Laboratory Manual for Undergraduates
(CY 1002)

DEPARTMENT OF CHEMISTRY
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FOREWORD

Twenty first century can be called as an era of “Materials”. Sustained efforts on new innovations and developments in Materials Science and Technology have made and continue to make notable contributions to the needs and comforts of both society and common man. *Chemistry which is primarily an experimental science is at the back of all this*.

Laboratory work and experiments are an integral part of learning chemistry. Experience of experimentation is a delightful way of appreciating concepts and consequences of chemistry and its practice.

The experiments included in this manual have been carefully designed and developed by a dedicated team from our department with a view to provide an opportunity to the young and enthusiastic first year undergraduate students, a taste of experimental chemistry, its range, variety, principles underlying in them and utility value.

I thank profusely the entire team of colleagues, staff and research scholars of my department who have worked for this and wish all the student participants a useful and enjoyable experience.

Head, Department of Chemistry

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Undergraduate Laboratory Safety Precautions

Safety Instructions
Safety signs and instructions from demonstrators and academic supervisors must be strictly obeyed.

Eating and Drinking
These activities are strictly forbidden in all laboratory areas or in adjacent rooms.

Supervision of Laboratory work
No one is to perform experimental work unless a demonstrator or a member of the academic staff is present in the laboratory.

Eye protection
All persons must wear approved safety spectacles or goggles in chemical laboratories.

Protective clothing
All persons carrying out experimental work must wear the appropriate protective clothing.

Reporting Accidents, Fire and Dangerous Occurrence
All accidents resulting in injury, property damage or fire that might cause injury, must be reported to the supervisor.

In the event of Fire
1. Leave the building immediately by the nearest exit.
2. Never use lifts.
3. The person or persons responsible for starting and /or discovering the fire must be available to brief with the Institute security officer.

Physical Chemistry Experiments

Expt:1

REACTION KINETICS - FIRST ORDER RATE CONSTANT

Aim: To determine the first order rate constant at room temperature for the hydrolysis of methyl acetate catalysed by hydrogen ions.

Glasswares Required:

- Stoppered bottle (250 mL).
- Burette (50 mL)
- Conical flask (250 mL)
- Pippette (2 mL and 5 mL).

Chemicals required:

- NaOH solution (0.2N)
- HCl Solution (0.5N)
- Methyl acetate (2 mL)
- Phenolphthalein (few drops)

Principle:

Various chemical reactions take place at different rates. The rate of any particular reaction depends on certain parameters, the most important one being the concentration and temperature. At constant temperature, study of the influence of concentration on the rate of a reaction enables us to determine the order of the reaction.
If the rate of a reaction (R) is given by $R = kC_1^p C_2^q C_3^r$, the overall order n = p + q + r and k is called the **rate constant**.

The hydrolysis of methyl acetate catalyzed by hydrogen ions (eqn.1) forms a convenient system for kinetic investigation at room temperature.

$$CH_3COOCH_3 + H_2O \overset{H^+(Catalyst)}{\rightleftharpoons} CH_3COOH + CH_3OH \quad (1)$$

In the above reaction, the concentration of water is practically constant throughout the reaction. The rate of the reaction is thus directly proportional to the concentration of the ester, i.e. the reaction is **pseudo first order**, since [H^+] is also a constant.

The progress of the reaction is followed by determining the change in the concentration of methyl acetate as a function of time. This is done in an indirect way as follows. For each mole of ester hydrolyzed, one mole of acetic acid is produced. The rate of hydrolysis of methyl acetate is equal to the rate of formation of acetic acid.
This is represented as:

$$\frac{d[CH_3COOH]}{dt} = -\frac{d[CH_3COOCH_3]}{dt} = k[CH_3COOCH_3] \quad (2)$$

In eqn. (2) k is the rate constant for the reaction and depends on temperature, [H^+] and the solvent used for the reaction. k can be calculated more easily from the integrated form of eqn. (2).

If "a" is the initial concentration of the ester, and x is the amount of the ester decomposed at any time t,

$$k = \frac{2.303}{t}\log\frac{a}{a-x} = \frac{1}{t}\ln\frac{a}{a-x} \quad (3)$$

From this kinetic equation (eqn. 3) for a first order reaction, the rate constant k can be calculated.

The determination of (a-x) at various time intervals (t) and substitution in the kinetic equation (eqn.3) would yield a set of values of k, which can be averaged.

Alternatively, a plot of log (a-x) against 't' would yield a straight line and the rate constant 'k' can be calculated from the slope.

Procedure

1. Pipette out 2 mL of methyl acetate into the given stoppered bottle and shake the bottle to ensure through mixing and start the stop watch as soon as the ester is added.
2. Immediately withdraw 5 mL of reaction mixture by means of a pipette and transfer it into the conical flask with about 20 mL of ice-cold distilled water.
3. Add a drop of phenolphthalein to the conical flask and titrate against standard NaOH (approximately 0.1 N) taken in the burette.
4. The first appearance of a pale pink color is taken as the end point. Note down the titre value (V_0). This value corresponds to time, t =0. Discard the reaction mixture in the conical flask and clean the conical flask for the next titration.
5. At definite intervals of time say, 10, 20, 30, 40, 50, 60 minutes' pipette out 5 mL of the reaction mixture, deliver it into ice-cold distilled water and titrate against standard NaOH.
6. Note the titre value (V_t) in each case into your record book.

(V_∞ - V_0) corresponds to the initial concentration (a) of the ester,

(V_∞-V_t) corresponds to the concentration of the ester at any time, i.e., (a-x). The kinetic equation in terms of the titre values reduces to

$$k = \frac{2.303}{t} \log \frac{v_\infty - v_0}{v_\infty - v_t} \qquad (4)$$

The titre values are used instead of concentration since they are directly proportional to each other.

Determination of V_∞ :

1. Add 100 mL of 0.5 N HCl in to a 250 mL stoppered bottle.

2. Pipette out 2 mL of methyl acetate into the given stoppered bottle
3. Keep the bottle in a water bath maintained at 80 °C for 30 minutes. This enables the entire methyl acetate to hydrolyze yielding an equivalent quantity of acetic acid (CAUTION: Hold the stopper while the bottle is being heated lest it would pop off).
4. Cool to room temperature and then withdraw 5 mL of the reaction mixture into the conical flask, add phenolphthalein indicator and titrate against the NaOH solution. Note the titre value (V_∞).

Graph:

1. Plot of log (V_∞-V_t) against 't'would yield a straight line. 'k'can be evaluated from the slope.

2. Plot (V_∞-V_t) versus time and from this graph, determine the time required for the concentration 'a' to drop to 90% a.

   Calculate the value of *k* from this and also by using eqn. (3).

Rough Observations

S.No.	Time (mins)	Burette Readings		Volume of NaOH (ml)
		Initial (mL)	Final (mL)	
1	0			
2	10			
3	20			
4	30			
5	40			
6	50			
7	60			

Expt: 2

Verification of Beer-Lambert law using gold nanoparticles

Aim : Preparation of gold nano particles using the citrate method and verification of Beer- Lambert law.

Glass wares Required:

- Standard Flask (5 mL) - 5 Nos.
- UV cuvette- 1 No.
- Flat bottomed flask (50 mL)- 1No.
- Measuring Cylinder (25 mL)- 1No.
- Graduated pipette (1 mL, 2mL and 5mL each one).

Chemicals required:

- Chloro auric acid solution
- Tri sodium citrate

Principle

The Beer-Lambert law, also known as the Beer's law or the Lambert-Beer law or the Beer-Lambert-Bouguer law in various books, is the linear relation between the absorbance and concentration of an absorbing species. It relates to the absorption of light to the properties of the material through which the light is traveling.

In practice, Beer's Law is accurate enough for a range of species, solvents and concentrations, and is a widely used relationship in quantitative spectroscopy. Most often, it is used in a quantitative way to determine concentrations of an absorbing species in solution.

Absorbance is measured in a spectrophotometer by passing a collimated beam of light at wavelength λ through a plane parallel slab of material that is normal to the beam.

For liquids, the sample is held in an optically flat, transparent container called a cuvette. Absorbance (A) is calculated from the ratio of light intensity passing through the sample (I_0) to the intensity that is incident on the sample (I).

$$A = \log_{10}\left(\frac{I_0}{I}\right)$$

According to this law, when light passes through a material, there exists a linear dependence between the absorbance (A) of light and the product of absorption coefficient (α) with the path length (l) of the light (distance though which light travels in the material).

Absorption coefficient of a material in turn depends on the molar absorptivity or molar extinction coefficient or molar absorption coefficient (ε) and the concentration (c) of the absorbing species in the material. Thus, according to Beer-Lambert law, absorbance is given by

$$A = \varepsilon c l$$

where ε is constant, for a given wavelength of light, and can be obtained from the absorbance values of solutions with different concentrations. Absorbance is unit less, while the units of ε, l, c are $L.mol^{-1}.cm^{-1}$, $mol.\ L^{-1}$ and cm^{-1}, respectively.

The Beer-Lambert law was independently discovered (in various forms) by Pierre Bouguer before 1729, Johann Heinrich Lambert in 1760 and August Beer in 1852. Beer extended the exponential absorption law to include the concentration of solutions in the absorption coefficient

.

Appearance of wine-red colour in step 6 indicates the formation of colloidal gold or gold nanoparticles. The recorded UV-Vis spectrum of the solution, step 10, shows the absorbance at a λ_{max} of ~520 nm, as shown in Fig. 1. This feature is attributed to the **plasmon resonance** of gold nanoparticles. Read more about plasmon resonance in a book on nanomaterials.

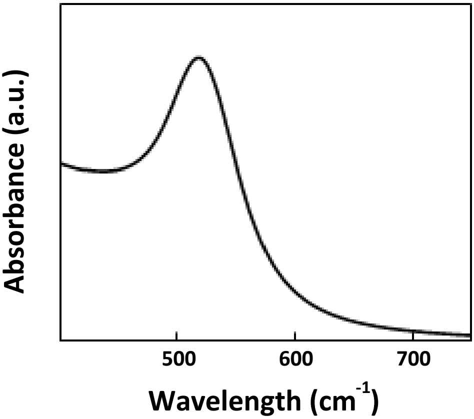


Fig. 1 UV-Vis spectrum of as prepared gold nanoparticles.

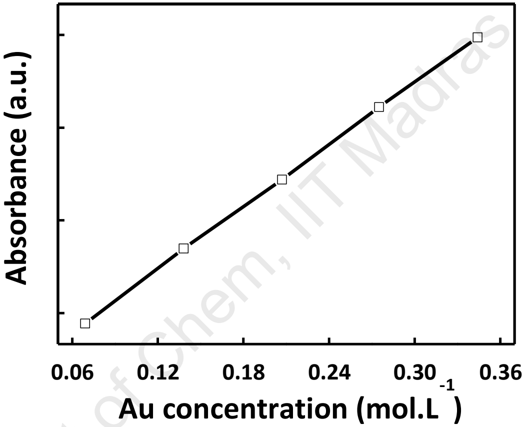


Fig. 2 Plot of absorbance vs. concentration of gold nanoparticles.

Fig. 2 shows the plot of absorbance vs. concentration for the gold nanoparticles of different dilutions. It can be observed that all the data points fit into a straight line, thus conforming to the Beer-Lambert law. Slope of the above plot gives the absorption coefficient of the gold nanoparticles.

For the calculation of absorption coefficient, path length is taken as equal to the width of the cuvette used for recording the UV-Vis spectrum (we have taken it as 1 cm). From the above data, the value of absorption coefficient is calculated as ~3.36 $L.mol^{-1}.cm^{-1}$. By making use of the above plot (Fig. 2), we can calculate the concentration of gold nanoparticles of an unknown dilution.

You may want to know that light attenuation in this particular case is not truly due to optical absorption alone as there is a scattering component. At the limit of small particles (as in this case, where the particle size is ~ 20 nm), scattering is disregarded.

Scattering and absorbance together is called extinction and the Y axis will be labeled as “**Extinction**” in the case of larger particles.

<u>Procedure</u>

1. Prepare 10 mM $HAuCl_4$ solution in a 5 mL standard flask by dissolving 17 mg of $HAuCl_4 \cdot 3H_2O$ in water. **(Stock Solution)**

2. Prepare ~0.5% solution of trisodium citrate in a 10 mL sample bottle flask by dissolving 25 mg of $Na_3C_6H_5O_7$ in 5 mL of water. **(Stock solution)**

3. Take 0.5 mL of 10 mM $HAuCl_4$ solution in a 50 mL flat bottomed flask using a 1 mL pipette and add 13 mL of distilled water to it using a 25 mL measuring jar.

4. Heat the solution over a heating mantle and bring the solution just to a boil (marked by the appearance of bubbles from the colourless solution).

5. To the boiling solution, add 1 mL of ~0.5 % trisodium citrate solution using a 1 mL pipette.

6. Continue heating (with boiling) till the colour turns to wine-red. (This may take about 1-2 minutes)

7. Remove the flask from the mantle and keep it for cooling in air, for about 15-20 minutes.

8. To account for loss of water during boiling, make the solution to 14.5 mL (i.e. to the original volume) in a 25 mL measuring jar.

9. Calculate the concentration of gold nanoparticles in the solution in terms of gold.

10. Prepare the following solutions of different concentrations.

    - 5 mL prepared solution
    - 4 mL prepared solution + 1 mL distilled water
    - 3 mL prepared solution + 2 mL distilled water
    - 2 mL prepared solution + 3 mL distilled water
    - 1 mL prepared solution + 4 mL distilled water

11. Collect the absorbance at 520 nm of undiluted solution and those of the above diluted ones, with distilled water as the reference, using a UV-Vis absorption spectrometer.

12. Plot a graph of absorbance vs. concentration with the obtained values and verify Beer-Lambert Law.

Rough Observation

S.No	Volume of prepared Solution (mL)	Volume of Distilled water (mL)	Gold concentration (moles/Litre)	Absorbance
1.	5	0		
2.	4	1		
3.	3	2		
4.	2	3		
5.	1	4		

Expt:3

ACID STRENGTH IN A CITRUS FRUIT

Aim: To determine the acid strength in a citrus fruit juice using conductometry.

Glass wares Required:

- Standard Flask (250 mL- 2Nos)
- Beakers (100 mL- 2 Nos.)
- Pipette (25 mL, 20mL and 10mL - one each).
- Burette (25 mL and 10mL - one each)

Chemicals required:

- NaOH solution
- HCl solution (0.02N)
- Citrus fruit Juice
- Buffer tablets (pH 4, pH 7 and pH 10)
- 0.01N KCl solution

Principle

Citrus fruits contain fairly a large amount of citric acid. It is often a good source of Vitamin C (Ascorbic acid) too. A simple acid-base titration with a strong base can be performed to determine its acid strength. However, some citrus fruits are colored and in such a case conventional titration using indicator-dyes is not possible. Various instrument based titrations like conductometric, pH metric or spectrophotometric methods then become essential.

The present experiment is aimed at introducing two instruments for carrying out acid-base titrations: a pH meter and a conductivity bridge.

First part of the experiment involves the use of pH meter for titration of a strong acid with a strong base of unknown strength. A pH meter is a potentiometer, which uses a glass electrode sensitive to H^+ ion concentration. If a strong acid is titrated against a strong base (in the burette), the pH of the solution varies as a sigmoidal curve with a very sharp increase of pH at the end point.

Second part of the experiment is the conductometric titration of the citrus fruit juice, (which, in all likely hood, is supplied to you after dilution in water), with a standard sodium hydroxide solution (standardized in the first part of the experiment. A solution containing ions conduct electricity due to movement of ions to oppositely charged electrodes under the influence of an electric field. More the concentration of free ions in a solution, more the conductance.

The conductance of a weak acid is usually low as free ions furnished in the solution is less. On titrating with a strong hydroxide solution (fully ionized), an initial decrease of conductivity is observed due to mutual suppression of ionization of the acid and the salt that is formed. However, after a few drops of titrant addition, the less ionizable acid is progressively replaced by its completely ionizable sodium salt, the conductance starts to increase linearly.

Once the end point is reached and all the acid has been converted to its salt, addition of further sodium hydroxide results in a much rapid increase in the conductance as OH^- ion conducts more than the acid anion. The end point is thus the point where this change in slope takes place.

Procedure: PART -I

Calibration of pH meter

1. Switch on the pH meter and allow it to stabilize for 10 minutes.

2. Adjust the temperature compensate to 25°C.

3. Insert the pH probe into the buffer solution of pH 4 and note the reading if not correct adjust the screw on the side of the pH meter.

4. Similarly do the same for pH 7.

Part I: Standardization of the Sodium hydroxide solution.

5. Wash the glass electrode by distilled water and immerse it into a standard acid solution and note the pH.

6. Add small aliquots of sodium hydroxide solution of unknown strength from a burette, stir the solution well, and note the pH and the burette reading. Continue further till there is almost no change in pH.

7. Plot "pH" vs. "volume of base". The point of inflection of the sigmoid curve, which is expected at pH 7, is the end point. Calculate the strength of the sodium hydroxide solution.

Rough Observation

Volume of NaOH(ml)	pH	Volume of NaOH (mL)	pH
0.0		8.0	
1.0		9.0	
2.0		10.0	
3.0		11.0	
4.0		12.0	
5.0		13.0	
6.0		14.0	
7.0		15.0	

Procedure: PART -II

Calibration of Conductometer:

1. Switch on the Conductometer and allow it to stabilize for 10 minutes.

2. Insert the conductometry probe into standard 0.01 N KCl solution and adjust the knob and set the reading to 1413 μS at 25°C).

Part-II: Determination of strength of the citrus fruit juice.

1. Switch on the conductivity bridge and allow it to stabilize for 10 minutes.

2. Take the given juice solution in a beaker and note its conductance.

3. Titrate with standard NaOH solution and note the burette readings and conductance after each addition.

4. An initial dip in conductance is observed which soon increases linearly with volume of base added. At the end point you will observe a faster increase of conductance with base addition.

5. Stop the titration after 8/10 readings after the end point.

6. Plot Conductance *Vs* Volume of base added. Find the end point and estimate the strength of the juice solution given.

Rough Observation

Vol. of NaOH (mL)	**Conductance Ohm^{-1}**	**Vol. of NaOH (mL)**	**Conductance ohm^{-1}**
0.0		2.75	
0.25		3.0	
0.50		3.25	
0.75		3.5	
1.0		3.75	
1.25		4.0	
1.5		4.25	
1.75		4.5	
2.0		4.75	
2.25		5.0	
2.50			

Inorganic Experiments

Expt:4a

Estimation of Cu^{2+} ion by colorimetry & Fe^{2+} ion by redox titration

Aim : To determine the concentration of Cu^{2+} ion by colorimetry.

Principle: The given Cu^{2+} ion solution is treated with excess 1:1 NH_3 solution to form complex and made up to a known volume. The absorbance of the complex in the visible region is measured at 610 nm using a colorimeter. From the calibration plot drawn with the absorbance of standard solutions, we can find out the concentration of copper in the given unknown solution.

Glass wares required:

- Standard flask (25 mL) – 6 Nos.
- Burette (25 mL) – 1 No.
- Colorimeter tube – 1 No.
- Measuring Jar (10 mL) – 1No.
- Beaker (100 mL) – 1 No.
- Dropper – 1No.

Chemicals required:

- Copper Sulphate (5000ppm) standard solution
- Ammonia Solution (1:1)

Procedure:

Five different known concentration of Copper – Ammonia complex solution were prepared by the following method:

Flask No.	Vol. of Cu^{2+} (5000 ppm) soln*****.	Vol. of 1:1 $NH_4OH^{\#}$	Vol. to be made up	Conc. in mg/L
1	1	5	25	
2	2	5	25	
3	3	5	25	
4	4	5	25	
5	5	5	25	

*****The quantity mentioned should be exactly taken from burette

$^{\#}$Ammonia should be taken only using measuring cylinder. **Caution!** **<u>Never use pipette for ammonia.</u>**

Their absorbance at 610 nm was measured using a colorimeter. A plot is drawn with absorbance in Y axis and Copper concentration in mg/l in X axis.

To the unknown solution given in the 25 mL standard flask 5 mL of 1:1 NH_3 solution is added and then it is made up to 25 mL mark using droppers.

After making up the solution is mixed well. Required quantity is transferred into the colorimeter tube and absorbance at 610 nm is measured.

Using the above calibration plot and the value of absorbance for the unknown, the concentration of copper ion in the given solution is calculated.

Expt:4b Estimation of Fe^{2+} ion by Redox Titration

Aim: To determine the normality of the given unknown Fe^{2+} ion solution.

Principle: The given unknown Fe^{2+} ion solution is made up to a known volume and then titrated with standard potassium dichromate solution in acid medium with *ortho* phosphoric acid and Diphenyl ammine as indicator. End point is the appearance of violet colour.

Glasswares required:

- Standard flask (100 mL) – 1 No.
- Pipette (10 mL) – 1No.
- Burette (25 mL) – 1No.
- Conical Flask (250 mL) – 1No.

Chemicals required:

- Standard Potassium Dichromate solution (0.05N)
- Unknown Ferrous ammonium sulphate solution (roughly 0.5N solution)
- Sulphuric acid (2N)
- Orthophosphoric acid
- Diphenylamine indicator (1% in conc. sulphuric acid)

Procedure:

The given Fe^{2+} ion solution is made up to 100 mL in a standard flask and shaken well.

10 mL of this solution is pipette out into a 250 mL conical flask. To this 5 mL of 2N Sulphuric acid, 2 mL of Ortho Phosphoric acid and 2 drops of Diphenylammine indicator were added.

This solution is titrated against standard 0.05N Potassium dichromate solution till violet color appears. From the titre value the normality of the given unknown Fe^{2+} solution is calculated.

Rough Observation

S.No	Volume of 5000 ppm Copper Solution (mL)	Concentration (mg/L)	Absorbance
1.	1	200	
2.	2	400	
3.	3	600	
4.	4	800	
5.	5	1000	
6.	Unknown sample	?	

Rough Observation

Sl.No.	Vol. of Fe^{2+} solution Pipetted out (V_2 mL)	Burette Reading		Volume of $K_2Cr_2O_7$ (V_1 mL)
		Initial	Final	
1	10			
2	10			
3	10			

Expt 5:

Gravimetric analysis

Aim: To estimate the Ni^{2+} ion in the given unknown solution by precipitating the Ni^{2+} ion as Nickel-DMG complex and performing gravimetric analysis.

Principle:

Nickel is precipitated by the addition of an ethanolic solution of 1% dimethyl glyoxime (DMG) to a hot, faintly acid solution of the nickel salt, and then adding a slightly excess of dilute ammonia solution (free from carbonate). The pink coloured precipitate is washed with cold very dilute (1:100) ammonia and then with cold water. Dry the nickel dimethylglyoximate after at 110-120^{o}C for 30 minutes. Cool in a desiccator to room temperature and then weigh. Repeat drying for constant weight.

Glasswares required:

- Beaker with glass rod (250 mL) – 1No.
- Watch glass – 1No.
- Measuring cylinder (10 mL) – 1No.
- Sintered Crucible (G3) – 1No.
- Desiccator – 1No.

Chemicals required:

- Hydrochloric acid (1:1)
- DMG solution (1% in absolute ethanol)
- Ammonia Solution (dilute)
- 1:100 very dilute ammonia for washing

Procedure:

1. Dilute the given unknown Ni^{2+} solution to 100 mL.

2. Heat the solution to 70-80°C, using electrical hot plate then carefully shift the solution to water bath and add a slight excess of the dimethylglyoxime (~10 mL) reagent and immediately add dilute ammonia solution (slightly excess) drop wise, directly to the solution with constant stirring until precipitation takes place.

3. Allow this to stand in steam bath for 15 minutes.

4. Cool to room temperature and filter through pre weighed sintered crucible (Empty weight of the crucible to be taken first).

5. Keep the G3 sintered crucible in the suction setup and first wash it by draining 20 mL very dilute ammonia (1:100) solution through it by applying vacuum. Then transfer the precipitate quantitatively into the sintered crucible carefully without any loss.

6. Wash the precipitate with very dilute 1:100 ammonia and then with cold water.

7. Dry it at 110-120°C for 20 min.

8. Cool the crucible in desiccator and then weigh the crucible with precipitate. Repeat until constant weight is attained.

9. Weigh as $Ni(C_4H_7O_2N_2)_2$, which contains 20.32% Ni.

Rough Observation

Weight of Sintered Crucible with NiDMG complex :
Weight of empty Sintered Crucible :
Weight of NiDMG precipitate :

Weight of Nickel in given unknown solution = weight of NiDMG X 0.2032 =

Expt:6

Synthesis of copper oxalate complex $K_2[Cu(C_2O_4)_2].2H_2O$

Aim : Synthesis of copper oxalate and determination of the its oxalate content.

Glasswares required:

- Beaker (100 mL) with glass rod – 2Nos.
- Sintered Crucible (G3) – 1No.
- Conical flask (250 mL) – 1No.
- Burette (50 mL) – 1No.

Chemicals required:

- Copper sulphate solid
- Potassium oxalate solid
- Acetone
- Sulphuric acid (4N)
- Potassium permanganate solution (0.05N)

Principle:

In acid solution, permanganate, MnO_4^- [Mn(VII)] is reduced to Mn(II). The half reaction is:

$$MnO_4^- + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O \qquad E^o = 1.51\ V$$

In basic or neutral solution, permanganate is reduced to manganese (IV) oxide. The half reaction is

$$MnO_4^- + 4H^+ + 3e^- \rightarrow MnO_2 + 2H_2O \qquad E^o = 1.70\ V$$

Sodium oxalate or oxalic acid is often used to standardize permanganate. The relevant half reaction is

$$C_2O_4^{2-} \rightarrow 2CO_2 + 2e^- \qquad E^o = -0.49\ V$$

Therefore, the reaction with permanganate is favourable, i.e.,

$$2\ MnO_4^- + 5C_2O_4^{2-} + 16\ H+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O,\ E^o = 1.02\ V$$

Procedure

1. Dissolve 2 g of $CuSO_4.5H_2O$ in 5 mL water and heat the solution to 70°C.
2. Add the hot solution with stirring to 15 mL solution of potassium oxalate, containing 6g of $K_2C_2O_4.H_2O$.
3. Allow the solution to cool and filter the precipitate through a pre weighed sintered (G-3) crucible. Wash with ice cold water, followed by ice cold acetone. Dry the product at 50-60^O C and weigh. Note the yield.

Analysis

1. Weigh accurately about 0.1g of the dried copper oxalate complex into a conical flask
2. Add 10 mL of 4N H_2SO_4, dilute to 50 mL. Heat to boiling.
3. Titrate the hot solution with 0.05N $KMnO_4$.and note the end point which is the appearance of permanent pink color.
   1000 mL 1N KMnO4 = 44.01g of $C_2O_4^{2-}$

$$\% \text{ of } [C_2O_4]^{2-} \text{ in the complex} = \frac{44.01}{1000} \times \frac{N_{KMnO4}}{W_c} \times V_{KMnO4} \times 100$$

(Wc = Weight of the copper oxalate complex)

Rough Observation

Sl.No.	Weight of complex taken (W_c gms)	Burette Reading		Volume of $KMnO_4$ (V_{KMnO4})	% of C_2O_4
		Initial	Final		
1					
2					
3					

Organic Experiments

Expt: 7

BASE CATALYZED ALDOL CONDENSATION

Aim: Synthesis of dibenzalpropanone by Aldol condensation

Glasswares required:

- Watch glass– 1No.
- Glass rod – 1No.
- Conical flask (250 mL) – 1No.
- Boiling tube – 1No.

Chemicals Required:

1. Acetone (0.7 mL)
2. Benzaldehyde (2.5 mL)
3. NaOH soln. (2.35g in 23 mL water)
4. Ethanol (15 mL)

Procedure:

1. To a 250 mL conical flask add 23mL of 10% NaOH and 15 mL of ethanol. Then cool the mixture in an ice bath.

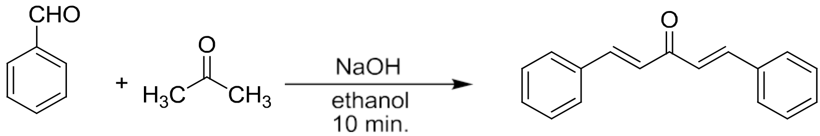
2. To the resultant solution, add 2.5 mL benzaldehyde, then add 0.7 mL acetone slowly over 15 minutes. Then, stir the reaction mixture with a glass rod vigorously till the product precipitate is formed.

3. A yellow solid will appear slowly and after 30 mins, it was filtered through by Buckner Funnel through Whattman's filter paper.

4. Wash with water, dry, weigh and recrystallize from ethanol.

5. Find out the melting point of recrystallized aldol product.

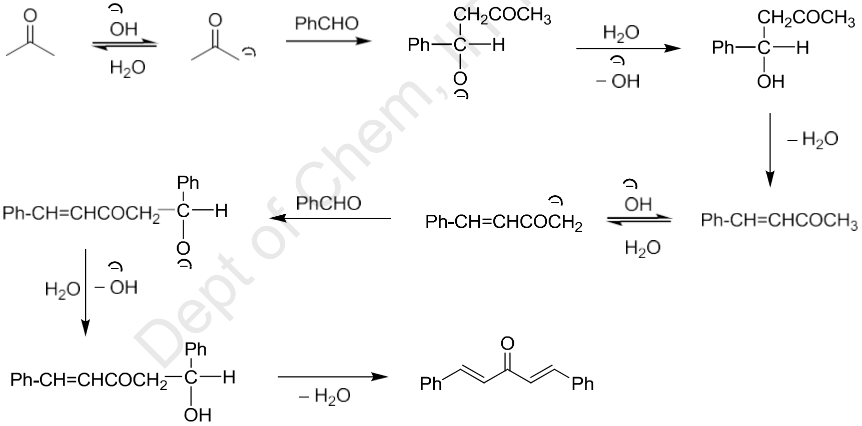
Reaction:



Benzaldehyde Acetone

Dibenzal acetone (or)
Dibenzylidene acetone

Mechanism:



Expt : 8

HALOGEN ADDITION TO C=C BOND

Aim : Bromination of *cis* or *trans*-stilbene

Glasswares required:

- Double neck RB flask (100 mL)
- Water condenser
- Dropping funnel (10 mL)
- Measuring cylinder (20 mL)

Chemicals Required:

- *Cis or trans*-Stilbene - 1.8 g
- 33% HBr in acetic acid - 5.2 mL
- 30% Hydrogen peroxide - 7 mL
- Ethanol - 15 mL

Procedure:

1. Add stilbene (*cis or trans)* (1.80 g) and ethanol (15 mL) to a two neck 100 mL round-bottom flask fitted with reflux condenser and a dropping funnel and reflux on a water bath.

2. Add 5.2 mL of HBr (33%) and hydrogen peroxide (H_2O_2, 30%) (7 mL) using dropping funnel sequentially to this refluxing solution of stilbene.

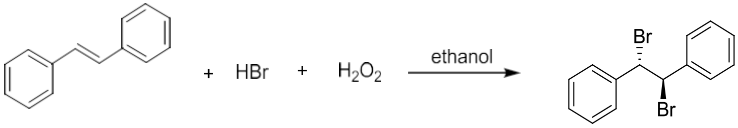
3. The colourless solution will become deep orange in colour, within 15-20 minutes, the orange colour will disappear. This indicates the bromination of stilbene.

4. Allow the solution to cool to precipitate stilbene dibromide.Filter the precipitate, wash with two or three small portions (3 mL or less) of ethanol until the organic color is removed and dry.

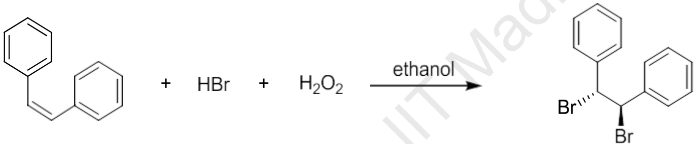
5. Find out the melting point of the formed product.

6. Based on the melting point of the product, identify the stereochemistry of the product and stilbene.

Reaction



trans- stilbene *meso*-1,2- dibromo-1,2- diphenylethane



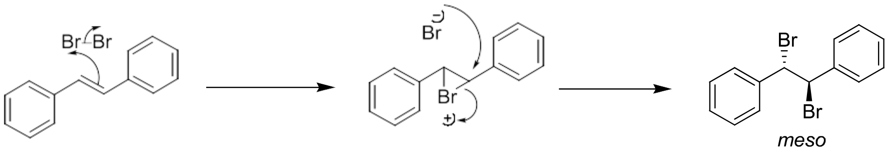
cis -stilbene *racemic*-1,2-dibromo-1,2-diphenylethane

Caution:

Care must be taken while handling the solution of hydrogen bromide and hydrogen peroxide. Avoid contact with skin. Do not inhale. In case of accidental touch, wash with plenty of water immediately.

Mechanism:

$$2HBr + H_2O_2 \xrightarrow[\text{oxidation}]{\text{in situ}} 2\,H_2O + Br_2$$



Expt: 9 QUALITATIVE ANALYSIS OF ORGANIC COMPOUND

TESTS FOR FUNCTIONAL GROUPS

Experiment	**Observation**	**inference**
Test for Acids: a) **$NaHCO_3$ test**: Sample + 1 mL of $NaHCO_3$ solution	A brisk effervescence with the evolution of CO_2.	Presence of Acid
b) NaOH test: 0.1 g of substance (3 drops) + 1 mL 10% NaOH solution. Shake well. All organic acids dissolve when shaken with NaOH.	On acidifying with conc. HCl, the acid is precipitated.	Presence of Acid
Test for Aldehydes and Ketones: a)**Borsche's test**: Substance + ~1 mL Borsche‟s reagent, heat and add 1 mL Conc.HCl.	Orange solution (or) precipitate	Presence of Aldehyde or Ketone
b) **Fehling's Solution Test: (for Aliphatic Aldehydes only)** Sample + ~1 mL each of Fehling‟s solution "A" & " B". Heat it for 2-3 mins.	Red Precipitate	Presence of Aliphatic Aldehyde or aldose (like Glucose)

c) Tollen's Reagent test:(Aldehyde only) Sample + ~1 mL Tollen‟s reagent, Heat the test tube in a water bath for 5 mins. **d) Legal's test: (for ketones only)** Sample + 2 mL water + 5 drops of Sodium nitroprusside solution + 5 drops NaOH+ 5 drops CH_3 COOH	Bright silver mirror forms on the sides of the test tube. Red colour changes to purple	Presence of Aldehyde or Aldose (like Glucose) Presence of ketone
Test for Phenol Sample +1 mL of neutral Ferric chloride	A violet colour	Presence of phenol
Test for Amines **a)Solubility test:** 0.1g substance +~ 1 mL dil HCl. Shake well. **b) Dye Test**: Sample +~ 3 mL dil. HCl, cool(in ice), add 1 mL $NaNO_2$ solution and 1 mL alkaline – β -naphthol	Substance is soluble and reappears on adding NaOH A Scarlet red dye	Presence of Amine A Primary Amine

APPENDIX-I

S.No	Solution	Weight/Volume Required
1.	NaOH (0.1 N)	8.0 g / 2 lit
2.	HCl (0.5 N) HCl (0.1N)	90 mL / 2 lit 18 mL / 2 lit
3.	NaCl (1%)	2g / 200 mL
4.	I_2 (0.005M)	2g KI+water+1.27g I_2, makeup to 1 lit
5.	KI (0.1 N)	8.3 g /500 mL
6.	$Na_2S_2O_3.5H_2O$ (1.0N)	248.18g / 1 lit
7.	Sat. I_2 in CCl4	10g I_2 /500 mL CCl4
8.	H_2SO_4 (0.5 N)	14 mL / 1 lit
9.	KNO_3 (0.1 N)	5.055 g / 500 mL
10.	HCOOH (0.5 M)	9.55 mL / 500 mL
11.	KBr (1.0 M)	59.5g / 500 mL
12.	$CuSO_4.5H_2O$ (0.1N)	24.971g/1 lit
13.	$FeSO_4.(NH_4)_2SO_4.6H_2O$ (0.5 N)	196.1 g / 1lit add 5 mL conc sulphuric acid
14.	KCl (0.01 N)	0.746 g / 1 lit
15.	$KMnO_4$ (0.1 N)	3.1606 g/1 lit
16.	$K_2Cr_2O_7$ (0.1 N)	4.903g/ 1 lit

APPENDIX -II

Preparation of Indicators and reagents

S.No	Indicator	Preparation of solution
1.	Diphenylamine	Dissolve 1 g of substance in 100 mL of Conc.H_2SO_4
2.	Phenolphthalein	Dissolve 5 g of Phenolphthalein in 100 mL of 50% alcohol
3.	Fehling"s A	A blue aqueous solution of copper(II) sulfate,
4.	Fehling"s B	A clear solution of aqueous potassium sodium tartrate (also known as Rochelle salt) and a strong alkali (commonly sodium hydroxide).
5.	Borsche"s reagent	Alcoholic solution of 2,4-Dinitrophenylhydrazine and con.H_2SO_4
6.	Tollen"s reagent	It is usually ammoniacal silver nitrate, but can also be other mixtures, as long as aqueous diamminesilver(I) complex is present.

NOTES