

## Acetylsalicylic acid






### Chemicals

- Salicylic acid, impurified
- Standard solution, known titre – sodium hydroxide-solution 0.1 M
- Indicator solution
- Glacial acetic acid
- Acetic anhydride
- Mixture of ethyl alcohol and water (1:2)
- Activated carbon
- Distilled water

### Materialien

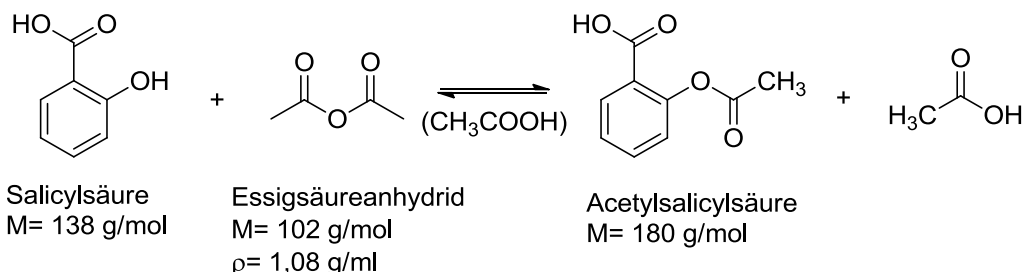
- Burette
- Erlenmeyer flask
- Measuring flask
- Bulp pipette with pipetting aid
- 500 mL multiple neck flask
- Lifting platform
- Beaker glasses
- Liquid funnel
- Measuring cylinder(2x 50 mL)
- Dropping funnel
- Glass stirrer
- Solid matter funnel
- Thermometer with ground joint
- Clamps and screwed joints
- Heating basket
- Stirring motor with stirrer and stirring locking
- Drying oven
- Analysis balance
- Reflux condenser

### Safety tips

<p><u>Acetic acid 100%:</u></p> <ul style="list-style-type: none"><li>• H226, H314</li><li>• P210, P243, P280, P301+P330+P331, P304+P340, P309+P310</li><li>• HAZARD!!</li></ul> 	<p><u>Acetic anhydride</u></p> <ul style="list-style-type: none"><li>• H226, H302+H332, H314, H335</li><li>• P210, P243, P280, P301+P330+P331, P304+P340, P309+P310</li><li>• HAZARD!!</li></ul> 
<p><u>Salicylic acid</u></p> <ul style="list-style-type: none"><li>• H302, H318</li><li>• P280, P305+P351+P338, P309+P310</li><li>• HAZARD!!</li></ul> 	<p><u>Ethyl alcohol:</u></p> <ul style="list-style-type: none"><li>• H225</li><li>• P210 P243 P280</li><li>• HAZARD!!</li></ul> 
<p><u>Acetylsalicylic acid:</u></p> <ul style="list-style-type: none"><li>• H302 H319 H335 H315</li><li>• P280 P302+P352 P305+P351+P338 P309+P311</li><li>• ATTENTION!!</li></ul> 	

## Reaction equation

Esterification by acidic catalysis:



## Experimental procedure

### **Purity determination of the salicylic acid:**

- Titrate the homogenized educt with the standard solution (known titre)
- Chose your own indicator solution
- Assumption:  $w(\text{salicylic acid}) = 75\text{-}85\%$  pure

### **Description of 1-mol preparation:**

- Place 1.2 mol acetic anhydride and 1.7 mol acetic acid in a 500 mL multiple neck flask apparatus with stirrer
- Add 1 mol salicylic acid at room temperature
- Stir the emerged suspension for two hours at  $100^\circ\text{C}$
- Hold the temperature for 10-15 minutes and within this time add drop wise 500 mL of water, then cool the mixture down to  $20^\circ\text{C}$  → stir for another 30 minutes
- Suction-filter the white suspension and wash two times with 150 mL water each
- Recrystallize the moist raw product with activated carbon from the mixture of ethyl alcohol and water
- Then dry the recrystallized product at  $105^\circ\text{C}$  and determine the melting point of the dry product

## Waste disposal:

- Dispose of the mother liquor in the container for non-halogenic solutions
- Dispose of the wastes of the titration in the container for alkaline solutions

## Analysis:

- Calculate the purity of the impurified salicylic acid
- Calculate the charging stock for the production of a minimum of 25 g acetylsalicylic acid with a gain ratio of 60-65%
- Calculation of the yield in grammes and percentage of theory
- Determination of the melting point of the prepared product

## Preparation list

### Chemicals:

- Salicylic acid w=75-85% ca. 50 g
  - impurified with sodium cholride
- Acetic acid anhydride ca. 50 mL
- Acetic acid ca. 100 mL
- Ethyl alcohol 500 mL
- Activated carbon
- NaOH solution c=0,1 M 150 mL
  - known titre
- phenolphthalein solution w(indicator)= 0,1% in ethyl alcohol

### Tools:

- Burette
- Funnel
- Beaker glasses
- Erlenmeyer flask
- Weighing glass
- Stirring motor
- Stirrer
- Stirrer with locking
- 500 ml multiple neck flask with joint
- Thermometer with joint
- Reflux cooler with cooling tubes
- Dropping funnel
- Heating unit
- Powder funnel
- Suction strainer with round filter
- Evacuation bottle with rubber collar
- Evacuation unit
- Measuring cylinder
- Cooling bath
- Glass funnel with fitting folded filter
- Porcelain bowl
- Glass stirrer
- Drying oven
- Precision balance
- Melting-point apparatus
- Melting-point tube

