25} +103.7^\circ$ (c 0.28 in CHCl_3); FTIR (KBr): $\nu = 2960(\text{C-H}_{\text{str}}, \text{CH}_3)$, $1730(\text{C=O}_{\text{str}})$, $1465(\text{C-H}_{\text{bend}}, \text{CH}_2)$, $1375(\text{C-H}_{\text{bend}}, \text{CH}_3)$ cm^{-1} ; $^1\text{HNMR}$ (300MHz, CDCl_3): $\delta = 2.21(\text{s}, 15\text{H}, \text{CH}_3)$, $4.11(\text{m}, 2\text{H}, \text{CH}_2)$, $4.29(\text{m}, 1\text{H}, \text{ring-CH})$, $5.19(\text{m}, 2\text{H}, \text{ring-CH})$, $5.49(\text{m}, 1\text{H}, \text{ring-CH})$, $6.31(\text{s}, 1\text{H}, \text{ring-CH})$ ppm; Calcd. C 49.23, H 5.68, O 45.09, Found: C 49.19, H 5.59, O 45.03.

RESULTS AND DISCUSSION

Acetylation is a most common method in synthetic organic chemistry using many reagents and catalysts. The synthetic procedure of penta-O-acetyl- α -D-glucopyranose is very well documented in Vogel's Textbook of Practical Organic Chemistry using D-glucose, acetic acid, acetic anhydride and perchloric acid, however we tried it for ten times and were not getting the product and hence modified the method as mentioned above. The obtained product was very pure and all the physico-chemical and spectral characterization data were highly consistent with the reported data.

CONCLUSION

The novelty and benefits of the present modification method can be reported as the use of small amount of acetic anhydride, no use of acetic acid, direct addition of perchloric acid and higher percentage yield. This method is very useful, effective and convenient. This current developed method can be used for the routine synthesis of α -D-glucose pentaacetate.

CONFLICT OF INTEREST

The authors declare no conflict of interest.



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