

Tittle: Preparation of acetylsalicylic acid

Work instructions

Task: Prepare acetylsalicylic acid (Aspirin) by acetylation of salicylic acid.

Theory

Acetylation of 2-hydroxybenzoic (salicylic) acid is the addition-elimination reaction of an aromatic alcohol with acetic anhydride in the presence of sulfuric acid to form Aspirin according to Eq:



Equipment: boiling flask (100 ml) with stopper, magnetic stirrer with stir bar, laboratory stand, graduated cylinder, Büchner funnel, suction flask, rubber adapter, water jet pump

Chemicals: salicylic acid, sulfuric acid (98%), acetic anhydride (99%)

Procedures:

1. All work is carried out with safety goggles!

Acetylation of salicylic acid

- Mix 2.5 g of salicylic acid, 5 ml of acetic anhydride and 2 drops of sulfuric acid in a 100 ml flask with a stir bar. Stir the reaction mixture on the magnetic stirrer. The temperature rises spontaneously to 70-80°C and the salicylic acid gradually dissolves. After 12 to 15 minutes, the temperature drops and the reaction mixture clears. Continue stirring for a further 20 min while the contents of the flask solidify.
- 2. Add 25 ml of water to the flask and mix well. Aspirate the product on a Büchner funnel, wash with more water, dry.

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Management of chemical substances

Chemicals	Form	H-statements	P-statements
Salicylic acid	Solid	H302, H318, H361d	P305+P351+P338,
			P310
H_2SO_4	Liquid, 98%	H290, H314	P260, P280, P303 +
			P361 + P353, P304 +
			P340 + P310, P305 +
			P351 + P338
Acetic anhydride	Liquid, 99%	H226, H302, H314,	P280, P305 + P351 +
		H332	P338, P310

Sources of risk and assessment of risk severity

There is no risk when following all the principles for working with chemicals and using personal protective equipment (gloves, goggles, lab coat).

Waste management method

Dispose of waste materials in a marked container. Do not return unconsumed residues to storage bottles. Dispose of broken glass in a marked container.

Risk reduction measures

Avoid direct exposure, use protective equipment. Do not expose to prolonged or repeated exposure. In the event of an accident or if you feel unwell, inform the teacher immediately. These substances or their containers must be disposed of as hazardous waste. Do not eat, drink, smoke. Wash your hands with warm water and soap or treat them with a reparative cream after work or when interrupting work.

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Worksheet

Calculations

1. Calculate the theoretical yield of acetylsalicylic acid (C₉H₈O₄). Start from the mass of salicylic acid (C₇H₆O₃).

 $M(C_7H_6O_3) = \dots g.mol^{-1}, M(C_9H_8O_4) = \dots g.mol^{-1}$

 $m(C_9H_8O_4) = \dots g$

2. Calculate the practical yield of acetylsalicylic acid v %. PV = g, TV = g

PV(%) =%

Observation

- 1. Describe the appearance and odor of acetylsalicylic acid.
- 2. Search for the basic physicochemical properties of acetylsalicylic acid.

Feature	Description
Solubility in water	
Solubility in other solvents	
Density	
Melting temperature	

Yield

1. Record the mass of acetylsalicylic acid prepared.

 $m(C_9H_8O_4) = \dots g$



Questions

- 1. Write the equation of the reaction for the preparation of acetylsalicylic acid.
- 2. Explain the function of sulfuric acid in this reaction.
- 3. Indicate how you would determine the purity of the product obtained.
- 4. Analyze the factors that can reduce the yield of the reaction.
- 5. Suggest how to increase the purity of synthesized acetylsalicylic acid.
- 6. State the practical significance of this synthesis and its industrial applications.

Conclusion

Briefly summarize the objective of the experiment, the main results and compare them with the expected values.

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