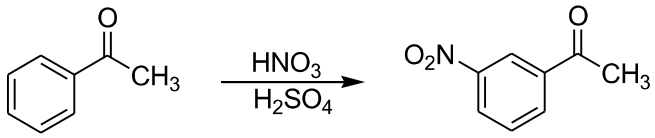
Synthesis of 3-nitroacetophenone

Reaction:



Procedure:

Take a 250 cm^3 flask with three necks with a mechanic stirrer, dropping funnel and a thermometer impinging the reaction mixture and pour 37 cm^3 of concentrated sulphuric acid. Switch on the stirrer, cool down the flask (cooling mixture: ethanol-dry ice) until the temperature in the flask drops to 0 °C.

Then carefully add drop by drop 0.125 mol of acetophenone so that the temperature inside the flask does not exceed 5 °C. Then cool the mixture down to -7 °C and be adding a cooled nitration mixture consisting of 15 cm^3 of concentrated sulfuric acid and 10 cm^3 of concentrated nitric acid a rate so that the whole operation does not take more than 30 minutes and the temperature of the reaction mixture is still within -5-0 °C. After adding the nitration mixture maintain the cooling and stirring for next 10 minutes. Then stir the mixture with a glass rod and during the stirring pour it on a crushed ice mixture of 165 g of ice and 375 cm^3 of water. By the stirring the product is precipitated as a yellow wax or solid substance.

Once the ice is melted suck off the nitro compound and remove water on a filter by pressing with a glass stopper. After bringing it into a glass beaker wash it out twice with 75 cm^3 of water and blend it in 8 cm^3 of ethanol. After each of these three washing suck up the product and squeeze it out perfectly with a glass stopper. Then re-crystallize the product from 25-35 cm^3 of ethanol with addition of activated carbon. Filter the hot solution into 250 cm^3 of cold water at intensive stirring with glass rod. After several minutes of standing suck up the product, wash it with 50 cm^3 of cold water and squeeze it to dry as much as possible. Do the second re-crystallization from about 30 cm^3 of ethanol at using activated carbon. Suck up the created yellow crystals and let them dry in the air. Determine the yield and measure the melting point (temperature). **Store the product in a marked vessel for next lab task – synthesis of 1-(3-nitrohpenyl)ethanol and 3-aminoacetophenone!**

Additional questions:

1. Why must be the reaction mixture cooled during the synthesis of 3-nitroacetophenone?
2. Why takes the substitution the position meta?
3. Why is acetophenone first dropped to sulphuric acid and afterwards the nitration mixture is added?