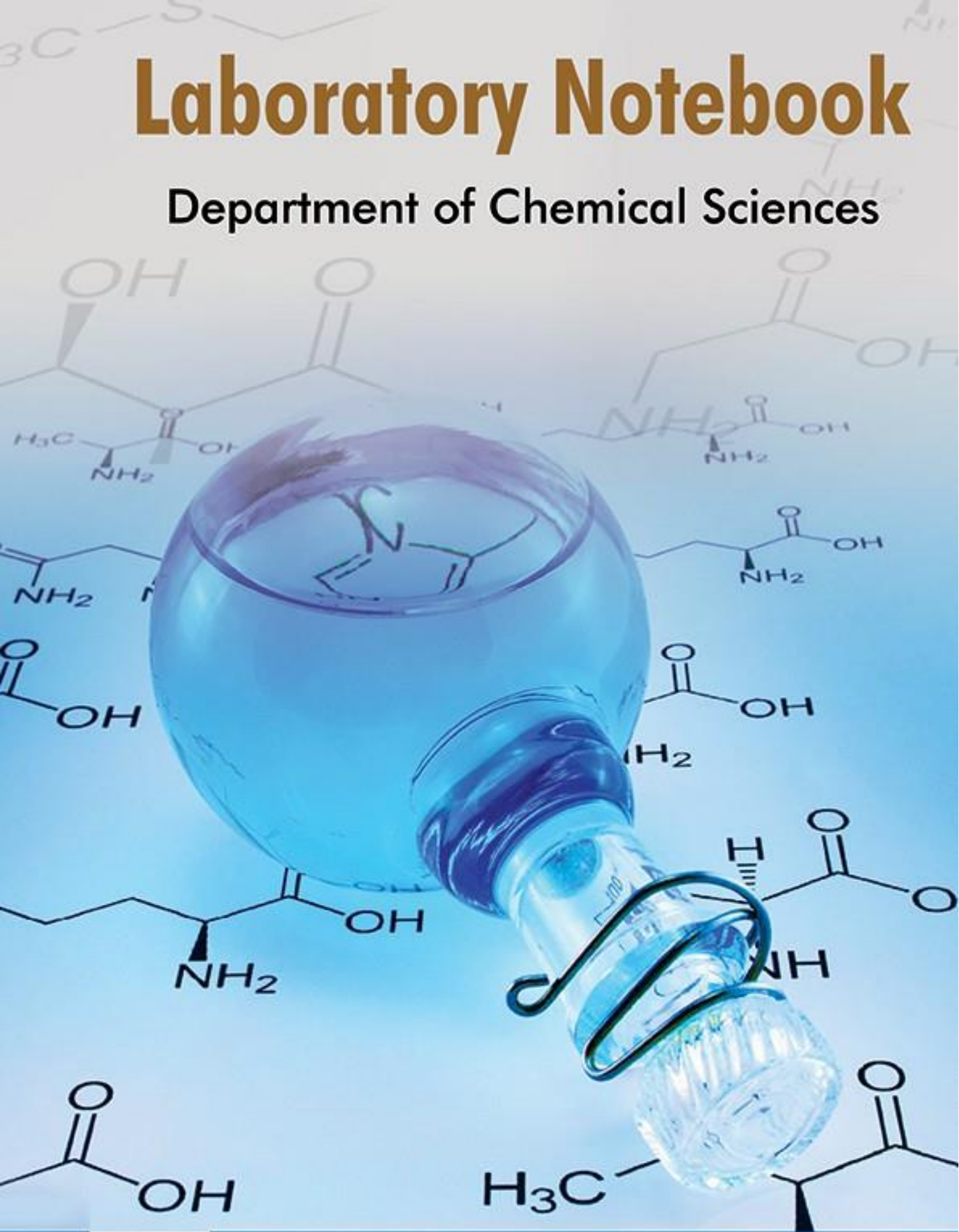
Laboratory Notebook

Department of Chemical Sciences



IISER KOLKATA

Indian Institute of Science Education and Research Kolkata
भारतीय विज्ञान शिक्षा एवं अनुसंधान संस्थान कोलकाता

Laboratory Notebook

Chemistry Lab I

Course Code:CH1202

Name:……

ID:……

Email:……

MobileNo.:……

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CONTENTS

	Experiment	Page	Instr.	Tentative date	TAs
1.	Acid Neutralization Power of Common Antacids.	6 - 13	RS	Jan 20-24	AS, AB, AnS, SSi
2.	Determination of Phosphoric Acid in Soft Drinks.	15 – 17	RS	Jan 27-31	JK, SB, SoS, RD
3.	Estimation Method for Calcium in Natural Milk using Standard EDTA Solution.	18 – 22	PD	Feb 3-07	RD, BB, AnS, JK
4.	Determination of Order for the persulphate-Iodide Reaction	23 – 28	PD	Feb 10-14	RD, BB, AnS, PH
5.	Determination of the pKIn Value of an Acid-Base Indicator by Spectrophotometric Method.	29 - 34	RV	March 31-April 4	SB, JC, SSi
6.	Molecular Modelling of Organic/Inorganic Molecules and Basics of Electronic Structure Theory.	35	SS	March 17-21	PB, AS, PC, SoS
7.	HOMO-LUMO Energy Optimization of a Few Organic/Inorganic Molecules Using Computational Calculation.	36	SS	March 24-28	PB, AS, PC, SoS
8.	Determination of Isoelectric Point of an Amino Acid	37 -43	RV	April 7-11	SB, JC, PB, SSa
9.	Replacement reaction: Synthesis of Iodoform (yellow solid) from ethanol, iodine and NaOH.	44 – 45	DH	Jan 6-10	PH, SSa, JL, AB
10.	Coupling: Coupling of benzene diazonium chloride with 2-naphthol (rose red dye).	46 - 47	DH	Jan 13-17	AB, PH, SSa, JL

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CH1202 Chemistry Lab I

Semester II, January 2024 - May 2025

Time: 2:00 p.m. – 5.00 p.m.

General Instructions

1. Attendance is mandatory. In case of illness etc. the student must contact the instructor. All labs must be completed in order to get a passing grade.
2. All data and results should be recorded directly in the lab notebook. The recording should include, title of the experiment, date of experiment, working procedure, results and calculations.
3. Instructor should review the data, before next experiment.
4. computer print outs may be directly pasted on the lab notebook.

Grading:

The marks distribution for the lab course will be as follows

1.	Lab book …….....	25
2.	Attendance ……	10
3.	Mid- SEM (quiz)…….…….….....	20
4.	End Sem Quiz …….....	25
5.	END SEM Exp.…….....	20

Mid Sem Quiz: Feb 17- 21 (any convenient day)
End Sem Quiz and Exp: April 21-25

Mandatory!!!

How to write/prepare a chemistry lab book

- Make sure you **always have your notebook when you are in lab**.
- **Write legibly using black permanent ink only. Do not use pencil.** Ball-point pens are recommended as they do not smear as easily.
- **Correct mistakes by drawing a single line ~~through~~ the error.** Do not erase, white-out, or scribble out mistakes. Do not remove pages from your notebook.
- **Include a table of contents at the front of the notebook.** Either use the inside flap or the first couple of pages to accommodate a TOC.
- **Use only the front side of each page.** Do not write on the back of each page.
- **If you are required to write what you plan to do in advance, write a summary of the procedure steps in your own words.** It is not necessary to rewrite every little detail from the lab manual.
- **Use past tense and write in 3rd person to describe what you did**. Your entries should indicate what you did in the past, not what you plan to do.
- **You can set up your lab notebook for an experiment ahead of lab, but be sure to record ALL *EXPERIMENTAL* details in the notebook DURING the time you are in lab.** Do not wait to record information after the lab.
- **Record what you actually did in lab. Do not simply copy your lab manual.** It is especially important to make note of anything you do that is different or in addition to your lab manual instructions, such as using different amounts of a substance or doing things in a different order.
- **Include all observations and measurements in your lab notebook. Always include units of measurement.** These notes should include if a substance changed colors, if a reaction produced bubbles, if a process took longer than expected, the weights/volumes of substances used, etc.
- **Be as detailed and descriptive as possible in all observations as well as titles and headings.** For example, use “Preparation of Alum" rather than "Chemical Synthesis”
- At the end of each lab, ask yourself, "Can I recreate this experiment/my results with what I have written down?"

General Page Structure:

- Top of each page
  - Date
  - Experiment Title
  - Name
  - If you are continuing from the previous page, write “continued from p. #”
- Bottom of each page
  - Page number (if the notebook is not labelled with page numbers)
  - Cross out all blank space at the bottom of the last page of each experiment; start a new page for each experiment

EXPERIMENT-1

Acid Neutralization Power of Common Antacids

Introduction: A few commercially available antacids are bases that stoichiometrically react with acid. These medicines are used to treat the symptoms of acid reflux. Aluminum and magnesium hydroxides are antacids that work quickly to lower the acid in the stomach. Liquid antacids usually work faster/better than tablets.The number of moles of acid that can be neutralized by a single tablet of a commercial antacid will be determined by back titration.

Purpose/Objective: •

- Understand standardization of acids and bases by titration and perform titration calculations.
- Reinforce the procedure of acid/base titration (using phenolphthalein)
- Introduce the concept of buffers
- Introduce the concept of back-titration
- Determine the acid neutralizing power of two commercial antacids
- Compare theoretical and experimental results.

How does the process work: Acid-base reactions and the acidity (or basicity) of solutions are extremely important in a number of differentcontexts — industrial, environmental, biological, etc. The quantitative analysis of acidic or basic solutions canbe performed by titration. In a titration, one solution of known concentration is used to determine theconcentration of another solution by monitoring their reaction. Recall that concentration is often reported in molarity, *M*.

An acid is a source of aqueous $H^+(aq)$. For example, HCl(aq) is the acid in your stomach: $HCl(aq) \rightarrow H^+(aq) + Cl^-(aq)$. In a healthy stomach, pH is regulated naturally and digestion functions properly when the pH is around 3 (recall neutral is pH = 7). Excess stomach acid can be combated with bases, or "antacids". Bases are $H^+(aq)$ acceptors; in water, they provide species that can react with $H^+(aq)$. Common ingredients in antacids are metal hydroxide and metal carbonate salts. The hydroxides provide hydroxide ion, OH^-, which can react with $H^+(aq)$ to form H_2O. Carbonates provide the carbonate ion, CO_3^{2-}, which can react with $H^+(aq)$ to form H_2O and CO_2. The reactions of interest in this lab are neutralization reactions

$$H^+(aq) + OH^-(aq) \rightarrow H_2O(l)$$

$$2\ H^+(aq) + CO_3^{2-}(aq) \rightarrow H_2O(l) + CO_2(g)$$

The active ingredients in the antacid used in this experiment are listed on the label as 110 mg of $Mg(OH)_2$ and 550 mg of $CaCO_3$. The balanced equations for the neutralization of acid with these active ingredients are:

$$Mg(OH)_2 + 2\ HCl \rightarrow Mg^{2+} + 2\ Cl^- + 2H_2O$$

$$CaCO_3 + 2\ HCl \rightarrow Ca^{2+} + 2\ Cl^- + CO_2(g) + H_2O$$

Notice the 2-to-1 mole ratio of HCl-to-base. To determine the amount of base in an actual tablet, ideally you would dissolve it in water and titrate with acid. In most titrations, solutions of the acid and the base are used.This is not an option here because $CaCO_3$ is quite insoluble in water. By the time the tablet completely dissolves, you will have added too much acid. To overcome this problem, the antacid tablet is dissolved in aknown amount of excess acid; the excess acid is neutralized with more base.

Tablet [$Mg(OH)_2$/$CaCO_3$] + HCl → neutralized tablet + excess acid → acidic solution

ExcessHCl + NaOH→ neutral solution

The excess HCl is titrated with NaOH(aq) until enough OH^- (from the NaOH solution) has been added tocompletely react with the excess H^+ (from the excess HCl in the solution). So, part of the added acid is neutralized by the antacid tablet; the remainder is neutralized by the NaOH added. This is called **back titration**.The equivalence point is when the number of moles of NaOH added equals the number of moles of HCl remaining after the reaction with the tablet. HCl is the $H^+(aq)$ source; NaOH is the $OH^-(aq)$ source. At the endpoint of the titration, the acid has been neutralized by the base.

$$V_{H^+} \times M_{H^+} = n_{H^+} = n_{OH^-} = V_{OH^-} \times M_{OH^-}$$

$$\text{or } n_{H^+} = V_{OH^-}[OH^-]$$

So:

$$\text{nHCl total} = \text{nHCl neutralized by tablet} + \text{nHCl neutralized by NaOH}$$

$$(V_{HCl} \times M_{HCl}) = (n_{HCl \text{ neutralized by tablet}}) + (V_{OH-} \times M_{OH-})$$

$$\text{or } (n_{HCl \text{ neutralized by tablet}}) = (V_{HCl} \times M_{HCl}) - (V_{OH-} \times M_{OH-})$$

One factor to consider: since the tablet contains a carbonate, the neutralization reaction produces carbon dioxide. Because CO_2 dissolves in water to produce carbonic acid, H_2CO_3, it can cause your

results to be off. You will drive off the CO_2 by heating the solution just below boiling for about 5 minutes to alleviate this problem. Another factor to consider: acidic and basic solutions are generally colorless. How can you tell when you have reached the endpoint of the titration? At the endpoint, the amounts of strong acid (e.g., H^+) and strong base (e.g., OH^-) are equal. The pH changes dramatically with addition of more acid or base. An *acid-base indicator* gives a visual indication of the acidity or basicity of a solution. The indicator is usually an organic dye that behaves as a weak acid or a weak base. The indicator's color depends on whether it is in the dissociated or undissociated form (which depends on the pH of the solution):

$$\mathbf{HIn \rightarrow H^+(aq) + In^-}$$

HIn is the undissociated form that is dominant at lower pH levels; In^- is the conjugate base (remains after dissociation) that is dominant at higher pH levels. HIn has one color and In^- another. The equilibrium constantfor this weak acid is:

$$\mathbf{K_a = [H^+][In^-]/[HIn]}$$

The pH of the solution changes by about 4 pH units around the equivalence point. This means that $[H^+]$ (and $[OH^-]$) changes by 10^4 at that point, so the ratio of the two colored forms of the indicator changes by 10^4. The solution transitions from 100 times as much HIn to 100 times as much In^- with just a few drops of titrant added. The color change occurs precisely at the end point ($nH^+ = nOH^-$). A drop or two of indicator called bromthymolblue (BTB) is all that is needed to observe the endpoint. At the endpoint, BTB changes from yellow (in acid) to a faint blue (in base). The appearance of the faint blue marks the endpoint of the titration

Materials and Apparatus:.

- Common antacid.
- Standardized HCl solution
- Standardized NaOH solution
- Bromomethyl blue (BTB)
- Burette, Pipette, Beaker, Conical.

Procedure

Follow the procedure outlined for buret usage. Be sure your buret is clean and the stopcocks are firmly seated.

- Put some water in the buret and practice controlling the stopcock. **Do not fill burets on the work-bench.Always keep all chemicals below eye level.** This decreases the chance of getting chemicals in your eye in the event of a spill.
- If you have air bubbles in the buret, gently knock the bottom of the buret to free them so they can rise to the surface.
- You will determine the volume of titrant delivered by *subtracting* the initial buret reading from the final (volume by difference).
- Mount the buret on the stand. In real titrations, you would put a white towel or piece of paper over the dark base of the ring stand so the color change of the indicator will be easy to see. Since this is a practice, your titrant is water. You're just practicing the stopcock control and volume reading. The goal is to get a feel for the buret.
- Practice reading the volume (liquid level at the bottom of the meniscus). Take readings to 0.01 or 0.02 mL.
- Record the initial volume of water. Add water to a collection flask and read the new volume. Find the volume of water added by difference.
- Practice by delivering a milliliter, a few drops, and one drop.
- Set up a 50-mL buret with the stock NaOH. It may help you to start with Part 3 because it takes some time for the solution to heat up and cool.

Part 1: Standardization of NaOH (*if necessary*)

Determine the concentration of the base, NaOH, by titrating a known mass of potassium hydrogen phthalate (KHP), to neutral (the equivalence point). The molar mass of KHP is 204.23 g/mol, and it has one acidic hydrogen per molecule.

Precisely weigh out approximately 1.000 g KHP. About 10 mL of NaOH should be used in the titrations. The NaOH solution's concentration is about 0.5 *M*. Put the KHP into 50–100 mL water in a 250-mL titrating flask. It does not need to dissolve completely, and you don't need to know how much water is in the flask. The KHP is functioning as a strong acid and will dissolve as it is titrated. You can warm the water to aid the dissolution if needed.

Use a few drops of BTB as indicator in the titration flask. Record the initial volume of NaOH from the buret and then begin the titration. As you turn the stopcock, push it into the barrel so it doesn't loosen and leak.Record the color change at the end point and the final volume on the buret. The volume of NaOH used = $V_{final} - V_{initial}$. Perform three titrations with the NaOH to obtain reproducible results.

Part 2: Standardization of HCl (*if necessary*)

To determine the precise molarity of the HCl solution, titrate it with the NaOH to the endpoint; use BTB as the indicator unless instructed otherwise.Use a volumetric pipet to transfer exactly 10 mL of stock HCl into a 125 mL Erlenmeyer flask.Record the initial volume of NaOH and titrate the HCl.

Record the color change at the end point and the final volume of NaOH. The volume of NaOH used = $V_{final} - V_{initial}$. Repeat to be sure you can get reproducible results.

STOP — if you were not instructed to do parts 1 and 2, record the molarities of the HCl and the NaOH in yournotebook. The molarities values listed on the bottles are to the ten-thousandth place (four decimal places).

Part 3: Determination of the Amount of Acid Neutralized by an Antacid Tablet

You will first react the antacid tablet with a known amount (volume) of the standardized HCl. Then you will titrate the remaining HCl with the standardized NaOH to determine the amount of acid that was not consumed by the antacid tablet. Please make sure that you have recorded the molarities of the NaOH and HCl (on the reagent bottles to four decimal places).

Rinse all the glassware you will be using. You must have data for at least four good trials. Please make sure you record the mass of four antacid tablets to the nearest 0.01 g (pan balance). Each tablet will weigh a different amount, so keep track of which tablet is in which flask. Label four 125 mL Erlenmeyer flasks. To each flask add about 25 mL of distilled water.Using a volumetric pipet, *accurately* add 25 mL of HCl and an antacid tablet. Make sure to record the molarity from the bottle if you did not standardize it. The 25-mL volumetric pipet has an uncertainity of ±0.03 mL. Heat gently to a near boil for about 5 minutes, carefully avoiding splattering. Be sure that the tablets are completely dissolved before titrating the solutions.Allow the solutions to cool (to touch).Add a few drops of BTB indicator. Record the molarity of the NaOH (if you did not standardize it). The first titration may be a trial to learn approximately what volume of NaOH is needed to reach the endpoint and to become familiar with the color change at the endpoint.

Record the initial volume of NaOH to 0.01 mL.Add NaOH in about 1 mL portions while swirling the solution. Stop between additions to swirl for a moment and observe the color. When you begin to see temporary faint color changes, add the NaOH in 0.5-mL increments. Near the endpoint, add the NaOH dropwise.

Record the final volume on the buret to 0.05 mL when you reach the endpoint. Save the solution in the flask as a reminder of the final color. The volume of NaOH required is $V_{final} - V_{initial}$; report the volume needed to 0.05mL. Accurately titrate the three remaining samples.

Dispose of your waste solutions in the waste containers in the back hood. Clean your bench top and rinse your glassware. Return any equipment that you borrowed (clean).

Calculate the number of moles of HCl, *n*H+, to four sigificant figures using the volume and molarity of the HCl solution. This is the total amount of acid requiring neutralization (by the tablet and the NaOH).

Calculate the number of moles of NaOH titrant that you added to four significant figures using molarity and volume. This is the number of moles of HCl neutralized by the NaOH.

Determine the number of moles of HCl not neutralized by the NaOH to four significant figures. This is the number of moles of HCl neutralized by the antacid. *n*acid neutralized by tablet = *n*acid initially in flask –*n*acid neutralized by NaOH. Find the average number of moles of HCl neutralized by the tablet and standard deviation.

Compare the average with the amount theoretically expected based on the label. Express this comparison as the % ratio of the actual amount of acid that a tablet neutralizes to the theoretical amount that it should neutralize (to three significant figures).

% = 100% × (*n* acid actually neutralized) / (*n* acid theoretically neutralized)

This could be less than 100% if the tablet does neutralize as much as expected or more than 100% if it exceeds what is claimed on the label.Use the average moles of HCl neutralized by the tablets and the average mass of the tablets to determine the moles of acid neutralized per gram of tablet (to three significant figures). This is a more universal neutralization expression (it is independent on the mass of the tablet).

Reporting Results:

Preparation of exactly 0.5(M) KHP solution

Weight taken:

Concentration of KHP solution:

Preparation of 0.5(M) NaOH solution

Preparation of 0.5(M) HCl solution

Standardization of NaOH with standard KHP

No.	Vol. of KHP	Vol. of NaOH
1.		
2.		
3.		

Concentration of NaOH

Standardization of HCl with previously standardized NaOH solution

No.	Vol of NaOH	Vol. of HCl
1.		
2.		
3.		

Concentration of HCl

Calculation of Antacid concentration by back titration

No.	Vol. of Antacid	Vol. of HCl	Vol. of NaOH
1.			
2.			
3.			

Amount of acid neutralizing by the antacid

Questions?

1. What you did, how you did it and what you determined?
2. What were possible experimental reasons for error (deviations from expected values)?
3. How consistent were your tablets in the amount of antacid they contained?
4. What do you know about primary and secondary standard?

Student'sComments

EXPERIMENT-2

Determination of phosphoric acid in soft drinks

Introduction:
Phosphoric acid is one of several weak acids that is present in carbonated beverages. It is a component of all cola soft drinks. Phosphoric acid has a much higher concentration than other acids present in a soft drink, so its concentration can be determined by a simple acid-base titration.

In this experiment, you will use a pH Sensor to monitor pH as you titrate. The region of most rapid pH change will then be used to determine the equivalence point. The volume of NaOH titrant required to reach the equivalence point will be used to determine the molarity of the H_3PO_4.

PURPOSE

The purpose of this experiment is to determine the amount of phosphoric acid,H_3PO_4, in a variety of soft drinks by titrating each sample with sodium hydroxide, NaOH.

How does the process work?

In this experiment, you will titrate a sample of a cola soft drink with a sodium hydroxide solution and determine the concentration of phosphoric acid, H_3PO_4. Hydrogen ions from the first dissociation of phosphoric acid react with hydroxide ions from the NaOH in a one-to-one ratio in the overall reaction:

$$\mathbf{H_3PO_4\,(aq) + OH^-(aq) \rightarrow H_2O\,(l) + H_2PO_4^-(aq)}$$

Chemicals

- Various cola soft drinks, decarbonated
- 0.050 M NaOH
- Deionized water
- Oxalic acid dihydrate

Apparatus

- 50-mL buret
- 100-mL graduated cylinder
- 250-mL beaker
- magnetic stirrer (if available)
- stirring bar (or stirring rod)
- pH Meter

Standardization of NaOH

Procedure:

1. Prepare 100 mL of 0.05N oxalic acid solution in a volumetric flask.
2. Pipette out 10 mL of 0.05N oxalic acid in a 250mL conical flask.
3. Add 2 – 3 drops of phenolphthalein indicator to the solution and titrate against the 0.05N NaOH solution.

Estimation of Phosphoric acid

Procedure:

1. Use a graduated cylinder to measure out 40 mL of a decarbonated cola soft drink and 60 mL of distilled water into a 250-mL beaker

2. Place the beaker on a magnetic stirrer and add a stirring bar and start the magnetic stirrer. If no magnetic stirrer is available, you need to stir with a stirring rod during the titration.

3. Immerse the tip of the pH electrode in the beverage solution and adjust its position so that it is not struck by the stirring bar.

4. Obtain a 50-mL buret and rinse the buret with a few mL of the 0.050 M NaOH solution. Dispose of the rinse solution as directed by your teacher. Fill the buret a little above the 0.00-mL level of the buret with 0.050 M NaOH solution. Drain a small amount of NaOH solution so it fills the buret tip *and* leaves the NaOH at the 0.00-mL level of the buret. Record the precise concentration of the NaOH solution in your data table.

5. You are now ready to perform the titration..

6. Before you have added any NaOH solution, Note 0 as the buret volume, in mL. Check the pH displayed and write it down. This is the first data pair for this experiment.

7. Add 0.5 mL of NaOH solution. When the pH stabilizes, note it down and enter the current buret reading. You have now the second data pair for the experiment.

8. Continue to add 0.5-mL increments, entering the buret level after each increment. When the pH has leveled off (near pH 10), STOP the data collection.

9. Examine the data to find the *equivalence point*—that is, the 0.5-mL volume increment that resulted in the largest increase in pH. Plot a graph of pH *vs*. Volume using MS-Excel or Origin etc. In the graph of pH *vs*. Volume go to the region of the graph with the large increase in pH. Find the NaOH volume (in mL) just *before* this jump. Record this value in the data table. Then record the NaOH volume *after* the 0.5-mL addition producing the largest pH increase.

10. Print a copy of the graph of pH *vs*. volume.

11. Dispose of the beaker contents as directed by your teacher. Rinse the pH electrode and return it to the storage solution.

Result:

Preparation of exactly 0.05(N) Oxalic acid solution

Weight taken:

Concentration of Oxalic acid solution:

Standardization of NaOH by OxalicAcid

S. No.	Vol. of Oxalic acid	Vol. of NaOH
1		
2		
3		

Use exact concentration of NaOH solution for the titration.

Vol. of NaOH	pH

Questions?

1. How many protons are produced from the? How does is deprotonate?
2. Why$H_3PO_4$is added in cold-drinks?
3. Why cold drinks are carbonated? How does this carbonate affect us?
4. What is the important of stirring during titration?
5. What is the pH of the cola drinks?

EXPERIMENT-3

Estimation Method for Calcium in Natural Milk Using Standard EDTA Solution

Introduction

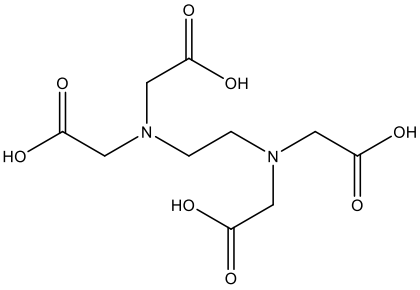
Metal ions form complexes. The formation of such complexes can serve as the basis of accurate and convenient titrations for such metal ions. Such determinations are referred to as complexometric titrations. The accuracy of these titrations is high and they offer the possibility of determination of metal ions at concentrations at the millimolar(mM) level. Many cations will form complexes in solution with a variety of substances that have a pair of unshared electrons (e.g. on N, O, S atoms in the molecule) capable of satisfying the coordination number of the metal. The metal ions act as Lewis acid (electron pair acceptor) and the complexing agent is the Lewis base (electron pair donor). The number of molecules of the complexing agent, called the ligand, will depend on the coordination number of the metal and on the number of donor atoms on the ligand molecule.

Simple complexing agents such as ammonia are rarely used as titrating agents because a sharp end point corresponding to a stoichiometric complex is generally difficult to achieve. This is true since the stepwise formation constants are frequently close together and not very large, and a single stoichiometric complex cannot be observed. Certain ligands that have two or more complexing groups on the molecule, however, do form well-defined complexes and can be used as titrating agents. One such reagent that is widely used is ethylenediaminetetraacetic acid (EDTA).

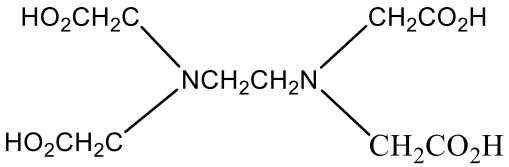
Purpose:

1. The hardness of the water sample due to $CaCO_3$(upto ppm level).
2. % of calcium in milk
3. % of calcium in egg shell
4. % Ca in solid samples

The classic method of determining calcium and other suitable cations is titration with a standardized solution of ethylenediaminetetraacetic acid (EDTA). EDTA has the structure shown below. Instead of repeatedly drawing this structure or writing out the chemical formula, the EDTA molecule is represented as "H_4Y".Each acid hydrogen on EDTA can be removed, producing H_3Y^{-1}, H_2Y^{-2}, HY^{-3}, and Y^{-4} ions. The disodium dihydrate of EDTA, $Na_2H_2Y.2H_2O$ is commonly used to prepare standard EDTA solutions. EDTA is readily available from many commercial sources, and often in such a high purity that solutions need not be standardized for routine work.



Primary standard zinc acetate dihydrate can be used to standardize EDTA solutions.Of the various EDTA species, only the Y^{4-} ion (the completely deprotonated anion of EDTA) forms a 1:1 complex with metal ions. To increase the fraction of Y^{4-}, the pH needs to be increased to 10 in this experiment.

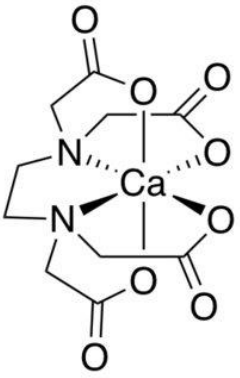


H_6Y^{2+}

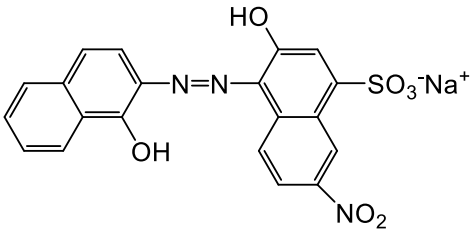
$pK_1 = 0.0\ (CO_2H)$ $pK_4 = 2.69\ (CO_2H)$

$pK_2 = 1.5\ (CO_2H)$ $pK_5 = 6.13\ (NH^+)$

$pK_3 = 2.00\ (CO_2H)$ $pK_6 = 10.37(NH^+)$



The endpoint of an EDTA titration is determined with a *metallochromic* indicator. These indicators are complexing agents that change color when combined with metal ions. A variety of indicators can be used for EDTA titrations. In this experiment, we will use Eriochrome black T (EBT) indicator, having the structure shown below.



Eriochrome black T

This indicator (shown as H_2In^- in the equations below) changes from blue to red when combined with a metal ion, forming a complex ion:

$$M^{2+} + H_2In^- + 2H_2O \leftrightarrow MIn^- + 2H_3O^+$$

blue red

EDTA is a stronger complexing agent than the indicator, and displaces the indicator from the metal ion allowing the indicator to return (through shades of

violet) to a pure blue color, indicating the end of the reaction.

$$MIn^{-} + Y^{4-} \leftrightarrow MY^{2-} + H_2In^{3-}$$

red blue

Calcium ion (Ca^{2+}) does not form a stable red complex with the EBT indicator; therefore the direct titration of Ca^{2+} by EDTA may not cause a sharp color change of EBT indicator at the end point. The magnesium complex with EBT is stable and the formation constant (K_f) of Mg^{2+} with EDTA is lower than the formation constant (K_f) of Ca^{2+} with EDTA. Thus, a displacement titration of Ca^{2+} by the mixture of $Mg^{2+}$and EDTA will help to determine the end point with the following mechanism:

$$CaIn^{3-} + MgY^{2-} \leftrightarrow CaY^{2-} + MgIn^{-}$$

To accomplish this displacement titration, a small amount of Mg^{2+} will be mixed with the EDTA solution. The EDTA-Mg^{2+} mixture will titrate the unknown Ca^{2+} solution. At the end point, Mg^{2+} will be released from the EBT indicator and complexed with EDTA, causing the color change from red to blue.

Chemical required:

EDTA, $Zn(OOCCH_3)_2.2H_2O$, NH_4Cl, *NH_3,* Buffer, EBT indicator

Apparatus required:

Conical, Pipette, volumetric flask (250ml), volumetric flask (100ml), pipette, measuring cylinder.

1. Standardization of EDTA solution:

Since EDTA is not primary standard substance. Its aqueous solution is required to be standardized against a primary standard solution. The most widely used primary standard substance for EDTA complexometric is zinc acetate dihydrate, $Zn(OOCCH_3)_2.2H_2O$.

(a) Standard (0.02M) zinc acetate solution (100ml): (Need to prepare)

Transfer ~2g of NH_4Cl into 100ml volumetric flask and dissolve the salt in 10 ml distilled water. Weigh out accurately 440 mg of zinc acetate dihydrate, $Zn(OOCCH_3)_2.2H_2O$ (F.W. 219.38) and transfer the same quantitatively into volumetric flask. shake to dissolve the salt, adding more distilled water as required. Dilute to mark and mix intimately.

(b) (0.02M) ETDA solution: (A solution of ~0.02 M EDTA will be provided)

(c) NH_4Cl-NH_3 buffer (pH 10.5): (A buffer solution will be provided)

Procedure:

Take 25ml of the standard (0.02M) zinc acetate solution into a 250 ml conical flask, dilute with 20ml of distilled water and add 5ml of NH_4Cl-NH_3 buffer solution (pH 10.5), 5 drops of of EBT indicator solution (will be provided) and homogenize it, the solution assumes a wine red colour. Titrate with EDTA solution, while swirling the flask gently, until the wine red colour changes to blue. Record the titre of the EDTA solution and calculate its strength.

2. Estimation of calcium

(d) Natural milk solution will be provided:

Transfer 25 mL of the given milk solution to 250 mL conical flask. Add 20 mL extra distilled water and 5 mL of buffer followed by 5 drops of EBT indicator. Stir the solution well to homogenize it and titrate it. While swirling the flask gently titrate it against standard EDTA solution, until the wine-red color changes to blue. Record the titer of the EDTA solution

Experimental Write-Up & Calculation

Strength(S) of zinc acetate solution, S = (w/1.0969). (M/50)

S. No.	Volume of Zinc Acetate Solution	Required volume of EDTA	Average required volume of EDTA(V1) ml
1	25		
2	25		
3	25		

S. No.	Volume of Milk Solution	Required volume of EDTA	Average required volume of EDTA (V3) ml
1	25		
2	25		
3	25		

Suppose, 25ml of Ca^{2+} solution ≡ (V_3-V_2) ml of the (S_1) EDTA solution

Strength of Ca^{2+} solution = $[(V_3-V_2)/25] \times [(25 \times S)/V_1]$

Compare the result with the strength of the prepared Ca^{2+} solution.

Strength of Ca^{2+} solution in grams per liter:

Questions

1. **What is the indicator used in this titration?**

Ans: Eriochrome Black T

2. **Why can Eriochrome Black T not be used directly as an indicator?**

Ans: Because Ca-Eriochrome Black T complex is not stable. So, [Mg(EDTA)]-EBT mixture is used as a indicator. (also see the introduction part)

3. **What is the color of the doubly ionized Eriochrome Black T indicator in slightly basic solution?**

Ans: Blue

4. **Why zinc acetate solution is prepared in 2% NH_4Cl solution?**

Ans: Acetate ion, being the conjugate base of a weak acid, is a strong base and render the solution sufficiently alkaline to cause precipitation of zinc hydroxide in water.

$$CH_3COO^- + H_2O \rightarrow CH_3COOH + OH^-$$

$$Zn^{2+} + 2OH^- \rightarrow Zn(OH)_2$$

To prevent the precipitation of zinc hydroxide, zinc acetate is dissolved in 2%NH4Cl solution, which acts as a buffer solution of pH (4.5-5), at which zinc hydroxide does not precipitate.

5. **Is it possible to use the sodium salt of EDTA as a primary standard?**

Ans: No, it is a secondary standard solution**.**

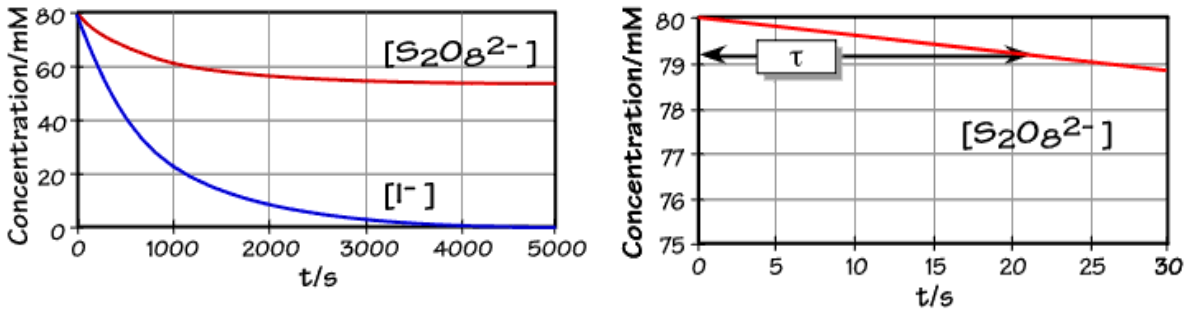
6. **At what pH is the Ca titration carried out? Why?**

Ans: Around pH10**.**

Student's Comments

EXPERIMENT 4

Determination of Order for the Persulphate-Iodide Reaction



PRINCIPLE:

The rate law for the reduction reaction of persulphate by iodide:

$S_2O_8^{2-}(aq) + 2\ I^-(aq) \longrightarrow I_2(aq) + 2\ SO_4^{2-}(aq)$ will be determined. The orders of reaction with respect to persulphate and to iodide will be experimentally obtained by measuring rates of the reaction for various concentrations of the reactants.

INTRODUCTION:

The overall oxidation of iodide ion by persulphate can be expressed as

$$2I^- + S_2O_8^{2-} \rightarrow 2SO_4^{2-} + I_2$$

Or more explicitly

$$S_2O_8^{2-} + I^- \rightarrow SO_4^{2-} + SO_4^- + I^0$$

$$SO_4^- + I^- \rightarrow SO_4^{2-} + I^0$$

$$I^0 + I^0 \rightarrow I_2$$

The rate of the reaction is followed by estimating the iodide formed at different time intervals, by titrating with sodium thiosulphate using starch as indicator. The volume of thiosulphate is plotted as a function of time. The initial slope of this plot gives the initial rate of the reaction. The values of initial rates obtained can be used to calculate the total order and individual orders with respect to iodide as well as persulphate ion.

APPARATUS: Burette 50 cm^3, pipettes 5, 25, and 50 cm^3, stoppered bottles, 250 cm^3 conical flasks, 250 cm^3 standard flasks, porcelain trough, porcelain tiles.

CHEMICALS: Potassium iodide solution (0.1 N); Potassium persulphate solution (0.1 N); Acetic acid (1.0 N); Sodium thiosulphate solution (0.01 N); Starch indicator, ice cold water.

EXPERIMENTAL PROCEDURE:

1) Prepare different reaction mixtures using the volumes given in the Table 1. For example, mix 1 mL of 0.1 N acetic acid and 20 mL of 0.1 N potassium iodide in a stoppered bottle.
2) Add the required amount of water (in this case 9 mL) so that the final volume is 40 mL.
3) Add 10 mL of 0.1 N potassium persulphate solution in the stoppered bottle and start the stopwatch when half the volume of persulphate is added.
4) Stir the reaction mixture and pipette out 5 mL of it into a conical flask containing ice cold water to quench the reaction and titrate the liberated iodide against thiosulphate solution using starch as indicator.
5) Repeat this every 5 minute for at least 40 minutes.
6) Carry out similar titrations with the other solution mixtures.

RESULTS:

Table 1. Typical reaction mixtures for determining the order of the reaction between iodide and persulphate ions.

Bottle no.	Volume of acetic acid(.1 N) (mL)	Volume of 0.1 N KI solution (mL)	Volume of 0.1 N $K_2S_2O_8$ solution (mL)	Volume of water in (mL)
1	1	20	10	9
2	1	10	5	24
3	1	20	5	14
4	1	10	10	19

Treatment of Data: Record all your observations systematically as follows:

- Construct separate tables for each reaction mixture as given in Table 2.
- Plot the titre value as a function of time and evaluate the initial slopes (this can be done either numerically, or graphically or by both the processes).
- The slopes obtained at the initial periods of the reaction can be taken to be initial rates. (Why are we interested in the initial rates and not the values of rates at any given time of the reaction?)

Table 2. Volume of thiosulphate consumed for a known aliquot of the reaction mixture at various time intervals.

Normality of thiosulphate solution taken =.....N

Bottle no.1

Time in minutes	Burette readings		Volume of thiosulphate(mL)
	Initial	Final	
0			
5			
10			
15			
20			
25			
30			
35			
40			

Bottle no.2

Time in minutes	Burette readings		Volume of thiosulphate(mL)
	Initial	Final	
0			
5			
10			
15			
20			
25			
30			
35			
40			

Bottle no.3

Time in minutes	Burette readings		Volume of thiosulphate(cm^3)
	Initial	Final	
0			
5			
10			
15			
20			
25			
30			
35			
40			

Bottle no. 4

Time in minutes	Burette readings		Volume of thiosulphate(cm^3)
	Initial	Final	
0			
5			
10			
15			
20			
25			
30			
35			
40			

Normally in a chemical reaction the rate of the reaction is proportional to the concentration of the reactants raised to the power m, where m is the order of the reaction with respect to the reactant. For the reaction between iodide and persulphate ions the rate expression can be written as

$$\text{Rate} \; \alpha \; [I^-]^m [S_2O_8^{2-}]^n = k[I^-]^m [S_2O_8^{2-}]^n$$

The ratio of the initial rate values obtained for bottles 1 and 4 can be written as

$$\text{rate1/rate4} = k[100]^m [50]^n / k[50]^m[50]^n$$

(Here the volumes taken are assumed to be proportional to concentration since the total volume of the reaction mixtures is kept constant.). Therefore,

$$\text{rate1/rate4} = [2]^m \quad ; \quad \text{Or, } \log \{\text{rate1/rate4}\} = m\log 2$$

So the value of m, the order with respect to iodide can be found out. Similarly the ratio of the rates for bottles 1 and 3 can be written as

$$\{rate1/rate3\} = k[100]^m[50]^n/k[100]^m[25]^n$$

$$\text{Or } \log \{rate1/rate3\} = n\log 2$$

So the value of n, the order with respect to persulphate can be calculated. The overall order of the reaction = m+n.

Also, the ratio $rate1/rate2 = k[100]^m[50]^n/k[50]^m[25]^n$

Or, $\log \{rate1/rate2\} = (m+n) \log 2$

Hence, the overall order of the reaction (m+n) can be calculated.

RESULTS:

Report the individual orders and overall order of the reaction. Comment how the observed reaction orders accounts for the mechanism of the reaction.

Note:

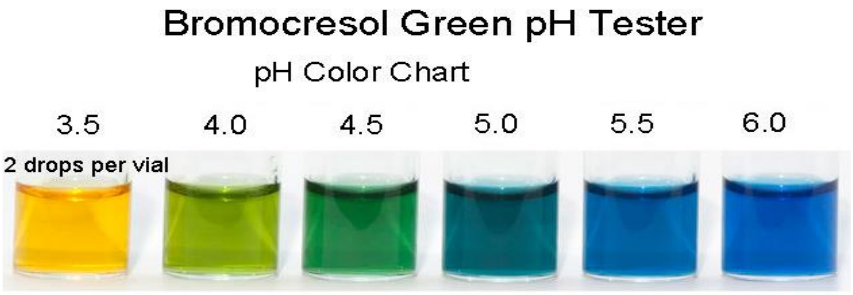
The reaction is also believed to occur in steps:

$$I^- + S_2O_8^{2-} \xrightarrow{\text{slow}} (S_2O_8I)^{3-}$$
$$(S_2O_8I)^{3-} \xrightarrow{\text{fast}} 2SO_4^{2-} + I_2$$

Suggest any alternative way of studying the kinetics of the reaction between iodide and persulphate ions.

EXPERIMENT-5

DETERMINATION OF THE pK_{In} VALUE OF AN ACID-BASE INDICATOR BY SPECTROPHOTOMETRIC METHOD

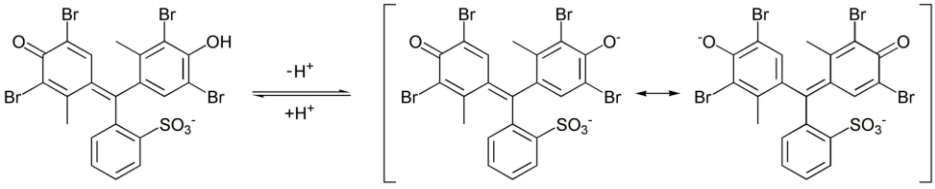


PRINCIPLE:

Spectrophotometric methods will be used to determine the acid dissociation constant of an acid-base indicator (Bromocresol green), the light absorption characteristics of its acid and base form. This experiment will provide you with opportunities to refine your understanding of absorption process while providing an opportunity to apply many aspects of acid-base chemistry.

INTRODUCTION:

Acid–base indicators are weak acids or bases having distinctly different colours in acidic and alkaline solution, and by virtue of change of colour they indicate the end points of acid-base titrations. To illustrate this point, consider the case for Bromocresol green (an organic acid):



As shown above, this proton can be donated/or received to water to obtain a hydronium ion. If we represent the acidic form of the bromocresol green as (HIn) and the conjugate base as (In-) then the dissociation reaction looks like:

$$HIn \leftrightarrow \quad H^+ \quad + \quad In^- \quad \Longrightarrow \quad H_3O^+ \quad + \quad In^-$$

The acid dissociation constant (Equilibrium constant) can be represented as:

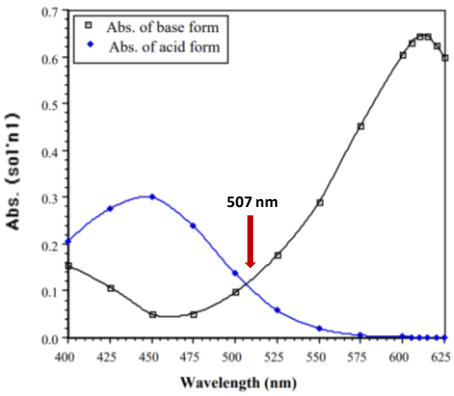
$$K_{In} = \frac{[H^+][In^-]}{[HIn]}$$

The strategy of this experiment is to adjust [H_3O+] to known values using a buffer and then to measure the ratio [In-]/[HIn] spectrophotometrically. Knowing this ratio and the value of [H_3O^+/pH] will allow us to calculate Ka using the above equation.

- The trick then is knowing how to determine the ratio [In-]/[HIn] using light absorption measurements .

ABSORPTION SPECTRAL CHARACTERISTICS OF BROMOCRESOL GREEN:

The acid form of bromocresol green (HIn) absorbs light in a different region of the spectrum than the basic form of bromocresol green (In-). From the figure below, you may see that the two species have distinctly different values for λmax. Acid-base indicators are useful for determining pH and indicating end-points because they change color as the pH of the solution changes. It is important to note that the two solutions used to measure these absorption spectra have the same concentration of the bromocresol green indicator. We want to select a wavelength that will allow us to determine the relative concentration of each species present. One very poor choice occurs at about 507 nm, because at this wavelength both the species absorbs equally well. The best choice is the wavelength which has the largest difference in absorbance for the two species. This may be at > 565 nm where the acid form hardly absorbs light.



THEORY: The ionization equilibria of a weak acid indicator (HIn) may be represented according to,

$$\underset{\textit{Acidic for}}{Hin} \leftrightarrow \underset{\textit{Alkaline form}}{H^+ + In^-} \quad \ldots\ldots\ldots\ldots (1)$$

for which the ionization constant (K_{In}) in dilute solution may be defined as the concentration quotient (2)

$$K_{In} = \frac{[H^+][In^-]}{[HIn]} \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

where, [ ]'s represent the molar concentrations of the respective species. Transforming the equation (2) in logarithmic form one obtains,

$$pH = pK_{In} + \log \frac{[In^-]}{[HIn]} \quad \ldots\ldots\ldots\ldots (3)$$

(where, $pK_{In} = -\log_{10} K_{In}$ and $pH = -\log_{10} [H^+]$ in dilute solution).

Thus, if a fixed amount of the indicator is placed in the same volume of a series of buffer solutions of different known pH values, the ratio, $[In^-]/[H_{In}]$, will increase with increase of pH. If the values of the ratio at different pH are determined by measuring the colour intensity of the indicator solutions then the pK_{In} value of the indicator can be found out if the pH of the buffer solutions is known.

If the alkaline form of the indicator (In^-) absorbs at a selected wavelength and Beer's law is obeyed in the range of concentration of the indicator used, then the absorbance (A) of the indicator solution at a particular pH will be proportional to its concentration, provided the acid form (H_{In}) does not absorb at this wavelength.

$$A = \varepsilon [In^-] \, l \qquad (4)$$

In a strongly alkaline solution, H_{In} is practically absent, and the absorbance (A) will correspond to the total concentration (T_{In}) of the indicator.

$$A' = \varepsilon [T_{In}] \, l \qquad (5)$$

Where, ε = molar extinction coefficient of In^- and l = optical path length in cm.

Mass balance equation of the indicator is,

$$T_{In} = [H_{In}] + [In^-] \qquad (6)$$

$$\therefore \quad [H_{In}] = T_{In} - [In^-] \qquad (7)$$

From (5) – (4) one obtains, $\dfrac{(A' - A)}{\varepsilon \, l} = [H_{In}]$ (8)

$$\frac{(A)}{\varepsilon \, l} = [In^-] \qquad (9)$$

Substituting these values of HIn and In^- in equation (3) one obtains,

$$pH = pK_{In} + \log_{10}\left(\frac{A}{A' - A}\right) \qquad (10)$$

A and A′ may be measured colourimetrically. Therefore, by plotting $\log_{10}$ [A/(A′ - A)] against pH of the buffer solutions a straight line of slope =1 will be obtained, of which the intercept on the pH axis will give pK_{In}.

APPARATUS: Burette 50mL, 10 mL pipette, 50 mL volumetric flask, Beaker, test tubes.

CHEMICALS: Oxalic acid 0.5N, Sodium hydroxide ~0.5N, acetic acid ~0.5N, bromocresol green, phenolphthalien indicator.

EXPERIMENTAL PROCEDURE:

You will be provided with ~0.5 N NaOH and~0.5 N acetic acid.

1. Prepare 50mL of 0.5 N oxalic acid in a volumetric flask.

2. Standardisation of NaOH (~ 0.5 N) using oxalic acid. Then standardise the acetic acid.
3. Prepare 100 mL of exact 0.4 N acetic acid (pK^H= 4.74 at 25°C) and 50 mL of exact 0.4 N NaOH solutions separately by usual procedure.
4. Take 6 hard glass test tubes of uniform dimensions and label them from 1 to 6. Prepare the following series of solutions by proper mixing (experimental pH values may be obtained from chart below, or, may be determined using a pH meter).

Test tube	Vol. of 0.4 N acetic acid (mL)	Vol. of 0.4 N NaOH (mL)	Volume of Water (mL)	pH (Expt.)	A	A/(A'-A)
1	5.0	0.5	4.5	3.72		
2	5.0	1.5	3.5	4.27		
3	5.0	2.5	2.5	4.63		
4	5.0	3.5	1.5	4.99		
5	5.0	4.5	0.5	5.57		
6	0	2.5	7.5		A' =	

5. Add a few drops of bromocresol green indicator to test tube number 6 using a dropper.
6. Set spectrophotometer at 570 nm, adjust the transmittance of water to 100%.
7. Measure the transmittance of the solution in test tube 6. If the transmittance is below 15% (i.e. Absorbance is above 0.82), take test tube 7 and add fewer number drops of the indicator to it and measure the transmittance. In this way by adjusting the number of drops of the indicator, adjust the transmittance of the alkaline form between 25 to 15 % (absorbance is above 0.60 but below 0.82) using test tube numbers 6 to 8 as required.
8. Add the same number of drops of the indicator as adjusted in step 5 to each of test tubes 1 – 5 and measure their transmittance.
9. Calculate the absorbance (A) values of solutions 1 - 5 and the absorbance (A′) of the alkaline solution of the indicator (6, 7 or 8) using the relation:

   $A = \log (100/T\ \%) = 2 - \log T$
10. Plot pH against $\log_{10}$ [A/(A′ - A)] and draw the best straight line of unit slope passing through the experimental points, using the same scale for pH and $\log_{10}$ [A/(A′ - A)] axis. Find pK_{In} from the intercept on the pH axis.

Table 1: Standardisation of NaOH

Sl. No	Vol. of Oxalic acid (mL)	Burette reading			Avg. Vol(mL)	Strength of NaOH
		Initial	Final	Diff.		

Table 2: Standardisation of Acetic Acid

Sl. No	Vol. of Acetic acid (mL)	Burette reading			Avg. Vol(mL)	Strength of Acetic Acid
		Initial	Final	Diff.		

CONCLUSION: pK_{In} of bromocresol green is .....

EXPERIMENT-6

Molecular Modelling of Organic/Inorganic Molecules and Basics of Electronic Structure Theory.

This experiment will give students an idea of drawing and visualizing molecules and show how to obtain optimized ground state structures of these molecules. A very basic theoretical knowledge will be provided before the hands on session.

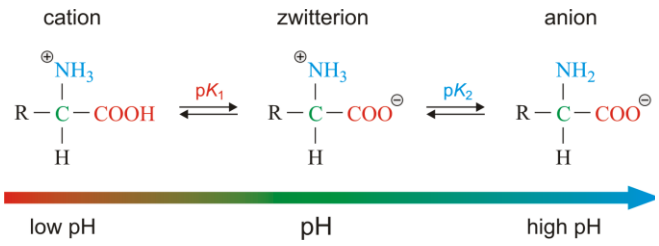
EXPERIMENT-7

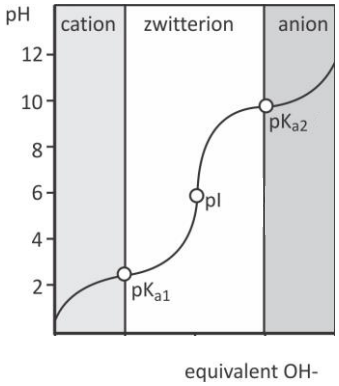
HOMO - LUMO Energy gap, Geometry Optimization of a Few Organic/ Inorganic Molecules Using Computational Calculation.

This experiment will discuss very brief what are the levels of theory available in Modern Quantum Chemistry Package.As such discussion needs knowledge of advanced quantum chemistry mostly we will discuss very elementary quantum chemistry.We will show how Highest Occupied Molecular Orbital (HOMO) and Lowest Unoccupied Molecular Orbital (LUMO) energy levels can be calculated for some molecules.

EXPERIMENT 8

DETERMINATION OF ISOELECTRIC POINT OF AN AMINO ACID





PRINCIPLE: Amino acids are molecules that contain both a base site (an -NH_2 group) and an acid site (a -COOH group). Individual amino acids differ only in the identity of the group, -R. On dissolution of an amino acid in water, the proton from the -COOH group gets transferred to the -NH_2 end of the molecule as the NH_2 group is a stronger base than -COO^- resulting a *zwitterion*. Depending on the pH of the solution the amino acids will be either in cationic form (low pH) or in anionic form (high pH).

- The pH at which the presence of these two types of ions in the same concentration is called the isoelectric point (**pI**). **At this pH, the amino acid does not migrate in an electric field**. (gel electrophoresis)
- **pI** is the pH at which the amino acid is neutral, *i.e.* the zwitterion form is dominant.

INTRODUCTION:

$$HA(aq) + H_2O = H_3O^+(aq) + A^-(aq)$$

The extent of this reaction is indicated quantitatively using the equilibrium constant, K_{eq}. The equilibrium constant is given as

$$K_{eq} = K_a = \frac{[H_3O^+(aq)][A^-(aq)]}{[HA(aq)]} \qquad \text{..... (1)}$$

The equilibrium constant for reaction of an acid with water is usually symbolized as K_a to remind us the type of reaction being dealt with.

- The concentration of water, since present in high concentration and thus essentially a pure liquid, is not included in eq. (1).
- The strength of an acid in aqueous solution is defined in terms of the magnitude of K_a. Strong acids have K_a values larger than 1 and that of weak acids is less than 1.

The equilibrium established when a weak acid reacts with water can be explored using the following pH titration: The pH of the solution must change as the titration proceeds.

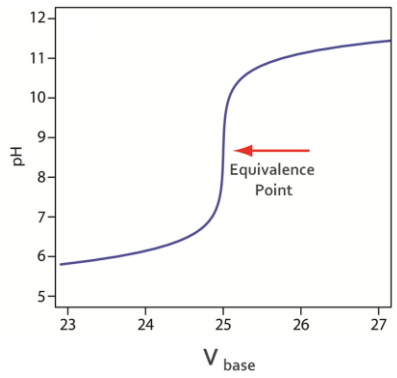
1. At the beginning of the process, before base is added, the pH of the solution is fairly low because it contains acid.
2. As titration proceeds, acid is neutralized by the added base, and pH rises.
3. Addition of base after all of the acid has been neutralized produces a basic solution, with a high pH.
4. The pH of the solution at each interval is monitored by a pH meter.

A plot of pH versus the volume of titrant added to the solution gives the so-called titration curve.

- The curve is shaped like "S". All titration curves have this characteristic shapes.

This provides the method for determining the equivalence point:

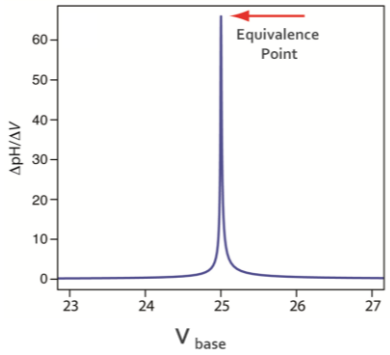
We successively add small volumes of base, measure pH after each addition, and plot the titration curve, from which we may find V_{base} at the inflection point (the equivalence point).



Moles of acid in the original aliquot is calculated as : Moles of acid = V_{base} at inflection point x M_{base}

A derivative plot needs to be created to determine the pK_a values accurately. The steps are as follows:

1. Calculate ΔpH/ΔV from the collected pH data for each addition of the titrant.
2. Plot ΔpH/ΔV against V (volume of titrant added).
3. The plot gives sharp peaks at the equivalence points corresponding to the sharp jumps in the titration plot.



In the case of amino acids, titration of the zwitterion with standard NaOH would provide the K_a value for the $-NH_3^+$ acid, which is expected to be similar to that of NH_4^+ ($pK_a = 9.25$). However, K_a value for the -COOH group could also be determined. It is possible to generate the acid form in solution by adding a strong acid to the zwitterion. The strong acid transfers a proton to the $-COO^-$ group of the zwitterion, resulting into a cation. Titration of a solution of this cation with standard NaOH should then yield two equivalence points, one for each acid. It should thus be possible to measure both the K_a values.

APPARATUS: pH meter, beaker, burette, pipette, glass rod, spatula.

CHEMICALS: Potassium hydrogen phthalate, glycine, alanine, HCl, NaOH, phenolphthalein.

EXPERIMENTAL PROCEDURE:

i) Standardisation of NaOH solution

1) Prepare 100 mL 0.1 *M* KHP (Potassium hydrogen phthalate) solution.
2) Standardize the supplied ~0.1*M* NaOH solution against KHP solution using phenolphthalein indicator (three results).

ii) Amino acid titration and estimation of equivalence point

1) Transfer exactly 10 mL of the supplied protonated amino acid solution to a clean 100 mL beaker.
2) Add 15 mL of distilled water to the beaker so that the total volume of the amino acid solution is 25 mL.
3) To homogenize the solution, place the beaker on the top plate of a magnetic stirrer and place a 1-inch stir bar in the beaker. Rinse the pH electrode and submerge it in the solution containing protonated amino acid. Make sure that the tip of the electrode is clear of the magnetic stir bar in the beaker before starting the stirrer. The rotation rate should be reasonably fast, but not so vigorous that splashing of the solution occurs.
4) Record the initial pH of the solution. Initiate the pH titration by adding 0.5 mL of NaOH solution from burette.
5) On each addition of base solution, note the pH of the solution. Continue this addition until you find larger gaps between two subsequent pH values. This indicates approach of the equivalence point. Reduce the volume of addition of the alkali solution to 0.1 mL until you comfortably cross the sudden jump in pH, indicating the equivalence point. After the equivalence point is passed, increase each volume of addition to 0.5 mL. Repeat this process if you expect more than one equivalence points.
6) Discard the solution on completion. Rinse the pH electrode with distilled water till pH meter reading is approximately equal to that of distilled water. Leave the pH electrode in beaker of distilled water and turn the meter off.

RESULTS:

Table 1. Preparation of 100 mL standard 0.1 *N* KHP solution

Weight taken (g)	Weight to be taken (g)	Strength of KHP solution

Table 2. Standardization of NaOH solution using standard KHP solution

Sl. No.	Volume of KHP (mL)	Burette reading (mL)			Average volume (mL)	Strength of NaOH solution
		Initial	Final	Difference		

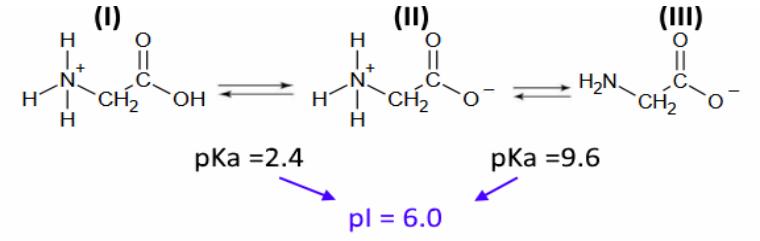
Table 3. Titration of amino acid solution using standard NaOH solution

Volume of amino acid (mL) =

Sl. No.	Volume of NaOH (mL)	pH	ΔV (mL)	ΔpH	ΔpH/ΔV

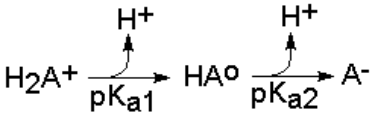
DISCUSSION:

Amino acids are more complicated than simple weak acids since amino acids have at least 2 ionizing groups. Glycine, for example, has both a carboxylic acid and an amino group that can ionize: If we dissolve the free base of glycine in pure water (ie neutral pH), it will ionize by protonating itself. The equilibrium is far to the right so most of the glycine is in the charged form called the zwitterion and glycine is still neutral because the +ve charge is netualized by the -ve charge. Glycine is always in the zwitterion form at neutral pH.



Glycine

Now if we put Glycine at an acid pH where it is fully protonated (i.e., it has all the protons bound to it which it bind), we can titrate it to reveal its 2 pK values for the alpha-carboxylic acid group and the alpha-amino group.

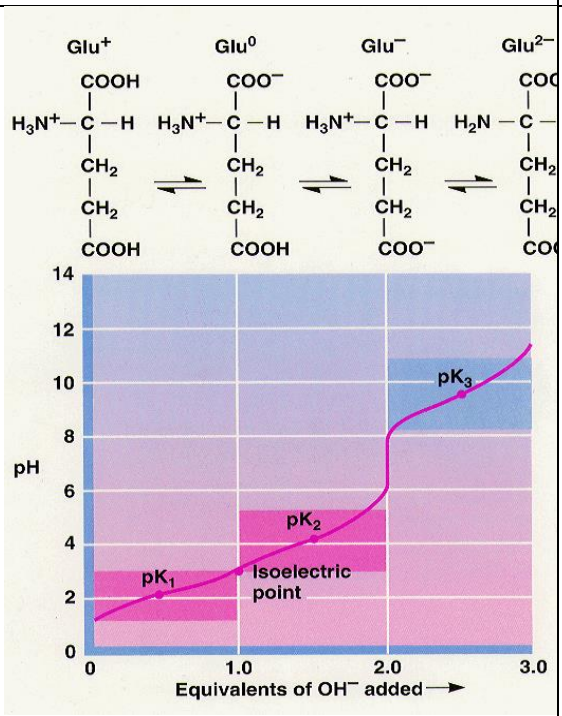
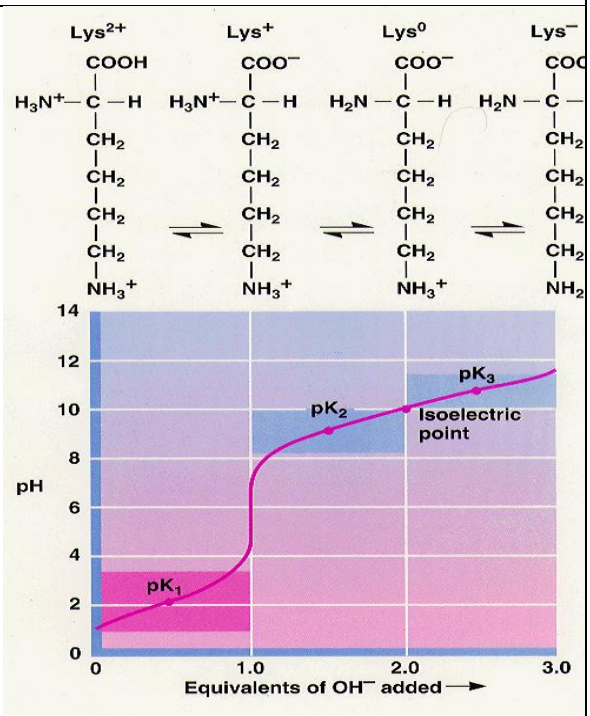


From the pK values, the pI (called the electric point or the place where Glycine has no net charge) can be calculated:

$pI = (2.4 + 9.6)/2 \approx 6$; ($pK_1 = 2.4$, $pK_2 = 9.6$)

Glycine is neutral at pH 6; it has no net charge here.

Some amino acids are classified as triprotic. This is because, in addition to the ionizable protons of the α-COOH and α-NH_3 groups, they also have a dissociable proton in their R group. Although triprotic amino acids can exist as zwitterions, under physiological conditions these amino acids will be charged. If the net charge under physiological conditions is **negative**, the amino acid is classified as an **acidic** amino acid because the R group has a proton that dissociates at a pH significantly below pH 7. The remaining triprotic amino acids are classified as **basic** amino acids due to a) their having a net **positive** charge under physiological conditions and b) an R group dissociable proton with a pKa near or greater than pH 7. Titration curves for triprotic amino acids generate the same information as those for the diprotic amino acids. The pI for a triprotic amino acid can be determined graphically, although this is somewhat more challenging.

Glutamic Acid	Lysine
	

EXPERIMENT-9

Replacement reaction: Synthesis of Iodoform (yellow solid)

Introduction

Iodoform (CHI_3) is the iodine analogue of chloroform. It is a pale yellow crystalline solid (m.p. 119°C), having a characteristic odour. It is used as a mild antiseptic and disinfectant. It is also used in the preparation of many medicinal ointments used as pain-relievers.

Purpose/Objective

- Replacement reaction.
- Synthesis of iodoform
- One pot synthesis
- Characterization

How does the process work

Iodoform can be prepared by treating any organic compound containing $CH_3CH(OH)$ - group (e.g., ethanol, 2-propanol, 2-butanol) or $CH3CO^-$ group (e.g., propanone, 2-butanone) with iodine in presence of sodium hydroxide. In the laboratory, it is usually prepared from ethanol. The chemical reactions involved are:

With ethanol

$$2\ NaOH + I_2 \rightarrow NaOI + NaI + H_2O$$

$$CH_3CH_2OH + NaOI \rightarrow CH_3CHO + NaI + H_2O$$

$$CH_3CHO + 3\ NaOI \rightarrow I_3CCHO + 3\ NaOH$$

$$I_3CCHO + NaOH \rightarrow CHI_3 + HCOONa$$

Materials and Apparatus

- NaOH
- Ethanol
- Iodine
- TLC plate

Procedure

1. Dissolve 5 g of iodine in 5 ml ethanol in a 100 ml conical flask.
2. Add 5% NaOH solution in small portions while continuously shaking the flask. Cool the flask from time to time under tap water. The addition of NaOH solution is further continued till the brown colour of iodine just disappears.
3. Allow the flask to stand for 5-10 minutes.
4. Decant off the clear supernatant liquid.

5. Filter the iodoform, wash with little cold water and then dry on a filter paper.

Crystallization of iodoform

1. Place the crude iodoform in a 100 ml conical flask.
2. Add small amount of rectified spirit and heat it on a water bath.
3. Add more rectified spirit slowly till the iodoform dissolves.
4. Filter the solution quickly through a fluted filter paper into a beaker.
5. Cool the solution in ice. The iodoform will crystallize rapidly.
6. Filter the crystals on a Buchner funnel, dry the crystals between the ibids of the filter paper.

Conclusion

This experiment describes an easy, safe, preparative procedure demonstrating a replacement reaction. Product isolation in the laboratory is straightforward. A solid product is formed in the reaction, which enables the students to manipulate the product easily and determine whether they made a small or large amount of product.

EXPERIMENT-10

Coupling of benzene diazonium chloride with 2-naphthol (rose red dye)

Introduction

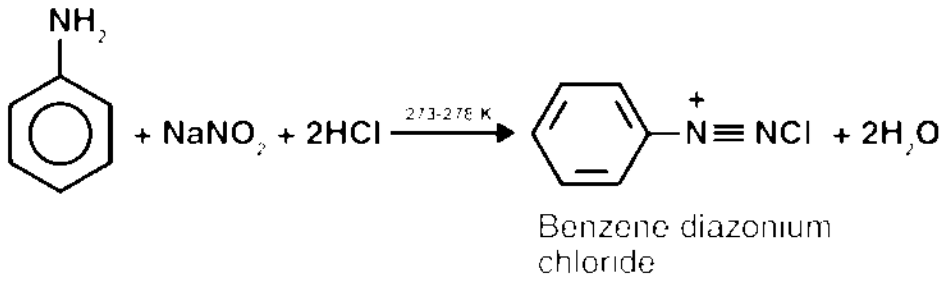
The azo products obtained when diazonium salts react with aromatic compounds have an extended conjugated system through N = N bond. This reaction is called a coupling reaction and the products formed are coloured. It is primarily used for textile dyeing.

Purpose/Objective

- Coupling reaction.
- Synthesis of rose red dye
- One pot synthesis
- Characterization

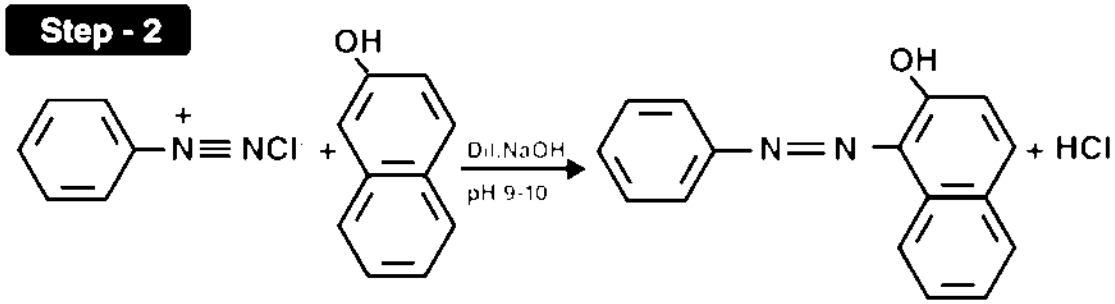
How does the process work

2- Naphthol aniline dye is a scarlet dye that can be prepared by coupling reaction. Aniline reacts with sodium nitrite in the presence of hydrochloric acid to form benzene diazonium chloride. Further benzene diazonium chloride reacts with 2-naphthol forms a bright orange colour 2-naphthol and forms aniline dye.



Benzene diazonium chloride

Step - 2



Benzene diazonium chloride | 2-Naphthol | 2-Naphthol aniline dye (orange colour)

Materials and Apparatus

- 2-Naphthol
- Aniline
- $NaNO_2$
- HCl

Procedure

1. Dissolve 5 ml of aniline in a mixture of concentrated hydrochloric acid and water.
2. Cool the solution in an ice bath between 0-5^oC.
3. Add a solution of 4 gm sodium nitrite in 15 ml of water dropwise with continuous shaking and controlling the temperature below 5^oC.
4. Take another flask to dissolve 8 gm of 2-naphthol in a solution of 5 gm sodium hydroxide solution in 50ml of water.
5. Cool the solution in the ice bath to 0-5^oC.
6. Now mix the two cold solutions slowly dropwise with constant stirring.
7. Continue the stirring for at least half an hour without allowing the temperature to rise above 10^oC.
8. An orange colour azo dye called 2-naphthol aniline separates out.
9. Filter the crude sample and wash it with cold water.
10. Dry and recrystallise it from ethanol.

Conclusion

This experiment describes an easy, safe, preparative procedure demonstrating a coupling reaction. Product isolation in the laboratory is straightforward. A solid product is formed in the reaction, which enables the students to manipulate the product easily and determine whether they made a small or large amount of product.

IISER KOLKATA

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