

## Experiment

**Aim:** To prepare and submit salicylic acid from methyl-salicylate by hydrolysis reaction.

**Reference:**

1. Green Chemistry Environmentally Benign Reaction by V.K. Ahluwalia; Published by Ane Books Pvt. Ltd. First Edition; Page No. 155

**Requirement:**

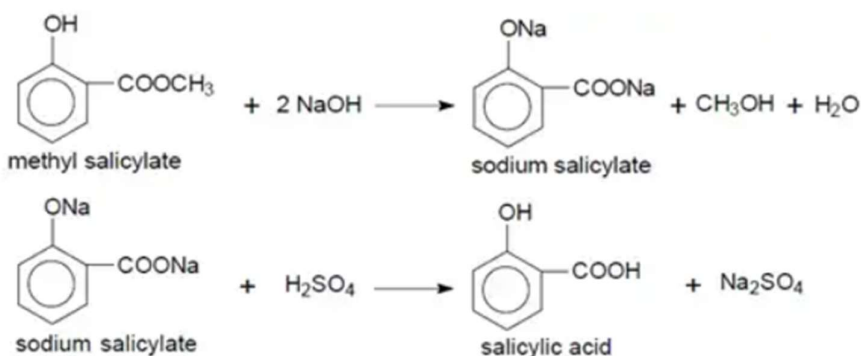
**Apparatus:** Beaker, Erlenmeyer flask, Filtration apparatus, reflux apparatus, RBF, Glass Road, filter paper, etc.

**Chemicals:** Methyl-salicylate, Sulfuric acid (6M), Sodium hydroxide (6M).

**Principle:**

Alkaline hydrolysis of esters is called saponification and is an irreversible process. Here, one mole of methyl salicylate (oil of wintergreen) reacts with two moles of sodium hydroxide to form sodium-salicylate with methanol and water, each of one mole. Sodium salicylate is reacted with sulfuric acid or hydrochloric acid to remove the sodium ion and forms salicylic acid with sodium sulphate as a byproduct.

**Reaction:**



**Use:** It is used to synthesize aspirin with benzoic acid as a topical product to treat skin irritation and inflammation caused by burns, insect bites, fungal infections, or eczema as a topical agent. Salicylic acid is widely used in skincare products, such as cleansers, toners, and spot treatments, to help treat and prevent acne.

**Procedure:**

1. Measure 3 ml of methyl salicylate (density – 1.184) and transfer it into a 200 ml round-bottom flask (RBF). Add 50 ml of 6M NaOH solution and a couple of boiling chips to the RBF.
2. Reflux the solution for 30-45 minutes after it begins to boil. If the reaction mixture still appears oily or cloudy, continue refluxing.
3. Check for clarity and homogeneity in the solution. If the wintergreen oil smell persists, reflux for additional minutes.
4. Remove the boiling chips and transfer the solution to a 250 ml beaker. Slowly add 6M H<sup>2</sup>SO<sup>4</sup> or hydrochloric acid to the solution while stirring until the pH reaches 2.0.
5. Cool the mixed solution in an ice bath for 10 minutes. Vacuum filter the solution and rinse it with ice-cold water.
6. Recrystallize the product from hot water, yielding 1.2 g with a melting point of 158-159°.

**Calculation:**

Molecular formula of methyl-salicylate = C<sub>8</sub>H<sub>8</sub>O<sub>3</sub>

Weight of methyl salicylate = 152 g/mole

Molecular formula of salicylic acid = C<sub>7</sub>H<sub>6</sub>O<sub>3</sub>

Weight of salicylic acid = 138 g/mole

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**Result:**

The salicylic acid was synthesis and found to be.....%.