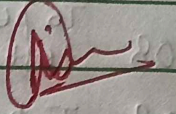
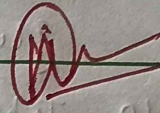
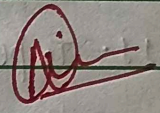
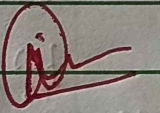
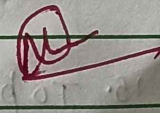
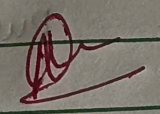
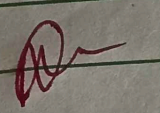


Pharmaceutical Chemistry

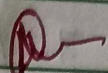
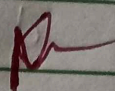
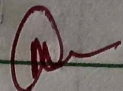
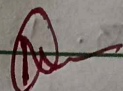
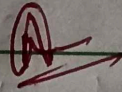
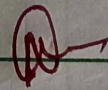
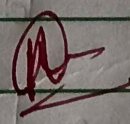
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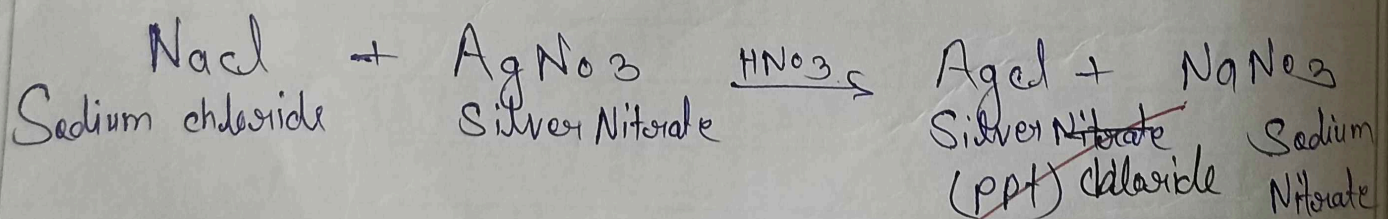
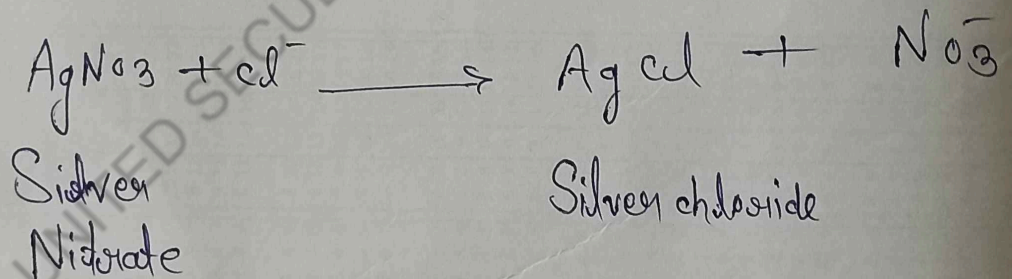
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Sr. No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
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2.	To perform a limit test for sulphate in a given sample of tap water.	3-4			
3.	To perform a limit test of Iron in the given sample.	5-6			
4.	To carry out identification test for Aspirin.	7-8			
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7.	To perform the identification test for				

Index

Sr. No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
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09	To perform the ident ⁿ test for Calcium gluconate	18-19			
10	To perform ident ⁿ test for Sodium carbonate	20-21			
11	To perform the ident ⁿ test for magnesium sulfate	22-24			
12	To carry out ident ⁿ test for sulfonamide	25-26			
13	To prepare and submit the crude product of Benzoic acid & calculate the Percentage Yield	27-28			
14	To prepare and submit picric acid and calculate its % yield	30-31			
15	To perform the ident ⁿ test and test for purity of Caffeine	32-33			

Reaction $\rightarrow$

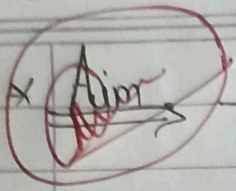


Experiment-1

Date _____

Experiment 01

Page No. 01



To perform the limit test for chloride in a given sample of tap water.

* Reference Parle, A. pharmaceutical chemistry
1. Laboratory Manual. I (10s published and
distributed prt. Ed Ist 2000 page No. 59.

* Requirements — chemical Required — HNO_3 , AgNO_3
 NaCl .

Apparatus — Measuring cylinder glass Rod Nessler
cylinder.

* Theory — Limit test are quantitative or semi
quantitative test designed to identify and
control a small number of impurities which
are likely to be present in the substance
this test involves the reaction of silver nitrate
with soluble chloride to form the ppt of
silver chloride which is insoluble in HNO_3 .



Teacher's Signature: _____

Sample Solution	Standard Compound
1. Dissolve specified quantity of substance in to 10 ml Distilled water.	1 ml Sodium chloride solution transfer in to Nessler cylinder
2. Add 10 ml Nitric acid (HNO_3)	Add 10 ml HNO_3
3. Make up the volume up to 50 ml with tap water	Make up the volume up to 50 ml of distilled water
4. Now Add 1 ml silver Nitrate (AgNO_3)	Add 1 ml silver Nitrate Solution.
5. Stir the solution with glass Rod and to stand for 5 minutes.	Stir the solution with glass Rod and allow to stand for 5 minutes.

* Observation -

The opalescence of the test solution was less than standard solution.

* Result

A Limit test for chloride was performed.

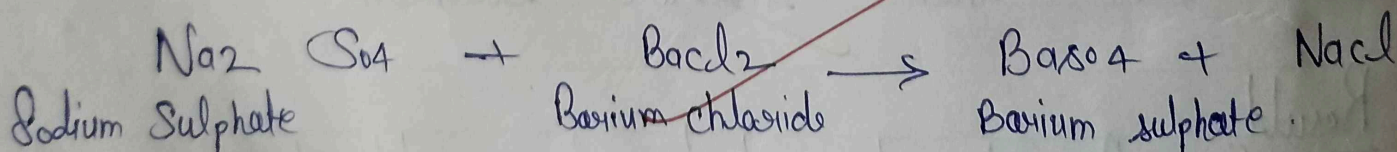
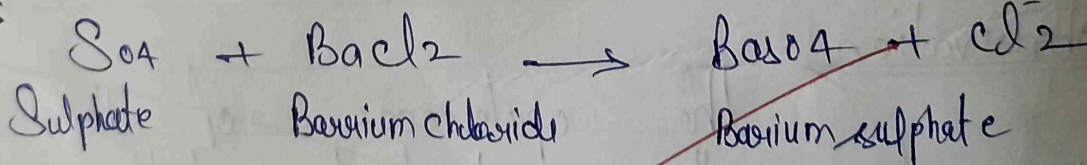


Teacher's Signature: _____

07/10/1

UNITED SECULAR SOCIETY

Reaction →



10/1/10

Experiment-2

Date _____

Experiment 02

Page No. 02

* ~~Aim~~ $\Rightarrow$

To perform a limit test for sulphate in a given sample of tap water.

* Reference -

part A " pharmaceutical chemistry Laboratory manual " CBS publishers and Distributors Pvt. Ltd. 1st 2008, pp 59.

* Requirements -

* Chemical Required - Hydrochloric acid Barium sulphate and Barium chloride.

* Apparatus Required - Measuring cylinder, glass Rod, pipette and Nessler's cylinder etc.

* Theory -

Limit test or ~~quantitative~~ or semiquantitative test designed to identify and control a small number of impurities which are likely to be present in the substance. This test involves the reaction of Barium chloride with soluble Sulphate to form the precipitate of barium sulphate which is insoluble in dilute hydrochloric acid. The Barium sulphate precipitate is white in colour.



Teacher's Signature: _____

* Procedure

Standard	Test
1. Take 1 ml of 0.1089 w/v of Na_2SO_4 / K_2SO_4 in Nessler cylinder.	Dissolve specific quantity of Sample in 10 ml of tap water.
2. Add 2 ml of dilute HCl.	Add 2 ml dilute HCl.
3. Make up the volume up to 45 ml with distilled water.	Make up the volume up to 45 ml with distilled water.
4. Add 5 ml of Barium Sulphate	Add 5 ml of solution Barium Sulphate.
5. Stirrer the solution and Allow to stand for 5 minute.	Stirrer the solution and allow to stand for 5 min.

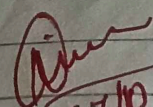
* Observation -

The opalescence of the test solution is less than the standard solution. Hence test will pass.

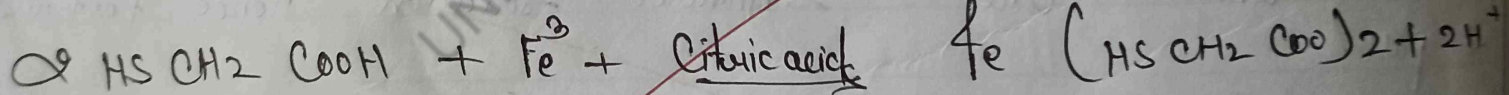
* Result -

A Limit test for Sulphate was performed.

Teacher's Signature: _____


 07/10

Chemical Reaction



Thioglycolic acid

ferrous thioglycolate.

Experiment-3

Date _____

Experiment 03

Page No. 05

* ~~AIM~~

To perform the limit test of Iron in the give sample.

* Reference

Singh H.R. Kapoor V.K. practical pharmaceutical chemistry. Vallabh, parakashan Edition 1st - 2000 page No - 59.

* Requirements —

* Chemical Reaction

Thioglycolic acid citric acid Ammonia
Solution A ferric Ammonium Sulphate

* Apparatus Required —

Nessler's cylinder. Measuring cylinder, glass Rod, pipette

* Theory —

The limit test of Iron based on the Reaction between Iron And thioglycolic acid and in the presence of citric acid a deep reddish brown colour is formed. thioglycolate is colourless in acidic medium but in alkaline medium it gives purple colour.



Teacher's Signature: _____

* Procedure.

Standard	Test.
1. Take standard 1.5 ml of Iron Solution.	Dissolve specific quantity of Sample in water.
2. Add 1.5 ml of iron free Citric acid.	Add 1.5 ml of Iron free citric acid.
3. Add 1.5 ml of thioglycollic acid.	Add 1.5 ml of thioglycollic acid
4. Volume Make up to 50 ml.	Volume make up to 50 ml.
5. Stirrer the solution and allow to stand for 5 minute.	Stirrer the solution and allow to stand for 5 min.

* Observation.

The opalescence produce should be less than standard. Hence Limit test will pass.

* Result.

The limit test of Iron was performed.

Experiment-4

Date: _____

Experiment - 04

Page No. 07

~~* Aim -~~

To carry out identification test for Aspirin.

* Reference

Singh H.R Kapor V.K practical pharmaceutical chemistry. Vallabh, prakashan Edition

* Requirement -

* Apparatus - Test tube, test tube stand, Burette pipette, Spetula, test tube Holder.

* Chemicals :-

Sodium Hydroxide, dil. H_2SO_4 ethyl alcohol
FeCl₃ Solution.

* Theory -

Aspirin is a group of medications called Salicylates. It works by stopping the production of certain natural substances that cause fever, pain, swelling and blood clots. Aspirin is also available in combination with other medication such as antacids, pain Relievers, and Cough and Cold medication.

* physical properties -

* Colour - white crystalline powder.

* Odour - odourless.



Teacher's Signature: _____

* Procedure —

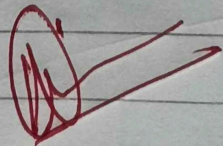
1. Boil about 0.5 g of Aspirin with 10 ml Sodium hydroxide Solution for 3-4 minute. Cool down the mixture. Add 10 ml dil. H_2SO_4 . An odour of Vinegar or acetic acid is produced and a white crystalline ppt is formed. The following test is performed on the ppt and perform the following test.

a. To the small amount of ppt add 3 ml of ethyl alcohol. 3 ml H_2SO_4 and Heat the mixture on a boiling water bath. A fruity smell is produced [Ether]

b. Dissolve the precipitate in water add $FeCl_3$ Solution. Deep Violet colour is obtained.

* Result —

Aspirin was identified in the given Sample.



Experiment - 5

Date: _____

Experiment 05

Page No. 09

* ~~Aim~~ →

To carry out the identification test paracetamol.

* Reference -

Singh H.R Kapoor V.K practical pharmaceutical chemistry. Vallabh, prakashan Edition.

* Theory -

Paracetamol (PCM), also known as acetaminophen, is a medication used to treat fever and mild to moderate pain.

* Physical properties -

Colour - white

State - solid.

Odour - odourless.

Procedure -

1. * Dissolve 0.1 g of sample in 10 ml water and feed solution to it violet-blue colour is obtained.

2. Boil 0.1 g sample with HCl for 3 minute. Add 10 ml of water. Cool it. Now add 0.05 ml of potassium dichromate violet colour is obtained (appears slowly.)

* Result - PCM was identified in the given sample.

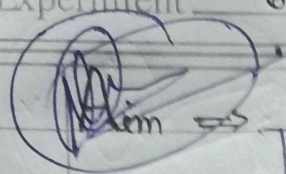
Teacher's Signature: _____

Experiment - 6

Date _____

Experiment 06

Page No. 10



To perform the identification tests for the given sample of ammonium chloride.

Reference $\Rightarrow$

Part A, "Pharmaceutical chemistry 1 Laboratory Manual," CBS publishers and Distributors Pvt. Ltd, Ed. Ist 2008, pp 59.

Requirements $\Rightarrow$

Chemical Required —

Ammonium chloride, Sodium hydroxide, red litmus paper, Sodium cobalt nitrite, Nitric acid, Silver nitrate, dilute ammonia solution, potassium dichromate, sulphuric acid, diphenyl carbazide solution.

Apparatus Required —

Test tubes, test tube stands, test tube holders, glass rod.

Theory $\Rightarrow$

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry, and solubility. In this regard, the identification of a single pure compound is

Teacher's Signature: _____

There four Very much simpler than the identification of a mixture. The experiment deals only with the identification of simple compound, i.e. those that contain only one cation and one anion.

Procedure.

Reactions of Ammonium ions:

S.No	Test	Observation.
1.	Take a few mg of sample and add sodium hydroxide solution. Heat. Bring the moist red Litmus paper near the mouth of the test tube.	Dark blue.
2.	Dissolve 10 mg of sample in water. Add 1 ml of freshly prepared 10% w/v solution of Sodium Cobalt Nitrate.	Clear yellow.

② Reaction of chlorides :-

S. No.	Test	Observation
--------	------	-------------

1.	Dissolve 2 mg of sample in 2 ml of water. Acidify with dilute Nitric Acid. Add 0.5 ml Silver nitrate solution. shake and allow to stand and add ammonia + Nitric acid	Produced white Precipitate
----	---	---------------------------------------

Result -

The identification test was performed successfully.

WIP

Experiment - 07

Date _____

Experiment 07

Page No. 13

~~Aim~~

To perform the identification test of Barium sulphate.

Reference:

1. Singh H. R. Kapoor V. K. "Practical pharmaceutical Chemistry" Vallabh Prakashan. Edition 1st, 2008 page no. 1920.
2. Chaturvedi G. R. "pharmaceutical chemistry inorganic" Himalaya publishing house. ed. 5th 2010 p.p 256-257.

Requirements:-

Chemical Required:

Barium sulphate, Barium chloride solution, dilute hydrochloric acid, Silver nitrate, solution, dilute ammonia solution, potassium dichromate, sulphuric acid etc.

Apparatus Required:

Test tube, test tube stand, test tube holder, pipette, Glass Rod etc.

Theory-

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of

Teacher's Signature: _____

acid / base chemistry, redox chemistry and solubility. In this regard the identification of a single pure compound is therefore every much simpler than the identification of a mixture.

This experiment deals only with the identification of simple compounds, i.e. those that contain only one cation and one anion.

Procedure

Reactions of Barium ions.

S.No	Test	Observation.
1.	Take 1 ml of filtrate, acidify with dilute HCl + add 0.2 ml of barium chloride solution	yellowish green colour.
2.	Add 0.1 ml of iodine solution to the suspension obtained in the above test. Add stannous chloride solution dropwise. Boil the mixture.	white precipitate

Reaction of Sulphate Ions

S. No	Test	Observation
1.	Dissolve 50 mg of sample in 5 ml of water. Acidify with dilute hydrochloric acid. Add 1 ml of barium chloride.	yellow colour.
2.	Dissolve 50 mg sample with 5 ml of water. Add 2 ml of lead acetate solution. Divide in to two parts. • To the first part add ammonium acetate solution • To the second part add sodium hydroxide solution	white precipitate

Result:

An identification test of Barium sulphate was performed.

~~NAIP~~

Experiment - 08

Date _____

Experiment 08

Page No. 18

~~Aim~~

To perform the identification test of iodine.

Reference: Singh H.R. Kapoor V.K. "practical pharmaceutical chemistry" Vallabh prakashan, Ed 1st 2000, pp 19-20.

Chaturvedi G.R. "Pharmaceutical chemistry inorganic" Himalaya publishing house, Ed 5th, 2010 pp 256-257.

Requirements -

Chemical required: Iodine, starch solution.

Apparatus required: Volumetric flask, conical flask, Burette, pipette, Glass Rod.

Theory -

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid base chemistry, Redox chemistry, and Solubility. In this regard the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, i.e. those that contain only one cation and one anion.

Teacher's Signature: _____

Experiment _____

Procedure -

S.No	Test	observation.
1.	Heat the sample gently in a test tube. Allow it to Condense.	Turn into deep purple vapors.
2.	prepare a saturated solution of iodine. Add starch solution. Heat . Cool.	Blue-Black Starch colour

Result :

Identification test of iodine was performed.

Plp +

Teacher's Signature: _____

Experiment - 09

Date _____

Experiment 09

Page No. 19

~~Aim:~~

To perform the identification test for Calcium gluconate.

Reference :-

Sudha. T. Saranya. Saranya. & practical book of pharmaceutical chemistry - I, edition 2022, medical publishers. publication, page no - 166

Requirements :-

apparatus :-

Test tube.

chemical :-

Calcium gluconate, ammonium bicarbonate water, ferric chloride.

Theory :-

Calcium Gluconate is the Calcium salt of glucium acid an oxidation product of glucose, and contains 9.3% Calcium, which is about one-third of the Calcium in strength of Calcium chloride USP. Since it is soluble to the extent of only one part in 30 parts of cold water, the 10% solution is super saturated and is stabilized by the addition of Calcium saccharate tetrahydrate 0.46% w/v (which supplies Calcium 6.02%) Each mL contains 100 mg of Calcium gluconate monohydrate.

Teacher's Signature: _____

4.6 mg of Calcium saccharate tetrahydrate, and water for injection U.S.P. eq. S. Each mL provides 9.3 mg (0.465 milliequivalents) of Calcium.

Procedure

Experiment.	Observation	Inference
Take 1 ml of required quantity of Calcium gluconate solution dissolved in hot or cold water and add a 5 ml of ammonium bicarbonate.	White precipitate was formed.	presence of Calcium.
Experiment.	Observation	Inference
Take 1 ml of solution of 3% w/v of Calcium gluconate and add few drops of ferric chloride solution.	Yellow Colour appears.	presence of gluconate.

Result - The giving sample was found to be Calcium gluconate because it contain anion (gluconate) and cation (Calcium)

Tip

Experiment-10

Date _____

Experiment

10

Page No. 20

~~Aim~~ To perform the identification test for Sodium bicarbonate.

Reference :-

Sudha T. Saranya S. practical book of pharmaceutical chemistry - 1 edition 2022. medical publishers, publication, page no - 167.

Requirement.

Apparatus required

Test tube, watch glass spirit lamp.
Chemical Apparatus - Concentrated Diuretics H_2SO_4 magnesium hydroxide.

Theory :-

Sodium bicarbonate, Commonly Known as baking soda or bicarbonate of soda, is a chemical compound with the formula $NaHCO_3$. It is a salt composed of a sodium cation and a bicarbonate anion. Sodium bicarbonate is a white solid that is crystalline but often appears as a fine powder.

Teacher's Signature: _____

Procedure —

Experiment.	Observation	Inference
A small amount sample is made into a paste with conc. HCL in a watch glass and introduced in a non-luminous part of the flame.	Golden yellow colour is formed	Presence of Sodium.
Take a required quantity of sample and dil. H ₂ SO ₄ in a test tube.	Brisk effervescence is evolved.	Presence of bicarbonate.
Take 2 ml sample solution and add 1 ml of magnesium hydroxide solution and heated	A white precipitate is formed.	Presence of bicarbonate.

Results —

The given sample was identified as Sodium bicarbonate.

~~NAIP~~

Teacher's Signature: _____

Experiment - 11

Date _____

Experiment 11

Page No. 22

~~Aim~~

To perform the identification test for magnesium sulphate.

Reference :-

Sudha. T. saranya. s practical book of pharmaceutical chemistry - I edition 2022, medical publishers. publication page no - 160 -

Requirements -

Apparatus Requirements -

Test tube, pipette, test tube stand.

Chemical Requirements -

Dilute ammonia magnesium sulphate sodium hydroxide HCl. Barium chloride solution. lead acetate. solution.

Theory :-

Magnesium Sulfate is a magnesium salt having sulfate as the counterion. It has a role as an anticonvulsant, a Cardiovascular drug, a Calcium Channel blocker, an anaesthetic, a tocolytic agent, an anti-arrhythmia drug, an analgesic and a fertilizer. It is a magnesium salt a metal sulfate and an organic magnesium sulfate.

Teacher's Signature: _____

Procedure —Test for Cations —

Experiment	Observation	Inference
Take few mg of the sample in a test tube and add 1 ml of dilute solution.	White precipitate is formed.	presence of magnesium.
To 0.5 ml of a neutral or slightly acid solution of the sample add 1 ml of a 0.18% w/v of thionin yellow and 1 ml of NaOH solution.	Bright red colour was formed.	presence of magnesium.

Test for Anions.

Experiment	Observation	Inference
Take few gram of test sample in a test tube and 1 ml of dil. HCl and 1 ml of barium chloride solution.	white precipitate is formed.	presence of Sulphate.



Test for Anions -

Experiment	Observation	Inference
Take few gram of test sample in a test tube and 1 ml of dil. HCl and 1 ml of barium chloride solution.	white precipitate is formed	presence of Sulphate.
Take few mg of test sample in a test tube and add 5 ml of water and 2 ml of lead acetate solution.	White precipitate is formed	presence of Sulphate.

Result: The identification test for magnesium sulphate was performed.

~~PHIP-1~~

Experiment-12

Date _____

Experiment 12

Page No. 25

~~Aim~~

To Carry out identification test of Sulfanilamide.

Reference -

Sudha T. Saranya's practical book of pharmaceutical chemistry I edition 2012 medical publishers publication page no-191

Requirement -

Apparatus Requirement :- Water bath, Volumetric flask, Beaker, measuring cylinder, Test tube.

Chemical Requirement:- HCl, Sulfanilamide, water, Sodium nitrite solⁿ, β -naphthol solⁿ, Sodium acetate.

Theory -

Sulfanilamide is an organic Sulfen Compound Structurally similar to p-aminobenzoic acid (PABA) with antibacterial property. Sulfanilamide competes with PABA for the bacterial enzyme dihydropteroate synthase, preventing the incorporation of PABA into dihydrofolic acid, the immediate precursor of folic acid. This leads to an inhibition of bacterial.

Teacher's Signature: _____

Folic acid Synthesis and de novo synthesis of purines and pyrimidines ultimately resulting in cell growth arrest and cell death.

Physical Properties —

Colour — yellowish — white or white Crystal or fine powder.

Procedure —

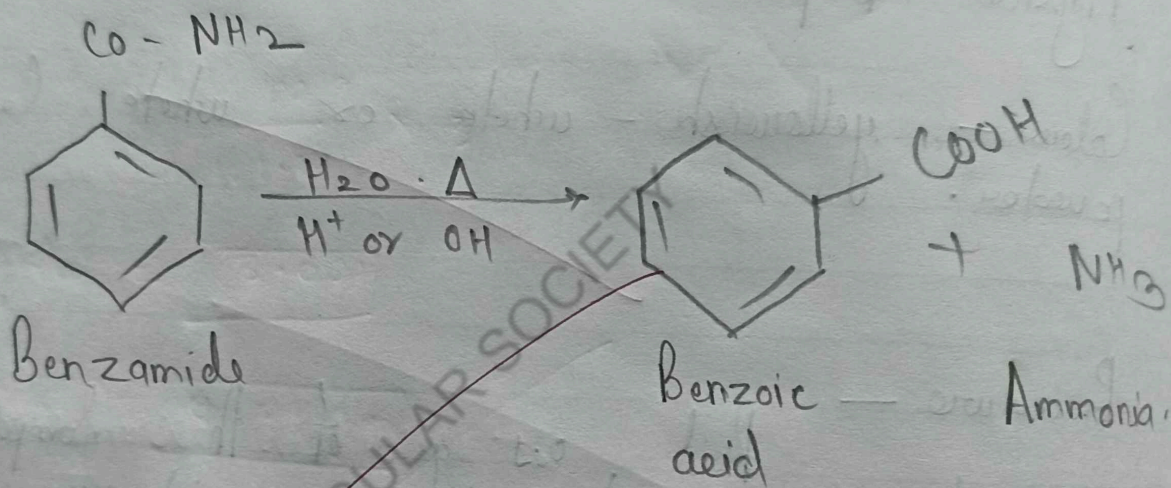
Boil 0.1 g of the sample in 10 ml of dilute hydrochloric acid for 15 minutes and cool. 2 ml solution of ice water was added and 4 ml of Sodium nitrite solution was added and cooled again, pour the mixture in to 2 ml of B-naphthol solution containing 1 g of Sodium acetate orange precipitate was produced.

Dissolved about 10g of sample in a mixture of 10 ml of water and 2 ml of 0.1 N Sodium hydroxide add 0.5 ml of Copper Sulphate solution, a pale turbidity appears at first but it gradually changes to brownish grey finally set as reddish brown precipitate.

Result —

Identification test of Sulfanilamide was done successfully.

Teacher's Signature: _____



Experiment - 13

Date _____

Experiment

Page No. 27

~~Aim~~

To prepare and submit the crude product of Benzoic acid and Calculate the percentage yield.

Reference - Sudha T., S. Savanya, "Practical Book of Pharmaceutical Chemistry, I- edition 2022, medical publisher, Page No - 184 - 185

Requirement : Benzamide, NaOH, HCl, Litmus paper.
WT Balance, Conical flask, Heating mantle, Beaker

Principle : Benzamide may be hydrolysed by aqueous acid or alkali, yielding benzoic acid or its salt.

Materials :

Benzamide - 3 g

0.5 M NaOH - 70 cm³

2 M HCl - ~ 10 cm³

Litmus paper - (red and blue), Ice.

Procedure -

Weigh out ^{3.00} 3.00 g of benzamide, place it in a 250ml Conical flask and add to it 0.5 M Sodium hydroxide solution. Fit the flask with a cork carrying glass tubing and boil the mixture for about 20 minutes. Test the gas evolved at the head of the glass tubing when the mixture begins to boil. Allow the reaction mixture to cool and then transfer it to a 100 ml beaker.



Teacher's Signature: _____

Amount of Benzamide taken for synthesis :- 3.00 gm

molecular wt of product - 122 g/mol

molecular wt of Reactant - 121.14 g/mol

$$\text{Theoretical yield} = \frac{\text{M.wt of Product}}{\text{M.wt of Reactant}} \times \text{wt of Reactant taken}$$

$$= \frac{122}{121.14} \times 3$$

$$\text{Theoretical yield} = \boxed{3.021}$$

$$\text{Practical yield} = \boxed{4.04 \text{ gm}}$$

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

$$= \frac{4.04}{3.021} \times 100$$

$$\% \text{ yield} = \boxed{133.7 \text{ gm}}$$

Add an excess of 1 M hydrochloric acid with a dropper, swirling continuously until a copious precipitate is obtained. Filter the resulting solution under suction, wash the crystals thoroughly with minimum amount of ice cold water. Filter to obtain pure but damp crystals of the product. Dry these crystals thoroughly between filter papers until. Measure the mass of the product, base on the mass of the crystals you obtained, Calculate the percentage yield.

uses :-

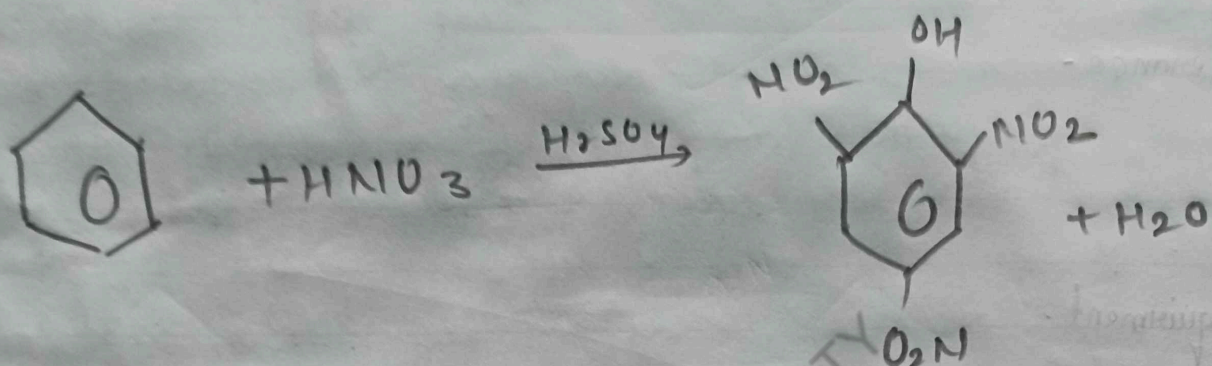
To manufacture a wide variety of product such as perfumes, dyes, topical medications and insect repellents.

Result:- The benzoic acid was prepared and submitted

- 1) Theoretical yield = 3.021 gm
- 2) Practical yield = 4.04 gm
- 3) Percentage yield = 133.7 gm.

~~Prepared~~





2,4,6-Trinitrophenol (picric acid)

Experiment-14

Date _____

Experiment

Page No. 30

~~Aim -~~ To prepare and submit picric acid and calculate its percentage yield.

Reference: Sudha T., S. Sarayana, "Practical Book of Pharmaceutical Chemistry, I- edition 2022, Medical Publishers, Page NO - 186-187

Requirement :-

Apparatus:- Water bath, Measuring cylinder, Balance, Conical flask.

Chemical Required.

phenol - 2.5 ml.

Conc. Sulphuric acid - 3.5 ml.

Conc. nitric acid - 9.5 ml.

Theory -

Picric acid is basically prepared from phenol by reacting phenol with concentrated Sulphuric acid and concentrated nitric acid. The nitro groups from nitric acid attacks on ortho and para positions which is the best suitable and stable position for the newly formed compound which is picric acid.

Principle -

Phenol on nitration with concentrated nitric acid in the presence of Con. Sulphuric acid gives picric acid (2,4,6-trinitro phenol)

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Calculation:-

Amount of Phenol taken synthesis = 2.5g

Molecular weight of Product = 229.1 g/mol

Molecular weight of Reactant = 94.11 g/mol

$$\text{Theoretical yield} = \frac{\text{M.wt of Product}}{\text{M.wt of Reactant}} \times \text{Wt of Reactant taken}$$

$$= \frac{229.1}{94.11} \times 2.5$$

$$\text{Theoretical yield} = \boxed{6.085}$$

$$\text{Practical yield} = \boxed{4.039}$$

$$\text{Percentage yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}}$$

$$= \frac{4.039}{6.085} \times 100$$

$$\text{Percentage yield} = \boxed{66.37\%}$$

Procedure —

2.5 g of phenol and 8.5 ml of Con. sulphuric acid were mixed and stirred well. This is then heated on a water bath for 30 minutes when a clear solution of o- and p-phenol sulphonic acids is obtained. Cool the flask in ice bath and then add 9.5 ml of Con. nitric acid with constant stirring. Allow the reaction mixture to stand for some time till the complete evolution of brown fumes stops, heat the flask for 2 hours with occasional shaking. Cool the reaction mixture and add 100 ml of H₂O. Filter the product of yellow crystals of picric acid. Wash thoroughly with water to remove all the nitric acid. Recrystallise the product with dilute alcohol.

uses —

Preparation of other derivatives.

Result:— Picric acid was prepared and submitted.

Theoretical yield = 6.085 gm

Practical yield = 4.039 gm

Percentage yield = 66.379 gm.

~~12/11/1~~



Teacher's Signature: _____

~~Aim~~

To perform the identification test and test for purity of Caffeine.

Reference - Sudha T, S. Sarayana, Practical Book of Pharmaceutical chemistry, I-edition 2022, Medical publishers, Page No -

Requirement: Apparatus: Test tube, Test tube stand, Porcelain dish, Waterbath.

Chemical: Tannic acid, Caffeine, Potassium chloride, HCl.

Theory:

The murexide test is an analytical technique to identify the presence of Caffeine and other purine derivatives in a sample. These compounds do not respond to the common alkaloid detecting tests such as Dragendorff's reagent. In this test the alkaloids are mixed with a tiny amount of potassium chlorate and a drop of hydrochloric acid. The sample is then evaporated to dryness and the resulting residue is exposed to ammonia vapour. purine alkaloids produce a pink colour in this test [1] [2] murexide (ammonium purpurate) with a purple colour is also produced in this test.



Identification test:

Experiment	Observation	Inference
To a saturated solution add few drops of ferric acid solution.	white precipitate is formed	presence of Caffeine.
pure crystals of Caffeine are mixed with potassium chlorate and Conc. HCl 2-3 drops in porcelain dish and evaporated to dryness on a water bath and ammonia solution.	A purple colour is formed.	presence of Caffeine.

Test for purity -Test for heavy metals:

Dissolve 1g in 5 ml of 0.1 ml HCl and dilute to 25 ml with water. The solution complies the limit test for heavy metals method A (20 ppm).

Test for Sulphated ash:

Not more than 0.1% determined by the method in I.P.

Acidity / alkalinity:

Dissolve 0.2g in 10 ml boiling water and Cool, add 0.1 ml of bromomethylene blue solution. The solution is coloured green/yellow. Titrate with 0.02 M NaOH to obtained blue

Teacher's Signature: _____

Experiment. _____

Colour, not more than 0.2 ml is required.

Result :-

The Identification Test and test for purity
was performed as per I.P.

~~Report~~

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