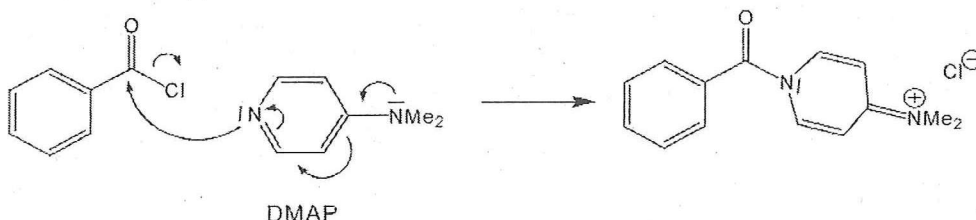


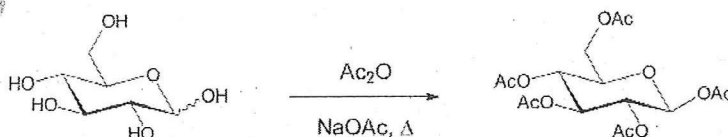
Glucose pentaacetat

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Benzoylations are normally carried out with benzoyl chloride in pyridine at room temperature. Acylations in pyridine can be accelerated by the addition of dimethylamino pyridine (DMAP, 'Steglich's base'¹²) to the reaction mixture acting as nucleophilic catalyst.



Synthesis of penta-O-acetyl- β -D-glucopyranose.¹³



Ausatz: 10% → A suspension of 25 g (0.3 mmol) anhydrous sodium acetate in 300 ml (3.2 mmol) of acetic anhydride is heated to reflux temperature in a round-bottomed flask. Then, the heating is removed and anhydrous D-glucose (50 g, 0.3 mmol) is added in portions, so that the reaction mixture continues to reflux. When all glucose is added, the reaction mixture is stirred under reflux for 30 min, poured onto 1 liter of crushed ice and stirred for 2 h. The precipitated product can then be filtered off, washed with water and recrystallized from ethanol to yield 53 g (50%) of colorless crystals melting at 135°C.

General acetylation procedure using acetic anhydride-pyridine. The compound to be acetylated is dissolved in dry pyridine (approx. 10 ml pyridine for 1 g of sugar), acetic anhydride (2 equivalents per OH-group) is added and the mixture is stirred at room temperature until the reaction is complete (observed using TLC). In the case of small scale reactions (less than approx. 500 mg starting material) for work-up, co-evaporation with toluene followed by flash chromatography is sufficient to obtain pure acetylated product. When larger amounts of acetic anhydride are used, an aqueous work-up is advisable prior to flash chromatography.

General benzoylation procedure. The compound to be benzoylated is dissolved in dry pyridine (approx. 10 ml pyridine for 1 g of sugar), benzoyl chloride (2 equivalents per OH-group) is added and the mixture is stirred at room temperature until the reaction is complete (observed using TLC). Water is added, and the solution is extracted three times with dichloromethane. The combined organic phases are successively washed with satd. aq. NaHCO₃ solution, 1N HCl, and water, dried over MgSO₄, filtered and concentrated *in vacuo*. Flash chromatography yields the pure benzoylated product.

Th. K. Lindhorst, *Essentials of Carbohydrate Chemistry and Biochemistry*, 3. Auflage, Wiley-VCH, Weinheim 2007, S. 62.