Technische Universität München
AuTUM
Monika Partsch

4-Nitroacetanilide

Chemicals

- Acetic acid, konz.
- Sulfuric acid, conz.
- Acetanilide
- Nitric acid, conz.
- Sodium chloride
- Ethyl alcohol

Material

- 500 mL- multiple neck flask
- Stirring motor
- Stirrer with locking
- Cooler with cooling tubes
- Thermometer with joint
- Beaker glasses
- Droping funnel
- Cooling bath
- Heating unit
- Measuring cylinder
- Erlenmeyer flask
- Round bottom flask
- Funnel with folded filter
- Powder funnel
- Suction filter with rubber cuff
- Feeding bottle
- Vacuum connection
- Spatula
- Porcelain stick, porcelain bowl
- Drying oven
- Precision balance

Safety tips

Acetanilide
- H272 H302 H410
- P210 P280 P273 P301+P312
- HAZARD!!

Nitric acid, conz.
- H272 H314
- P210 P280 P301+P330+P331 P304+P340 P309+P310
- HAZARD!!

Ethyl alcohol:
- H225
- P210 P243 P280
- HAZARD!!

Acetic acid, conz.
- H226 H314
- P210 P243 P280 P301+P330+P331 P304+P340 P309+P310
- HAZARD!!

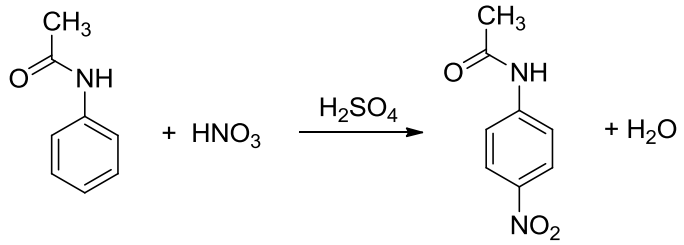
Sulfuric acid, conz.
- H314, H290
- P280, P301+P330+P331, P305+P351+P338, P309+P310
- HAZARD!!

4-Nitroacetanilide
- H319 H335 H315
- P280 P302+P352 P304+P340 P305+P351+P338 P309+P311
- ATTENTION!!

This project has been funded with support from the European Commission. This publication reflects the views only of the author, and the Commission cannot be held responsible for any use which may be made of the information contained therein.

Reaction Equation:

Nitration/ Electrophilic substitution at aromates:



Acetanilid
M=135,17 g/mol

4-Nitroacetanilid
M=180,16 g/mol

Experimental procedure

- Prepare a suspension of 25 g of acetanilide and 25 mL of glacial acetic acid in a 500 mL multiple neck flask
- Stir vigorously and ad drop wise 50 mL of conc. sulfuric acid (in the beginning make sure that the velocity of drops is faster to cause a rise of temperature to 40° C)
- After cooling with a mixture of ice and sodium chloride below 10° C, add drop wise to the clear solution a mixture of 13 mL conc. nitric acid and 8 mL conc. sulfuric acid (carefully produced previously using ice cooling)
- The temperature must stay below 10° C during the drop wise addition
- After removing the cooling mixture, stir for another hour at room temperature
- Then pour the contents of the flask onto 250 g of crushed ice and stir
- Under repeated stirring leave the crystal pulp for 15 minutes and suction-filter sharply
- Suspend the filter cake in a beaker glass two times with 300 mL of water each, then suction-filter and wash acid-free (approx. 6x100mL)
- Suction-filter the raw product as dry as possible
- Then recrystallize with ethyl alcohol
- Cool the filtrate to 20° C and suck off
- Wash the filter cake two times with 15 mL of cold ethyl alcohol each
- Dry the product to mass consistency at 100° C

Waste disposal:

- Dispose of the filtrates in the container for acid wastes

Analysis:

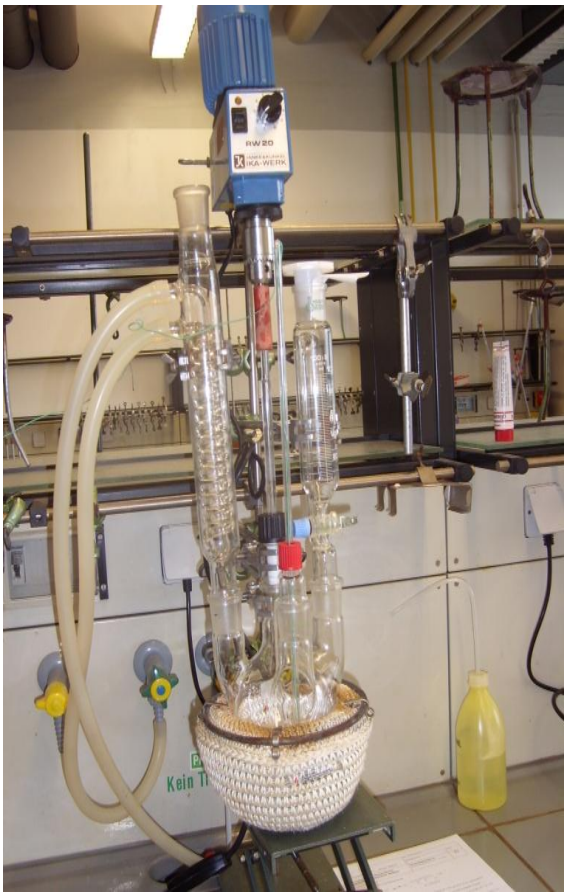
- Calculate the yield of product regarding acetanilide in grammes and percentage of theory

This project has been funded with support from the European Commission. This publication reflects the views only of the author, and the Commission cannot be held responsible for any use which may be made of the information contained therein.

Preparation list

Chemicals:

- Acetic acid, conc. 25 mL
- Sulfuric acid, conc. 58 mL
- Acetanilide 25 g
- Nitric acid conc. 13 mL
- Sodium chloride for the ice mixture
- Ethyl alcohol approx.. 350 mL



Tools:

- 500 mL- multiple neck flask
- Stirring motor
- Stirrer with locking
- Cooler with cooling tubes
- Thermometer with joint
- Beaker glasses
- Droping funnel
- Cooling bath
- Heating unit
- Measuring cylinder
- Erlenmeyer flask
- Round bottom flask
- Funnel with folded filter
- Powder funnel
- Suction filter with rubber cuff
- Feeding bottle
- Vacuum connection
- Spatula
- Porcelain stick, porcelain bowl
- Drying oven
- Precision balance

This project has been funded with support from the European Commission. This publication reflects the views only of the author, and the Commission cannot be held responsible for any use which may be made of the information contained therein.