

CHAPTER-16

Experiment: 16



PREPARATION OF PICRIC ACID FROM PHENOL

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Aim:

To synthesize Picric acid from Phenol and to find out its % practical yield and melting point.

Requirements:

A. Glassware & Instruments

1. Flat bottom flask (500 ml)
2. Beaker
3. Reflux condenser
4. Buchner's flask
5. Vacuum pump, 25 ml
6. Measuring cylinder
7. Thiel's tube
8. Capillary

B. Chemicals & Reagents

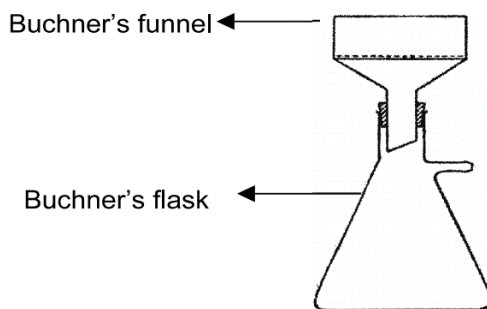
1. Phenol
2. Conc. H_2SO_4
3. Conc. HNO_3
4. Alcohol:Distilled water (DW) mixture (2:1)
5. Ice

Theory:

- **Nitration:** The process of nitration is the replacement of a hydrogen atom with a nitro group (NO_2). The nitration, sulphonation, and halogenation reactions are extraordinarily facile due to the phenol's activated nucleus. Nitration occurs at room temperature with diluted HNO_3 . When phenols are treated with concentrated HNO_3 in the presence of concentrated H_2SO_4 , nitration occurs at both the ortho and para positions, producing picric acid. When $-\text{NO}_2$ (nitro) groups are to be substituted for $-\text{SO}_3\text{H}$ (sulphonic acid) groups, it is preferable

to convert phenol to phenol sulphonic acid with H_2SO_4 and then nitrate the compound with concentrated HNO_3 .

- **Synthesis:** It is a chemical reaction that produces a new product with a distinct structural formula, molecular weight, and melting point.
- **Purification:** Purification is the removal of contaminants from a product. Recrystallization, washing, and drying the product in an oven at a specific temperature and for a specified amount of time are included in the purification of the product. Buchner's funnel and flask assembly:



- **Recrystallization:** Recrystallization is the process by which a compound is dissolved in a solvent of choice through heating, and then slowly cooled to form a saturated solution from which pure compound crystallises.
- **Yield:** It refers to the amount of product obtained from a synthesis. They include the theoretic yield, the practical yield, and the percentage yield.
- **Theoretical Yield** is the weight of the product one should obtain based on the stoichiometric quantities of the reagents, assuming that the reaction is carried out to 100 percent efficiency.
- **Practical Yield** is the actual amount of product obtained after product purification.
- **Percentage yield** is calculated from the formula given below:

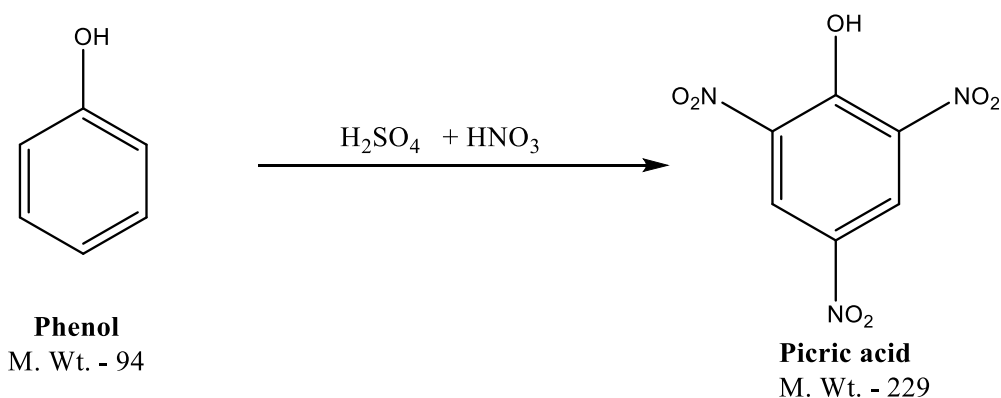
$$\% \text{ Yield} = \frac{\text{Practical yield} \times 100}{\text{Theoretical Yield}}$$

Procedure:

1. Place 4 g of phenol (or 5 ml of phenol solution) in a 500 ml flask with a flat bottom.

2. Add 5.0 ml of concentrated H_2SO_4 and thoroughly mix. Since the reaction is exothermic, the mixture heats up.
3. For thirty minutes, heat the flask in a water bath to complete the formation of phenol sulphonic acid. The flask must be thoroughly chilled in an ice-water mixture.
4. Place the flask on a wooden surface in a fume cabinet, add 15 ml of con nitric acid, and shake vigorously for a few seconds
5. A vigorous chemical reaction occurs, and harmless red nitrous fumes escape the flask.
6. After the reaction has subsided, heat the flask in a bath of boiling water for 1 to 2 hours while continuously shaking. Initially, a thick oily layer forms, which then transforms into a crystalline mass.
7. Add approximately 50 ml of cold water and chill the mixture in ice water.
8. Collect the product using Buchner's funnel and vacuum filtration, then wash it with cold water until it is free of acidity and drain.
9. Recrystallize the product from a mixture of alcohol and water (2:1) (about 50 ml solvent is required).
10. At the pump, filter the crystalline material and dry it by pressing filter paper.

Reaction:



Observations:

- Amount of Phenol taken for synthesis = 4 g
- Practical yield of the recrystallised product =
- Melting point of the product =

Calculations:

Theoretical Yield of the product.

From the reaction,

94 g of Phenol gives 122 g of Picric acid

4 g of Phenol will give 9.74 g of Picric acid

Percentage Practical Yield

$$\% \text{ Yield} = \frac{\text{Practical yield} \times 100}{\text{Theoretical Yield}} = \dots\dots\dots\%$$

Result:

Percentage yield of Picric acid =%

Melting point of Picric acid is =°C

Viva questions:

- What is the product's chemical name?
- Utilize the picric acid.
- What role does sulfuric acid play in the nitration process?
- What is electrophilic replacement?
- How is phenol's nucleus activated for nitration?
- Give at room temperature the product of nitrating phenol with diluted nitric acid.
- Phenols are acidic/basic or neutral? Give justification
- What is ionised nitronium? How is it formed in the nitrating mixture (nitric acid and sulphuric acid)? Why is nitration typically conducted at a lower temperature?
- What precautions must be taken when handling concentrated sulfuric and nitric acids?
- What is the definition of electrophile? Include two additional electrophiles.