# 4-Aminobenzene sulphonic acid

# **Chemicals**

- Acetic anhydride
- Sulfuric acid, conc.
- Acetanilide
- Hydrochloric acid, conc.
- Ethyl alcohol
- Activated carbon

## **Materials**

- • 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!!

### Hydrochloric acid, conc. H314 H335

- P280 P301+P330+P331 P305+P351+P338 P309+P310
  - HAZARD!!





- H225
- P210 P243 P280
- ATTENTION!!



### Acetic anhydride

- H226, H302+H332, H314, H335
- P210, P243, P280, P301+P330+P331, P304+P340, P309+P310
- HAZARD!!



## Sulfuric acid, conc.

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

# 4-Aminobenzene sulphone acid:

- H319 H315 H317
- P280 P302+P352 P305+P351+P338
- ATTENTION!!







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# **Reaction equation**

$$CH_3$$
 $+H_2SO_4$ 
 $-H_2O$ 
 $CH_3$ 
 $+HCI, +H_2O$ 
 $-CH_3COOH$ 
 $-CH_3COOH$ 
 $-CH_3COOH$ 
 $-CH_3COOH$ 
 $-CH_3COOH$ 

## **Experimental procedure**

- Place 83 mL of acetic anhydride in a 500 mL multiple neck flask and add dropwise 14 mL conc.sulfuric acid at 20°C
- After that add 30 g Acetanilide whilst stirring
- Heat the mixture to 85-90°C and hold for 15 minutes
- Then cool to 20°C and suction-filter sharply free from water
- Return the raw 4-Sulfacetanilide into the flask, suspend it in 100 mL water and heat to boiling whilst stirring
- Within 5 minutes add 35 mL conc. hydrochloric acid and boil for another 5 minutes under reflux
- Then cool to 20°C and suction-filter sharply (washing liquid = mother liquor)
- Wash the filter cake two times with 30 mL ethyl alcohol each, then pre-dry in a porcelain bowl for 30 minutes at 80°C
- Recrystallize the raw product with 350 mL of water using activated carbon, then suction-filter at 20°C and dry at 110°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





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# **Preparation list**

## Chemicals:

Acetic anhydride
Sulfuric acid, conc.
Acetanilide
Hydrochloric acid, conc.
Ethyl alcohol
Activated carbon
83 mL
14 mL
30 g
55 mL
60 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
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