Karpagam Academy Of Higher Education (Established Under Section 3 0f UGC Act,1956) COIMBATORE – 641021

DEPARTMENT OF CHEMISTRY

Syllabus

Semester - I

19CHP112 ORGANIC CHEMISTRY PRACTICAL-II 4H 2C
(QUANTITATIVE ANALYSIS AND DOUBLE STAGE PREPARATIONS)

Instruction Hours/week:L: 0 T:0 P:4 Marks: Internal:40 External: 60 Total:100

Scope

This practical course deals with the quantitative analyses of organic compounds and double stage preparation of organic compounds by standard organic reactions.

Objectives

On successful completion of the course the students should have

1. Learnt about the quantitative analyses of organic compounds.
2. Learnt the two stage preparation of organic compounds by important name reactions.

Methodology

Blackboard teaching and Demonstration.

Contents

Estimation of phenol, ethyl methyl ketone, glucose, nitro, amino and methoxy groups, unsaturation in an organic compound.

Analysis of oils Reichert- Meissl value, Iodine value, saponification value and acetyl value.

Double stage preparations- Anthranilic acid and pthalimides.

Extraction and estimation of active constituents

Only for learning purpose and demo (Not for exam)

a. Lactose from milk.
b. Caffeine from tea.
c. Nicotine from tobacco extract.
d. Citric acid or ascorbic acid from a tablet or from a natural source.
e. Curcumin from turmeric.
f. Lycopene from tomato.

SUGGESTED READINGS:

Text Books:

1. Arun Sethi, (2003). *Laboratory experiments in Organic Chemistry*. New Delhi: New Age Publisher.
2. Bansal, R. K, (2008). *Laboratory Manual of Organic Chemistry* (IV Edition). New Delhi: New Age Publishers.
1. Furniss, B. S., Hannaford, A. J., Smith, P. W. G., & Tatchell, A. R., (2004). *Vogel's Textbook of Practical Organic Chemistry* (V Edition). Singapore: Pearson Education Ltd.

Reference Books:

1. Lepse, P. A., & Lyle B. P., (1986). *Lab Manual for Lingren's Essentials of Chemistry*. New Delhi: Prentice Hall.
2. Mendham, J., Denney, R. C., Barnes, J.D., & Thomas, M. (2002). *Vogel's textbook of quantitative Chemical Analysis* (VI Edition). Singapore: Pearson Education Ltd.

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DEPARTMENT OF CHEMISTRY

Name of the Staff	:	**Dr. M.R.EZHILARASI**
Department	:	Chemistry
Title of the Paper	:	**ORGANIC CHEMISTRY PRACTICAL-II**
Paper Code	:	**19CHP112**
Class	:	I- M. Sc-Chemistry
Year and Semester	:	2019–2020 and I-Semester

No.	**Duration Hours**	**Name of the Experiments**	**Supporting Materials**
1	4	Preparation of anthranilic acid from Phthalic anhydride	R1-241-242
2	4	Preparation of Benzanilide from Benzophenone	R2-1049-1050
3	4	Preparation of aspirin	R3-191
4	4	Estimation of aniline	R1-131-135
5	4	Estimation of phenol	R1-131-135
6	4	Estimation of glucose	R1-148-149
7	4	Estimation of Ketone	R1-138-139
8	4	Determination of	R1-150-151

		saponification of oil	
9	4	Isolation of Lactose from Milk and citric acid from lemon	R1-233
10	4	Viva-voce questions discussion	
	4	Model Practical Examination	

REFERENCES

R1:Bansal, Raj K, 2001. Laboratory Manual of Organic Chemistry. IV Edition, New Age Publishers, New Delhi

R2:Mendham J., R.C.Denney, J.D.Barnes and M.Thomas, 2002. Vogel's textbook of quantitative Chemical Analysis. VI Edition, Pearson Education Ltd., Singapore

R3: Pandey.O.P, Bajpai.D.N, Giri.S. 2001.Practical Chemistry,S.Chand and Company , New Delhi

ORGANIC CHEMISTRY PRACTICAL-II
Laboratory Manual
Quantitative Analysis

M.Sc Chemistry

DEPARTMENT OFCHEMISTRY

KARPAGAM ACADEMY OF HIGHER EDUCATION
COIMBATORE – 21

Dr. M.R.Ezhilarasi

Assistant Professor,

Department of Chemistry

CONTENTS

S.No	NAME OF THE EXPERIMENTS	Page. No.
	DOUBLE STAGE ORGANIC PREPARATIONS	
1	Acetanilide from p-Bromoacetanilide	3
2	Benzanilide from Benzophenone (Beckmann Rearrangement)	6
3	Preparation of Anthranilic Acid from Phthalic Anhydride	9
4	Aspirin from Methylsalicylate	12
	ORGANIC ESTIMATIONS	
5	Estimation of Phenol	16
6	Estimation of Aniline	19
7	Estimation of Glucose	20
8	Saponification Value of an Oil	25
9	Estimation of Ethyl Methyl Ketone	28
	EXTRACTION OF NATURAL PRODUCTS	
10	Isolation of Lactose from Milk	32
11	Isolation of Citric acid from Lemon	34
12	Isolation of Lycopene from Tomato	36

DOUBLE STAGE PREPARATIONS

1. ACETANILIDE FROM p-BROMOACETANILIDE:

Stage-1:Acetanilde from Aniline:

Principle

Aniline can be acetylated easily by refluxing with a mixture of acetic anhydride and sodium acetate.

$$\underset{\text{Aniline}}{C_6H_5NH_2} + \underset{\text{Acetic anhydride}}{(CH_3CO)_2O} \xrightarrow{CH_3COONa} \underset{\text{Acetanilide}}{C_6H_5NHCOCH_3} + \underset{\text{Acetic acid}}{CH_3COOH}$$

Mechanism:

Aniline acts as a nucleophile. The lone pair of electrons present on the nitrogen atom attacks the carbonyl group of acetic anhydride, and the resulting product is acetanilide.

Requirements:

Chemicals	
Chemical Name	**Quantity**
Aniline freshly distilled	3ml
Acetic anhydride	4 ml
Glacial acetic acid	4 ml
Apparatus	
Apparatus Name	**Nos**
Round bottom flask (250 ml)	1

Reflux condenser	1
Wire-gauze	1
Glass beaker (250 ml)	1
Test tube (10 ml)	1

Procedure:

The glacial acetic acid is mixed with acetic anhydride in a dry test tube and shaken well. Aniline is taken in a round bottom flask and the above mixture is added slowly to aniline with constant shaking. A bit of broken porcelain is added to prevent bumping. The flask is fitted with a reflux condensed, and heated gently for about 30 minutes over a wire-gauze. The hot liquid is then poured in a thin stream, with stirring, on crushed ice in a beaker. The white solid that separates is filtered, washed with cold water, dried with filter paper, and the yield is noted.(Expected yield:4.5 g).

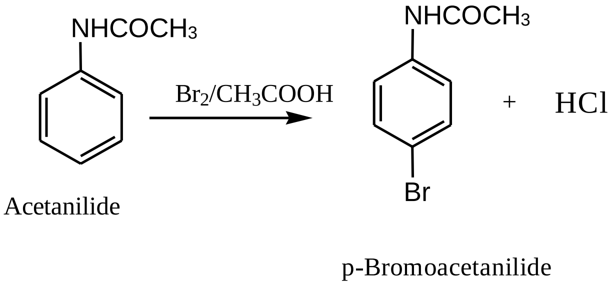
Recrystallisation:

About 1 g of the crude acetanilide sample is dissolved in the minimum amount of hot water; 1 g of animal charcoal is added and then heated. The hot liquid is rapidly filtered (through hot water funnel, if available).The filtrate is allowed to cool. The acetanilide crystallizes out in rhombic plates.

Stage-2: p-Bromoacetanilide from Acetanilide:

Principle:

The anilide group present in acetanilide is a moderate activating group, which directs the incoming brominium ion to *ortho* and *para* positions. Practically, only the *para*-product is formed, due to the steric hindrance of the bulky functional group.



Reqirements:

Chemicals	
Chemical Name	**Quantity**
Acetanilide	2 g
Bromine	3ml
Glacial acetic acid	15 ml
Rectified spirit	10ml
Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Cork	1
Boiling test tube	1
Dropper	1
Glass rod	1

Procedure:

Acetanilide (2 g) is dissolved in 5ml glacial acetic acid in a conical flask, fitted with a good cork. Bromine (3 ml) is dissolved in 10 ml glacial acetic acid and taken in a boiling test tube. Using a dropper, about 1ml of bromine in acetic acid is added at a time to the contents of the flask and shaken well. The addition of bromine is continued till the color of the solution in the flask becomes yellow. The flask is corked well and shaken vigorously for about 10-15 minutes. The flask is kept aside for 15 minutes. The contents of the flask are then poured in a thin stream in to crushed ice in a beaker, with stirring. The colorless crystals of *p*- Bromoacetanilide that separates out are filtered, washed and dried. (Expected yield: 2.5 g)

Recrystallization:

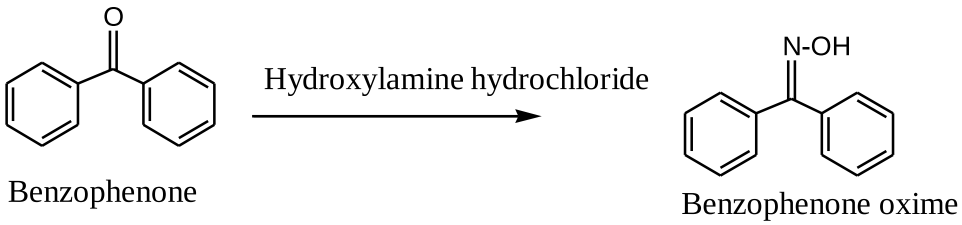
About 0.5 g of crude acetanilide is recrystallized from hot rectified spirit, and the melting point is determined. (Correct melting point: 167°C)

2. BENZANILIDE FROM BENZOPHENONE (BECKMANN REARRANGEMENT)

Stage-1: Benzophenone oxime from Benzophenone:

Principle

When benzaldehyde is condensed with hydroxyl amine, one water molecule is removed and gives benzophenone oxime or benzophenoxime. This undergoes Beckmann rearrangement followed by hydrolysis gives benzanilide.



Reqirements:

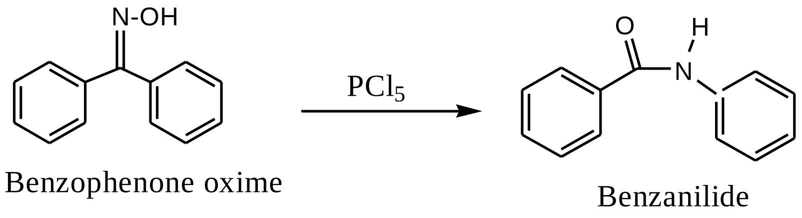
Chemicals	
Chemical Name	**Quantity**
Benzophenone	5 g

Hydroxylamine Hydrochloride	3 g
Rectified spirit	50 ml
Sodium hydroxide	8 g
Concentrated Hydrochloric acid	15 ml
Apparatus	
Apparatus Name	**Nos**
Round bottom flask (250 ml)	1
Reflux condenser	1
Wire-gauze	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Water bath	1
Funnel	1

Procedure:

A mixture of benzophenone, hydroxylamine hydrochloride, about 10 ml of water and rectified spirit is placed in a round-bottom flask. Sodium hydroxide pellets are then added in portions with constant shaking to the mixture in the flask. When the reaction becomes too vigorous the flask is cooled with tap water. When all the sodium hydroxide has been added, the flask is fitted with a reflux condenser and heated on a water bath for 15 minutes. The flask is then cooled, the contents poured into 100 ml of water containing conc.HCl. The precipitate is filtered at the pump, washed with water and dried in vacuum desiccators over sulphuric acid. (Expected yield: 5 g; Melting Point: 142°C)

Stage-2: Benzanilide fromBenzophenone oxime:



Reqirements:

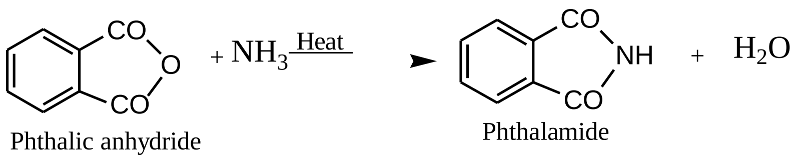
Chemicals	
Chemical Name	**Quantity**
Benzophenone oxime	2 g
Dry Ether	3 g
PCl_5	50 ml
Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Wire-gauze	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Water bath	1
Funnel	1

Procedure:

A solution of 2 g of benzophenone oxime in a 20 ml of dry ether is taken in a 250ml conical flask. 3 g of PCl_5 is added well. Ether is evaporated on the water bath about 20 ml of water is added. The solution is boiled for 30 minutes .The supernatant liquid is recrystallized from methanol. (Expected yield: 1.6 g; Melting Point: 163°C)

3. PREPARATION OF ANTHRANILIC ACID FROM PHTHALIC ANHYDRIDE:

Stage-1: Preparation of phthalamide from Phthalic anhydride:



Reqirements:

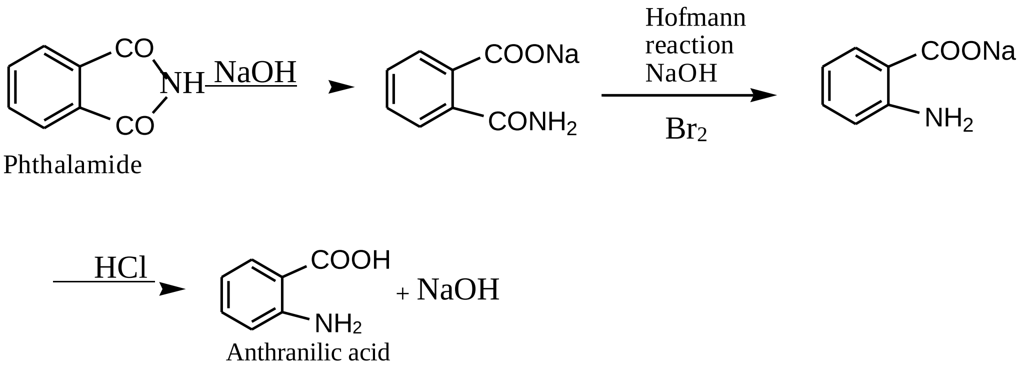
Chemicals	
Chemical Name	**Quantity**
Phthalic anhydride	10 g
Concentrated ammonia solution	10.5 mlg
Apparatus	
Apparatus Name	**Nos**
Round bottom flask (250 ml)	1
Air condenser	1
Wire-gauze	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Water bath	1
Funnel	1

Procedure:

10 g of phthalic anhydride and 10.5 ml of the concentrated ammonia solution is heated in around-bottom flask with an air condenser; heating is continued until it forms a homogenous

mixture. During heating the flask is shaken occasionally poured the content of the flask while hot in a mortar ground well and recrystallized from the rectified spirit. (Expected yield: 4.5 g; Melting point: °C)

Stage-2: Preparation of Anthranilic acid from phthalamide:



Reqirements

Chemicals	
Chemical Name	**Quantity**
Phthalamide	2.4 g
Bromine	1 ml
Sodium Hydroxide	5.5 g
Acetic acid	Few drops
Apparatus	
Apparatus Name	**Nos**
Wire-gauze	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Water bath	1
Funnel	1

Procedure:

Preparation of a solution for 3 g of NaOH in 12 ml of water in conical flask and cooled 0^0C and 1 ml of bromine in the portion and shaken well so that bromine reacts completely. Prepare a solution 2.5 g of NaOH in 8 ml of water and added 2.4 g of phthalamide that was finely powder. Added one portion of cold hydrobromide solution stirred vigorously, the solid dissolved the temperature, 70°C, warm the mixture to 280^0C for about 20 minutes cooled and added con.HCl slowly until the solution was neutral, the solid precipitate completely by adding acetic acid filtered and washed with cold water and recrystallized from hot water.

4. ASPIRIN FROM METHYLSALICYLATE

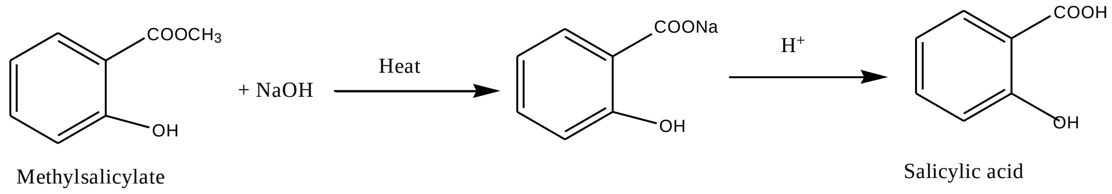
Stage-1

SALICYLIC ACID FROM METHYL SALICYLATE

The same experimental set-up discussed above can be used for the preparation of salicylic acid from methyl salicylate.

Principle:

Methyl salicylate is hydrolysed to sodium salicylate and methanol when heated with aq.NaOH.From sodium salicylate,salicylic acid is precipitated by the addition of con.HCl.



Requirement:

Chemicals	
Chemical Name	**Quantity**
Methyl salicylate	10 g
Aq.NaOH soln. (10%)	30 ml
Concentrated Hydrochloric acid	20 ml
Apparatus	
Apparatus Name	**Nos**
Round bottom flask (250 ml)	1
Reflux condenser	1
Wire-gauze	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Water bath	1
Funnel	1

Procedure:

The procedure to be adopted is the same as that given for preparing benzoic acid from ethyl benzoate.

Methyl salicylate (4 ml) and 10% aq.NaOH (25 ml) are mixed in a round bottom flask. A bit of broken porcelain is added to prevent bumping. The round bottom flask is fitted with a reflux condencer, and heated over wire-gauze steadily till the oily layer of ester disappear (about 30-45 minutes). Then, the contents of the flask are cooled and transferred into a 250 ml beaker. The flask is rinsed with about 5 ml water and the rinsings are also added to the beaker. To free the benzoic acid from the salt solution, on HCL is added, drop by drop, with constant stirring; till the solution is distinctly acidic (test with litmus paper).Salicylic acid is liberated in the solid form. The content of the beaker are cooled, and the precipitated benzoic acid is filtered off at the pump (water pump or vacuum pump), washed with cold water, dried using filter paper and the yield of the dry crude product is noted. (Expected yield: 3 g)

Recrytallization:

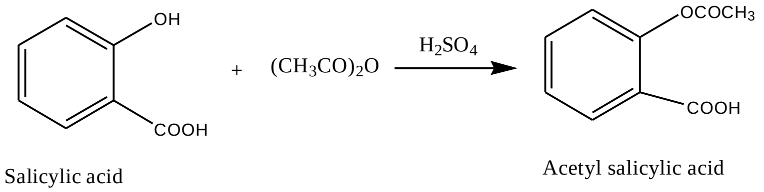
About 0.5 g of the crude benzoic acid is dissolved in 5 ml of boiling water,filtered, poured into a watch glass and allowed to cool slowly. After the crystals are formed the liquid is poured off and the crystals are dried with bits of filter paper. The melting point is determined. (Correct melting point: 121^0C.)

Stage-2

ACETYL SALICYLIC ACID (ASPIRIN) FROM SALICYLIC ACID

Principle:

Salicylic acid is a phenolic acid. The Phenolic group can easily be acetylated using acetic anhydride or acetyl chloride. When salicylic acid is boiled with acetic anhydride, acetyl salicylic acid is obtained.



Requirements:

Chemicals	
Chemical Name	**Quantity**
Salicylic acid	10 g
Acetic anhydride	30 ml
Concentrated Sulphuric acid	20 ml
Ethanol (rectified spirit)	

Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Wire-gauze	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Water bath	1
Funnel	1

Procedure:

Salicylic acid (3 g) is mixed with acetic anhydride (5 ml) in a 100 ml conical flask, and 3 drops con. Sulphuric acid is added. The mixture is shaken well, and heated on a water-bath at 50-60° C for about 10-15 minutes, with occasional shaking. Then the conical flask is removed, the contents are cooled under the tap, and then poured in a thin stream into 100ml of water in a beaker. The acetyl salicylic acid that separates out is filtered off at the pump, washed, dried with filter paper and weighed. (Expected yield: 4 g)

Recrystallization:

The crude acetyl salicylic acid is recrystallized as follows: About 1 g of the sample is dissolved in the minimum amount of hot ethanol and then poured into 40 ml of water in a 250 ml beaker. The turbid solution is then warmed to get a clear solution. The resulting clear solution is filtered and allowed to cool slowly, when needles of acetyl salicylic acid separates out. It does not possess a definite melting point (decomposes at 128-135°C)

Note: Acetyl salicylic acid can also be recrystallized from a mixture of equal volumes of acetic acid and water.

ORGANIC ESTIMATIONS

INTRODUCTION

Quantitative estimations of organic compounds plays an important role in the analytical laboratory,e.g., to find out the purity of the chemicals and the extent adulteration, if any. The quantitative estimation is based on standard chemical reaction and they generally involved volumetric methods. Since most of the organic reactions are slow, instructions regarding mixing of reagents, conducting the reactions, and the time interval, required for titration must be followed strictly.

Knowledge of the chemical reactions involved in the estimation is very essential, in order to understand the calculation involved.

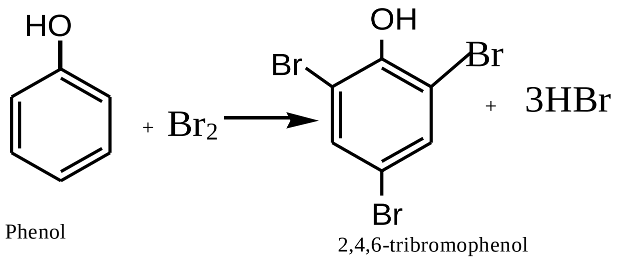
A standard conversion table for glucose estimation is also including.

An error of 2-3 %is generally allowed in organic estimation.

1. ESTIMATIONS OF PHENOL

Principle

Phenol on bromination in aqueous solution gives 2, 4, 6-tribromophenol according to the following equation:-



Instead of using a standard solution of bromine water, a bromate-bromide mixture, which readily liberates bromine in the presence of an acid, is preferred, because its strength does not very with time.

Equivalent weight of phenol =Molecular Weight/6=94/6 =15.67

The bromate-bromide mixture readily liberates bromine, on acidification; this bromine is used for the bromination of phenol.

$$BrO_3^{-} + 5Br^{--} + 6H^{+---} \rightarrow 3Br_2 + 3H_2O$$

The aqueous solution of phenol is treated with an excess of the brominating agent, and the unreacted bromine is back-titrated against standard sodium thiosulphate using starch as a indicator.

1,000 ml of 1N thiosulphate =1,000ml of 1N Br =15.67 g of phenol.

1 ml of N/10 Br =0.001567 g of phenol

Reqirements (Chemicals)

Chemicals	
Chemical Name	**Quantity**
AR potassium dichromate	1.2 g /100 ml (about N/10)
Potassium bromide	3 gdissolved in 200 ml of water
Potassium bromate	0.6 g} (about N/10)
Sodium thiosulfate	6 g /250 ml water (about N/10)
Potassium iodide	10 g/100 ml water (about 10%)
Fresh distilled phenol	8 g /1 litre water (about N/2). (20 ml volume range may be given to each student in a bottle).
Concentrated Hydrochloric acid	100 ml

Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Iodine flask(250 ml)	1
Standard measuring flask (100 ml)	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Burette (50 ml)	1
Pipette (20 ml)	1
Funnel	1

Procedure

Titration 1: *Standardisation of sodium thiosulfate solution*

A standard solution of N/10 potassium dichromate is prepared thus**:**about 1.2 g of analar (pure) crystals (equivalent weight =49), accurately weight in a chemical balance, is dissolved in water and the solution is made up to 250 ml in a standard flask.

20 ml of standard $K_2Cr_2O_7$ solution is pipetted out into a clean conical flask. About 5 ml of conc.HCl is added, followed by 10 ml of 10% aq.KI solution. The liberated iodine is immediately titrated against thiosulfate solution taken in the burette. When the solution becomes pale yellow in colour, 1 ml of freshly prepared starch solutionis added,and the titration is continued,addingthiosulfate solution in drops, with constant shaking. The end point is the change of color from blue to green (due to Cr^{3+}).The titration is repeated to get concordant values. The strength of thiosulfate is calculated.

$$CrO_7^{2--} + 14H^+ + 6I^{--} \rightarrow 2Cr^{3+} + 7H_2O + 3H_2$$

$$2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$$

Titration 2: *Standardisation of* BrO_3*/Br mixture*

20 ml of the bromate-bromide mixture is pipetted out into a clean 250 ml conical flask.About 10 ml of 10%aq.KI soln. is added, followed by 5 ml con.HCl. The liberated iodine is titrated against thiosulfate solution taken in the burette, till the solution becomes yellow in colour.About1 ml of freshly prepared starch solutionis added, and the titration is continued (in drops) with constant shaking. The end point is the disappearance of blue color (blue to colourless). The titration is repeated to get concordant values.

Titration 3: *Estimation of phenol*

The given phenol solution in the bottle is quantitatively transferred and made up to 100 ml on a standard flask.20 ml solution is pipetted out into a clean conical flask. The soln. is diluted by adding 50 ml of distilled water. 5 ml con.HCl.is also added. To this a known volume (40 ml) of bromate-bromide mixture is added from the burette) slowly with constant shaking.(The solution should attain a permanent yellow color, due to excess of liberated bromine; otherwise a further known volume of the bromate-bromide mixture is added).Then, about 10 ml of 10% aq.KI is added. The liberated iodine is titrated against thiosulfate solution using starch as an indicator, which is added near the end point. The end point is the the disappearance of blue color (blue to colorless). A duplicate is also conducted.

Calculation

Calculate the strength of sodium thiosulphate from the standard$K_2Cr_2O_7$ (Titration 1)

Let 20 ml of BrO_3/Br mixture require A ml of thiosulfate solution (from Titration 2).

Therefore, 40 ml* of BrO_3/Br should requireA/20 X 40 *= 2A ml .thio.

(* or,actual volume used).

Let (20 ml phenol + 40 ml brominating mix) required B ml thio (Titration3).

20 ml phenol reacted with (2A-B) ml thio.

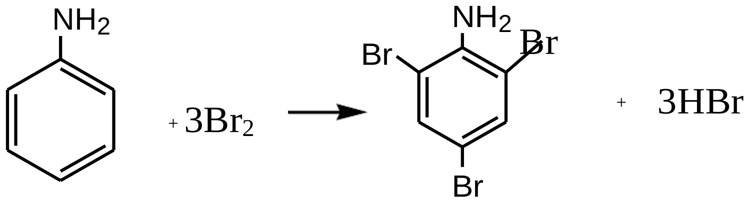
Strength of phenol solution =2 A-B str.of thio/20

Amount of phenol in 100 ml of made up solution = (2A-B)str.of thio/20 **x**15.67/10

=---------g

Note;

Aniline can also be estimated by following the same method as that given above for the estimation of phenol. Aniline reacts with bromine as follows:



2,4,6-tribromoaniline

Equivalent weight of aniline = Molecular weight/ 6x 93/6 =15.5

2. ESTIMATION OF GLUCOSE (BERTRAND'S METHOD)

Principle

Glucose is reducing sugar. When glucose is boiled with an excess of alkaline, copper hydroxide, it is oxidized to gluconic acid and Cu (OH) $_2$ gets reduced to cuprous oxide.

CH_2OH -$(CHOH)_4$—CHO +2 $Cu(OH)_2 \rightarrow CH_2OH$ --- $(CHOH)_4$---COOH + $Cu_2O \downarrow$ +H_2O

The required red--coloured Cu_2O is dissolved in a warm acidic solution of ferric alum.Cu_2Oreduced ferric sulphate (in ferric alum) to ferrous sulphate.The reduced $FeSO_4$is titrared against standard $KMnO_4$.

$$Cu_2O + Fe\,(SO_4)_3 + H_2SO_4 \rightarrow 2CuSO_4 + 2FeSO_4 + H_2O$$

$$10FeSO_4 + 8H_2SO_4 + 2KMnO_4 \rightarrow 2MnSO_4 + 5\,Fe\,(SO_4)_3 + 8\,H_2O$$

2Cu= $2FeSO_4$=2 litres 1 N $KMnO_4$

1atom of copper (63.6.g) = 1 litre 1 N $KMnO_4$

1000 ml of 1 N $KMnO_4$ =63.6.g.of copper

1ml of N/10 $KMnO_4$= 0.00636.g of copper

= 63.6.g.of copper

From the amount of copper, the equivalent amount of sugar is calculated from the given conversion table.

Requirements:

Chemicals	
Chemical Name	**Quantity**
AR Oxalic acid	1.6 g/250 ml water

Potassium permanganate	0.75 g/250 ml water (About N/10)
Cupric sulfate (Fehling A)	8 g/200 ml water
Rochelle salt – 40 g Caustic soda – 30 g} (Fehling B)	dissolved in 200 ml water
Ferric alum –24 g Con.H_2SO_4 –20 ml}	dissolved in 200 ml water
AR Glucose	13 g/1 litre of water. (20 ml volume range may be given to each student in the bottle.)
dil.H_2SO_4	100 ml
Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Iodine flask(250 ml)	1
Standard measuring flask (100 ml)	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Burette (50 ml)	1
Pipette (20 ml)	1
Funnel	1
Wire gauze	1

Procedure

Titration 1: *Standardization of Potassium permanganate solution*

A Standard soln. of N/10 oxalic acid is prepared by weighing about 1.6 g. of analar acid crystals (equivalent weight: 63) accurately in a chemical balance and dissolving in water. The soln. is made up to 250 ml in a standard flask.

20 ml Standard oxalic acid soln. is pipetted out into a clean 250 ml conical flask.20 ml dilute sulphuric acid is added 60^0C. (Bearablewarmth).The hot soln. is then titrated against $KMnO_4$soln.taken in the burette. The end point is the appearance of pale permanent pink colour. The titration is repeated to get concordant values. The strength of $KMnO_4$ is calculated.

$$2MnO_4^{--} + 16\ H^+ + 5CrO_4^{2--} \rightarrow 2Mn^{2+} + 10CO_2 + 8\ H_2O$$

Titration 2*: Estimation of Glucose*

The given glucose solution in the bottle is quantitatively transferred and made up to 100 ml in a standard flask.20 ml of $CuSO_4$solution (Fehling solution—A)is added, followed by the addition of 20 ml of tartrate (Fehling solution—B). The solution is then heated to boiling, at maintained at this temperature for 2 to 3 minutes. The red cuprous oxide precipitate formed is allowed to settle. The clear supernatant liquid is carefully decanted.

Amount of Copper precipitated by different amounts of Glucose

Glucose in mg	Copper in mg	Glucose in mg	Copper in mg	Glucose in mg	Copper in mg
10	20.4	41	79.3	71	31.4
11	22.4	42	81.1	72	33.1
12	24.3	43	82.9	72	34.7
13	26.3	44	84.7	74	36.3
14	28.3	45	86.4	75	37.9
15	30.2	46	88.2	76	39.6
16	32.3	47	90.0	77	41.2
17	34.2	48	91.8	78	42.8

18	36.2	49	93.6	79	44.5
19	38.1	50	95.4	80	46.1
20	41.1	51	97.1	81	47.7
21	42.0	52	98.9	82	49.3
22	43.9	53	100.6	83	50.9
23	45.8	54	102.3	84	52.5
24	47.7	55	104.1	85	54.0
25	49.6	56	105.8	86	55.6
26	51.5	57	107.6	87	157.2
27	53.4	58	109.3	88	158.8
28	55.3	59	111.1	89	160.4
29	57.2	60	112.8	90	162.0
30	59.1	61	114.5	91	163.6
31	60.9	62	116.2	92	165.2
32	62.8	63	117.9	93	166.7
33	64.6	64	119.6	94	168.3
34	66.5	65	121.3	95	169.9
35	68.3	66	123.0	96	171.5
36	70.1	67	124.7	97	173.1
37	72.0	68	126.4	98	174.6
38	73.8	69	128.1	99	176.2
39	75.7	70	129.8	100	177.8
40	77.5				

The precipitate is washed with water with water to remove the excess of $CuSO_4$ solution (It is advisable to filter the precipitate through G-4 sintered glass crucible.) To the precipitate, acidified ferric alum is added slowly with shaking, till the red precipitate completely dissolves. About 20 mlof dil.H_2SO_4 is added and the solution, is titrated against $KMnO_4$ taken in the burette.The end point is the appearance of pale permanent pink colour. The duplicate is also conducted.

Calculation

Calculate the strength of $KMnO_4$ from the standard oxalic acid (Titration 1)

Volume of sugar solution taken = 20 ml

Volume of $KMnO_4$ required to react with the reduced $FeSO_4$ =A ml

1 ml of N/10 $KMnO_4$ = 6.36 ml of copper

$$A \text{ ml of N/10 } KMnO_4 = \frac{6.36 x A\ x\ str.\ of\ KMnO4}{0.1}$$

= B mg of copper

From the conversion table, the glucose equivalent is noted, corresponding to copper equivalent.

B mg of Cu = Z mg of sugar

20 ml glucose solution contains Z mg of sugar

Amount of glucose present in 100 ml of the made- up solution = 100/20 x Z=5 Z mg

3. SAPONIFICATION VALUE OF OIL

Principle

The saponification value is defined as the number of milligrams of KOH required for the hydrolysis of 1 g of oil or a fat. To determine the saponification value, a weighed quantity of the given oil is refluxed with a known volume of alcoholic potassium hydroxide solution.The unused alkali is then back- titrated against standard acid.

Saponification values of some common oils are given below

S.No	**Oil Name**	**Saponification values**
1	Mustard oil	174
2	Castor oil	175-183
3	Olive oil	185-196
4	Groundnut oil	186-194
5	Linseed oil	188-195
6	Gingerly oil	188-193
7	Cotton seed oil	194-196
8	Coconut oil	253-262

Requirements:

Chemicals	
Chemical Name	**Quantity**
AR sodium carbonate crystals	7 g/250 ml water
Alcoholic potash (KOH)	6 g dissolved in 200 ml rectified spirit(about 0.5 N)
Phenolphthalein	
HCl (about N/2)	10 ml con. HCl in 200 ml water

Concentrated Hydrochloric acid	100 ml
Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Round bottom flask (250 ml)	1
Reflux condenser	1
Standard measuring flask (100 ml)	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Burette (50 ml)	1
Pipette (20 ml)	1
Funnel	1

Procedure

Titration 1: *Standardisation of HCl*

About 7 g of analar sodium carbonate crystals (eq.wt=53) are weighed accurately in a chemical balance ,dissolved in water ,and the solution in made up to 250 ml in standard flask.20 ml is pipetted out into a clean conical flask. About 2 drops methyl orange indicator are added and titrated against hydrochloric acid taken in the burette (till the light pink colour appears). The titration is repeated to get concordant values. The strength of HCl solution .is calculated.

Titration 2: ***Determination of saponification value of an oil***

About 1-2 g of the given oil is weighed accurately in a chemical balance and transferred into 250 ml round- bottom flask.25 ml of alcoholic potash (about N/2) is added from the burette slowly into the flask. It is then titrated with an air condenser, and heated on a water- bath for about 30 minutes. A blank is run simultaneously with the same quantity of alcoholic potash, but without the oil. Both the flasks are cooled, and titrated against standard HCl solution using about 1 ml of phenolphthalein indicator. The end point is the disappearance of pink colour.

Calculation

Calculate the strength of HCl solution(Titration1)

Let w.g be the weight of oil taken.

Let A ml be the volume of HCl required for excess alcoholic potash.

Let B ml must be the blank titre value

1000 ml of 1 N HCl = 56.1 g of KOH

1 ml of 1 N HCl =56.1 mg of KOH

Saponification value = $56.1\ x\ (B-A) xstr.of\ HCl$ / w

4. ESTIMATION OF ETHYL METHYL KETONE

Principle

Acetone (or any ketone containing CH_3 –CO group) forms iodoform,when shaken well with a mixture of iodine and aq.KOH solution according to the following equations

$$I_2 + 2KOH \rightarrow KI + KOI + H_2O$$

$$CH_3COC_2H_5 + 3KOI \rightarrow CI_3COC_2H_5 + 3KOH$$

$$CI_3COC_2H_5 + KOH \rightarrow CHI_3 + C_2H_5COOK$$

3 molecules of iodine =1 molecules of acetone

3 molecules of iodine =58 g of acetone

6 litre of 1Niodine= 58 g of acetone

1 ml of 1 N iodine =0.0097 g of acetone

The excess of iodine (present as KI and KOI) is decomposed in the presence of an acid, according to the following equation

$$KI + KOI + H_2SO_4 \rightarrow K_2SO_4 + I_2 + H_2O$$

The liberated iodine is titrated against standard thio using starch as s indicator.

$$I_2 + 2Na_2S_2O_3 \rightarrow 2NaI + Na_2S_4O_6 \text{ (sod.tetrathionate)}$$

Requirements:

Chemicals	
Chemical Name	**Quantity**
AR potassium dichromate	1.2 g
Sodium thiosulfate	6 g /250 ml of water (about N/10)

Potassium iodide	10 g/ 100 ml water
Potassium hydroxide	11 g/ 200 ml water (about 1 N)
Aq.iodine	2.5 g/ 200 ml water (about N/10)
Acetone	5 g/1 litre water (about N/2)(20 ml volume range may be given to each student in a bottle.)
Starch solution (1 %)	10 ml (freshly prepared)
Concentrated Hydrochloric acid	20 ml
Apparatus	
Apparatus Name	**Nos**
Conical flask (250 ml)	1
Iodine flask (250 ml)	1
Standard measuring flask (100 ml)	1
Glass beaker (250 ml)	1
Dropper	1
Glass rod	1
Burette (50 ml)	1
Pipette (20 ml)	1
Funnel	1

Procedure

Titration 1: *Standardisation of Thio*

A standard solution of N/10 $K_2Cr_2O_7$ is prepared by weighing about 1.2 g of analar crystals (eq.wt = 49) accurately in a chemical balance, dissolving in water, and making up to 250 ml in a standard flask.

20 ml of standard $K_2Cr_2O_7$soln.is pipetted out into a 250 ml clean conical flask. About 5 ml con.HCl is added, followed by 10 ml of 10% aq.KI.The liberated iodine is immediately titrated against thio taken in the burette. When the solution acquires straw yellow colour, 1 ml of freshly

prepared starch is added, and the titration is continued (in drops) with constant shaking. The end point is the change in colour from blue to green (due to Cr_{3+}). The titration is repeated to get concordant values .The strength of thio is calculated.

$$Cr_2O_7^{2} + 14H^{+} + 6I \rightarrow 2Cr^{3+} + 7H_2O + 3I_2$$

$$2S_2O_3^{2} + I_2 \rightarrow 2I + S_4O_6^{2}$$

Titration 1: *Standardisation of Iodine solution*

20 mlof iodine solution is pipetted out into a clean 250 ml conical flask. About 1 ml of starch is added. The solution is titrated against thio taken in the burette. The end point is the disappearance of blue colour (blue to colourless). The titration is repeated to get concordant values.

Titration 2: *Estimation of acetone*

The given acetone solution in the bottle is quantitatively transferred and made up to 100 ml in a standard flask. 20 ml of the made up solution is pipetted out into a clean 500 ml stopper bottle.50 ml of 1 Naq.KOH is added to the bottle. A known volume of aqueous iodine (100 ml of N/10) is added from the burette,slowly,into the bottle, and the mixture is shaken well for 15 minutes. It is then acidified with about 20 ml of dil.H_2SO_4 and the liberated iodine is titrated against thio sulfate solution taken in the burette using starch as indicator. The end point is the the disappearance of blue colour.

A blank titration is carried out without acetone, using the same volume of KOH iodine against thio sulfate, starch as a indicator.

Calculation

Calculate the strengths of thio and iodine solutions (Titrations 1 and 2)

Let the volume of thio required by(acetone +I_2) solution = A ml

Blank titre value (100 ml of iodine + KOH) =B ml

B ml of thio is required for 100 ml iodine.

A ml of thio is required for100 /B x A = *x* ml of iodine

Unreacted iodine = *x*ml

Reacted iodine = (100—*x*)ml

1 ml of N iodine =0.0097 g of acetone.

(100—*x*) ml of iodine =0.0097 x (100—*x*) x (str. of iodine) g of acetone

100 ml of made-upsolution contains = 0.0097 x (100-x) x (str. ofI_2) x= 100/20

= ------ g of acetone.

EXTRACTION OF NATURAL PRODUCTS

1. ISOLATION OF LACTOSE FROM MILK

Requirements

Chemicals	
Chemical Name	**Quantity**
Milk	500 ml
Dilute acetic acid	100 ml
Calcium carbonate	2 g
Rectified spirit	50 ml
Apparatus	
Apparatus Name	**Nos**
Beaker(1 liter)	1
Centrifuging tubes and machine	1
Wire gauze	1
Watch Glass	1
Dropper	1
Glass rod	1
Funnel	1

Procedure

About 500 ml of milk is diluted with water and centrifuged to remove the fat. The fat appears on the top of the centrifuging tube is removed. The clear thin milk obtained after centrifugation is called skim milk. To the clear skim milk about 50 ml of dilute solution of acetic acid is added slowly with constant stirring. A white solid separates out calledcasein. The precipitated casein is filtered off, and the filtrate is neutralized with dilute aqueous sodium hydroxide solution. The solution is made very slightly acidic by adding 2 or 3drops of acetic acid to the neutralized solution. The solution is boiled on wire-gauze with 2 g of calcium carbonate solid. The mixture stirred well and filtered hot. The solution is concentrated by boiling and converted into one third volumes. About 3 volumes of rectified spirit is added and the solution is filtered. White crystals of lactose separate out. The white solid is filtered off at the pump, washed with alcohol and dried in air.

(Expected yield: 2 g)

2. ISOLATION OF CITRIC ACID FROM LEMON

Requirements:

Chemicals	
Chemical Name	**Quantity**
Lemon juice	100 ml
Calcium chloride (10%)	2 ml
Dilute ammonia	15 ml
Dilute Sulphuric acid	10 ml
Apparatus	
Apparatus Name	**Nos**
Beaker(1 liter)	1
Wire gauze	1
Watch Glass	1
Dropper	1
Glass rod	1
Funnel	1

Procedure

About 100 ml of either lemon juice or pineapple juice is taken and diluted with double the amount of water. The juice is neutralized by adding dilute ammonia slowly with constant stirring. The citric acid present in the juice is now converted into ammonium citrate. To the solution of ammonium citrate, about 2 ml of a 10% aqueous solution of calcium chloride is added. The resulting solution is heated to boiling and set aside for 30 minutes. A white precipitate of tricalcium citrate is formed. The precipitate is washed well with water and the washing is decanted. From the tricalcium citrate, citric acid can be obtained by treating with dilute sulphuric acid. The white precipitate of calcium sulphate formed is filtered and the filtrate is concentrated where crystals of citric acid appear.

4. ISOLATION OF LYCOPENE FROM TOMATO

Lycopene is a red pigment which is extracted from tomatoes

Requirement:

Chemicals	
Chemical Name	**Quantity**
Tomato	200 g
Benzene	30 ml
Ether	15 ml
Apparatus	
Apparatus Name	**Nos**
Beaker(1 liter)	1
Wire gauze	1
Watch Glass	1
Dropper	1
Glass rod	1
Funnel	1

***Procedure**:*

Mash 200g of ripe tomatoes in to a fine paste. Stir the paste with two separate portions (30 ml each) of warm benzene (40°C). Decant the benzene layer and filter to remove any solids. Distil off completely under vaccum. Recrystallize the residue from ether. The yield is 15 mg, m.p. 174-175°C.