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Experiment NO. - 1

Aim- To prepare or submit chlorobutanol from acetone & calculate the percentage yield.

Chemical required-

Chloroform - 1ml

Potassium hydroxide - 1gm

Acetone - 14ml

Principle -

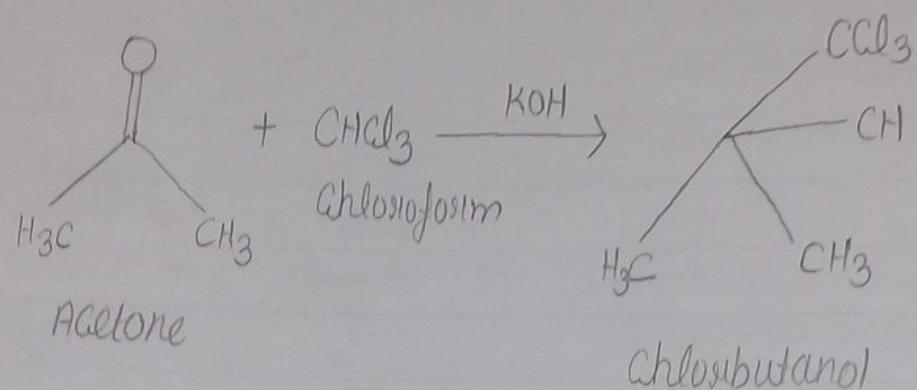
Chlorobutanol is also known as chloroketone. It is a trichloro derivative of tertiary butyl alcohol. It is prepared by combination with acetone chloroform in the presence of solid potassium hydroxide chlorobutanol is used as local anaesthetic in dental preparation also antiseptic.

Procedure -

About 1ml of chloroform and 1gm of solid potassium hydroxide are taken in a round bottom flask. Shake until the potassium hydroxide dissolves. To this 14ml of acetone was added for half an hour and crystal of chlorobutanol was separate out.

Melting point- $95^{\circ}\text{C} = 99^{\circ}\text{C}$

Reaction



Use - Local anaesthetics

Result -

Chlorobutanol was prepared and submitted and reported the following

Theoretical yield -

Practical yield -

Percentage yield -

Experiment No. - 2

Aim — To prepare & submit hexamine from formaldehyde and calculate its percentage yield.

Chemical required-

Formaldehyde — 4.7 gm

Ammonia solution — 7 gm

Principle —

Hexamine is heterocyclic organic compound $(\text{CH}_2)_6\text{N}_4$. It has symmetrical tetrahedral cage like structure. It is prepared by condensation reaction between formaldehyde and ammonia.

Procedure —

About 4.7 gm of 30% formaldehyde solution was taken in a beaker and add 7 gm of 24% ammonia solution until the solution is slightly alkaline. The mixture was heated on a water bath for 5 minutes. The solution allowed to stand for 15 minutes. The solution was filtered and then evaporated on a direct flame using China dish to a thick paste.

The hexamine crystals are obtained and dried. It was recrystallized from water or alcohol hexamines from colorless, odourless crystals, which are soluble in water and 90% alcohol.

Uses - Anti-infective agent

Result - Hexamine was prepared and submitted.

Experiment No. 3

Aim — To prepare & submit dalbutamide from p-toluene sulphonamide and calculate percentage yield.

Reference —

Dr. Abhishek Tiwari, Dr. Rajeev Kumar. A practical book of medicinal chemistry IIIrd, 1st edition 2019 published by Nirali Prakashan.

Chemical required —

P-toluene — 1gm
 Butyl Isocyanate — 2gm
 Triethylamine — 1.2 ml
 Tetrahydrofuran — 10ml

Principle — The synthesis of dalbutamide involves addition reaction of P-toluene sulphonamide and butyl isocyanate in the presence of triethylamine and tetrahydrofuran.

Procedure —

About N-butyl isocyanate (1 mol) and triethylamine (1.2 mol) in a round bottom flask containing 10ml of tetrahydrofuran kept in an ice bath.

To the above mixture p-toluene sulphonamide (1 mol) was added drop wise at 0°C . After completing the addition the temperature was suddenly raised to $35-45^\circ\text{C}$ and

boiled for 3-4 hrs. Then the solution was filtered the product was separated and dried. Then it was recrystallised by using Diethyle ether.

Meeting point -

128°C to 129°C

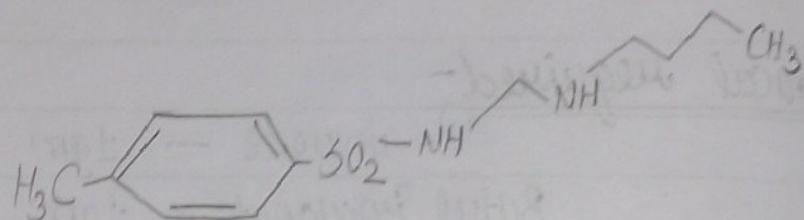
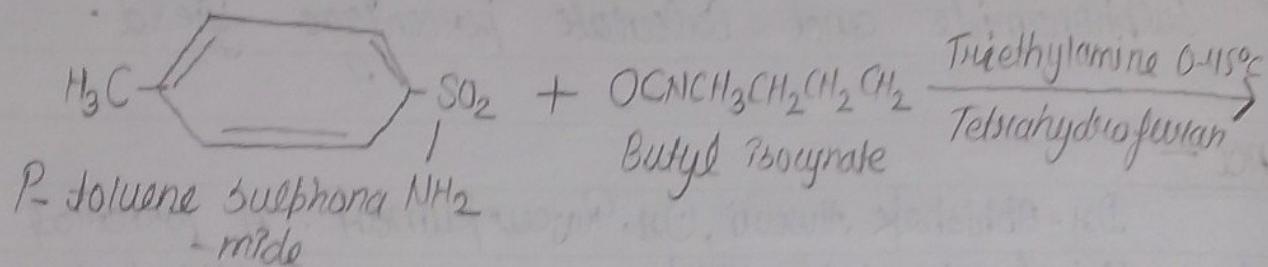
Uses -

As anti-diabetic

Result -

Tolbutamide was prepared & submitted.

Reaction



Talbutamide

Experiment NO. - 4

Aim- To synthesis and submit sulphonamide from p-acetamide benzene sulphanilamide and calculate its percentage yield.

Reference- Dr. Abhishek Tiwari, Dr. Rajeev Kumar. A practical book of medicinal Chemistry IIIrd, 1st Edition 2019 published by Nirali Prakashan.

Chemical required -

Resorcinol - 1.2 gm
Ethyle acetate - 2.4 ml
Conc. Sulphuric acid - 7.5 ml

Procedure -

1.5gm of 4-acetamide benzene sulphonamide is treated with a mixture of 1ml of Conc. sulphuric acid diluted with 2ml water. This mixture is gently heated under reflux for 1 hour. Then 3ml of water is added and the solution is filtered. While ~~is added~~ and the bottom hot solution is boiled again with the addition of a small quantity of activated charcoal. The solution is filtered while hot & the filtered is neutralized with powder solution sodium carbonate with stirring until all effervescence ceases and the sulphonilamide is precipitated as a white powder. The solution is

It was filtered the sulphonamide wash with water and dried finally crude sulphonamide is recrystallized from hot water to get colourless crystals.

Melting point — 163°C

Uses — Bacteriostatic agent

Result — The sulphonamide was synthesized and submitted.

Experiment NO. - 5

Aim -

To prepare and submit acetanilide from aniline using zinc dust.

Reference - Dr. Abhishek Tiwari, Dr. Rajeev Kumar. A practical book of medicinal Chemistry IIIrd, B+ Edition 2019 published by Nirali Prakashan.

Requirements -

Aniline, Glacial acetic acid, zinc dust, round bottom flask, glass rod, beaker.

Theory -

Green chemistry is defined as environmentally friendly chemistry which has objectives to design new chemical methods / products that can decrease environmental pollution at the design stage, even before its begins. If chemists are taught to synthesize the products and materials without using hazardous substance, then much waste hazards and cost can be evaded.

Procedure -

Heat a mixture of aniline (10ml) and zinc dust (0.5g) in acetic (30ml) in a 100ml round bottom flask over a gentle flame using water condenser.

- Continue heating for about 2 hours.
- Then carefully pour reaction mixture in cold water (100 ml) in a 250 ml beaker with vigorous stirring. slowly separate acetanilide crystals after 15 min. Filter the mixture with Buchner funnel, wash with water and dry (yield = 10 gm)

Uses -

Acetanilide is an example of ~~not~~ analgesic and antipyretic drug.

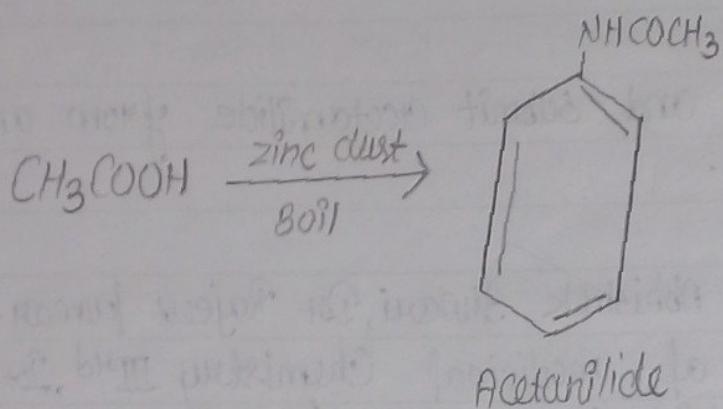
Caution -

Glassware should be clean and dry before being used.

Result -

The yield of obtained product was found to be gm and

3 - on bromine



Experiment No. - 6

Aim - To prepare and submit triphenyl imidazole from benzyl and benzaldehyde.

Reference - Dr. Abhishek Tiwari, Dr. Rajeev Kumar. A practical book of medicinal chemistry 3rd, 1st Edition 2014 published by Nihali Prakashan.

Requirements -

Benzil, ammonium acetate, benzaldehyde, glacial acetic acid, round bottom flask, glass rod, beaker.

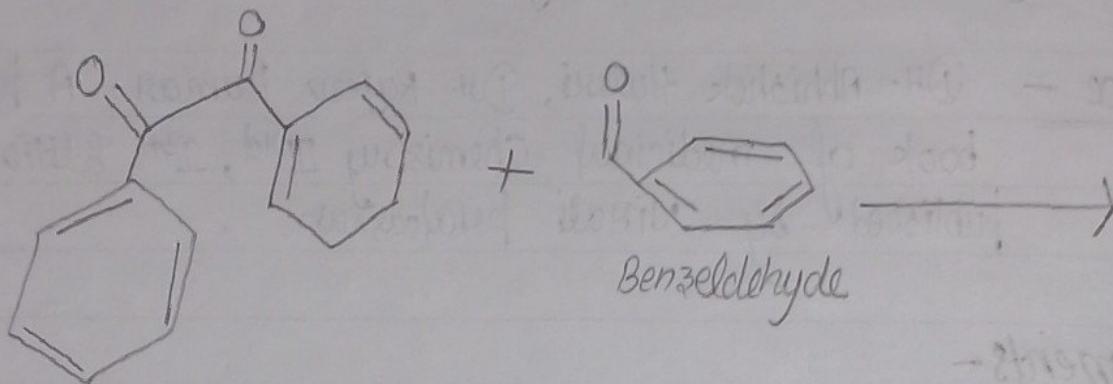
Theory -

2,4,5 triphenyl 1H-Imidazole was prepared from benzyl, ammonium acetate and benzaldehyde in the presence of glacial acetic acid.

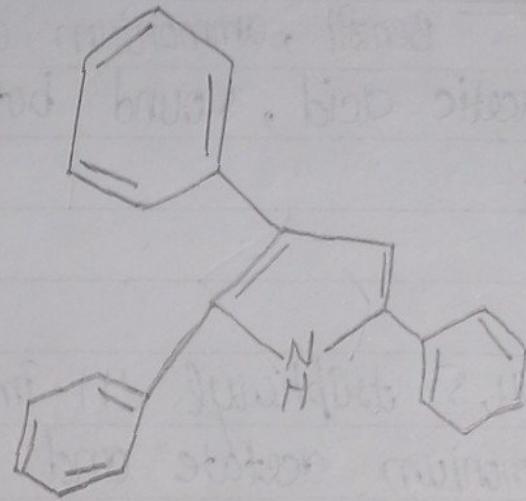
Procedure -

- Take benzil (\pm gm), ammonium acetate (\pm gm), benzaldehyde (2ml), glacial acetic acid (2ml) in round bottom flask and reflux for 3 hours.
- Allow reaction mixture to stand to room temperature to attain room temperature.
- Add 150 ml of water and filter.

Reaction



Benzil



(opt) states minimum, (opt) fixed w/for

hours of (opt) has been known. (opt) hydrolysis
based e not suffer, has specificity metal
of methanol or base of without addition with

methanol also

- The filtrate is neutralized with ammonium hydroxide or sodium carbonate to give solid bulky mass and again filter.
- Then the solid mass is washed with toluene and recrystallize from methanol.
- Meeting point of 2,4,5-triphenyl-1H-imidazole is $274 - 278^{\circ}\text{C}$.

Uses - 2,4,5-triphenyl-1H-imidazole has antibacterial and anti-inflammatory activities.

Caution - Glassware should be clean and dry before being used.

Result -

The yield of obtained product was found to be ___ gm and percentage yield was ___.

Experiment No. - 7

Aim- To prepare and submit pyrazoline from acetophenone by Claisen Schmidt reaction.

Reference- Dr. Abhishek Tiwari, Dr. Rajeev Kumar · A practical book of medicinal chemistry ^{3rd, 1st edition 2019 published by Nirali Prakashan.}

Requirements- Acetophenone, benzaldehyde, sodium hydroxide, ethanol, phenyl hydrazine.

Theory- Claisen - Schmidt reaction is carried out between acetophenone and substituted benzaldehydes in presence of basic medium andiline respective Chalcone. When Chalcone reacts with phenyl hydrazine, then substituted pyrazoline is formed.

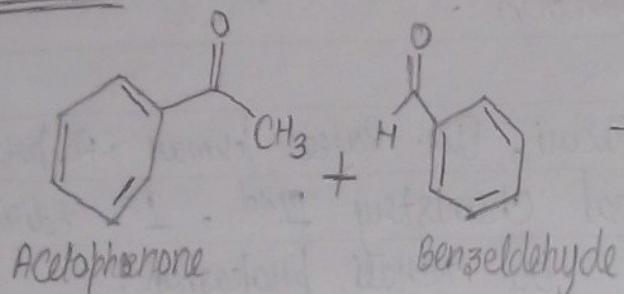
Procedure -

Step 1 - Synthesis of chalcone (Benzalacetophenone)-

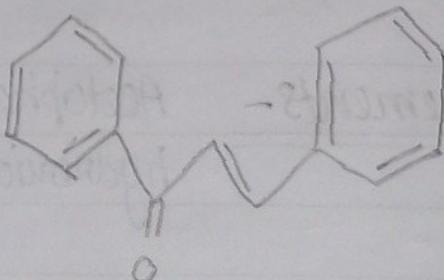
- Take a solution of 2.7g of sodium hydroxide in 100 ml of distilled water and 61.25 ml of rectified spirit in a 500 ml bolt head flask attached with mechanical stirrer.

Reaction -

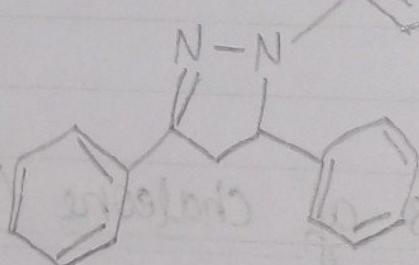
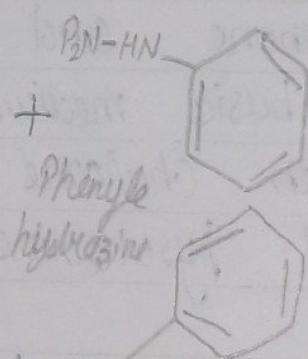
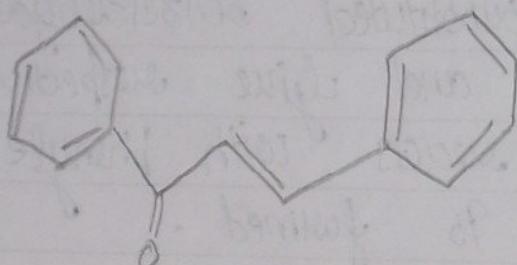
Step - 1



sodium hydroxide
Ethanol



Step - 2



- Immerse the flask in a crushed ice bath.
- Then add 26g freshly distilled acetophenone.
- Add 22ml pure benzaldehyde with stirring.
- Maintain temperature of the mixture at about 25°C and stir vigorously until the mixture is so thick but that stirring is no longer effective (2-3 hours).
- Remove the stirrer and place mixture in a refrigerator to overnight.
- Filter the product with solution on a Buchner funnel, wash with cold water until the washing are neutral to litmus and then with 10ml of ice cold rectified spirit.

Step-2 - Synthesis of pyrazoline -

Dissolve Collected 25 ml of Ethanol, Add diobutyl phenyle hydrazine (1.8gm) into the mixture. Then subject to microwave evaporation at 320 watts for 30 min.

Cool the mixture at room temperature which results in precipitation of pyrazoline.

Recrystallize the crude product from Ethanol.

Result -

The pyrazoline was synthesized & submitted successfully.

Experiment No.-8

Aim- To prepare and submit Aspirin from salicylic acid and calculate the percentage yield.

Reference- Dr. Abhishek Tiwari, Dr. Rajesh Kumar - A practical book of medicinal chemistry 3rd, 1st Edition 2019 published by Nikali Prakashan.

Requirements- Aspirin, salicylic acid, acetic anhydride, sulphuric acid.

Principle-

The microwave region of electro magnetic spectrum light between IR Irradiation and Frequency corresponding to the wavelength of 1cm -1 to 3m. The organic compound can be heated by applying energy in the form of microwave high frequency IR irradiation. The use of this technique can have substantial saving time for laboratory synthesis of drugs and chemicals.

Aspirin is prepared from salicylic acid in this method salicylic acid react with acetic anhydride in the presence of sulphuric acid to form Crystalline nature of aspirin.

Procedure -

Two reaction consist of a two neck flask with magnetic stir bar, temperature sensor and intensive cooler. A mixture of salicylic acid and acetic anhydride is filled in the reaction flask and three drop of Conc. sulphuric acid are added the apparatus is installed by means of a glass tube in the microwave system.

Standard refluxing apparatus for microwave system. The reaction mixture is heated under stirring for 90 sec with 90°w to 140°C. During the following cooling down the clear yellowish solidified to a compact white crystalline mass.

Melting point - 135°C

Use - Anti-inflammatory agent

Result - Aspirin was synthesized and reported the following -

Theoretical yield -

Practical yield -

Percentage yield -

Experiment NO. 9

Aim — To determine the percentage purity of given sample of tablet.

Reference — Dr. Abhishek Tiwari, Dr. Rajeev Kumar. A practical book of medicinal chemistry IIIrd, 1st edition 2019 published by Nirali Prakashan.

Requirements—

Sodium nitrate = 7.5 g

Sulphuric acid = 0.3 g

Potassium bromide = 3 g

Sodium nitrate = 0.1 M

Principle—

Debsone is a diamine o-phenylene sulphone. It is assayed by direct diazotization titration. The free primary amino group present in debsone is diazotized by nitrous acid and hydrochloric acid to form azoic diazonium compound. The end point can be determined by using External Indication i.e. starch iodide paper.

During titration with sodium nitrate an aromatic primary amine is diazotized. After the end point the drug solution containing a slight excess of nitroso comes in contact with starch iodide paper. It oxidizes iodide into iodine which gives blue colour with

with starch indicating the end point.

Procedure -

- Preparation of 0.1M sodium nitrate solution -

7.5 gm of sodium nitrate was dissolved in sufficient water to produce 1000 ml.

- Standardization of 0.1M sodium Nitrate -

0.3 g of solution sulphuric acid was dissolved in 50 ml of 2M hydrochloric acid, 3g of potassium bromide was added. Cool in ice and titrate with 0.1M sodium nitrate solution using starch iodide paper as internal indicator.

Each ml of 0.1M sodium nitrate = 0.01732g of $C_6H_7NO_3S$.

Assay of dapson tablet -

1. 20 tablets were weighed and powdered.
2. Tablet powder equivalent to 0.25gm dapson was weighed and dissolved in a mixture of 15ml of 2M hydrochloric acid.
3. The solution was cooled to about $15^{\circ}C$ and carry out

Sodium nitrite titration using starch iodide paper as an external indicator.

End point is immediate appearance of blue colour.

Each ml of 0.1M Sodium Nitrite = 0.01241 g of $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_2\text{S}$

Result -

Experiment NO. - 10

Aim

To determine the percentage purity of given sample of Benzyl penicillium tablet.

Reference- Dr. Abhishek Tiwari, Dr. Rajeev Kumar a practical book of medicinal chemistry IIIrd, Ist edition 2019 published by Nirmal Prakashan.

Requirements-

Sodium Sulphate - 0.02 N

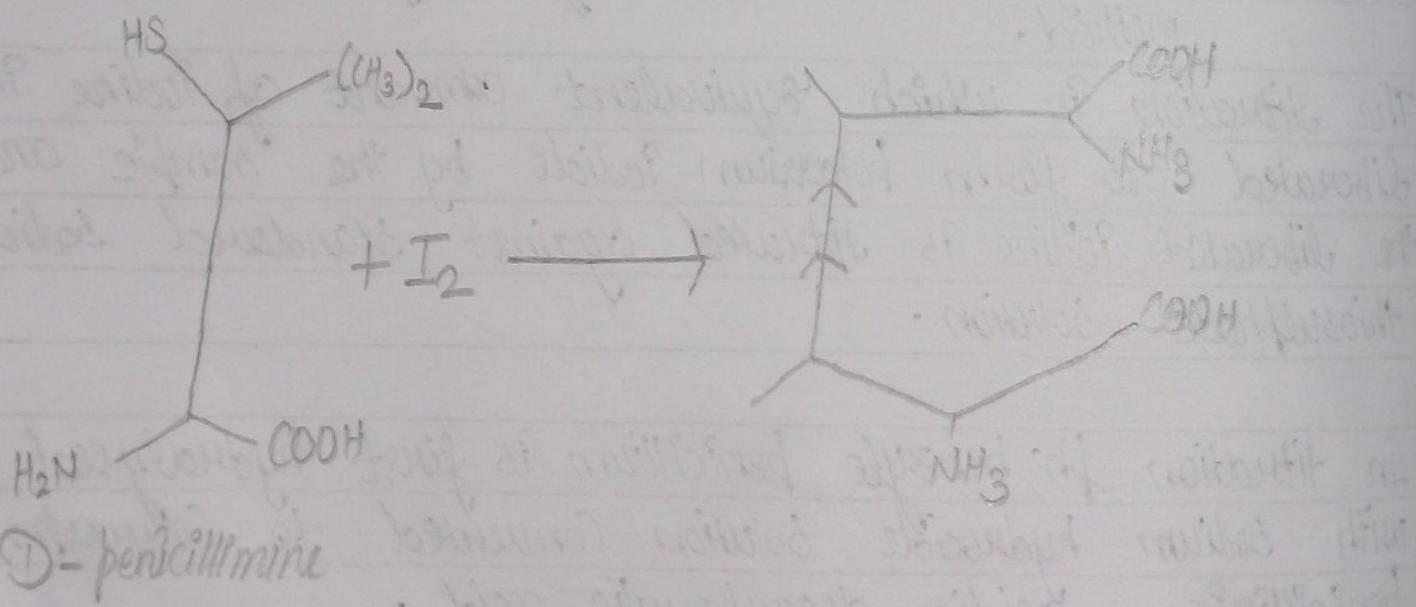
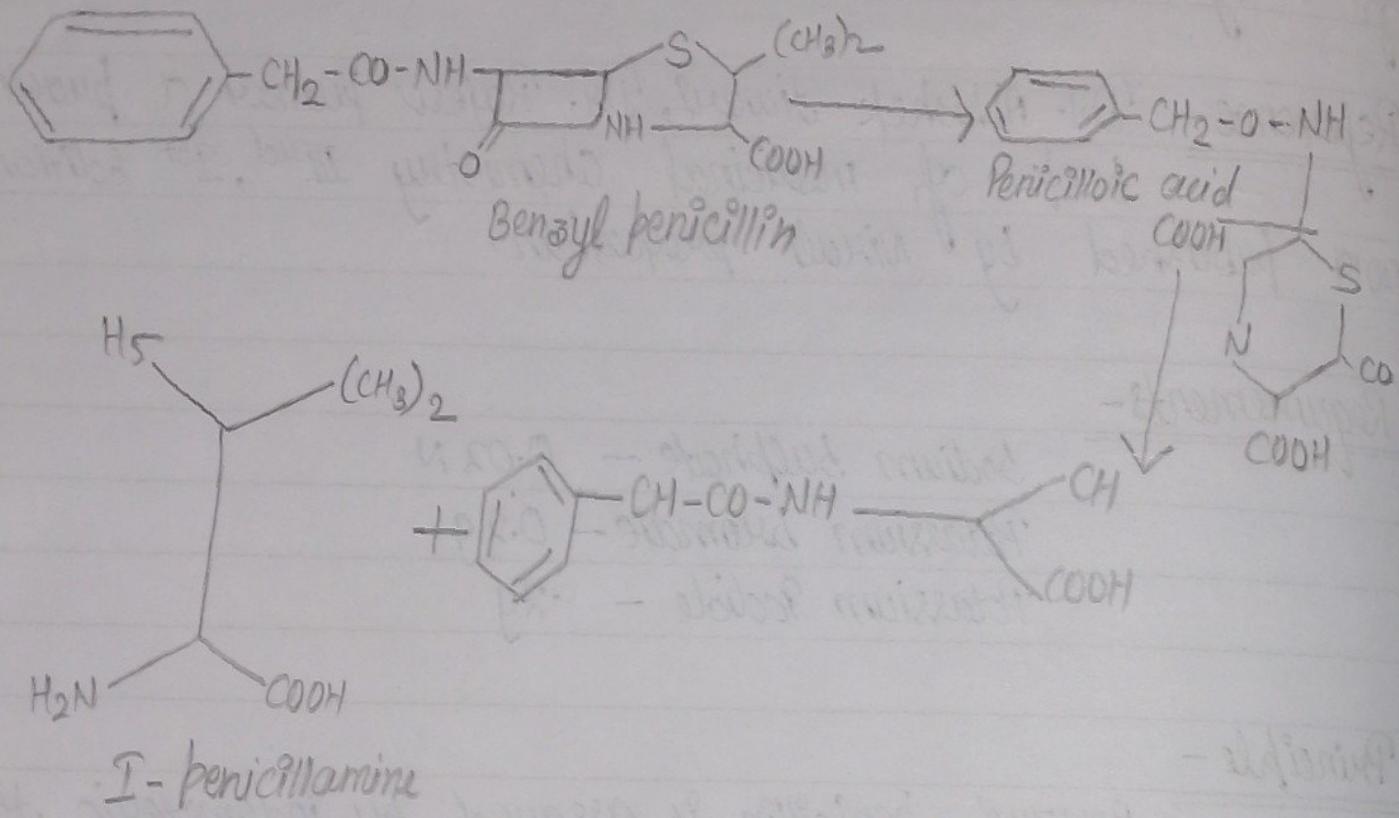
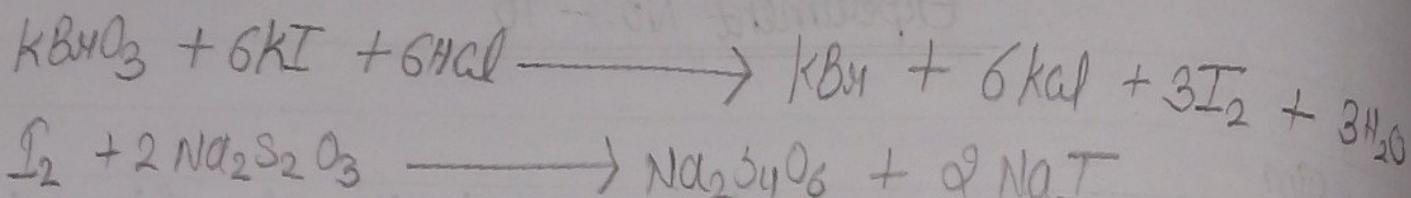
Potassium bromate - 0.2 g

Potassium iodide - .2 g

Principle-

- 1) Benzyl penicillin is assayed by iodometric titration method.
- 2) The titration in which equivalent amount of iodine is liberated to form potassium iodide by the sample and the liberated iodine is titrated against standard sodium thiosulphate solution.
- 3) In titration ~~for~~ benzyl penicillium is first hydrolysed with sodium hydroxide solution converted to corresponding penicillenic acidic decarboxylic acid.

Reaction



- 4) This type of indirect titration determination of compound is called iodometric titration.
- 5) Then penicilloic acid further treated with mineral acid to form D-penicillamine and benzyl penicillic acid.
- 6) An obtained D-penicillamine is further oxidized quantitatively iodine to give disulphide mevers of iodine is back titrated with 0.02M sodium thiosulphate, equivalent amount of liberated iodine can be measured by titration with sodium thiosulphate using starch as an indicator which is added near the end point as it gets hydrolysed by hydrochloric acid and iodine gets trapped in the matrix of starch.
- 7) Due to this there is no continuous liberation of iodine.
- 8) An end point is blue to apple green.

Procedure —

Preparation of 0.02N sodium thiosulphate —

0.02N sodium thiosulphate dissolved in 100ml of water then make up to 1000ml with water.

Standardization of 0.02N sodium thiosulphate -

Dissolved accurately weighed 0.2g of potassium bromate in 250ml of water taken in a ^{ch} conical flask.

From this take 50ml of the solution add 2g of potassium iodide, 3ml of 2m HCl and titrate with sodium thiosulphate solution using water starch as an indicator until the blue colour is discharged.

Each ml of 0.01N sodium thiosulphate = 0.002784 g of KBr.

Assay of benzyl penicillium -

- 1] Weighed accurately about 0.1gm sodium salt of benzyl penicillin taken in a stoppered flask dissolved in 10ml of water and dilute to 100ml.
- 2] 10ml of solution transferred into iodine flask 5ml of 1N solution sodium hydroxide was added, allowed to stand for 20 minutes.
- 3] The freshly prepared buffer solution, 5ml of 1N hydrochloric acid and 25ml of excess of 0.02N iodine solution were added to the stoppered flask and kept aside for 20 min in a dark place.

- 4) The end point is dissolved discolouration of blue colour.
- 5) To another 10ml of initial solution add 20ml of the buffer solution, allowed to stand for 20 minutes in the dark place and titrate with the same.
- 6) The difference between two titration represent the volume of 0.02N iodine equivalent to the total amount of penicillin equivalent present in the given sample of benzyl penicillin.

Result