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Diploma in Pharmacy 1st Year
Pharmaceutical Chemistry
Experiment

To prepare and submit picric acid from phenol.

Aim:

To prepare and submit picric acid from phenol.

Reference :

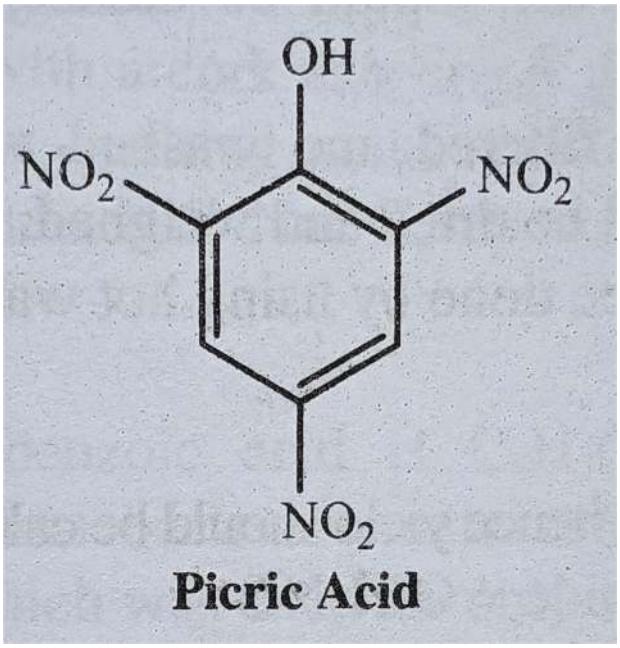
‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 64 - 68

Requirements:

Erlenmeyer flask (250 mL), volumetric flask, pipette, glass stirring rod,buchner funnel, suction pump, filter papers, measuring cylinder, rubber clock,fume hood, phenol (7.5 ml), nitric acid (22.5 ml), sulphuric acid (20 ml) andcold water (100 ml).

Theory:

Picric acid is a derivative of 2, 4, 6-trinitrophenol. Its molecular formuls is C_6H_3N3O7 It is yellow in colour. Because of the presence of phenol, it employed as a topical anti-infective and disinfectant, as well as a cleaning age and preservative. Picric acid is obtained from phenol that has been nitrated. Piene acid is often used to mark rats and mice in pharmacology experiment.

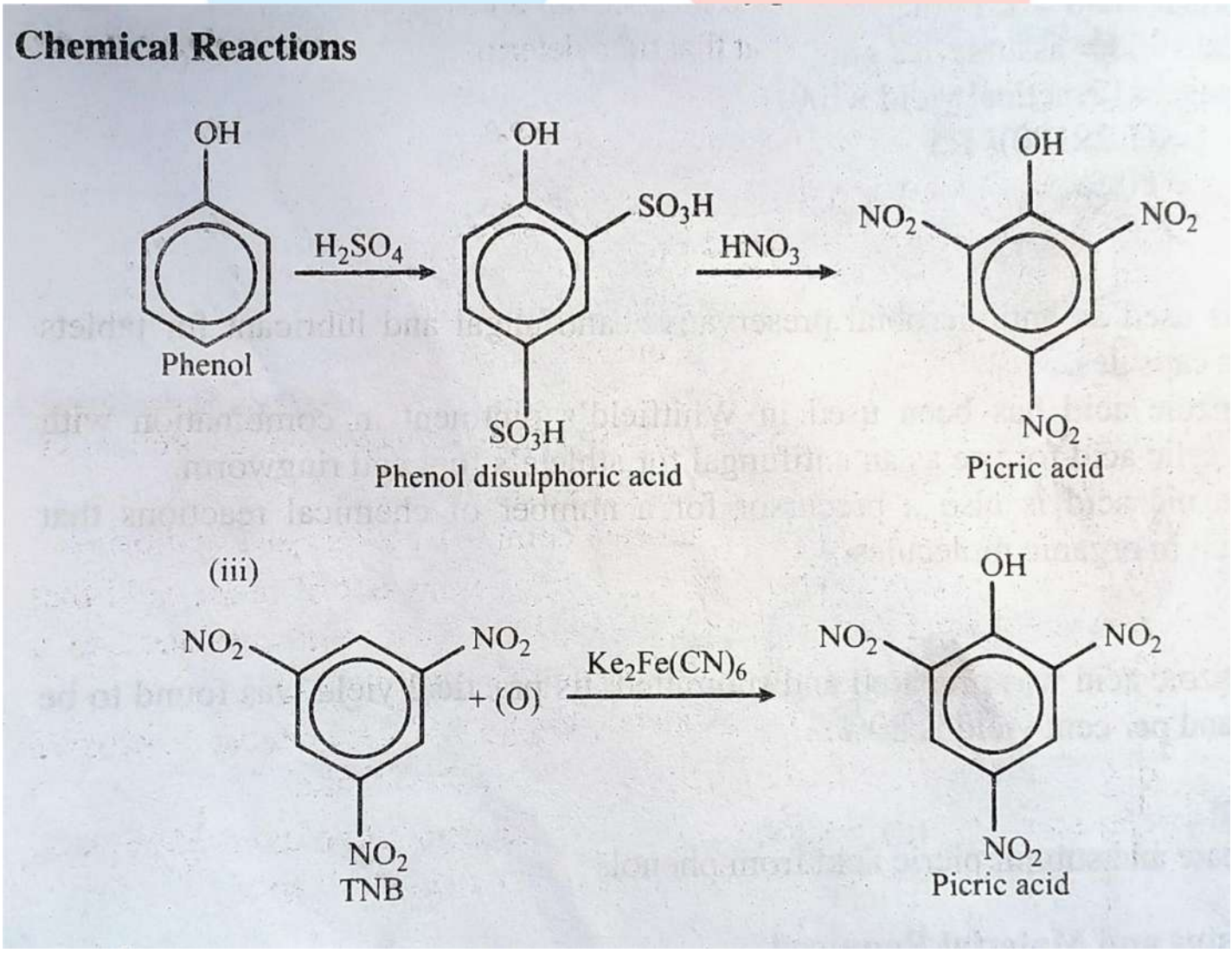


Picric Acid

Aromatic hydrocarbons such as 2, 4, 6-trinitrophenol or their substituted derivatives react with con Nitric acids undergo the nitration reaction (electrophilic substitution reaction) in the presence of strong sulphuric acid (e hydrogen atoms are replaced by nitro group). Even in the absence of sulphurie acid, nitration can be done in some cases.

An important class of organic compound synthesis is the electrophilic aromatic substitution reaction. The position and extent of substitution of new incoming groups are determined by substituents which are already present in the benzene nucleus.

Chemical Reactions



Procedure:

1) 7g of phenol and 20 ml of conc. Sulphuric acid should be taken in 500 ml dried round bottomed flask. The mixture should be shaken and heated on a water bath for half an hour.
2) A clear solution of o- and p-phenol sulfonic acids should be obtained during this time. The flask should be then cooled in an ice-bath.
3) 22.5 ml of conc. HNO, should be gradually added with continuous shaking. An exothermic reaction occurs, releasing vast amounts of red fumes (nitrogen oxide) and turning the liquid a deep red colour.
4) The flask should be heated in water bath for 2 hour with infrequent shaking. The fiask should be cooled and 100 ml of water should be added to it and then cool again.
5) The solid should be filtered and washed completely with cold water. The crude picric acid should be recrystallized from aqueous hot alcohol.

Calculation

Here limiting reagent is phenol; hence yield should be calculated from its amount taken.

Molecular formula of phenol = C_6H_6O

Molecular formula of picric acid = $C6H3N3O$

Molecular weight of phenol = 94 g/mol

Molecular weight of picric acid = 229.10 g/mol

94 g of phenol reacts with benzoyl chloride to give 229.10 gm of picric acid. Therefore, 7.5 of phenol will give x gm of picric acid

x= (229.10×7.5)/94 = 18.27g

Theoretical yield = 18.27 g

Practical yield = assume 15.3 gm

Percentage (Practical yield x100)

=(15.3x100)/18.27

83.74%

Uses :

- ✓ It is utilised as a yellow dye for silk and wool, as an explosive as well as an antiseptic in burn therapy.
- ✓ By far, the most common application has been in weaponries and explosives.
- ✓ Explosive D. also called as Dunnite, is an ammonium salt of picric acid that is more powerful than but less stable than TNT (which is produced in a similar process to picric acid but with toluene as the feedstock).
- ✓ Picramide can be further marinated to produce the very stable explosive TATB by aminating picric acid (usually starting with Dunnite)
- ✓ It has been used in organic chemistry to make crystalline salts of organic bases (picrate's) for the sake of identification and characterization.

Result: Picric acid was prepared and submitted. Its practical yield was found to be 15.3 gm and percent yield about 83.74%.

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