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Preparation of Salicylic Acid	عنوان المحاضرة باللغة الانجليزية
تحضير حامض الساليساليك	عنوان المحاضرة باللغة العربية
١	رقم المحاضرة
Ullmann's Encyclopedia of Industrial Chemistry	المصادر والمراجع
The Beneficial Biological Properties of Salicylic Acid(٢٠١٥) "	
Salicylic acid as a peeling agent: a comprehensive review" (2010)	

محتوى المحاضرة

Preparation of Salicylic Acid

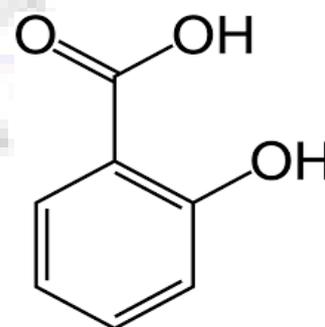
1- Introduction

Salicylic acid : is a natural phenolic compound.

It is one of the oldest compounds used in traditional medicine as an analgesic and antipyretic.

It forms the chemical basis for the development of aspirin (acetylsalicylic acid).

- -٢ -Hydroxybenzoic acid (C₇H₆O₃)
- -Naturally present in willow bark
- -Basis for Aspirin synthesis



2-Chemical Structure

Molecular formula: C₇H₆O₃

- -Contains -COOH (carboxyl group)
- -Contains -OH (phenolic group, ortho position)

- Molar mass: 138.12 g/mol.
- -Aromatic benzene backbone

This structure gives it acidic and phenolic properties.

3-Physical Properties

- State: White crystalline solid.
- Solubility:
Slightly soluble in water (approximately 2 g/100 ml at 20°C).
Soluble in alcohol, ether, and acetone.
- Melting point: approximately 158–161°C.

Odor: Mild and distinctive

4-Chemical Properties (Reactions)

- Esterification: With alcohol to form salicylate acetate (such as aspirin).
- Acidification: When reacted with bases, it produces salicylate ions.
- Oxidation: Can be slowly oxidized to salicyloquinone.
- Sulfate-phosphate reaction: Can form soluble salts.

5-Preparation

A) Natural Preparation

Extracted from the glycoside salicin found in willow.

Enzymatic hydrolysis → yields salicin → oxidation → salicylic acid

B) Industrial Preparation (Kolbe–Schmitt Reaction)

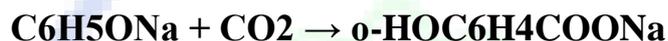
Sodium phenolate is used to prepare salicylic acid via the Kolbe-Schmitt reaction.

The reaction is carried out by heating the compound under pressure in the presence of carbon dioxide gas, producing sodium salicylate, which is then treated with sulfuric acid.

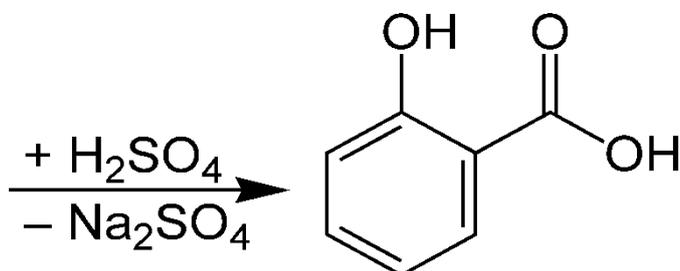
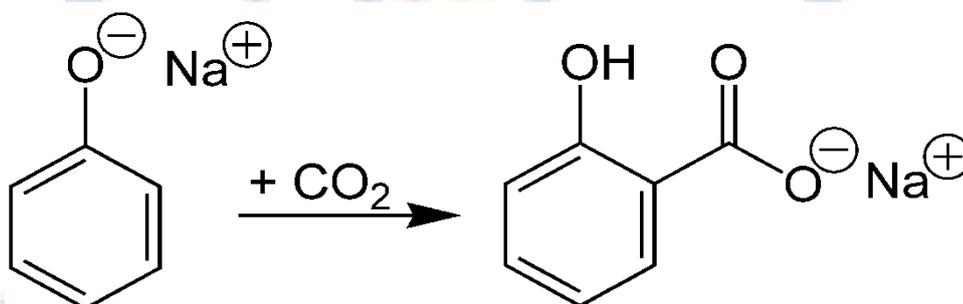
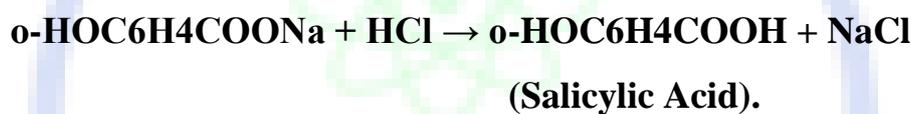
- First step: Convert phenol to sodium phenolate.



- Second step: Reaction of phenols with CO_2 under pressure and temperature (125°C , 5–7 atm).

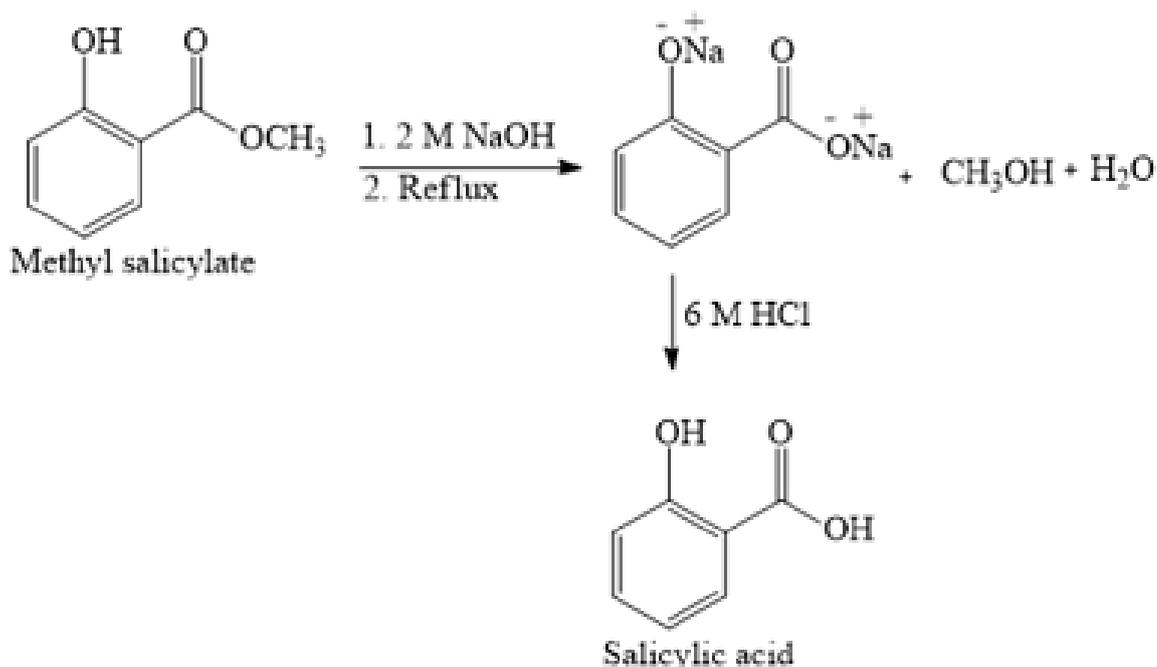


- Step 3: Acidify the product with mineral acid (HCl).



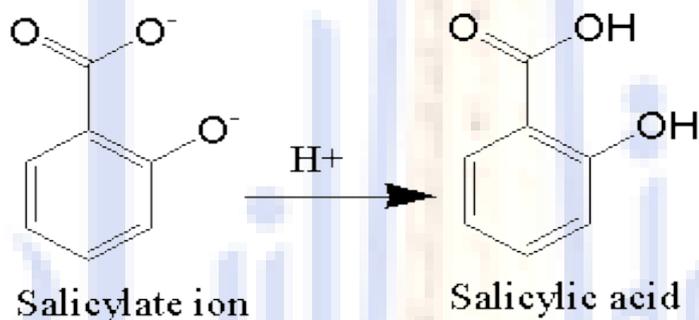
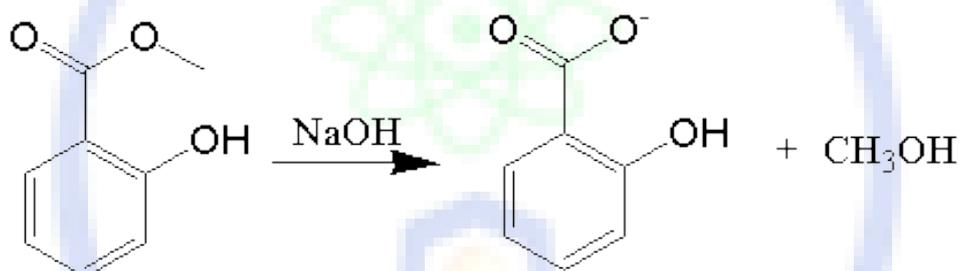
C) Preparation of salicylic acid by hydrolysis of methyl salicylate

- This document describes the preparation of salicylic acid by the hydrolysis of methyl salicylate under basic conditions. The method involves treating methyl salicylate with sodium hydroxide and sulfuric acid, yielding salicylic acid and methanol as the main products.
- Methyl salicylate, commonly known as oil of wintergreen, is an ester easily identified by its distinctive aroma.
- - When treated with an aqueous base, this ester undergoes hydrolysis, producing methanol, water, and the sodium salt of salicylic acid.
- - Organic salts like this sodium salt typically dissolve well in water or can be made to dissolve with gentle heating.
- - During the subsequent work-up, adding sulfuric acid converts the sodium salt into salicylic acid by protonation.
- - As a result, the primary organic products obtained from this reaction are methanol and salicylic acid.



6-Reaction Mechanism

- Sodium salicylate first, then, upon addition of HCl, converts to salicylic acid.
- Mechanism:
- Attack of OH^- on the carbonyl carbon \rightarrow formation of a tetrahedral intermediate.
- Dissociation of the R-OH group \rightarrow formation of salicylate.
- Acidification: Sodium salicylate + HCl \rightarrow Salicylic acid + NaCl



7-Applications

- Precursor in Aspirin (Acetylsalicylic Acid) synthesis
- Cosmetics and skincare (0.5–2% as exfoliant)

- **Topical anti-inflammatory agent**
- **Intermediate in dyes and preservatives industry**

8-Preparation of Salicylic Acid

- **1- Add 2.1 ml of methyl salicylate to 25 ml of 20% sodium hydroxide in a round flask, then heat the mixture under reflux for 15–30 minutes.**
- **2- Cool the mixture, then add 35 ml of 5% sulfuric acid**
- **3- Filter the precipitate.**

QUESTIONS

- **1. What causes the immediate formation of the white precipitate when methyl salicylate is added to the sodium hydroxide aqueous solution?**
- **2. Provide a clear and concise definition of a catalyst. In this experiment, does sodium hydroxide act as a catalyst? Please justify your answer.**
- **3. What is the reason behind the conversion of the phenolic hydroxyl group into its sodium salt during the process of basic hydrolysis?**

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