Chapter: 17

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# AIM: TO PERFORM THE SYNTHESIS OF OIL OF WINTERGREEN. CALCULATE PERCENTAGE YIELD OF SYNTHESIZED PRODUCT

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## Requirements

#### Chemicals

- 1. Salicylic acid
- 2. Dry methanol
- 3. Sulfuric acid
- 4. Carbon tetra chloride
- 5. Magnesium sulphate

## Apparatus

- 1. Round bottom flask
- 2. Reflux condenser
- 3. Thermometer
- 4. Separating funnel
- 5. Beaker
- 6. Buchner funnel
- 7. Measuring cylinder

# Theory

Methyl salicylate (oil of wintergreen) contains ester function group. Ester can be prepared by corresponding acid and alcohols using esterification process. Esterification is a type of condensation process where water type small molecules eliminate to afford final products.

### **Physical Properties**

## Salicylic Acid

- It is a white crystalline solid having boiling and melting point 211°C and 315°C respectively
- It contains acrid taste and exhibit limited solubility in aqueous medium
- Salicylic acid has the pocket for three hydrogen bond acceptors and two hydrogen bond donors

#### Methanol

- Methanol is a liquid substance and completely soluble in aqueous medium
- Methanol is first compound of alcohol series which has explosive property

# Methyl Salicylate

- It has limited solubility in water and highly soluble in organic medium
- It is yellowish liquid having 220°C boiling point

# **Chemical Properties**

#### Methanol

- Methanol is an alcoholic substance having molecular formula CH<sub>3</sub>OH
- Methanol can be obtained from distillation of wood
- Methanol can also be synthesized from hydrogen gas and carbon monoxide gas

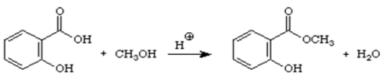
# Methyl Salicylate

- Methyl salicylate produces salicylic acid and methanol upon hydrolysis in basic medium.
- Methyl salicylate contains methylated carboxylic group with free phenolic group

# Procedure

- 1. Add 28g (0.2mol) of salicylic acid, 64g (81ml, 2mol) dry methanol and 8ml of conc. Sulfuric acid in a RBF of 500ml capacity.
- 2. Attach a reflux condenser assembly with the reaction mixture and boil it for 5hrs.
- 3. After completion of reaction, pour the residue in cold water and rinse the RBF 2-3 times.
- 4. Add 10-15ml of carbon tetrachloride and shake vigorously. After some time, methyl salicylate separates at the bottom of the separatory funnel making a distinct layer.
- 5. Collect the oil of wintergreen/methyl salicylate in a separate beaker and discard the water layer. Wash the product with sodium bicarbonate to remove any free acids.
- 6. Wash again with water and dry the product using magnesium sulphate powder.
- 7. Filter the product using filter paper and collect in RBF fitted with thermometer and an air condenser.
- 8. Boil the filtrate to remove carbon tetrachloride to afford pure methyl salicylate.
- 9. Weigh the product and calculate percentage yield.

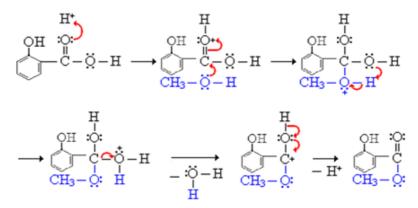
#### Reaction



salicylic acid

methyl salicylate

#### Mechanism



# Applications

- Used as a flavouring agent in pharmaceuticals and chewing gums, toothpaste.
- Has analgesic and anti-inflammatory potential which is useful in relieving joint pains.
- Used in the form of a fragrance in liniments, beverages, and food.
- Used as a cooling agents in various formulations.
- Can also be used as an insecticide in various preparations as well as in mouthwash solutions.

### Percentage Yield

- 138g salicylic acid forms 152g methyl salicylate
- Therefore, 128g salicylic acid will form.....? (X) g methyl salicylate
- X = (152x128)/138 = 30.84g
- Theoretical yield = 30.84g
- Practical yield =.....g
- Percentage yield = (Practical yield/Theoretical yield) x100

**Result:** Oil of wintergreen was successfully synthesized from salicylic acid and methanol.