MANUAL FOR CHEMISTRY LABORATORY

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School of Sciences

DEPARTMENT OF CHEMISTRY

Contents

1. INTRODUCTION:

A chemistry laboratory is one of the best places in the world where one can see miracles happening. It is a workshop for the chemists and the place where the students can learn the basics of applications of chemistry. Here, the students can learn the techniques of identification, estimation and preparation of chemical substances. The aims and objectives of practical work in the laboratory can be stated as follows:

- Better understanding of scientific concepts and principles.
- Promotion of basic skills and competencies (procedural skills, observational skills, drawing skills, reporting and interpretation skills).
- Awakening and maintaining curiosity in the learning environment.

But the chemistry laboratory is such a place where people should be very careful. It is very important for a student to know how to work with equipment and what precautions should be taken. Different type of chemicals present in the laboratory may cause hard on exposure, contact or mishandling. This type of information including do's and don'ts will be discussed in this manual.

2. LABORATORY FACILITIES:

University has established its first own Chemistry laboratory which was set up at School of Sciences, Kalyani Regional Campus of this University in 2016 with modern instrumentation facility to cater to the needs of the students in their practical classes in a centralized manner. Along with general facilities, University also has the following sophisticated instruments facility in its own chemistry laboratory at Kalyani Campus:

i) Fluorescence spectrophotometer (Agilent; Cary Eclipse)
ii) FTIR spectrophotometer (Agilent. Cary 630)
iii) UV-VIS Spectrophotometer (Shimadzu; UV-1900)
iv) Microwave-Ultraviolet-Ultrasonic Synthesis system (Microwave Reactor; NuTech)
v) Rotary evaporator with Chiller (BUCHI; R100)
vi) 3-digit balance (Shimadzu) and 5-digit balance (Shimadzu)
vii) P^H meter
viii) Conductometer

ix) Centrifuge machine (Remi)

x) Temperature controlled Hot-air oven

xi) Digital Melting Point

xii) Magnetic Stirrer (Remi 1mL & 2mL)

xiii) Fume chamber

3. RULES TO MAINTAIN IN THE CHEMISTRY LABORATORY:

A list of do's and don'ts can be a helpful reminder of laboratory safety issues. These are as follows

Do's:

- Presence in the Laboratory classes (LCES for BDP students) is mandatory.
- You have to bring **a Laboratory Coat** (Apron) and **Safety Goggles** and also have corresponding Study material, calculator and a **laboratory notebook** (not loose-leaf or spiral) while performing experiments in the laboratory. Without them you are not allowed to enter the laboratory session.
- Read the study material before entering the laboratory to build overall concept about the laboratory experiments present in the syllabus.
- Eating, drinking, smoking, and cell phones are forbidden in the laboratory at all times. Do not chew gum during laboratory sessions. Avoid unnecessary movement and talk in the laboratory.
- Cheating will lead to a zero point for Laboratory reports and in the notebook. If it is repeated second time, you will fail from the course**.**
- Read the label on the bottle carefully before using the required chemical. Always bring your container to the reagent shelf and do not take the bottles to your desk.
  Note: NEVER RETURN UNUSED CHEMICAL TO THE REAGENT BOTTLE.
- The laboratory is equipped with fire extinguisher, fume chamber and first aid supplies. Learn the locations and proper use of these items.
- Insert glass objects into rubber stoppers and corks with extreme care.
- You have to check your data sheet with your Laboratory Counsellor at the end of the

Laboratory period.

- Any accident involving even the most minor injury must be reported to the Laboratory Counsellor.
- At the end of the experiment, clean and dry the glass apparatus and wipe off the top of the working table. All electrical apparatus should be switched off and unplugged. Ensure that the gas, taps should be turned fully off and all waste should be placed in the appropriate waste container before you leave the laboratory.

Don'ts:

- Do not attempt to modify the written procedures unless instructed to do so. Perform only assigned experiments.
- Do not through sodium metal (Na) in water/basin. It can make explosion leading to an accident.
- Do not attempt any unauthorized experiment. Perform only lab operations and activities given by the Laboratory Counsellor.
- Do not work alone in any laboratory.
- Do not insert the pipette or dropper into the reagent bottles; this helps in avoiding any possible contamination.

  Note: ALWAYS HANDLE PRIMARY STANDARDS AND STOCK SOLUTIONS WITH CARE. CONTAMINATION WILL LEAD TO POOR RESULTS FOR YOU AND OTHERS.
- Graduated cylinders and bottles are not to be heated because these break very easily and their volume also changes.

4. LABORATORY SAFETY:

At all times when you are working in the chemistry laboratory you should use prudent practices. **Recognize that safety is, ultimately, everyone's individual responsibility.** Following safety information must remember into chemistry laboratory:

- When diluting sulphuric acid, **pour the acid slowly and carefully into the water** with constant stirring. Never add water to the acid as it may result in the liberation of a lot of heat.
- Use a **fume hood** when directed to do so.
- Student must not reach across lighted burners as it may result in an accident.
- While heating a substance in a test tube, care should be taken to ensure that the mouth of the **test tube is not pointing at anyone**. A student should never look down into a test tube that is being heated.
- **Never touch or taste a chemical** or solution as most of chemicals are either corrosive or poisonous.
- Exercise care when picking up potentially hot objects.
- Always **wash your hands** prior to existing the lab and before eating.

5. LABORATORY FIRST-AID:

If a corrosive substance falls on your skin, should be reported immediately to the supervisor and immediately wash the spot with large quantities of water, followed by remedial action indicated below:

- **Spurting of corrosive chemical**: Protect your eyes from any spurting of acid or a corrosive chemical. In case of such spurting into the eyes, immediately wash with lot of water and inform the Laboratory Counsellor immediately.
- **Acid spill**: Treat with sodium bicarbonate or ammonium carbonate (2M) solution; then apply vaseline or a soothing cream.
- **Base spill**: Treat with acetic acid (1M) followed by vaseline or a soothing cream
- **Bromine**: Treat with 2M ammonia; keep the affected part dipped in dilute sodium bisulphite solution till bromine is washed off. Finally apply vaseline.

- **Phenol**: Wash with ethanol and then take hospital treatment.

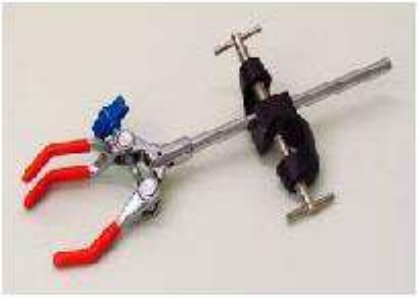
The most common accidents in the chemistry laboratory involve cuts, burns or fire. The first-aid to be given in each case is below:

- **Cuts**: If you have a cut, wash the wound well with cold water immediately. If bleeding is severe, apply pressure directly on to the wound to stop the bleeding. Then an antiseptic cream can be applied to the wound; it should be followed by proper dressing of the wound.
- **Burns**: Wash the burnt part with cold water for some time and then apply Burnol to it.
- **Fire**: In the event of fire, the flames should be extinguished with one of the extinguishers in the laboratory and the supervisor notified immediately. A small fire in a beaker, caused by the vapours of an inflammable liquid can be extinguished by covering it with a watch glass. If the clothes catch fire, one should lie on the flow and, fire can be put off by wrapping a thick blanket around the body.

6. IDENTIFYING SOME COMMON CHEMISTRY LABORATORY EQUIPMENTS:

		
Separating Funnel	**Pipette**	**Weighing Bottle**
		
Evaporating Dish	**Petri Dish**	**Filtering Flask**
		
Distilling Flask	**Watch Glass**	**Mortar Pestle**

		
Beakers	Erlenmeyer Flask	Graduated Cylinder
		
Pisette	Thermometer	Burette
		
Test Tube	Condenser	Volumetric Flask

		
Funnel	Test Tube Rack	Test Tube Holder
		
Curicible	Tongs	Balance
		
Clamp	Lab Stand	Bunsen Burner

7. COMMON REAGENTS REQUIRED FOR CHEMICAL ANALYSIS:

7.1. STRENGTH OF CONCENTRATED ACIDS AND BASES:

Name	Specific Gravity	Normality (Approximate)
Hydrochloric Acid	1.19	12 N
Sulphuric Acid	1.84	36 N
Nitric Acid	1.42	16 N
Glacial Acetic Acid	1.05	17 N
Syrupy Phosphoric Acid	1.71	15 N
Liquor Ammonia	0.83	18 N

7.2. PREPARATION OF DILUTE ACIDS AND BASES SOLUTIONS:

Name	Preparation of Solution	Strength
Hydrochloric Acid	Dissolve 83.3 ml of conc. HCl in 416.7 ml of distilled water to prepare 500 ml solution	2N
Hydrochloric Acid	Dissolve 166.6 ml of conc. HCl in 333.4 ml of distilled water to prepare 500 ml solution	4N
Sulphuric Acid	Dissolve 83.3 ml of conc. H_2SO_4 in 416.7 ml of distilled water to prepare 500 ml solution	6N
Sulphuric Acid	Dissolve 55.5 ml of conc. H_2SO_4 in 444.5 ml of distilled water to prepare 500 ml solution	4N
Acetic Acid	Dissolve 117.6 ml of glacial acetic acid in 382.4 ml of distilled water to prepare 500 ml solution	4N
Acetic Acid	Dissolve 58.2 ml of glacial acetic acid in 441.8 ml of distilled water to prepare 500 ml solution	2N
Ammonium Hydroxide Solution	Dissolve 111 ml of liquor NH_3 in 389 ml of distilled water to prepare 500 ml solution	4N
Sodium Hydroxide Solution	Dissolve 50 g of NaOH in 500 ml of distilled water to prepare 500 ml solution	10%; 0.6N

7.3. PREPARATION OF AQUA REGIA:

It is prepared by mixing 1 volume of conc. HNO_3 and with 3 volume of conc. HCl.

7.4. PREPARATION OF SOME COMMON INDICATORS:

Name	Preparation of Solution	Strength
Ba – diphenylamine Sulphonate	Dissolve 0.2 g of the dye staff in 100 ml of distilled water	0.2%
Methyl orange (pH range 3.1 – 4.4)	Dissolve 0.05 g of the dye staff in 100 ml of distilled water	0.05%
Phenolphthalein (pH range 8.3 – 10)	Dissolve 0.5 g of the dye staff in 100 ml of 50% of ethanol	0.5%
Starch Solution	Prepare a paste of 1 g of soluble starch with a little water and pour it into 100 ml of boiling water with constant stirring. Boil the mixture 2-3 minutes more.	1%

7.5. EQUIVALENT WEIGHT OF SOME COMMON REAGENTS:

Name	Molecular Weight	Equivalent Weight
Potassium permanganate $KMnO_4$	158	158/5 = 31.6
Potassium dichromate $K_2Cr_2O_7$	294.18	294.18/6 = 49.03
Oxalic Acid $H_2C_2O_4.2H_2O$	126	126/2 = 63
Mohr's Salt $(NH_4)SO_4.FeSO_4.\ 6H_2O$	392.13	392.13/1 = 392.13
Sodium Carbonate Na_2CO_3	106	106/2 = 53
Hydrochloric Acid HCl	36.5	36.5/1 = 36.5
Sulphuric Acid H_2SO_4	98	98/2 = 49
Sodium Hydroxide NaOH	40	40/1 = 40

7.6. SOME COMMON RACK REAGENTS:

Sl. No.	Name	Preparation of Solution	Strength
1	Ammonium carbonate	Dissolve 80 g of $(NH_4)_2CO_3$ in 430 ml of distilled water and add 70 ml of liquor NH_3.	4 N
2	Barfoed's Reagent	Dissolve 5 g of cupric acetate in 100 ml of distilled water and add 1 ml of acetic acid.	
3	Benedict's solution	Dissolve 17.3 g of $CuSO_4.5H_2O$ in 100 ml of distilled water. Dissolve 173 g of sodium acetate and 100 g of anhydrous Na_2CO_3 in 800 ml of	

		distilled water. Mix this two solution, a bluish green solution is obtained.	
4	Br_2 – water	Dissolve 11 ml of Br_2 in 1000 ml of distilled water.	
5	Calcium chloride	Dissolve 13.25 g of $CaCl_2$ in distilled water and then diluted to 250 ml with distilled water.	0.5 N
6	Denige's Reagent	Dissolve 5 g of yellow HgO in 20 ml of conc. H_2SO_4. Add 80 ml of distilled water carefully. Cool and filter.	
7	2,4 Dinitrophenyl hydrazine sulphate	Dissolve 2 g of 2,4-DNP in 100 ml of methanol, add 4 ml of conc. H_2SO_4	
8	Fehling's solution-A	Dissolve 34.5 g of $CuSO_4.5H_2O$ in 500 ml of distilled water and add few drops of conc. H_2SO_4.	
9	Fehling's solution-B	Dissolve 175 g of Rochelle salt (Potassium-Sodium tartaret) with 70 g of NaOH in 500 ml of distilled water.	
10	Ferric chloride solution	Dissolve 1 g of $FeCl_3$ in 100 ml of distilled water.	1%
11	Iodine solution	Dissolve 20 g of KI in 50 ml of distilled water and add 8 g of I_2. Dilute the to 1000 ml.	0.1 N
12	Lime water	Shake 0.6 g of lime with 250 ml of distilled water and filter the mixture. Keep the filtrate in well-stoppered bottle.	0.4 N
13	Molisch's Reagent	Dissolve 10 g of - Naphthol in 100 ml of alcohol	10%
14	Reinhardt solution	Dissolve 67 g of $MnSO_4.4H_2O$ in 250 ml of distilled water, add this solution to the mixture of 133 ml conc. H_2SO_4 and 250 ml of distilled water then add 133 ml of H_3PO_4.	
15	Schieff's Reagent	Dissolve 0.5 g of Rosaniline hydrochloride in 250 ml of distilled water. Cool and saturated the solution with SO_2 gas till the solution becomes colourless.	
16	Silver nitrate	Dissolve 1.7 g of $AgNo_3$ in 100 ml of distilled water and add 1-2 drops of liquor NH_3.	0.1 N
17	Starch- KI solution	Dissolve a paste of 1 g of starch in 100 ml of boiling distilled water. Add 1 g of KI and few drops of $CHCl_3$.	1%
18	Tollen's Reagent	Add 5-6 drops of 10% NaOH solution to 25 ml of 0.1 N $AgNO_3$ solution. Mixed thoroughly and allow to stand. Decant of the supernatant liquid and just dissolve the precipitate with strong NH_3 solution by adding dropwise.	

8. PREPARATION OF LABORATORY NOTEBOOKS:

One of your goals in this laboratory course should be to learn to keep proper records of your work. Your laboratory reports will be based on the data in your notebook, and the more complete the data are the more likely it is that you will be able to prepare a good report. **In a broader sense, a notebook is essential in laboratory work**.

You will be required to keep your laboratory records in a hard-cover, bound notebook. Careful notes should be taken during each laboratory lecture and recorded in ink at the time they are obtained. The Laboratory Counsellor may guide about the details format for writing the experiment in the notebook, as well as advice on the techniques that you will use. Observations in the notebook must be signed by the Laboratory Counsellor daily. The following are the requirements for your notebook preparation:

- Each day's work should be dated. Title of the experiment being performed should be indicated clearly.
- No erasures should be made; mistakes should be crossed out with a single line but remain legible.
- Pages must not be removed from the notebook.
- Use tables and graph to organize data whenever possible.
- The laboratory notebook should include all experimental data, such as masses, burette readings, temperature, notable occurrences (especially phase or color changes) all mathematical computations during and after the laboratory session.
- A separate conclusions section may be requested for each experiment. This is a short restatement of the main finding of the report.

9. SAMPLE EXERCISES BASED ON SELECTED TOPICS:

9.1. EXCERCISE 1: Preparation of coordination compound e.g. Potassium tris(oxalato) chromate(III)trihydrate; $K_3[Cr(C_2O_4)_3].3H_2O$

Principle:

Potassiumtrioxalatochromate(III)trihydrate is made by adding potassium dichromate in small portions to a hot solution of oxalic acid:

$$K_2Cr_2O_7 + 7H_2C_2O_4 + 2K_2C_2O_4 = 2\ K_3[Cr(C_2O_4)_3] + 6CO_2 + 7H_2O$$

Chemicals required:

(a) Oxalic acid, $H_2C_2O_4.2\ H_2O$: 7.8g

(b) Potassium oxalate, $K_2C_2O_4.\ H_2O$: 3.5g

(c) $K_2Cr_2O_7$: 3.0g

(d) Absolute alcohol : q.s.

Method of Preparation:

Dissolve 7.8g oxalic acid dihydrate in 20mL warm water in a 250mL beaker. To the solution add 3.0g $K_2Cr_2O_7$ in portions. When the vigorous reaction (due to the effervescence CO_2) subsides, heat to boil for 5 minutes and then add 3.5g of potassium oxalate monohydrate to it. Allow to cool under tap to room temperature and add 10.0mL ethanol. Stir and allow stand for 20 -30 minutes. Filter through suction, wash with 50% alcohol and dry in the air.

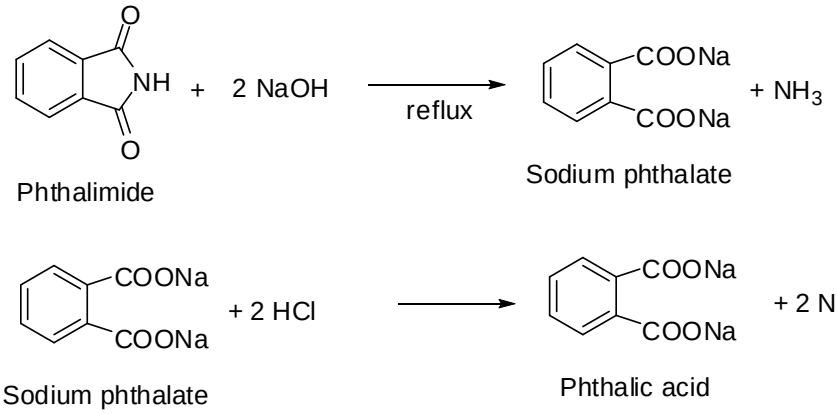
Yield: 7.2g.

Submit the product to your instructor in a paper wrapped and labelled including your name(s). Note down the experimental results following the chart given below.

Weight of $K_2Cr_2O_7$ taken	Theoretical Yield of $K_3[Cr(C_2O_4)_3].3H_2O$	Weight of $K_3[Cr(C_2O_4)_3].3H_2O$ obtained	percentage yield of product

9.2. EXCERCISE 2: Application of Hydrolysis reaction in Organic synthesis; Preparation of Phthalic acid

Reaction: Phthalimide on hydrolysis with 10% aqueous NaOH solution produces Sodium phthalate. Aqueous solution of sodium phthalate on treatment with conc. HCl produces Phthalic acid.



Instruments required:

- Round-bottomed flask (100ml)
- Beaker
- Condenser
- Stand and clamp
- Bunsen burner
- Buchner funnel with suction pump

Chemicals required:

- Phthalimide (5 g)
- NaOH (5 g)
- conc. HCl

Experimental Procedure: Take 5 g. of Phthalimide in a 100 ml r.b. flask fitted with a condenser. Pour 50 ml 10% NaOH (5 gm NaOH in 50 ml water) in it and put some glass beads into the solution. Heat the mixture to reflux on a Bunsen burner for 30 minutes. Then cool the reaction mixture at room temperature and transfer the mixture in a 250 ml beaker. Cool the beaker in ice. After cooling acidify the mixture strongly by adding conc HCL with stirring. Filter the white solid Phthalic acid on a Buchner funnel and wash it with water to make it acid free. Recrystallize the crude phthalimide from hot water and note the weight of obtained product, calculate its percentage yield and check its melting point.

Submit the product to your instructor in a paper wrapped and labelled including your name(s). Note down the experimental results following the chart given below.

Weight of phthalimide taken	Theoretical Yield of Phthalic acid	Weight of crude Phthalic acid obtained	Weight of recrystallized Phthalic acid obtained	percentage yield of product	Melting Point of recrystallized Phthalic acid

9.3. EXCERCISE 3: Estimation of Fe(II) and Fe(III) in a given mixture using $K_2Cr_2O_7$ solution

Principle:

The estimation is done by two steps. Direct titration of the mixture with standard $K_2Cr_2O_7$ after maintaining proper condition gives the amount of Fe^{2+}. Again Fe^{3+} of the mixture is first reduced to $Fe^{2+}$with $SnCl_2$ adding dropwise in hot 6 (N) HCl medium followed by the addition of drop of $SnCl_2$ in excess. After cooling the solution to room temperature excess $SnCl_2$ is removed by adding $HgCl_2$ solution when a silky white ppt appears. This ensures the completeness of the reduction.

$$2Fe^{+3} + Sn^{+2} \rightleftharpoons 2Fe^{+3} + Sn^{+4}$$

$$Sn^{+2} + 2HgCl_2 \rightleftharpoons Hg_2Cl_2 \downarrow + Sn^{+4} + 2Cl^-$$

After maintaining proper condition, this is titrated with the same standard $K_2Cr_2O_7$ solution. This titre value corresponds to the total iron [Fe^{3+} + Fe^{2+}]. The difference of the titre values will give the amount of Fe^{3+}. $Cr_2O_7^{2-}$ oxidises Fe^{2+} to Fe^{3+} in acid medium and itself gets reduced to Cr^{3+}

$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightleftharpoons 2Cr^{3+} + 7H_2O + 6Fe^{3+}$$

1 mole $Cr_2O_7^{2-}$ $\equiv$ 6 moles Fe^{2+}

or 1/6 mole $Cr_2O_7^{2-}$ $\equiv$ 1 mole Fe^{2+} $\equiv$ 1 Equivalent

Hence, 1 g equivalent of $K_2Cr_2O_7$ $\equiv$ 55.847 g of Fe

Or, 1000 ml 1 (N) $K_2Cr_2O_7$ solution $\equiv$ 55.847 g of Fe

Indicator: The estimation of Fe^{2+} is done by using Barium or Sodium diphenylamine sulphonate (BDS) in presence of H_3PO_4 or F^-.

Chemicals Required:

i) Standard~0.1 (N) $K_2Cr_2O_7$ solution

ii) Saturated aqueous solution of Barium-diphenylaminesulphonate (BDS) indicator salt

iii) Conc HCl

iv) 15 % $SnCl_2$ solution

v) 5% $HgCl_2$ solution

vi) Syrupy H_3PO_4

vii) Fe^{2+} and Fe^{3+} mixture (Unknown)

Procedure:

i) Determination of Fe (II):

An aliquot of 25 ml Fe^{2+} and Fe^{3+} mixture is pipetted out in a 500 ml conical flask, 100 ml 2 (N) H_2SO_4, 3 ml syrupy H_3PO_4, 4-5 drops of Ba-diphenylaminesulphonate indicator are added and titrated with the standard $K_2Cr_2O_7$ solution until the colour of the solution just changes from green to reddish-violet. The titration is repeated twice.

ii) Determination of total iron (Fe^{2+}+ Fe^{3+}):

An aliquot of 25 ml from the given Fe^{2+} and Fe^{3+} mixture is pipetted out in a 500 ml conical flask, 25 ml conc HCl is added, heated nearly to boiling and then reduced with $SnCl_2$ solution adding dropwise with constant shaking until the yellow colour of the solution is just discharged. One drop of $SnCl_2$ is added in excess. The flask is rapidly cooled under tap to room temperature. 10ml 5% $HgCl_2$ solution is added at a time, shaken and allowed to stand for 5 minute when a slight silky white ppt. of Hg_2Cl_2 appears.This indicates the completeness of the reduction of Fe^{3+} to Fe^{2+}. The solution is diluted with 100ml of distilled water, 5ml syrupy H_3PO_4 and 4-5 drops of Ba-diphenylaminesulphonate indicator are added. It is then titrated with the standard $K_2Cr_2O_7$ solution until the colour of the solution just changes from green to reddish-violet. The titration is repeated twice.

Experimental Results:

Table1: Estimation of Fe^{2+}

No. of Titrations	Volume of Fe^{2+} and Fe^{3+} mixture taken in mL	Burette reading of $K_2Cr_2O_7$		Volume of $K_2Cr_2O_7$ solution required in mL	Mean volume of $K_2Cr_2O_7$ required in mL
		Initial	Final		

Table2: Estimation of total iron (Fe^{2+}+ Fe^{3+}) after reduction with $SnCl_2$

No. of Titrations	Volume of Fe^{2+} and Fe^{3+} mixture taken in mL	Burette reading of $K_2Cr_2O_7$		Volume of $K_2Cr_2O_7$ solution required in mL	Mean volume of $K_2Cr_2O_7$ required in mL
		Initial	Final		

Calculation:

1) Let the strength of $K_2Cr_2O_7$ solution = S (N)

2) Estimation of Fe^{2+}:

25mL mixture ≡ x mL S (N) $K_2Cr_2O_7$ solution

≡ xS mL 1(N) $K_2Cr_2O_7$ solution

We have, 1000mL 1 (N) $K_2Cr_2O_7$ solution ≡ 55.847g of Fe

xS mL 1 (N) $K_2Cr_2O_7$ solution ≡ (0.055847 × x × S)g of Fe2+/25mL mixture

≡ (0.055847 × x × S × 40)g/L of Fe^{2+}

≡ A g/L

∴ Amount of Fe^{2+} ion in the given mixture ≡ A g/L

3) Estimation of total iron (Fe^{2+}+ Fe^{3+})

25mL mixture ≡ y mL S (N) $K_2Cr_2O_7$ solution

≡ y S mL 1(N) $K_2Cr_2O_7$ solution

∴ yS mL 1 (N) $K_2Cr_2O_7$ solution ≡ (0.055847 × y × S)g of Fe^{2+}/25mL mixture

≡ (0.055847 × y × S × 40)g/L of total Fe

≡ B g/L

∴ Amount of total iron (Fe^{2+}+ Fe^{3+}) = B g/L of the mixture

∴ Amount of Fe^{3+} ion in the given mixture = (B-A) g/L

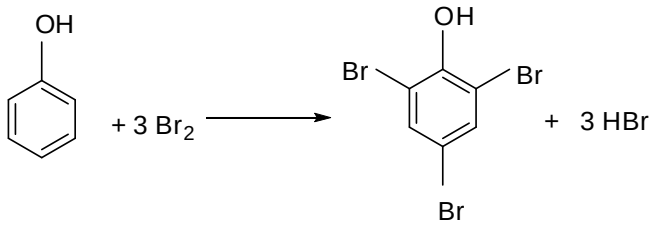
9.4. EXCERCISE 4: Estimation of Phenol by Bromination (Bromate- Bromide) method:

Principle:

Phenol can be estimated by the reaction with measured excess of standard $KBrO_3$ – KBr solution in presence of acid.

The bromine so liberated reacts quantitatively with phenol to form 2,4,6- tribromo phenol. The excess bromine is made to reacts with KI to liberate iodine which is then titrated with standard sodium thiosulphate solution using starch as indicator. The reactions are as follow –

$$KBrO_3 + 5KBr + 6\,HCl \longrightarrow 3\,Br_2 + 6\,KCl + 3\,H_2O$$



$$Br_2 + 2\,KI = I_2 + 2\,KBr$$

$$2\,Na_2S_2O_3 + I_2 = 2\,NaI + Na_2S_4O_6$$

$\therefore KBrO_3 = 3\,Br_2 = C_6H_5OH = 3\,I_2 = 6\,Na_2S_2O_3$

$\therefore$ 1 mole $Na_2S_2O_3 \equiv 3$ moles $Br_2 \equiv 3$ mole $I_2 \equiv 1$ equivalent

$\therefore$ 1000 ml (N) $Na_2S_2O_3$ solution $\equiv$ 94.112 g of phenol

In acid medium BrO_3^- react as

$$BrO_3^- + 6\,H^+ + 6\,e \rightleftharpoons Br^- + 3\,H_2O$$

$\therefore$ Equivalent weight of $KBrO_3 \equiv$ M. Wt./6 = 167/6 = 27.8333

Thus, 1000 ml (N) $KBrO_3$ solution contain 27.8333 g of $KBrO_3$

$\therefore$ 250 ml 0.1 (N) $KBrO_3$ solution contain 27.8333/40 = 0.6958 g of $KBrO_3$

Chemicals Required:

i) 0.1 (N) $KBrO_3$ – KBr solution:

ii) 10% KI solution

iii) 0.1 (N) $Na_2S_2O_3$. $5H_2O$ in 250 ml of distilled water.

iv) Starch solution

v) Phenol solution (Supplied) [Dissolve 2.5 g of Phenol in distilled water in a 250 ml volumetric flask upto the mark and supply 9 - 11 ml to each student]

Procedure:

1. **Preparation of 0.1 (N) $KBrO_3$ – KBr solution:** Dissolve 0.6958 g of $KBrO_3$ and 5 g KBr in 250 ml volumetric flask and dilute upto the mark with distilled water.
2. **Preparation of 10% KI solution:** Dissolve 10 g of KI in 100 ml of distilled water.
3. **Preparation of 0.1 (N) $Na_2S_2O_3$. $5H_2O$ solution:** Dissolve ~ 6.25 g of $Na_2S_2O_3$. $5H_2O$ in 250 ml distilled water.
4. **Peparation of phenol solution:** Diluted the supplied phenol soution with distilled water in a 100 ml volumetric flask upto the mark.
5. **Sandardisation of $Na_2S_2O_3$ solution:** Pipette out 25 ml of the $KBrO_3$ – KBr solution in 500 ml conical flask. Add 10 ml of distilled water, 10 ml conc. HCl and 15 ml of 10% KI solution and shake the mixture. Dilute the mixture with 180 ml of distilled water [keeping the acidity of the solution is about 0.5 (N)] and titrate the liberated I_2 with $Na_2S_2O_3$ solution, till pale yellow colour appears. Then add 2 ml of starch solution and continue the titration until the blue colour just disappears. Repeat the process three times.
6. **Estimation of phenol solution:** Pipette out 25 ml supplide aniline spolution in 500 ml conical flask. Add 50 ml of of $KBrO_3$ – KBr solution and 10 ml of conc. HCl. Shake the solution to mix the components intemately. Add 10 ml of 10% KI solution and 150 ml of distilled water [to keep the acidity of the solution is about 0.5 (N)] . Titrate the liberated I_2 with standard $Na_2S_2O_3$ solution, till pale yellow colour appears. Then add 2 ml of starch solution and continue the titration until the blue colour just disappears. Repeat the process three times.

Experimental Results:

Table – 1: Preparation of standard KBrO3 – KBr solution:

Initial weight of $KBrO_3$ (g)	Final weight of $KBrO_3$ (g)	Amount of $KBrO_3$ taken (g)
W_1	W_2	$W = W_1 - W_2$

Table – 2: Standardisation of $Na_2S_2O_3$ solution against standard $KBrO_3$-KBr solution:

No. of obs.	Volm. of $KBrO_3$ – KBr (ml)	Burette reading		Volm. of $Na_2S_2O_3$ soln. (ml)	Mean volm. of $Na_2S_2O_3$ soln. (ml)
		Initial	Final		
1.	25	0	...	...	
2.	25	...	...	...	V
3.	25	...	...	...	

Table – 3: Estimation of phenol solution:

No. of obs.	Volm. of Phenol solution + $KBrO_3$ – KBr (ml)	Burette reading		Volm. of $Na_2S_2O_3$ soln. (ml)	Mean volm. of $Na_2S_2O_3$ soln. (ml)
		Initial	Final		
1.	25 + 50	0	...	...	
2.	25 + 50	...	...	...	V_1
3.	25 + 50	...	...	...	

Calculation:

Strength of $KBrO_3$ – KBr solution ≡ W / 0.6958 (N/10)

Strength of $Na_2S_2O_3$ solution = S (N) , say

Applying the formula; $V_1 \times S_1 = V_2 \times S_2$ i.e., $25 \times W/0.6958\ (N/10) = V \times S_2$

∴ S_2 = (25 X W)/ (0.6958 X V x 10) (N) = S (N)

25 ml $KBrO_3$ – KBr solution ≡ V ml S (N) $Na_2S_2O_3$ solution

25 ml phenol + 50 ml $KBrO_3$ – KBr solution ≡ V_1 ml S (N) $Na_2S_2O_3$ solution

∴ 25 ml phenol solution ≡ $(2V – V_1)$ ml S (N) $Na_2S_2O_3$ solution

Since, 1000 ml of (N) $Na_2S_2O_3$ solution ≡ 93.066/6 g of Phenol

∴ $(2V – V_1)$ ml S (N) $Na_2S_2O_3$ solution ≡ 0.093066 X $(2V – V_1)$ X S /6 g of Phenol in 25 ml solution

∴ The amount of Phenol in supplied sample solution ≡ 0.093066 X $(2V – V_1)$ X S X 40 /6 g in 1000 ml

= 0.62044 X $(2V – V_1)$ X S g / lit.

Note: ***During the estimation of Aniline or Phenol the flask always be stopped after the addition of reagents to prevent the loss of bromine due to its high volatility.***

Details of such laboratory experiments and protocols are available in the Self Learning Materials (SLM) of the University and a learner may receive exposure of these laboratory procedures in PCP/ LCES programmes of the University.

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