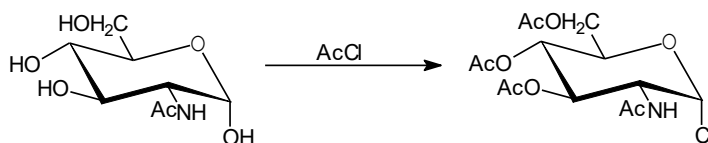


2. Synthesis of 2-acetamido-3,4,6-tri-*O*-acetyl-2-deoxy- α -D-glucopyranosyl chloride



In a 100 mL round-bottomed flask equipped with a magnetic stirrer bar and a reflux condenser protected by a tube of calcium chloride is placed 6 mL of acetyl chloride; this operation and the subsequent reaction are conducted in a hood. The condenser is temporarily removed, and 3 g. of dried 2-acetamido-2-deoxy-D-glucose (*N*-acetylglucosamine) is added in the course of 2 or 3 minutes with good stirring. The mixture is stirred for 72 hours without external heating at a room temperature of approximately 25 °C. The mixture boils spontaneously during the first hour of reaction. It is a clear, viscous, amber liquid at the end of the reaction.

Through the condenser there is added 30 mL of methylene chloride and the solution is poured with vigorous stirring onto of ice and of water. The mixture is transferred to a separatory funnel and shaken. The organic solution is drawn off without delay into a beaker containing ice and saturated sodium bicarbonate solution. The mixture in the beaker is stirred, and the neutralization is completed by shaking the mixture in the separatory funnel. The organic layer is run directly into a flask containing anhydrous magnesium sulfate. The entire washing procedure should be completed within 15 minutes. The solution is shaken or stirred with the drying agent for 10 minutes. The drying agent is separated on Büchner funnel and is well washed methylene chloride.

The filtrate is concentrated on a rotary evaporator at 50 °C, and dry ether is rapidly added with swirling to the warm solution. Crystallization usually begins after about 30 seconds.

Analytically pure 2-acetamido-3,4,6-tri-*O*-acetyl-2-deoxy- α -D-glucopyranosyl chloride is obtained; weight 1,5 g; m.p. 122–124 °C.

The obtained chloride is stored over calcium chloride in the freezer.