Practical Handbook for S.Y.B.Sc. Chemistry (Major)

Under Choice Based Credit System (CBCS)

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Preface

The present practical course in Chemistry (major) for the semester III and IV has been thoughtfully designed to provide students with a comprehensive understanding of fundamental concepts in Physical, Inorganic, Organic, and Analytical Chemistry through hands-on laboratory experience. The experiments included in this course aim to bridge the gap between theoretical knowledge and practical application, thereby fostering scientific temperament, analytical thinking, and problem-solving skills among learners.

In semester III, the Group A section emphasizes principles of physical chemistry and basic analytical techniques such as conductometry, chemical kinetics, colorimetry, and gravimetric analysis. It also introduces students to essential tools and instruments used in analytical laboratories, along with their principles, construction, and maintenance.

The Group B section focuses on inorganic and organic chemistry, including volumetric and gravimetric estimations, as well as organic synthesis and purification techniques. These experiments are carefully selected to develop accuracy, precision, and a clear understanding of reaction mechanisms and analytical methods.

Special emphasis has been placed on data interpretation, error analysis, calibration methods, and the development of laboratory skills such as observation, recording, and reporting. This course is expected to equip students with the necessary competencies required for higher studies, research, and professional careers in chemistry and allied fields.

Semester IV is also comprising with Group A and Group B in which Group A componentry focuses on electrochemistry, chemical kinetics, and analytical methods. Experiments such as the determination of standard emf and free energy change of the Daniell cell, potentiometric estimations, conductometric titrations, buffer capacity measurements, and

chromatographic separations are included to strengthen the conceptual understanding of physicochemical principles. Additionally, the study of distribution laws and extraction efficiency introduces students to important separation techniques widely used in chemical analysis.

A significant portion of the course is devoted to familiarizing students with essential analytical instruments and laboratory apparatus, including filtration systems, distillation assemblies, electrodes, conductivity cells, and chromatographic equipment. Emphasis is placed on their principles, construction, operation, care, and maintenance, ensuring safe and effective laboratory practices.

The Group B section deals with inorganic synthesis and organic qualitative analysis. Preparations of coordination compounds and inorganic complexes are included to enhance understanding of reaction chemistry and synthesis techniques. Organic qualitative analysis trains students in systematic identification of compounds through preliminary tests, solubility behavior, elemental detection, and functional group analysis. The determination of physical constants further reinforces the importance of purity and characterization of compounds.

Overall, this course aims to cultivate scientific rigor, observational skills, and methodical experimentation, preparing students for higher education, research, and professional applications in chemistry and allied disciplines.

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Semester - III

Chemistry Practical (Major)

(24_USCH304)

Instrumental Analysis (Conductometry)

Experiment No. 1

Aim: To verify *Ostwald's dilution law* for weak acid by conductometry.

Theory:

(1) Ostwald's dilution law states that at constant temperature, degree of dissociation of a weak electrolyte is inversely proportional to the square root of its concentration. The mathematical form of the law is represented as:

$$\alpha = \frac{1}{\sqrt{C}}$$ where, α the degree of dissociation

(2) In this experiment, the conductance of the acid solution is measured at different concentrations (C).

(3) Using the conductance of the solution, it is possible to calculate the specific conductance, equivalent conductance of the solution (λ_c) = $\frac{1000K}{c}$.

(4) It is possible to calculate the degree of dissociation α using the equation,

$$\alpha = \frac{\lambda_c}{\lambda_\infty}$$

λ_∞ Equivalent conductance at infinite dilution and is obtained from **Kohlrausch's law**. (5) The dissociation constant (K_a) can be obtained from Ostwald's law. The constant value of K_a (within reasonable limits) verifies Ostwald's law.

$$K_a = \frac{\alpha^2}{1-\alpha}$$

Requirements: Conductometer and conductivity cell, conductivity water, 100 cm^3 standard measuring flask, 100 cm^3 beakers, 25 cm^3 pipette, 10 cm^3 graduated pipette, 0.1N acetic acid solution, 0.1N KCl solution etc.

Procedure: [If cell constant is provided, proceed to Part II. If it is not provided, then proceed as given in Part 1.]

Part I: Determination of Cell constant:

(1) Wash the conductivity cell and a 100 cm^3 beaker using conductivity water. (2) Rinse both the cell and the beaker with a small quantity of 0.1N KCl solution. (3) Take about 50 - 60 cm^3 of supplied 0.1N KCl solution in the rinsed beaker and dip the conductivity cell in it, so that platinum plates dip in the solution completely. (4) Connect the cell to the conductometer and determine the conductance of 0.1 N KCl solution. Using this conductance and specific conductance value at measured room temperature (Table 2), determine the cell constant of the cell. (as shown in the calculations).

Part II: Measurement of conductance of the prepared acetic acid dilutions: (1) Wash three 100 cm^3 standard measuring flasks with conductivity water. Pipette out suggested volume of 0.1N acetic acid in the flasks as given in the Table 1 and dilute each flask with conductivity water up to the mark. Shake the solutions well.

(2) Wash the conductivity cell in a 100 cm^3 beaker with conductivity water carefully and then rinse it with 0.1N acetic acid solution.

(3) Take about 50 - 60 cm^3 of 0.1N acetic acid in the beaker and dip the cell in it to determine the conductance of this solution.

(4) Prepare 0.05N, 0.025N and 0.0125N acetic acid solution from 0.1N acetic acid solution and dilute it with conductivity water to 100 cm^3 in three different numbered standard measuring flasks.

Table: 1 Preparations of acid solutions

Flask number	Normality (N)	Volume of 0.1N Acetic acid in cm^3	Final volume with conductivity water
1	0.05	50.0	100
2	0.025	25.0	100
3	0.0125	12.5	100

Instrumental Analysis (Conductometry)

(5) Wash the conductivity cell and the beaker with conductivity water carefully. (6) Determine the conductance of 0.05N, 0.025N and 0.0125N acid solutions in the same manner. (7) Calculate the dissociation constant for each acid solution as shown in calculations. Show all the calculations systematically.

Observations and Calculations:

Temperature=_____ °C [_____°K].

Part I: Determination of cell constant:

(1) Conductance of 0.1N KCI solution = _____*mhos* [S].

(2) Specific conductance of 0.1N KCI solution at experimental temperature. (Refer to Table 2) = _____*mhos* cm^{-1}[S cm^{-1}]

(3) Cell constant = $\frac{\text{Specific conductance of KCI}}{\text{Conductance of KCI}}$ = _____ cm^{-1}

Part II: Calculation of dissociation constant:

(1) Equivalent conductance at infinite dilution (λ_∞) = 390.7 *mhos* cm^2 eq^{-1}[S cm^2 eq^{-1}].

(2) Cell constant (from Part I) = _____cm^{-1}.

Observation Table:

Concentration g.eq.dm^{-3}	Conductance *mhos* [S]	Specific conductance K of solution *mhos* cm^{-1} [S cm^{-1}]	Equivalent conductance *mhos* cm^2eq^{-1} [S cm^2 eq^{-1}]	Degree of dissociation	Dissociation constant
C	L x 10^{-3} *mhos* or S	K = cell constant * conductance	$(\lambda_c) = \frac{1000 * K}{C}$	$\alpha = \frac{\lambda_\alpha}{\lambda_\infty}$	$K_a = \frac{\alpha^2}{(1-\alpha)}$
0.1					
0.05					
0.025					
0.0125					

Results:

Concentration of solution g.dm^{-3} C	Value of Dissociation constant (K_a)
0.1	
0.05	
0.025	
0.0125	

Conclusions:

Since the values of K_a are fairly constant, the Ostwald's dilution law is verified.

(***Note*:** Dissociation constant of acetic acid at 25ºC or 298K is 1.74 x 10^{-5}).

Instrumental Analysis (Conductometry)

Table: 2 Specific Conductance of 0.1N KCl in aqueous solution in mhos.cm^{-1}

Temperature (℃)	Specific Conductance of 0.1N KCl solution in mhos.cm^{-1} or siemens cm^{-1}
10	0.00932
15	0.01048
20	0.01167
21	0.01197
22	0.01215
23	0.01239
24	0.01264
25	0.01288
26	0.01313
27	0.01337
28	0.01362
29	0.01387
30	0.01412
31	0.01437
32	0.01462
33	0.01488
34	0.01513

35	0.01559

Chemical Kinetics

Experiment No. 1

Aim: To determine the energy of activation of an acid catalysed hydrolysis reaction of methyl acetate. **Theory**: This hydrolysis is a very slow reaction. During its kinetic study it is catalysed by a mineral acid. The reaction is represented as:

$$CH_3COOCH_3 + H_2O \xrightarrow{H^+ \text{ catalyst}} CH_3COOH + CH_3OH$$

In most of the reactions, the reactants have to be supplied a certain minimum amount of energy for the reaction to occur. This energy is generally supplied in the form of heat, due to which the effective collisions between the reactant molecules increase giving rise to increase of energy of the molecule and they form an activated state or activated complex. This is a state of maximum energy and is very unstable. The activated state comprises of a transition state or an intermediate state which immediately gets converted into products.

Reactant must pass through this activated state before the products can be obtained. The minimum energy required for reactants to reach activated state is known as the Energy of activation (E_a).

Specific reaction rate is related the energy of activation the Arrhenius equation

$$K = A e^{-E_a/RT}$$

$$\log_{10} K = \log_{10} A - \frac{E_a}{2.303RT}$$

Where, K: Specific reaction rate, *A*: Constant, Ea: Energy of activation, R: Gas constant & T: Temperature in degree kelvin.

A hydrolysis reaction is studied kinetically at different temperatures and K value at each temperature is determined. A graph of log K is then plotted against 1/T, thus, E_a can be calculated from the slope of such a graph (Slope = - E_a/2.303*R*).

Requirements: 0.5N HCl solution, 0.25N NaOH solution, methyl acetate 1%, phenolphthalein indicator, clean glass stoppered bottles, 5 cm³ and 25 cm³ pipettes, 100 cm³ conical flask, burette, ice pieces etc.

Procedure:

This experiment is carried out in two Sets:

Set I: Hydrolysis at room temperature:

(1) Take 100 cm³ of 0.5N HCl in a clean glass stoppered bottle, and about 05 cm³ of methyl acetate in another bottle. Place both the bottles in a water bath to attain room temperature.

(2) Fill a burette with 0.25N NaOH solution.

(3) At a known time, carefully pipette out and add 5 cm³ of methyl acetate to the bottle containing 100cm³ HCl. Gently mix this reaction mixture and immediately pipette out 5cm³ of it transfer it to a conical flask containing a few ice pieces and a drop of 1% phenolphthalein indicator. (4) Titrate it against 0.25N NaOH solution from the burette. The end point is colourless to light pink. (pink colour should disappear after about 15-20 seconds). ***Note this burette reading as T***. (5) At 5th minute from the zero time pipette out 5 cm³ of the same reaction mixture and transfer it to a flask containing a few ice pieces and a

drop of 1% phenolphthalein indicator.

(6) Titrate this against 0.25N NaOH solution from the burette and record the reading in a tabular form. This is the reading for 5 minutes.

Chemical Kinetics

(7) Similarly titrate 5 cm³ of the reaction mixture at intervals of 10, 15, 20 and 25 minutes from the zero time respectively. Record these readings.

Set II: Hydrolysis at 10°C above room temperature:

(1) Take 100 cm³ of 0.5N HCl in a clean glass stoppered bottle.

(2) Place this bottle in a thermostat maintained at a temperature about 10°C above room temperature. (Thermostat is essentially a water bath which is heated electrically and has provisions for maintaining a constant temperature).

(3) Take 25-30 cm^3 of methyl acetate in a conical flask or a bottle. Stopper it carefully using a cork and place it also in the same thermostat to attain a constant temperature

(4) At a known time, pipette out and add 5 cm³ of methyl acetate (from the flask in the thermostat) to the bottle containing HC1. Note the time of mixing as zero time. Gently mix the contents of the bottle and immediately pipette out 5 cm³ of it transfer it to a conical flask containing a few pieces and a drop of 1% phenolphthalein indicator.

(5) Titrate this against 0.25N NaOH solution from the burette. End point will be from colourless to light pink colour (pink colour should disappear after about 15-20 seconds) ***Note this reading as T_t at respective time in min.***

(6) similarly, titrate 5 cm³ of the reaction mixture at intervals of 5, 10, 15, 20 and 25 minutes from the zero time respectively against 0.25N NaOH solution from the burette. Record these readings in the tabular form.

Observations and Observation Table:

Set I: At room temperature T_1 = _____° C or _____°K, T_∞= _____ cm^3 (Given), a = T_∞- T_0 cm^3

Time (t) min.	Titre / Burette Reading T_t (cm^3)	a = (V_∞ – V_0)	(a-x) = V_∞ – V_t (cm^3)	log(a-x)	V_∞ (V_∞ – V_t)	log V_∞ (V_∞ – V_t)	V_∞ = 2.303 / V_t log V_∞ (V_∞ – V_t)
00	T_0	Zero					
05							
10							
15							
20							

25							
						Mean K_1	

Similarly, prepare the observation Table for Set II and calculate mean K_2 value at temp T_2 i.e., $T_2 = (T_1 + 10)$ K.

Graphs and Calculations:

Chemical Kinetics

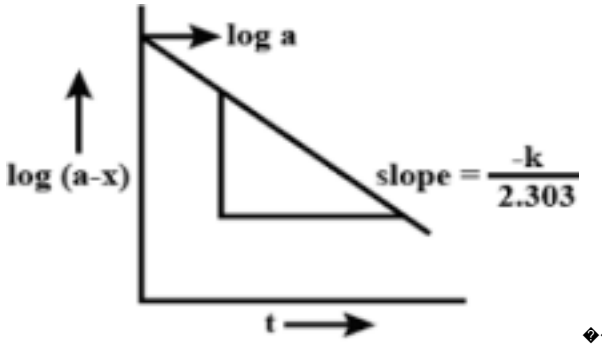
$$k = \frac{2.303}{t} \log \frac{a}{a-x}$$

$$k = \frac{2.303}{t}[\log a - \log(a-x)]$$

$\therefore \log (a-x) = \log a - (k.t / 2.303)$

Thus Plot the graph of log (a-x) against time t for both the Sets and obtain values of *K* from the slope.

Note: Sometimes T_0 need not be taken, but it can be calculated from the formula, $T_0 = 20/21 \times V$ whose V = volume of 0.25N NaOH solution required for neutralisation of 5 cm of 0.5N HCI solution.



$$k = \frac{2.303}{(t_2 - t_1)} \log_{10} \frac{V_2}{V_1}$$

Set No.	*Temperature*		*Specific reaction rate (k) min^{-1}*	
	°C	*°K*	*By calculation*	*By graph*
I : K_1				
II : K_2				

$k = 2.303 \log_{10} \frac{V_2}{V_1}$ where,

$(t_2 - t_1)$

E_a = Energy of activation for the acid catalyzed hydrolysis of methyl acetate. R= Gas constant = 8.314 JK^{-1} $mole^{-1}$

K_1 = Specific reaction rate at temperature T_1 (degrees kelvin) i.e., of Set I K_2 = Specific reaction rate at temperature T_2 (degrees kelvin) i.e., of Set II **Results**:

(1) Values of specific reaction rate at different temperatures. K_1 = *K (room temperature)* =_____ min^{-1}(by calculation) =_____ min^{-1}(by graph)

K_2 = *K (room temperature + 10°C)* =_____ min^{-1}(by calculation) =_____ min^{-1}(by graph)

(2) Energy of activation (E) = _____*J/mole.*

Chemical Kinetics

Experiment No. 2

Aim: To investigate the reaction between $K_2S_2O_8$ and KI with equal initial concentration of the reactants.

Theory: (1) $K_2S_2O_8$ oxidises KI to liberate I_2,

$K_2S_2O_8 + 2KI \; 2K_2SO_4 + I_2$

(2) The progress of the reaction is studied by titrating liberated I_2 against standard $Na_2S_2O_3$ solution using freshly prepared starch as indicator at specific time intervals.

The reaction is

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

(3) The specific reaction rate is determined graphically as well as by using the integrated rate equation. Also, the order of reaction is determined by fractional change method, i.e. by using the equation:

$$\frac{\log a_1 - \log a_2}{\log a_2 - \log a_1} = 1 +$$

where, a_1 represents volume at time t_1, for Set I and a_2 represents volume at time t_2, for Set II.

Requirements: 0.05N $K_2S_2O_8$ solution. 0.05N KI solution, 0.002N $Na_2S_2O_3$, freshly prepared starch indicator, crushed ice, water bath glass stoppered bottles, 10 cm^3 pipette, 100 cm^3 conical flask etc.

Procedure:

Set I:

(1) Carry out the experiment, preparing set as follows:

50 cm^3 of $K_2S_2O_8$ (Reagent stopper Bottle - I)	50 cm^3 of 0.05N KI (Reagent stopper Bottle - II)

(2) Keep both reagent bottles in water bath to attain the room temperature for about five minutes. (***Note***: Level of water in water bath should be more than that of solution present in reagent bottles). (3) Mix both the solutions carefully and note the time of mixing as zero time. Do not remove the reaction mixture bottle from the water bath throughout the experiment.
(4) Fill the burette with 0.002N $Na_2S_2O_3$ solution.
(5) Carry out the experiment by titrating 10 cm^3 of reaction mixture in 100 cm^3 conical flask at intervals of 5, 10, 15, 20, 25 and 30 minutes from the time of mixing (i.e., zero time) against standard 0.002N $Na_2S_2O_3$, solution from the burette using freshly prepared 1- 2 cm^3 starch solution as an indicator and small piece of ice (to arrest the reaction). The end point is from blue to colourless.

Set II:

25 cm^3 of $K_2S_2O_8$ + 25 cm^3 of distilled water (Reagent stopper Bottle – I)	25 cm^3 of 0.05N KI + 25 cm^3 of distilled water (Reagent stopper Bottle – II)

Follow exactly the same procedure as explained in step Nos. 2, 3, 4 and 5 of Set I.
Calculations for initial concentration a and b
Set I:
N_1 : Normality of the $K_2S_2O_8$ taken = 0.05N
V_1 : Volume of $K_2S_2O_8$ taken = 50 cm^3 N, Normality of K_2S_2O8 in the mixture (effective concentration, to be calculated)
V_2 = Total volume of the reaction mixture in Set-I = 100 cm^3

Chemical Kinetics

$\therefore N_1V_1 = N_2V_2$
$0.05 \times 50 = N_2 \times 100$
$N_2 = 0.05 \times 50 / 100$
Effective concentration of $K_2S_2O_8$ in mixture 0.025N
(2) a = V_3, volume of 0.002N $Na_2S_2O_3$, corresponding to $K_2S_2O_8$ in 10 cm^3 of the reaction mixture
$K_2S_2O_8 = Na_2S_2O_3$
Similarly, volume of $Na_2S_2O_3$ corresponding to KI = b = 125 cm^3
Since the concentration of KI and $K_2S_2O_8$, equal in the reaction mixture a = b = 125 cm for Set - I

Set II:
N_1 : Normality of the $K_2S_2O_8$ taken = 0.05N
V_1 : Volume of $K_2S_2O_8$ taken = 25 cm^3
N_2 Normality of K_2S_2O8 in the mixture (effective concentration, to be calculated) V_2 = Total volume of the reaction mixture in Set - II = 100 cm^3
$\therefore N_1V_1 = N_2V_2$
$\therefore 0.05 \times 25 = N_2 \times 100$
$N_2 = (0.05 \times 25) / 100$

Effective concentration of $K_2S_2O_8$ in mixture 0.0125N
(2) a = V_3, volume of 0.002N $Na_2S_2O_3$, corresponding to $K_2S_2O_8$ in 10 cm³ of the reaction mixture
$K_2S_2O_8 = Na_2S_2O_3$
$\therefore N_2V_2 = N_3V_3$
$\therefore 0.0125 \times 10 = 0.002 \times V_3$
$\therefore V_3 = (0.0125 \times 10) / 0.002 = 62.5\ cm^3$
$\therefore a = 62.5\ cm^3$
Similarly, volume of $Na_2S_2O_3$ corresponding to KI = b = 125 cm^3
Effective concentration of $K_2S_2O_8$, in mixture 0.0125N
∴a = b = 62.5 cm³ for Set - II.

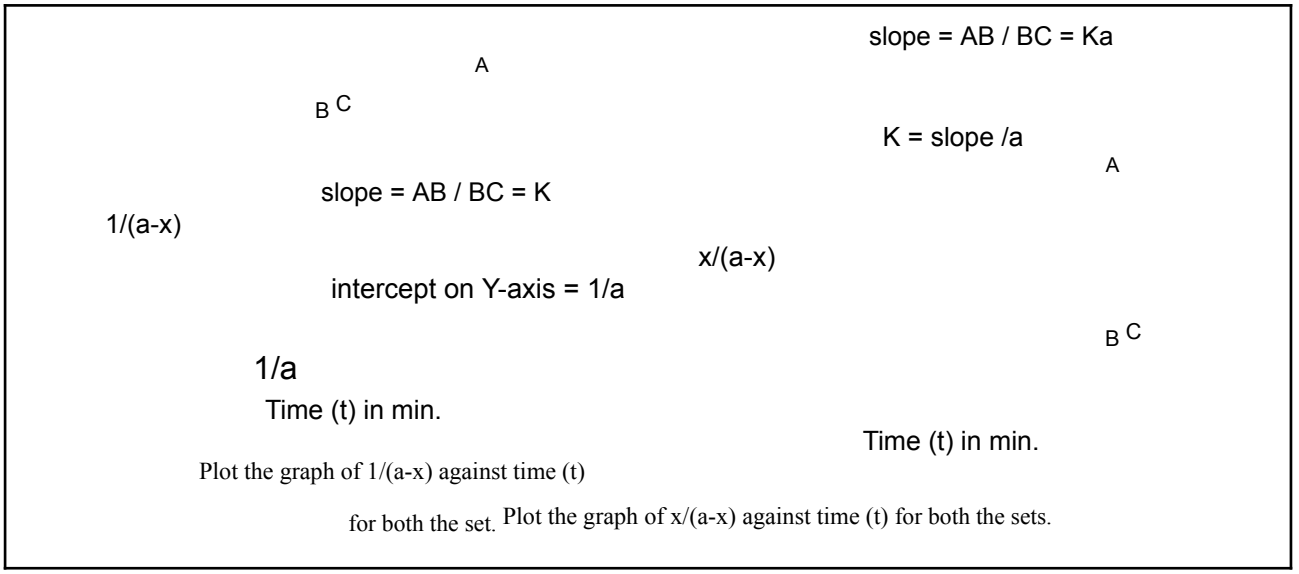
Observations, Observation Table & Calculation:
Set I: temperature = _____ °K, T_∞ = _____ cm^3 (Given), a = b = 125 cm^3

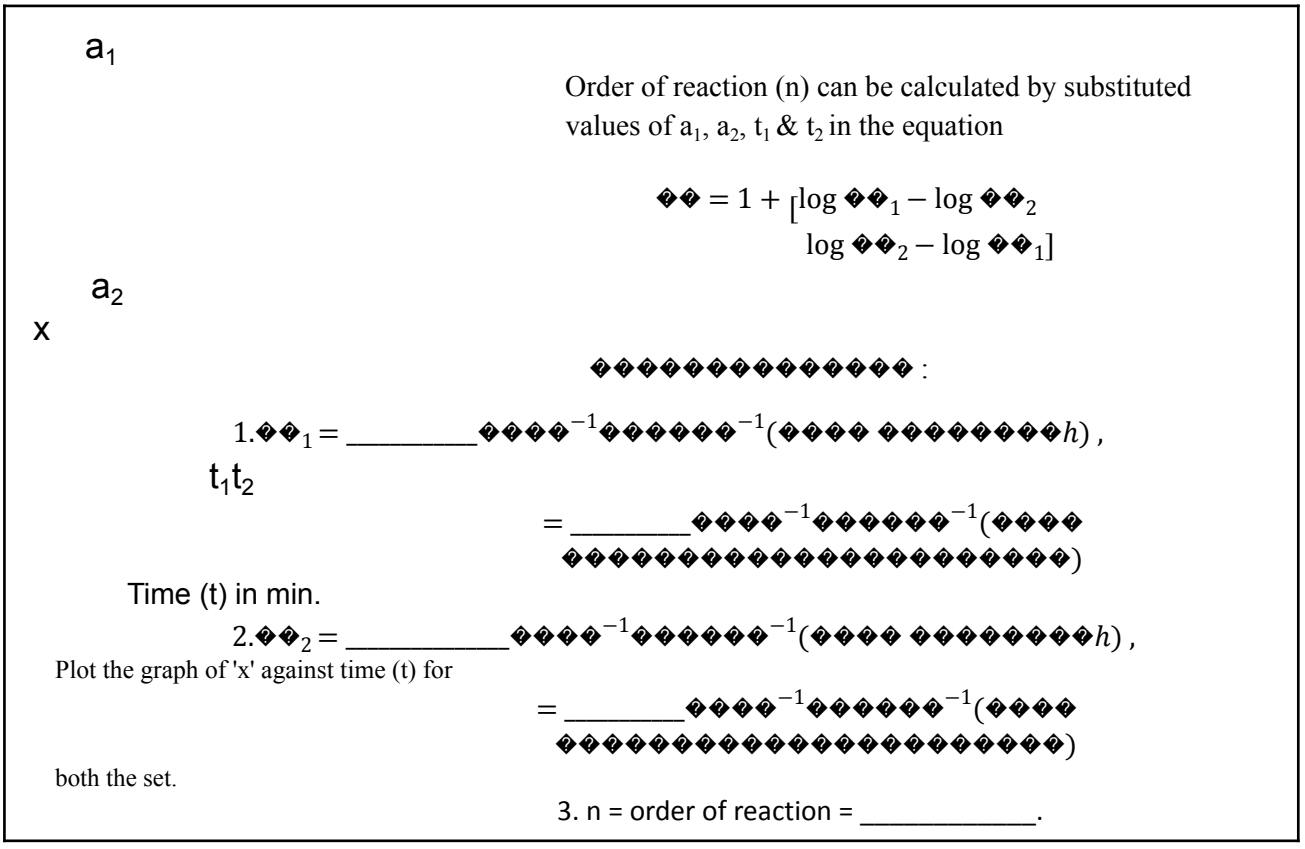
Time (t) min	Titre / Burette Reading x (cm^3)	(a – x)	log (a – x)	1 / (a – x)	$\frac{x}{a(a-x)}$	$\log\frac{x}{(a-x)}$	$k_1 = \frac{1}{t}\log\frac{a}{(a-x)}$
05							
10							
15							
20							
25							
30							
						Mean k_1	

Show the calculation of A systematically (one complete calculation)
Set II: temperature = _____ °K, T_∞ = _____ cm^3(Given), a = b = 62.5 cm^3 (1) a = b = 62.5 cm³ (in terms of 0.002N $Na_2S_2O_3$,).

Chemical Kinetics

(2) Observation Table and Calculation is similar to that shown in Set - 1.
Graph:





Conclusion: The graph of '1 / (a-x)' against 't' **OR'** x / (a-x)' against 't' showing straight line graph indicates the order of reaction is ***second order***.

Instrumental Analysis (Colorimetry)

Determination of Copper

Aim: Colorimetric determination of Copper ions in the given solution by using calibration curve method and calculate the percentage error.

Theory:

When a beam of monochromatic light having intensity I_0 is incident on a transparent medium, a part, I_a of it is absorbed, a part, I_r is reflected and the remaining part, is transmitted I_t. $I_0 = I_a + I_r + I_t$

For a glass-air interface I_r is negligible and therefore, $I_0 = I_a + I_t$ where $I_t/I_0 = T$ called the transmittance, $\log 1/T = \log I_0$. It is called the absorbance or optical density. The relation between absorbance A, concentration, c (expressed in $mol.dm^{-3}$) and path length, t or l (expressed in cm) is given by Beer-Lambert's law,

$A = \log I_0 / I_t = \varepsilon ct$, where, ε is the molar extinction coefficient, t is the path length and is constant for given substance at given wavelength. If the path length, l, is kept constant, then, $A \alpha C$. Hence, a plot of absorbance against concentration gives a straight line.

A series of standard copper sulphate pentahydrate solutions is treated with ammonia to get blue cuprammonium complex, and is diluted to a definite volume. The absorbance of each of these solutions is measured at 590 nm ($\lambda_{max.}$) since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration to get a calibration curve.

The given test solution is treated with strong ammonia and diluted to the same volume as above. The absorbance of this solution at 590 nm is measured and its concentration is determined from the calibration curve.

Requirements: $100cm^3$ standard measuring flasks, $10cm^3$ graduated pipette, burette, colorimeter, ammonia (AR) grade, 1000 ppm copper sulphate solution, distilled water, 590 nm filter etc.

Procedure:

(1) Prepare 1000 ppm solution of copper by dissolving 0.393 g of copper sulphate pentahydrate in 100 cm^3 of distilled water in a 100 cm^3 standard measuring flask.

(2) From the stock solution, pipette out 0, 2, 4, 6, 8, 10 and 12 cm^3 of into seven serially numbered standard measuring flasks.

(3) To each flask, add 5 cm^3 of ammonia (AR) solution and immediately cork the flask. (4) Dilute the solution in flask up to the mark using distilled water (Keep all the flasks in ice bath for 10 min.).

(5) Repeat step Nos. 3 and 4 for the given unknown solution (x).

(6) Measure the absorbance of these all solutions at 590 nm including unknown solution (x).

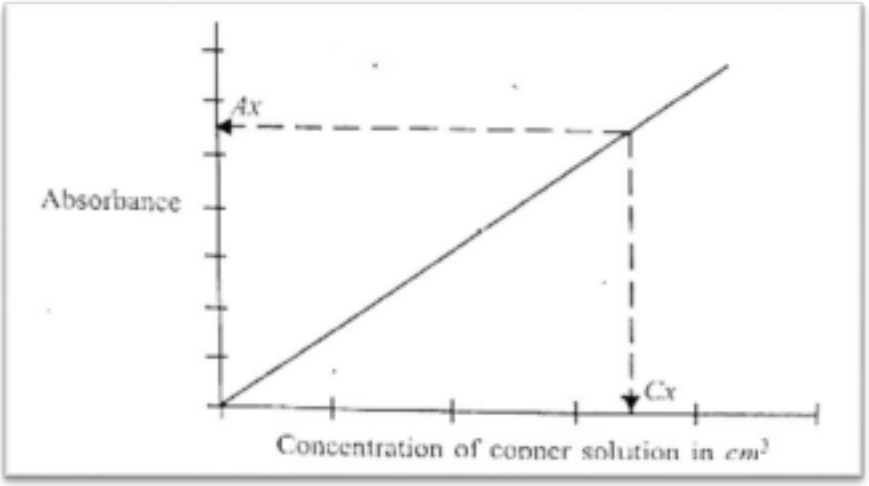
(7) Plot a graph of absorbance (y-axis) against concentration of Copper ion solution (x-axis). **Observation Table:**

Sr.	1000 ppm	Ammonia	Final	Concentration	Absorbance /

No.	Cu(II) solution (cm^3)	Solution (cm^3)	Volume (cm^3)	of Cu(II) in ppm	O.D. at 590 nm
1	0	5	100	0.0 (Blank)	0.0
2	2	5	100	20	
3	4	5	100	40	
4	6	5	100	60	
5	8	5	100	80	
6	10	5	100	100	
7	12	5	100	120	
8	Unknown (Cx)	5	100	C_x =	A_x =

Instrumental Analysis (Colorimetry)

Graph:
Plot a graph of absorbance against concentration.



Calculations:
From graph of concentration of Copper (II) in unknown solution = _____A ppm.
i.e. concentration of Copper (II) in unknown solution (x) = _____A ppm The given concentration of Unknown solution (x) = _____B ppm (Given)
Percentage Relative error = $|^{A-B}$

$_{\diamond\diamond}|$ x 100 = _____ %.

Results:
(1) Concentration of Copper (II) ions in given solution (A) = _____ ppm.
(2) Percentage Relative error = _____ %

Instrumental Analysis (Colorimetry)

Estimation of Ferrous ions

Aim: Colorimetric estimation of ferrous ions in the given solution by using calibration curve method and calculate percentage error.

Theory: A series of standard ferrous ammonium sulphate solutions is treated with 1, 10 phenanthrolline in presence of sodium acetate and hydroxyl ammine hydrochloride to get brick red colored complex, and is diluted to a definite volume. The absorbance of each of these solutions is measured at 520 nm since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against concentration to get a calibration curve.

The given test solution is treated in similar fashion and diluted to the same volume as above. The absorbance of this solution at 520 nm is measured and its concentration is determined from the calibration curve.

Requirements: 100 cm^3standard measuring flasks, 10cm^3graduated pipette, burette, colorimeter, 2M sodium acetate, 10% hydroxyl ammine hydrochloride, 0.25 % 1, 10 phenanthrolline solution, 250 ppm ferrous ion solution, distilled water, 520 nm filter etc.

Procedure:

(1) Prepare 250 ppm solution of ferrous ion by dissolving 0.216 g of ferrous ammonium sulphate in 100 $cm^3$of distilled water in a 100 cm^3standard measuring flask (Stock Solution). (2) From the stock solution, pipette out 0, 1, 2, 3, 4 and 5 $cm^3$of into six serially numbered standard measuring flasks.

(3) To each flask, add 10 cm^3 hydroxyl ammine hydrochloride, 1 cm^3 of sodium acetate solution and 10cm^3 1, 10 phenanthrolline immediately.

(4) Dilute the solution in flask up to the mark using distilled water.

(6) Treat the given unknown solution (x) with same step Nos. 3 and 4.

(7) Measure the absorbance of these all solutions including (x) at 520 mm.

(8) Plot a graph of absorbance (y - axis) against concentration of ferrous ions (x - axis).

Observation Table:

Sr. No.	250 ppm Fe (II) ion Solution (cm^3)	10% Hydroxyl Ammine Hydrochloride	2M Sodium Acetate	0.25 % 1,10 Phenanthroline	Total Volume	Concentration in ppm	Absorbance / O.D. at 520 mm
1	0	10	1	10	100	0.0 (Blank)	0.0
2	1	10	1	10	100	2.5	
3	2	10	1	10	100	5.0	
4	3	10	1	10	100	7.5	
5	4	10	1	10	100	10.0	
6	5	10	1	10	100	12.5	
7	X	10	1	10	100	Unknown (C_x) =	A_x =

Graph:

From graph of concentration of Fe(II) ions in unknown solution = _____ A ppm. i. e. concentration of Fe(II) ions in 10 cm^3 of unknown solution = _____ A ppm Concentration of Fe(II) ions in 100 cm^3 of unknown solution = 10 A ppm The given concentration of Unknown solution = _____ B ppm (Given)

Calculation:

Percentage Relative error = $|^{A-B}$

$$\text{◆◆}| \times 100 = \text{\_\_\_\_\_}\ \%.$$

Results:

(1) Concentration of Fe(II) ions in given solution = _____ ppm.

(2) Percentage Relative error = _____ %.

Gravimetric Analysis

Estimation of SO_4^{2-} ions as $BaSO_4$

Aim: Gravimetric estimation of sulphate ions using Barium as precipitant.

Theory: In gravimetric analysis an ion under estimation is converted to precipitate of known composition. It can be dried or ignited. From the weight of precipitate, quantity of ion under analysis can be determined.

Requirements: 10% $BaCl_2$ solution sample, $Na_2SO_4.10H_2O$, 250 cm^3 beaker, watch glass, glass rod, whatmann filter paper No. 42, water bath, distilled water, 100 cm^3standard measuring flask, 250 cm^3 beaker, 25 cm^3pipette etc.

Procedure:

(1) Dilute the given $Na_2SO_4.10H_2O$ solution 100 cm^3 in a standard measuring flask with distilled

water.

(2) Pipette out 50 cm^3 of this solution in 250 cm^3 breaker. Add 2 test tubes of distilled water and warm the solution.

(3) Add dropwise 30 cm^3 of 10% barium chloride solution to the beaker with constant stirring, to precipitate SO_4^{2-} ions as $BaSO_4$.

(4) Digest the precipitate on a boiling water bath for 30 minutes.

(5) Filter the precipitate through previously-weighed Whatman filter paper No. 42. (W_1 g) Wash the precipitate with distilled water till the filtrate is free from Cl and SO_4^{2-} ions (6) Dry the filter paper along with the precipitate on a drying cone.

(7) Cool it and weigh the precipitate along with the filter paper (W_2 g).

Reaction:

$$BaCl_2 + Na_2SO_4 \rightarrow BaSO_4 + 2NaCl$$

Observations:

(1) Weight of filter paper = _____ (W_1) g.

(2) Weight of filter paper + precipitate ($BaSO_4$) = _____ (W_2) g.

(3) Weight of precipitate ($BaSO_4$) = $W_2 - W_1$ = _____(x) g.

Calculations:

Let the weight of residue ($BaSO_4$) be (x) g.

Now 50 cm^3 of the diluted solution gave x g of $BaSO_4$

∴100 cm^3 of the diluted solution gave 2x g of $BaSO_4$

$BaSO_4 = SO_4^{2-} = Na_2SO_4 = Na_2SO_4 .10H_2O$

233.36 g of $BaSO_4$ = 96 g of SO_4^{2-} = 142 g of Na_2SO_4 = 322 g of $Na_2SO_4 .10H_2O$

2x g of $BaSO_4 = \frac{96 \times 2x}{233.36}$ = A g of Barium & $\frac{142 \times 2x}{233.36}$ = B g of Na_2SO_4 or $\frac{322 \times 2x}{233.36} = Na_2SO_4 .10H_2O$

Gravimetric Analysis

Percentage Error:

$$\% \text{ Error} = \left| \frac{\text{Experimental value} - \text{Accepted / Theoretical value}}{\text{Accepted value}} \right| \times 100$$

∴ % Error of SO_4^{2-} in $BaSO_4 = \left| \frac{A - y}{\diamond\diamond} \right| \times 100$ = _____ %

% Error of Na_2SO_4 / $Na_2SO_4 .10H_2O$ in $BaSO_4 = \left| \frac{B - z}{\diamond\diamond} \right| \times 100$ = _____ %

['y' (Theoretical accepted value) of SO_4^{2-} ions in g and 'z' (Theoretical accepted value of Na_2SO_4 /

Na_2SO_4 .$10H_2O$ in g will be provided)].

Results:

(1) Percentage error of SO_4^{2-} = _____ %.

(2) Percentage error of Na_2SO_4 / $Na_2SO_4.10H_2O$ = _____ %.

Gravimetric Analysis

Estimation of Ba^{+2} ions as $BaCrO_4$

Aim: Gravimetric estimation of barium ions using K_2CrO_4 as precipitant.

Theory: In gravimetric analysis, ion under estimation is converted into precipitate of known composition. The precipitate after digestion can be dried or ignited and weighed. From the weight of precipitate, the quantity of ion under estimation can be determined. Gravimetric analysis can be further sub-divided into direct and indirect gravimetric analysis.

In direct gravimetric analysis, a sparingly soluble salt is precipitated from the solution by using reagent followed by digestion, drying or ignition and weighing. This experiment is an example of direct gravimetric analysis, where barium is precipitated as barium chromate.

Requirement: 250 cm³ beaker, 100 cm³ standard measuring flask, glass rod, Whatman filter paper No. 42, $BaCl_2$ solution, 1% Methyl red indicator, 10% K_2CrO_4 solution, 2N CH_3COOH solution, 25 cm³ pipette, distilled water, cone etc.

Procedure:
(1) Dilute the given $BaCl_2$ solution to 100 cm³ in a standard measuring flask using distilled water. (2) Pipette out 50 cm³ of this solution in 250 cm³ beaker. Add 2 test tubes of distilled water and warm the solution to around 60°C.
(3) Add 2 drops of 1% methyl red indicator, add 2 N acetic acid solution dropwise till the colour of solution changes to red.
(4) Add dropwise 30 cm³ of 10 % potassium chromate solution to the beaker with constant stirring to precipitate Ba^{2+} as $BaCrO_4$.
(5) Digest the precipitate on a boiling water bath for 30 minutes.
(6) Filter the precipitate through previously weighed (say W_1 g) Whatman filter paper No. 42. Wash with distilled water till the colour of filtrate turns from yellow to colourless. (7) Dry the filter paper along with precipitate on a drying cone.
(8) Weigh the precipitate of $BaCrO_4$ along with the filter paper. (say W_2 g).

Reaction: $BaCl_2 + K_2CrO_4 \rightarrow BaCrO_4 + 2KCl$

Observations:
(1) Weight of filter paper = _____ (W_1) g.
(2) Weight of filter paper + precipitate ($BaCrO_4$) = _____ (W_2) g.
(3) Weight of precipitate ($BaCrO_4$) = $W_2 - W_1$ = _____ (x) g.

Calculations:
Let the weight of $BaCrO_4$ residue be x g.
Now 50 cm³ of the diluted solution gave x g of $BaCrO_4$
∴100 cm³ of the diluted solution gave 2x g of $BaCrO_4$
$BaCrO_4 = Ba^{2+}$ ions = $BaCl_2.2H_2O$
253.36 g of $BaCrO_4$ = 137.36 g of Barium = 244.36 g of $BaCl_2.2H_2O$
2x g of $BaCrO_4$ = $\frac{137.36 * 2x}{253.36}$ = A g of Barium & $\frac{244.36 * 2x}{253.36}$ = B g of $BaCl_2.2H_2O$

Percentage Error:
% Error = $\left|\frac{\text{Experimental value} - \text{Accepted (Theoretical) value}}{\text{Accepted value}}\right| \times 100$

∴ % Error of Ba^{2+} ions in $BaCrO_4$ = $\left|\frac{A - y}{\text{••}}\right| \times 100$ = _____ %

Gravimetric Analysis

% Error of $BaCl_2.2H_2O$ in $BaCrO_4$ = $\left|\frac{B - z}{\text{••}}\right| \times 100$ = _____ %
['y' (Theoretical accepted value) of Ba^{2+} ions in g and 'z' (Theoretical accepted value of $BaCl_2.2H_2O$ in g will be provided)].

Results:

(1) Percentage error of Ba (II) = _____ %.

(2) Percentage error of $BaCl_2.2H_2O$ = _____ %.

Tools of Analytical Chemistry – I

(A) ANALYTICAL GLASSWARES LIKE BURETTES, PIPETTES, STANDARD FLASKS, SEPARATING FUNNELS

Burettes:

Fig. 8.1

Burette

A burette is a graduated glass tube with a stopcock at the bottom, used for precise dispensing of liquids, especially in titrations. The graduations along the tube allow for accurate measurement of the dispensed volume. By carefully controlling the stopcock, chemists can add titrant drop by drop to reach the endpoint of a reaction. Burettes come in various sizes and types, including volumetric, piston (digital), and electronic, each offering different levels of precision and ease of use for quantitative chemical analysis.

Pipettes:

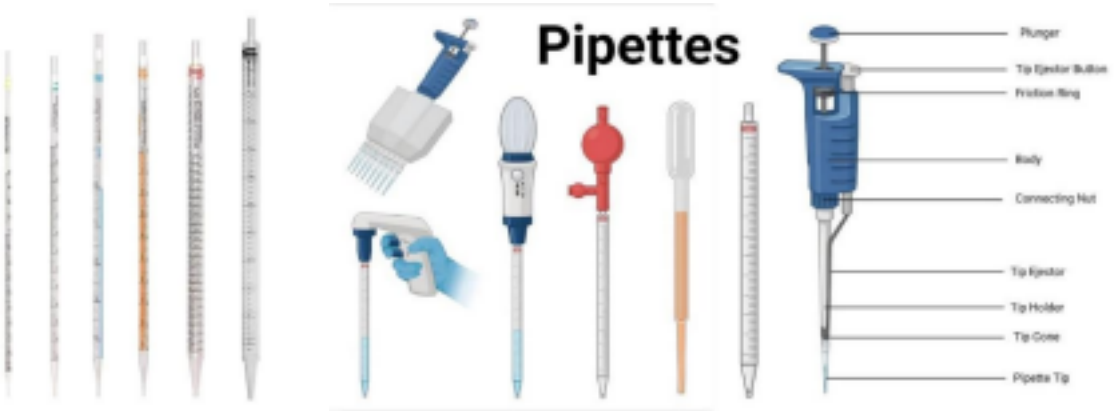


Fig. 8.2 Pipette A pipette is a laboratory instrument used to accurately transfer a specific volume of liquid. They come in various forms, each designed for different levels of precision and volume ranges. **Types of Pipettes:**

· **Volumetric Pipettes (or Bulb Pipettes):** These are designed to deliver a single, highly accurate volume. They have a bulb in the middle and a single calibration mark on the narrow neck. They are used when precise volumes are critical, such as in preparing standard solutions for titrations.

· **Graduated Pipettes (or Measuring Pipettes):** These are straight tubes with volume markings (graduations) along their length, allowing for the dispensing of variable volumes. Common types include:

a. Mohr Pipettes: Calibrated between two marks on the stem. The volume is measured between these marks.

b. Serological Pipettes: Graduated to the tip, meaning the last drop is intended to be blown out using a pipette bulb or controller to deliver the full volume. They often have etched rings near the top to indicate they are "blow-out" pipettes.

· **Micropipettes:** These are used to measure and dispense very small volumes, typically in the

microliter (µL) range (1 µL = 0.001 mL). They are commonly used in molecular biology, biochemistry, and biotechnology labs and often employ disposable tips to prevent contamination. Micropipettes are usually adjustable within a specific volume range.
· **Pasteur Pipettes (or Droppers):** These are long, slender glass tubes tapered to a narrow opening and often have a bulb at the top. They are used for transferring small volumes of liquids without a specific volume requirement, typically drop by drop.
How to Use a Pipette (General Principles):
1. **Selection:** Choose the appropriate type and size of pipette for the required volume and accuracy. 2. **Aspiration:** Use a pipette bulb, pump, or controller to draw the liquid into the pipette, carefully drawing it up to the desired mark (meniscus level for volumetric and graduated pipettes). Ensure there are no air bubbles in the liquid column.
3. **Delivery:** Allow the liquid to drain out of the pipette vertically into the receiving container. For volumetric pipettes, allow the liquid to drain under gravity, and the small amount remaining in the tip should not be blown out unless specifically instructed. For "blow-out" serological pipettes, gently blow out the remaining liquid after the free flow stops. For micropipettes, the plunger mechanism controls aspiration and dispensing.
Importance:
Pipettes are essential tools in various scientific disciplines for accurately measuring and transferring liquids, which is crucial for the reliability and reproducibility of experiments and analyses. **Standard flasks:**

Fig. 8.3 Standard flasks
A **standard flask** in chemistry most commonly refers to a **volumetric flask**. This is a piece of laboratory glassware designed to contain a precise volume of liquid at a specific temperature, indicated by a calibration mark on its long, narrow neck.

Key characteristics and uses:
· **Accurate Volume Measurement:** The primary purpose is to prepare solutions with highly accurate concentrations for tasks like titrations and standard solutions.

· **Calibration Mark:** A single etched line on the neck indicates the exact volume when the meniscus of the liquid aligns with it.
· **Various Sizes:** Available in a wide range of volumes, from a few milliliters to several liters.
· **Material:** Typically made of glass (borosilicate for better chemical and thermal resistance) or sometimes plastic. Amber-colored flasks are used for light-sensitive substances. ·

Stopper: Fitted with a stopper (glass or plastic) to prevent evaporation and contamination. · **Classes:** Volumetric flasks come in different accuracy classes (e.g., Class A for high precision, Class B for general use).

While "standard flask" often implies a volumetric flask, the term could broadly refer to other common laboratory flasks used as standards in certain procedures or as standard storage containers (like reagent flasks). However, in the context of precise measurements, the **volumetric flask** is the quintessential "standard flask."

(B) WEIGHING TOOLS SUCH AS TWO PAN BALANCE AND MONOPAN BALANCE, DIGITAL BALANCES:

Balances are essential tools in chemistry for accurately determining the mass of substances. The type of balance used depends on the required precision and the amount of material being weighed. Here are some common types:

Types of Balances:

· **Analytical Balances:** These are highly precise, measuring mass to a high degree of accuracy (typically to 0.0001 g or even better). They usually have a draft shield to protect the weighing pan from air currents and dust. Analytical balances are crucial for quantitative analysis, preparing standard solutions, and when high accuracy is paramount.

· **Precision Balances (Top-Loading Balances):** Less precise than analytical balances (readability from 0.1g to 0.001g) but have a higher capacity. They are faster and more convenient for routine weighing tasks where extreme accuracy isn't required.

· **Microbalances and Ultra-microbalances:** These are used for measuring extremely small masses in the microgram (µg) range or even smaller. They are highly sensitive and used in specialized applications like particle size analysis and filter weighing.

· **Triple-Beam Balances:** Commonly found in educational settings, these mechanical balances use a series of sliding weights along beams to determine mass. They are less precise than electronic balances but are durable and do not require a power source.

· **Moisture Analysers:** Specialized balances that determine the moisture content of a sample by measuring the mass loss upon heating.

Uses of Balances in Chemistry:

· **Quantitative Analysis:** Accurate weighing is fundamental for determining the amounts of reactants and products in chemical reactions.

· **Preparing Solutions:** Precisely weighing solutes is necessary to create solutions of specific concentrations (molarity, molality, etc.).

· **Gravimetric Analysis:** A quantitative technique where the amount of an analyte is determined by measuring the mass of a precipitate or residue.

· **Titrations:** Accurate weighing of primary standards is crucial for standardizing titrant solutions. · **Research and Development:** Balances are used for measuring reactants, products, and samples in various research experiments.

· **Quality Control:** In industrial settings (e.g., pharmaceutical, food), balances ensure the correct amounts of ingredients and the quality of final products.

· **Density Determination:** Precise mass measurements are needed to calculate the density of substances.

Calibration:

To ensure the accuracy of measurements, balances must be calibrated regularly using standard weights with known masses. Calibration verifies that the balance readings are consistent with these known standards and adjustments are made if necessary. Calibration

should be performed: · Upon initial installation.
· After moving the balance.
· Regularly, according to the manufacturer's recommendations and the frequency of use. · If there are concerns about the accuracy of the readings.
Proper use and regular calibration are essential for obtaining reliable and accurate mass measurements in any chemistry laboratory.

Two pan Balances:

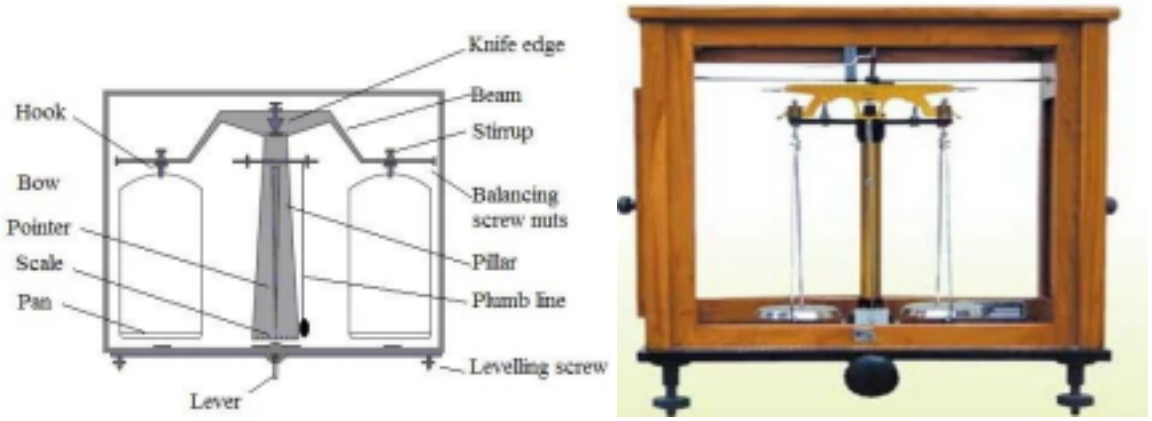


Fig. 8.4 Two-pan balance

Two-pan balance, also known as a **beam balance** or **equal-arm balance**, is a traditional type of weighing scale that compares the mass of an unknown object to a known mass. It consists of a horizontal beam balanced on a central pivot point (fulcrum). Two pans are suspended from the ends of the beam. **How it works:**
The object to be weighed is placed on one pan, and known standard weights are added to the other pan until the beam is level or balanced. At the point of balance, the mass of the object is equal to the total mass of the standard weights. Some two-pan balances also have graduated beams with sliding riders for finer adjustments to achieve balance.
Key features:
· **Direct comparison:** It directly compares the mass of the unknown with known masses, offering a fundamental method of measurement.
· **Mechanical:** It operates mechanically and does not require a power source. · **Durability:** Often robust and can last for a long time.
· **Educational value:** Clearly demonstrates the principle of mass comparison. While largely superseded by more convenient and often more precise electronic balances in modern laboratories, two-pan balances are still used in some educational settings and for specific applications where their fundamental principle is advantageous or where electronic balances are not practical. They were historically crucial for accurate measurements in chemistry and other sciences.

Digital balances:

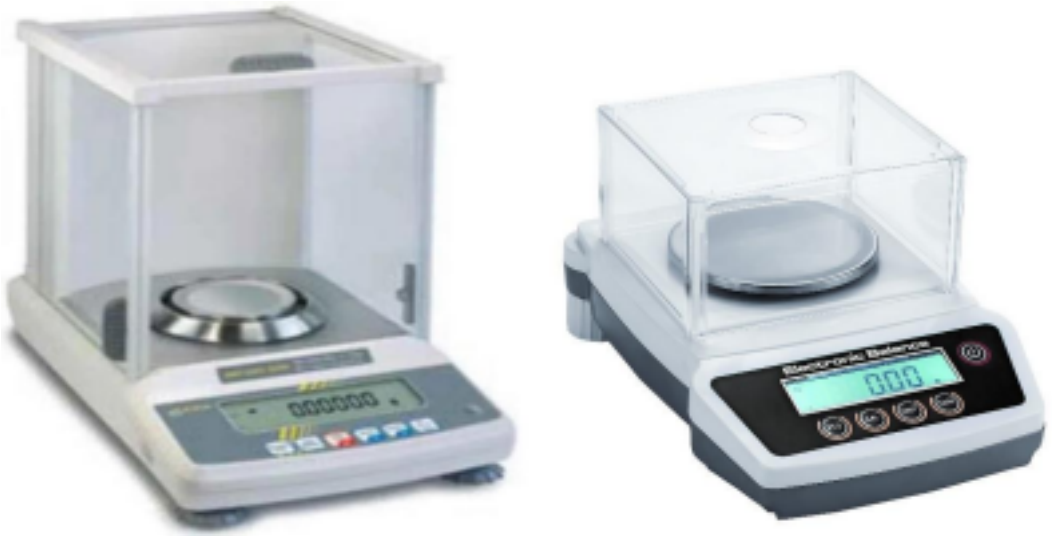


Fig. 8.5

Digital balance

Digital balances, also known as electronic balances or scales, are instruments used to accurately measure the mass of an object and display the result on a digital screen. They have largely replaced traditional mechanical balances in many applications due to their ease of use, speed, and often higher precision. **Principle of Operation:**
Digital balances work by measuring the force exerted by an object's mass, typically using a **load cell** based on **electromagnetic force compensation** or **strain gauge** technology.
1. **Load Cell:** When an object is placed on the weighing pan, it exerts a downward force due to gravity. The load cell, a transducer, detects this force and converts it into an electrical signal. 2. **Electromagnetic Force Compensation:** In some high-precision balances, an electromagnetic
force is generated to counteract the force of the unknown mass, keeping the weighing pan in a stable position. The current required to produce this counteracting force is directly proportional to the mass and is measured and converted into a digital reading.
3. **Strain Gauge:** In other balances, the force from the object deforms a strain gauge (a thin wire or semiconductor). This deformation causes a change in the electrical resistance of the strain gauge, which is measured and converted into a weight reading.
4. **Microprocessor and Display:** The electrical signal from the load cell is processed by a microprocessor, which converts it into a mass value based on calibration data. This value is then displayed on a digital screen, often with the option to show the mass in various units (e.g., grams, milligrams, ounces).
5. **Tare Function:** Most digital balances have a "tare" function that allows you to zero the balance with a container on the pan, so you can measure the net weight of the contents only.
Types of Digital Balances:
Digital balances come in various types, each designed for specific applications and offering different levels of precision and capacity:
· **Analytical Balances:** Offer very high precision (readability down to 0.0001 g or 0.1 mg) and are used for quantitative chemical analysis and research. They often have a draft shield to minimize the effects of air currents.

· **Precision Balances (Top-Loading Balances):** Provide good accuracy (readability from 0.1

g to 0.001 g) and higher capacity than analytical balances, suitable for routine lab work and industrial applications.
· **Microbalances and Ultra-microbalances:** Used for measuring extremely small masses (microgram range or lower) with very high sensitivity.
· **Compact Balances:** Portable and lightweight, used for basic weighing tasks where high precision is not critical.
· **Industrial Balances:** High-capacity balances designed for weighing larger quantities of materials in industrial and production settings.
· **Moisture Analysers:** Specialized balances that determine the moisture content of a sample by measuring mass loss upon heating.

Accuracy of Digital Balances:

The accuracy of a digital balance refers to how close its measurement is to the true value. It is influenced by factors such as:
· **Calibration:** Regular calibration with standard weights is crucial to ensure accuracy. · **Environmental Conditions:** Temperature fluctuations, air currents, and vibrations can affect readings.
· **Linearity:** The ability of the balance to provide accurate readings across its entire weighing range.
· **Repeatability:** The balance's ability to provide the same reading when the same mass is weighed multiple times.
· **Readability (Resolution):** The smallest increment of mass that the balance can display. While high readability doesn't guarantee accuracy, it indicates the level of detail in the measurement. Digital balances are indispensable tools in modern chemistry labs, providing rapid, convenient, and often highly accurate mass measurements for a wide range of applications.

(C) INCINERATION DEVICES: BURNERS

Burners are essential components of some **incineration devices**, providing the heat source necessary to initiate and sustain the combustion process. In the context of incinerators, burners serve several critical functions:
· **Start-up:** Burners are used to preheat the incineration chamber to the required ignition temperature before waste material is introduced.
· **Auxiliary Fuel:** They can supply additional heat during the incineration process, especially when the waste has a low calorific value or high moisture content, ensuring complete combustion. · **Temperature Control:** Burners help maintain the desired operating temperature within the incinerator to optimize combustion efficiency and minimize the formation of harmful emissions. · **Post-Combustion:** In some incinerator designs with secondary combustion chambers, burners ensure the complete oxidation of unburnt gases and particulate matter from the primary chamber. **Types of Burners Used in Incineration:** Incinerators can utilize various types of burners depending on the fuel source (gas, oil, or dual fuel), the size and design of the incinerator, and the specific requirements of the waste being incinerated. Some common types include:
· **Gas Burners:** These are often used when natural gas or propane is readily available, offering clean and efficient combustion.
· **Oil Burners:** Used when liquid fuels like diesel or heavy oil are the primary or auxiliary fuel source. These burners atomize the oil for efficient burning.
· **Dual Fuel Burners:** Offer flexibility by being able to operate on either gas or oil, providing a backup fuel option.

· **High-Temperature Burners:** Designed to achieve and sustain very high temperatures

required for the incineration of specific hazardous wastes.
· **Laminar Air Flow Burners:** Can be used for cleaner combustion with reduced NOx emissions in certain applications.
The design and control of the burners are crucial for the overall efficiency and environmental performance of the incineration device. Proper burner operation ensures thorough waste destruction and minimizes the release of pollutants into the atmosphere.

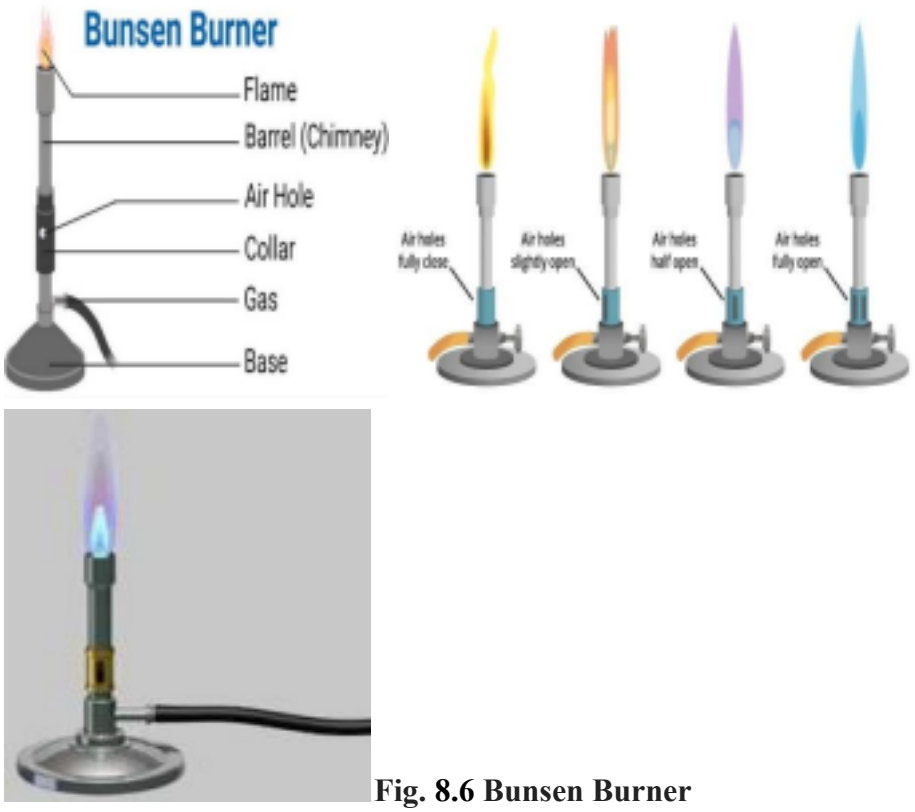


Fig. 8.6 Bunsen Burner

One of the most familiar apparatuses in the chemical laboratory is the Bunsen burner. The simple Bunsen burner consist mainly of three parts: (1) A base provided with a side tube and with a spud, (2) A metal barrel with a hole, (3) A metal cover which serve as an air regulator. Two kinds of flames can be obtained.

(1) Luminous: It is produced when the air hole is closed. (Yellow flame).
(2) Non-luminous: It is produced when the air hole is open and air supply is sufficient. (blue flame). **Incinerators:**
Incinerators are basically used to heat the residue or the precipitate obtained during gravimetric estimation, Incinerators are heaters having the facility to heat the silica crucible containing residue/Precipitate along with or without filter paper. After heating the residue in the incinerator at required temperature to convert it to known composition of the sample to be estimated.
In chemistry, **incinerators** are specialized devices used for the controlled burning of chemical waste. This process is employed to:
· **Destroy hazardous and toxic substances:** High temperatures break down complex and dangerous chemical compounds into less harmful substances like carbon dioxide and water. · **Reduce waste volume:** Incineration significantly decreases the volume and mass of chemical waste, minimizing disposal needs.
· **Treat various waste types:** Incinerators can handle solid, liquid, and gaseous chemical wastes, including solvents, contaminated materials, and by products.
· **Recover energy (in some cases):** The heat generated during incineration can sometimes be harnessed to produce steam or electricity.
Types of incinerators used for chemical waste include:

· **Rotary Kiln Incinerators:** Versatile for various waste types (solid, liquid, sludge) and offer good mixing and residence time.

· **Liquid Injection Incinerators:** Designed for burning pumpable liquid wastes through atomizing nozzles.
· **Fluidized Bed Incinerators:** Efficient for specific waste streams by suspending waste particles in a hot, turbulent bed of inert material.
Modern chemical incinerators are equipped with sophisticated air pollution control systems to remove harmful emissions like particulate matter, acid gases, and heavy metals, ensuring environmental safety. They are crucial for managing hazardous chemical waste streams from laboratories, industrial processes, and environmental remediation efforts.
Muffle Furnace:

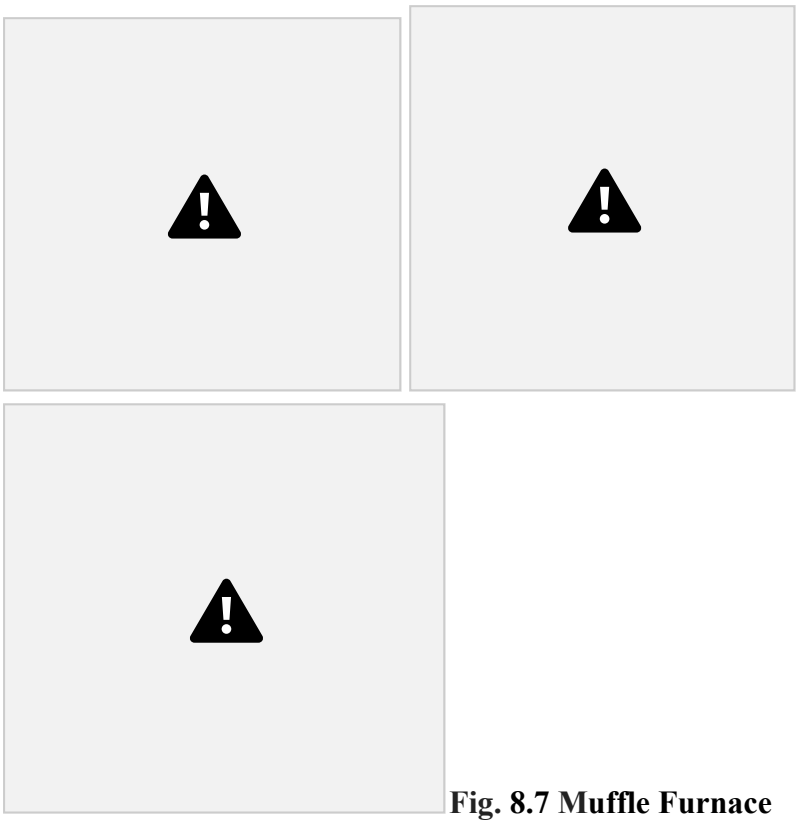


Fig. 8.7 Muffle Furnace

A **muffle furnace** is a type of furnace, crucial in chemistry and other scientific fields, designed to heat materials to high temperatures **without direct contact with a flame or combustion products**. The sample is placed inside an enclosed chamber, or "muffle," which is heated externally by heating elements. This indirect heating ensures a clean and controlled thermal environment, preventing contamination of the sample by ash or combustion gases.
Key features of a muffle furnace:
· **Isolated Heating Chamber:** The defining characteristic is the muffle, which isolates the sample. · **High Temperature Capability:** Muffle furnaces can reach temperatures ranging from a few hundred degrees Celsius up to **1800°C or even higher**, depending on the model and heating elements. Standard muffle furnaces typically operate between **300°C and 1200°C**. · **Temperature Control:** Equipped with precise digital controllers for setting and maintaining the desired temperature, often with programmable ramping and soaking cycles.
· **Heating Elements:** Typically, electric resistance wires (like Kanthal) or silicon carbide (SiC) rods are used as heating elements surrounding the muffle.
· **Insulation:** High-quality refractory materials insulate the chamber to ensure efficient

heating and minimize heat loss.

Common uses of muffle furnaces in chemistry include:

· **Ashing:** Determining the non-combustible inorganic content (ash) of a sample by burning off organic matter. This is vital in food chemistry, environmental analysis, and material science. · **Thermogravimetric Analysis (TGA):** Studying the thermal stability and composition of materials by monitoring their mass change as a function of temperature. · **Heat Treatment:** Annealing, sintering, and tempering of various materials. · **Drying:** Removing moisture from samples.

· **Ignition of precipitates:** In gravimetric analysis, precipitates are often ignited in a muffle furnace to obtain a stable, weighable form.

· **Fusion reactions:** Melting samples with a flux for subsequent chemical analysis (e.g., in X-ray fluorescence).

· **Materials research:** Studying the behaviour and properties of substances at high temperatures. · **Drug testing:** Analysing the stability and composition of pharmaceuticals.

In essence, the muffle furnace is a versatile piece of equipment in a chemistry lab, providing a controlled high-temperature environment for a wide range of analytical and preparative procedures.

(D) DRYING DEVICES: HOT AIR OVEN, MICROWAVE OVEN, DESICATORS, VACUUM DESSICATORS:

Hot Air Ovens:

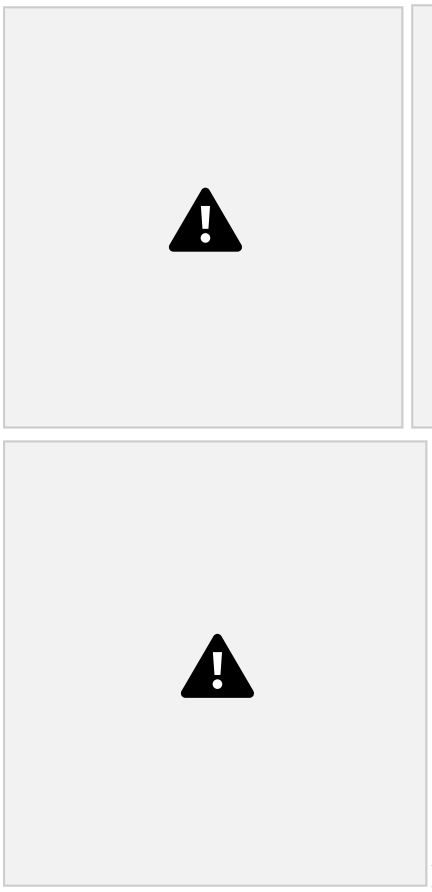


Fig. 8.8 Hot Air Oven

Hot air ovens in chemical analysis use dry heat for essential tasks like drying glassware and chemical samples to ensure accurate quantitative results by removing moisture. They also

sterilize certain heat stable materials and heat samples for specific analyses. Both gravity and forced convection ovens provide controlled temperatures, crucial for reliable outcomes. Vacuum ovens cater to heat-sensitive substances. These ovens are indispensable for maintaining sample integrity and equipment cleanliness in chemical laboratories.

Microwave ovens:

Fig. 8.9 Microwave Ovens

Microwave ovens have become increasingly valuable tools in chemical analysis due to their ability to provide **rapid and efficient heating** through the direct interaction of microwave energy with polar molecules and ions within a sample. This offers several advantages over traditional heating methods. **Key applications in chemical analysis include:**

· **Sample Digestion:** Microwave digestion is a widely used technique for preparing various

sample matrices (e.g., food, environmental samples, polymers) for elemental analysis. Using strong acids in closed vessels under microwave irradiation significantly accelerates the breakdown of the matrix, leading to faster and more complete digestion while minimizing analyte loss and contamination.

· **Solvent Extraction:** Microwaves can enhance the efficiency of solvent extraction by heating the solvent and sample directly, leading to faster extraction rates and reduced solvent consumption. This is used for extracting analytes from solids or liquids.

· **Drying:** Microwave ovens can be used for rapid drying of samples, often more quickly than conventional ovens. However, careful control is needed to prevent overheating or decomposition.

· **Ashing:** Microwave ashing offers a faster alternative to traditional muffle furnace ashing for determining the inorganic content of samples. Specialized microwave muffle furnaces provide efficient and uniform heating.

· **Chemical Synthesis:** While more related to chemical synthesis than strictly analysis, microwave heating accelerates reaction rates, improves yields, and can enable novel reaction pathways, which can be crucial in preparing standards or labelled compounds for analysis.

Advantages of using microwave ovens in chemical analysis:

· **Reduced reaction times:** Microwave heating is often significantly faster than conventional heating.

· **Higher yields:** More efficient energy transfer can lead to improved product formation or analyte extraction.

· **Reduced solvent consumption:** Faster reactions and extractions can minimize the need for large volumes of solvents.

· **Enhanced reproducibility:** Controlled microwave systems offer better temperature and pressure control, leading to more consistent results.

· **Energy efficiency:** Direct heating of the sample can be more energy-efficient than heating an entire oven or heating bath.

Specialized laboratory microwave systems are designed with safety features, precise temperature and pressure control, and materials resistant to corrosive chemicals, making them suitable for the demanding requirements of chemical analysis. While offering numerous benefits, careful optimization of parameters and understanding the interaction of microwaves with different matrices are crucial for successful applications.

Vacuum desiccators:

Fig. 8.10 Vaccum Desiccators

It is used in the laboratory for drying substances. It consists of a glass vessel closed by a lid. The lower part of which contains the dehydrating agent such as line, Sulphuric acid of anhydrous calcium chloride. The substance to be dried is kept in the upper part and the desiccator is closed by a lid. The drying agent absorbs the moisture from the air and

substance cools slowly. While opening or closing the desiccator, the lid should not be lifted from the desiccator. It should be closed or opened by sliding carefully the lid along the rim of the glass vessel. By making use of a desiccator the hot objects such as crucibles containing substances are cooled without moisture being absorbed by them from the air. In gravimetric crucibles should always be cooled before weighing in a desiccator. **(E) MONOCHROMATORS, FILTERS, SAMPLE HOLDERS, DIFFRACTION GEATINGS, PHOTOEMISSIVE CELLS, PHOTOMULTIPLIER TUBES**
Monochromator:

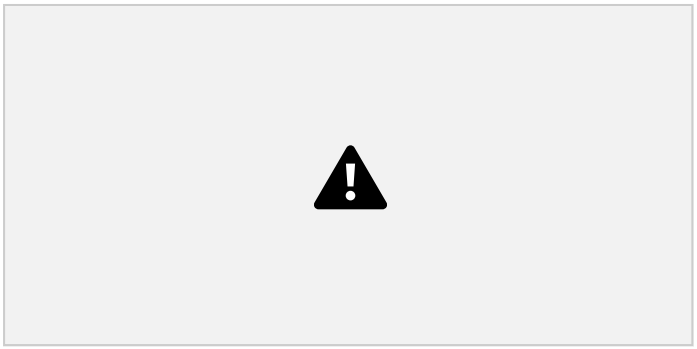


Fig. 8.11 Grating Monochromator

A **monochromator** is an optical device that isolates a narrow band of wavelengths (essentially "single color" light) from a broader spectrum light source. The name comes from the Greek words "mono" (single) and "chroma" (color).

Working Principle:
1. **Light Input:** Polychromatic light (containing many wavelengths) enters the monochromator through an **entrance slit**.
2. **Collimation:** The diverging light from the slit is made into a parallel beam using mirrors or lenses.
3. **Dispersion:** The parallel light then strikes a **dispersive element**, which separates the different wavelengths. The two main types of dispersive elements are:
○ **Prisms:** These refract (bend) different wavelengths of light at slightly different angles due to the wavelength dependence of the refractive index of the prism material. ○ **Diffraction Gratings:** These have a surface with many closely spaced parallel grooves. Light diffracts (bends and spreads out) as it reflects or passes through the grating, and constructive and destructive interference cause different wavelengths to emerge at different angles. Diffraction gratings are more commonly used in modern instruments due to their better dispersion and linear wavelength separation.
4. **Wavelength Selection:** After dispersion, the separated wavelengths are focused onto a plane containing an **exit slit**. By adjusting the position of the dispersive element (e.g., rotating the grating or prism), a specific narrow band of wavelengths is allowed to pass through the exit slit. The width of the exit slit determines the **bandwidth** (range of wavelengths) of the monochromatic light exiting the device. A narrower slit provides more monochromatic light but reduces intensity.
5. **Light Output:** The monochromatic light exiting the slit can then be directed towards a sample or a detector for further analysis.
Types of Monochromators:
Based on the dispersive element:

· **Optical filters** are devices that selectively transmit light of different wavelengths, usually implemented as plane glass or plastic devices in the optical path which are either dyed in the bulk or have interference coatings. The optical properties of filters are completely described by their frequency response, which specifies how the magnitude and phase of each frequency component of an incoming signal is modified by the filter

Fig. 8.12 Optical Filters with their mechanism in Optical Instruments

· **Prism Monochromators:** Utilize the refractive properties of prisms.

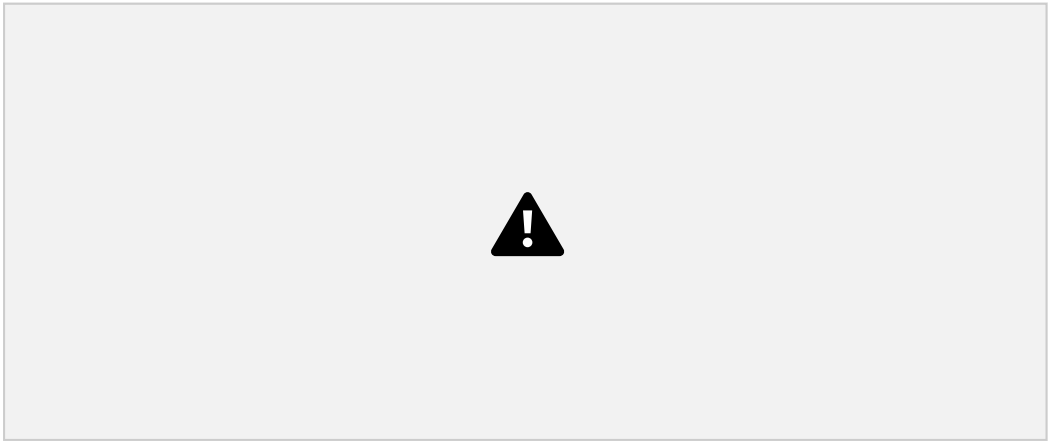
Fig. 8.13 Prism

· **Grating Monochromators:** Employ diffraction gratings. Common designs include Czerny Turner and Fastie - Ebert monochromators, which use mirrors to collimate and focus the light onto and from the grating.

Applications in Chemistry:

Monochromators are crucial components in various analytical instruments used in chemistry, including: · **Spectrophotometers (UV-Vis, IR, Fluorescence):** To select specific wavelengths of light to pass through a sample and measure its absorbance, transmittance, or emission at that wavelength. This is fundamental for quantitative analysis, identifying substances, and studying molecular properties.

· **Spectrofluorometers:** Use two monochromators, one to select the excitation wavelength and another to select the emission wavelength of a fluorescent sample.
· **Atomic Absorption Spectrometers (AAS):** To isolate the specific wavelength of light absorbed by the element being analyzed.
· **Raman Spectrometers:** To select the excitation laser wavelength and to analyze the wavelengths of the scattered Raman light.
· **Microplate Readers:** Used in biochemical and immunological assays to select specific wavelengths for measuring absorbance or fluorescence in multiple samples simultaneously. · **Circular Dichroism (CD) Spectrometers:** To select specific wavelengths of circularly polarized light to study the chiral properties of molecules.
· **Chromatography Detectors:** In some advanced detectors for HPLC or GC, a monochromator can be used to select specific wavelengths for compound identification and quantification. In essence, the monochromator is a vital tool for controlling the wavelength of light used in a wide range of chemical analyses, enabling precise and selective measurements



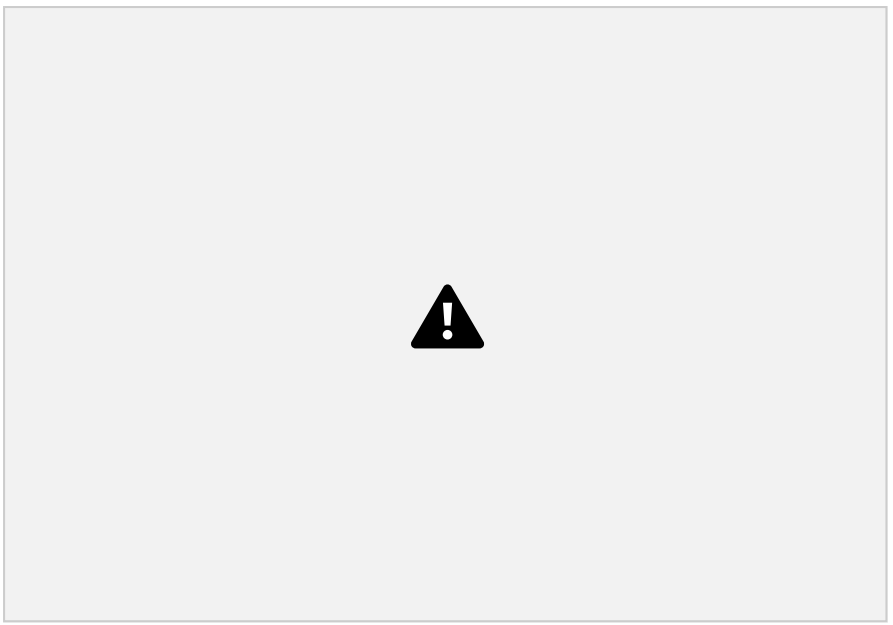


Fig. 8.14 Diffraction Grating

Sample holders:

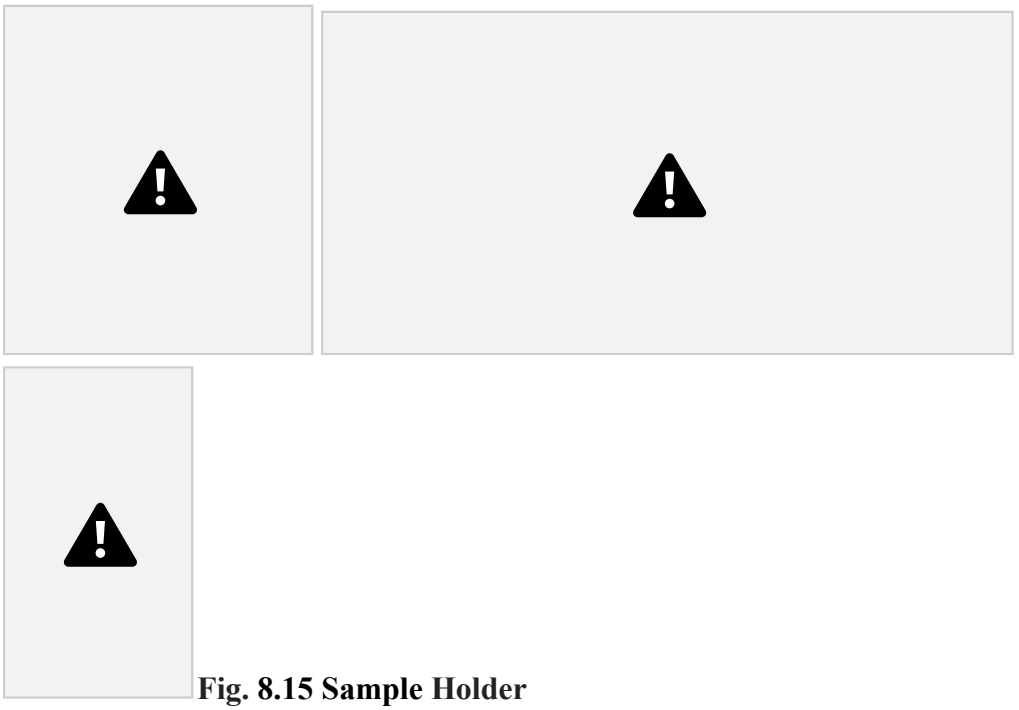


Fig. 8.15 Sample Holder

Most samples studied using visible and ultraviolet spectroscopy are liquid. The sample mast therefore be placed in a transparent container to allow measurement. These containers are called cuvettes. Cuvettes are generally nude from transparent plastic, gloss, or quartz. Different cuvettes have different optical properties. Plastic cuvettes are increasingly popular because they do not shatter when dropped, and because their low price makes them disposable. However, plastic cuvettes tend to have considerable absorbance in the ultraviolet region. For the measurements in the far ultraviolet region (below 250 nm) requires relatively

expensive quartz cuvettes. All cuvettes have the same path length in all orientations.
In the spectrophotometer layout generally the cuvette has a 1 cm path length as drawn. However, if the cuvette were inserted into the instrument in a different orientation, the path length would be considerably shorter (about 0.3 cm). Since absorbance depends on path length (the 'l' in A= εcl), the absorbance will also be smaller than expected for a sample of a given concentration.

Photo Emissive cell:

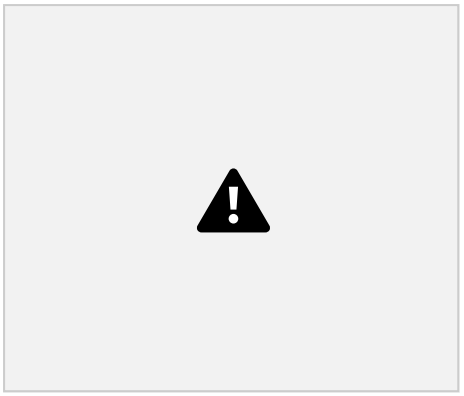


Fig. 8.16 Photo Emissive cell

A **photoelectric cell**, also known as a **photocell**, is a device that converts light energy into electrical energy. This conversion is based on the **photoelectric effect**, a phenomenon where electrons are emitted from a material's surface when light of sufficient frequency shines on it.

Key aspects of photoelectric cells: They typically consist of two electrodes within an evacuated or gas filled enclosure: a **photocathode** coated with a photosensitive material and an **anode** to collect the emitted electrons.

· When photons (light particles) strike the photocathode with enough energy (above the material's work function), they eject electrons (photoelectrons) from the surface.

· An applied voltage between the anode and cathode causes these photoelectrons to flow, creating an electric current proportional to the intensity of the incident light.

Types of Photoelectric Cells:

· **Photoemissive Cells (Phototubes):** These are vacuum or gas-filled tubes where electrons are directly emitted from the photocathode and collected by the anode.

· **Photovoltaic Cells (Solar Cells):** These generate a voltage directly when light falls on a semiconductor material, without needing an external voltage source.

· **Photoconductive Cells (Photoresistors):** These change their electrical resistance when exposed to light. The conductivity increases (resistance decreases) with increasing light intensity. Photoelectric cells have numerous applications, including light sensors in automatic doors, streetlights, exposure meters in cameras, solar panels for electricity generation, and detectors in scientific instruments like spectrophotometers (though modern instruments often use more sensitive detectors). **Photomultiplier tubes:**

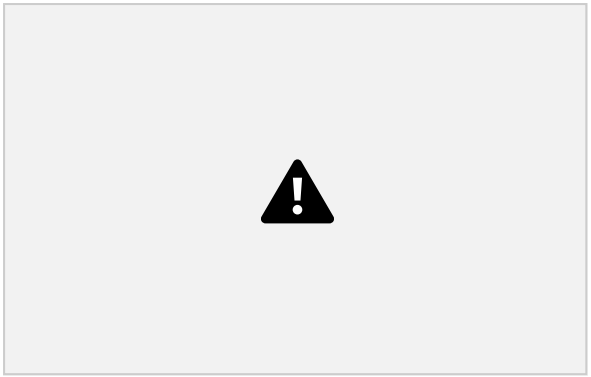


Fig. 8.17 Photomultiplier Tube (PMT)

A **photomultiplier tube (PMT)** is an extremely sensitive **vacuum tube** that converts incident photons (light particles) into an electrical signal. It is capable of detecting very weak light signals, even down to individual photons.

Working Principle:

1. **Photocathode:** Light enters the PMT through a window and strikes a **photocathode**, a photosensitive surface. The photocathode material (e.g., alkali metals or semiconductor compounds) is chosen for its sensitivity to specific wavelengths.
2. **Photoelectric Effect:** When a photon with sufficient energy hits the photocathode, it ejects an electron (photoelectron) into the vacuum inside the tube.
3. **Dynodes:** These photoelectrons are then accelerated by an electric field towards a series of electrodes called **dynodes**. Each dynode is held at a progressively higher positive voltage. 4. **Secondary Emission:** When a photoelectron strikes the first dynode, it has enough energy to dislodge multiple **secondary electrons** from the dynode's surface.
5. **Electron Multiplication:** These secondary electrons are then accelerated towards the next dynode, where the process of secondary emission is repeated, releasing even more electrons. This cascade effect continues through a series of dynodes (typically 6 to 14 stages), resulting in a significant **amplification** of the initial photoelectron signal. Gains of 106 to 108 are common.
6. **Anode:** The multiplied electrons are finally collected by the **anode**, producing a measurable electrical current pulse. The magnitude of this current is proportional to the number of photons that initially struck the photocathode.

Key Features:

· **High Sensitivity:** PMTs can detect extremely low light levels due to the electron multiplication process.
· **Fast Response Time:** They can respond to rapid changes in light intensity, often in the nanosecond range.
· **Low Noise:** When properly operated, PMTs exhibit low dark current (signal in the absence of light).
· **High Gain:** The multi-stage dynode system provides a large amplification factor. · **Wavelength Selectivity:** The spectral response is determined by the photocathode material.

Applications in Chemistry:

Photomultiplier tubes are widely used as detectors in various analytical instruments in chemistry, including:

· **Spectrophotometry (UV-Vis, Fluorescence, Atomic Absorption):** For detecting the intensity of light transmitted, emitted, or absorbed by a sample at specific wavelengths. Their high sensitivity is crucial for measuring weak signals, especially in fluorescence spectroscopy.
· **Scintillation Counting:** Detecting ionizing radiation by measuring the light emitted from a scintillator material when it interacts with radiation.
· **Chemiluminescence and Bioluminescence Assays:** Measuring the faint light produced by chemical or biological reactions.
· **Raman Spectroscopy:** Detecting the weak Raman scattered light from samples. · **Flow Cytometry:** Measuring the fluorescence and light scattering properties of individual cells. · **High-Performance Liquid Chromatography (HPLC) and other detectors:** For detecting analytes that exhibit fluorescence or luminescence.
· **Gamma Ray Spectrometry:** In conjunction with scintillators for detecting and measuring gamma radiation.
In summary, the photomultiplier tube is a highly sensitive and versatile light detector that plays a crucial role in many spectroscopic and analytical techniques in chemistry, enabling the measurement of very weak light signals with high speed and amplification.

Volumetric Analysis (Complexometry)

Determination of Total Hardness

~~Aim:~~ To determine the hardness of the given water sample.

Volumetric Analysis (Complexometry)

Theory: Hard water contains dissolved salts of calcium and magnesium in the form of bicarbonates. Hardness of water is of two types: (a) Temporary hardness & (b) Permanent hardness Hardness is the sum of calcium and magnesium ion concentration-both expressed as $CaCO_3$ in parts per million (ppm) or mg/dm^3 or indistinct end points . Interference of some metal ions leading to fading is avoided by adding inhibitors like Na_2S, $NH_2OH.HCl$, etc. A direct method of estimating the total hardness involves EDTA titrations using Eriochrome Black-T indicator at pH 10. This value gives total hardness of Ca^{2+} and Mg^{2+} ions.

Requirements: EDTA salt, water sample, buffer solution (pH = 10), 2% Eriochrome Black-T indicator, 25 cm^3 **Procedure:** standard measuring flask etc. burette, 100 cm^3 conical flask, 100 cm^3

(1) Prepare 100 cm^3 of 0.01M EDTA solution (0.372 g of EDTA dissolved in 100 cm^3 of distilled water in a 100 cm^3 standard measuring flask). Fill the burette after rinsing.

(2) Pipette out 25 cm^3 of the supplied water sample in a 100 cm^3 conical flask. Add 2 - 3 cm^3 of buffer solution (pH = 10), 3-4 drops (or a pinch) of 2% Eriochrome Black-T indicator to it. (3) Shake well and titrate against 0.01M EDTA solution from the burette. End point will be from wine red to blue colour (x).

Reactions:

(1) $CaR_2 + Na_2C_{10}H_{14}O_8N_2$ HCO_3 **Observations &** d. End Point:

$CaC_{10}H_{14}O_8N_2 + 2NaR$ (2) **Observation Table:**

$MgR_2 + Na_2C_{10}H_{14}O_8N_2$ a. Solution in Burette: $^-$, Cl^- or SO_4^{2-})

$MgC_{10}H_{14}O_8N_2 + 2NaR$ (R = b. Solution in Conical Flask:
c. Indicator:

	Burette level	Burette reading in cm^3	C.B.R. in cm^3
Calculations:	Initial	Final	I II III (x_1)

25 cm^3 of the water sample required x cm^3 of 0.01 M EDTA solution.

1000 cm^3 1M EDTA = 100 g

Difference of $CaCO_3$

∴ 1 cm^3 of 1M EDTA = 100 mg of $CaCO_3$

Volumetric Analysis (Complexometry)

Calculations:

25 cm^3 of the water sample required x cm^3 of 0.01 M EDTA solution.

1000 $cm^3$1M EDTA = 100 g of $CaCO_3$

∴ 1 cm^3 of 1M EDTA = 100 mg of $CaCO_3$

∴ ⬜⬜ cm^3 of 0.01M EDTA = 100 x (⬜⬜) x 0.01 = Z mg of $CaCO_3$. The

total hardness of water sample is obtained as $CaCO_3$ mg / dm^3 or ppm.

Since 25 cm^3 of the water sample = Z mg of $CaCO_3$.

∴ 1000 cm^3 _____ = $\frac{Z * 1000}{25}$ _____ ppm.

Results:

(1) 25 cm^3 of the water sample required (��) _____ cm^3 of 0.01 M EDTA solution. (2) Total hardness of given water sample = _____ $CaCO_3$ ppm (mg/ dm^3)

Volumetric Estimations

Estimation of Drugs

Assay of Commercial Sample of Aspirin

Aim: To carry out the assay of a commercial sample of aspirin (acetyl salicylic acid) using phenol red as indicator.

Theory:

Acetyl salicylic acid is an acid and hence can be titrated with a standard base with the help of a proper indicator.

Aspirin alone is determined by refluxing the neutralized sample solution with excess standard sodium hydroxide to hydrolyse the acetoxy group, cooling & back titrating the excess base with standard acid. The difference between the volume of standard sodium hydroxide solution consumed in the first and second titration is a measure of the salicylic acid and acetic acid.

Acetyl salicylic acid undergoes hydrolysis when treated with warm solution of sodium hydroxide producing sodium ethanoate.

An excess of sodium hydroxide is used; the unreacted alkali may be determined by back titration with standard hydrochloric acid.

Aspirin is liable to be contaminated with salicylic acid which may be present as a result of incomplete conversion to aspirin during manufacture or through hydrolysis on storage. Hydrolysis also releases acetic acid which can often be detected in samples of aspirin by odour. The quality of aspirin is therefore, controlled by a combination of double assay procedure and a separate colorimetric limit test for salicylic acid based on the reaction of its free phenolic group with iron (III) chloride. In the assay the sample is dissolved in ethanol (95%) because of its low solubility in water (1 in 300) and titrated directly with sodium hydroxide using phenolphthalein as indicator. Aspirin and contaminating acetic acid and salicylic acids are titrated in this procedure. However, for calculation purposes in determining compliance with the standard, it is assured that all material titrated is aspirin.

Requirements: 0.5N NaOH solution, 0.5 N HCI solution, phenol red indicator, succinic acid, distilled water, aspirin powder, 100 cm^3 standard measuring flask, 10 cm^3 and 25 cm^3 pipette, 150 cm^3 conical flask etc.

Procedure:

Part I: Standardization:

(1) Prepare 0.1 N Succinic acid (0.59 g dissolved in 100 cm^3 of distilled water in a 100 cm^3 standard measuring flask.

(2) Use 25 cm^3 of Succinic acid for titration. Let the reading be x_1 cm^3.

Part II: Estimation:

(1) Weigh accurately about 0.5 g (W_{sample}) of powdered commercial sample of aspirin and add 20 cm^3 of 0.5 N NaOH and boil gently for 10 minutes.

(2) Titrate the excess of alkali with 0.5 N HCI solution using phenol red as indicator. The end point will be from red to yellow orange colour. Say x_3 cm^3. Repeat the experiment with (20 cm^3) without the sample (Blank reading). Let reading be x_2.

(3) The difference between the two readings (x_2.- x_3) cm^3 corresponds to the amount of 0.5 N NaOH required by acetyl salicylic acid.

Volumetric Estimations

Observation Table:

(1) Standardization of NaOH: 25 cm^3 Succinic acid against NaOH

Burette level	Burette reading in cm^3			C.B.R. in cm^3 (x_1)
	I	II	III	
Initial				
Final				
Difference				

(2) Blank titration – 20 cm^3 NaOH against HCl

Burette level	Burette reading in cm^3			C.B.R. in cm^3 (x_2)
	I	II	III	
Initial				
Final				
Difference				

(3) Estimation of Aspirin – Unreacted NaOH against HCl

Burette level	Burette reading in cm^3			C.B.R. in cm^3 (x_3)
	I	II	III	
Initial				
Final				
Difference				

Calculations:

(1) Standardization of NaOH solution:

$V_{succinic\ acid} \times N_{succinic\ acid} = V_{NaOH} \times N_{NaOH}$

$$N_{NaOH} = \frac{25 * 0.1}{x_1}$$

(2) Standardization of HCI solution:

$V_{HCl} \times N_{HCl} = V_{NaOH} \times N_{NaOH}$

$$N_{HCl} = \frac{20 * N_{NaOH}}{x_2}$$

(3) Estimation of Aspirin - Unreacted NaOH against Standardized HCl solution:

1 cm^3 of 0.5 N HCI = 0.04504 g of aspirin

V cm^3, i.e., (x_2 - x_3) cm^3 of N_{HCl} = 0.04504 * N_{HCl} x V = _____ g of Aspirin (W)

0.5

(4) %purity = W * 100 = _____ %. (W_{sample} = weight of unknown sample)

W_{sample}

Results: Percentage purity of given commercial sample of Aspirin = _____ %

Gravimetric Analysis

Estimation of Nickel (II) as Ni-dmg

Aim: Estimate the amount of nickel present in the given Nickel Chloride / Nickel Sulphate solution.

Theory: Nickel ions are precipitated as a chelate complex of dimethylglyoxime reagent in an alkaline medium. Hence, Nickel is precipitated by adding solution of dimethylglyoxime to a hot faintly acidic solution of nickel salt and adding a slight excess of ammonia to neutralize the acid produced (the precipitate is soluble in mineral acid). The scarlet red coloured precipitate of nickel dimethylglyoxime is washed with cold water to remove the water soluble impurities. Such a chelate is stable up to 110°C hence can be dried safely. This method is the most rapid and accurate for the estimation of nickel ion.

Requirements: Ni^{2+} salt solution, 1% dimethylglyoxime reagent solution, 6N or 1:1 NH_4OH solution, sintered glass (G_3) crucible, electric oven, distilled water, 100 cm^3 standard measuring flask, 100 cm^3 beakers, 25 cm^3 pipette, water bath, ethyl alcohol etc.

Procedure:

(1) Dilute the given solution to 100 cm^3 in a standard measuring flask with distilled water. Pipette out 50 cm^3 of it in a 100 cm^3 borosil beaker.

(2) Add 25-30 cm^3 of 1% dimethylglyoxime reagent and stir it well. Add 1:1 NH_3, (or 6N NH_4OH) dropwise with constant stirring to precipitate Ni^2 as Ni-dmg (Once the precipitation is complete, smell of ammonia should be distinct to ensure the alkaline medium).

(3) Digest the precipitate on a hot water-bath for about half an hour. Test the supernatant solution for complete precipitation with 1-2 drops of the DMG reagent as well as with 6N NH_4OH solution.

(4) Filter the precipitate through a previously weighed sintered glass crucible (G_3). Wash the precipitate with distilled water, till the filtrate is free from Cl and SO_4^{2-} ions. (5) Finally wash it once with 2-3 cm^3 of 50% ethyl alcohol. Dry the precipitate at 110°C in an electric oven and weigh as Ni-dmg (x).

Reaction:

$NiCl_2 + 2NaC_4H_7N_2O_2 \rightarrow Ni(C_4H_7N_2O_2)_2 + 2NaCl$

Structure: Calculations:

Let the weight of residue (Ni-dmg) be (x) g. Now, 50 cm^3 of the diluted solution gave (x) g of Ni-dmg.

∴ 100 cm^3 of the diluted solution gave 2x g of Ni-dmg

From the equation,

Ni-dmg = $NiCl_2.6H_2O$

288.71g of Ni-dmg = 58.71 g of Nickel = 237.46 g of $NiCl_2.6H_2O$

∴ 2x g Ni-dmg = $\frac{58.71 \times 2x}{288.71}$ = _____ 'A' g of Nickel & $\frac{237.46 \times 2x}{288.71}$ = _____ 'B' g of $NiCl_2.6H_2O$

Percentage Error:

$$\% \text{ Error} = \left| \frac{\text{Experimental value} - \text{Accepted / Theoretical value}}{\text{Accepted value}} \right| \times 100$$

∴ % Error of Ni^{2+} in Ni-dmg = $|^{A-y}$

$_{\diamond\diamond}|$ x 100 = _____ %

∴ % Error of $NiCl_2.6H_2O$ in Ni-dmg = $|^{B-z}$

$_{\diamond\diamond}|$ x 100 = _____ %

['y' (Theoretical accepted value) of Ni^{2+} ions in g and 'z' (Theoretical accepted value of $NiCl_2.6H_2O$) in g will be provided].

Result:

(1) Percentage error of Ni (II) = _____ %.

(2) Percentage error of $NiCl_2.6H_2O$ = _____ %.

Short Organic Preparations and Their Purification

I. Oxime of a Ketone

Aim: To prepare Cyclohexanone Oxime from Cyclohexanone.

Theory: Reaction of aldehydes and ketones with hydroxylamine gives oximes. The nucleophilicity of the nitrogen on the hydroxylamine is increased by the presence of the oxygen. Successive proton transfer allows for elimination of water.

Reaction:

hydroxylamine
cyclohexanone → Cyclohexanone oxime

Procedure:

(1) Place 1cm^3or supplied quantity of cyclohexanone in a 100 cm^3 conical flask. (2) Add a solution of 2 g of hydroxylamine hydrochloride in 5 cm^3 of distilled water. Cool the mixture in cold water and add 3 cm^3 of 30% sodium carbonate solution slowly to the reaction mixture with constant stirring. Shake the flask.

(3) Heat it on a boiling water bath for 20 - 30 minutes while stirring mixture with a glass rod.

(4) Cool, filter the solid product and wash it with cold water.

(5) Recrystallize the product from alcohol and dry it.

(6) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

Density of cyclohexanone = 0.948g / cm^3

1cm^3x 0.948 g of cyclohexanone $= \frac{113 * 1 * 0.948}{98} = 1.09g$

98 g of cyclohexanone = 113 g of cyclohexanone oxime

(II) Percentage yield

$= \frac{\text{Weight of the product}}{\text{Theoretical yield}} * 100 =$ _____ %

Results:

(1) Melting point of the product: _____ °C (Expected melting point = 88 - 91°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

****

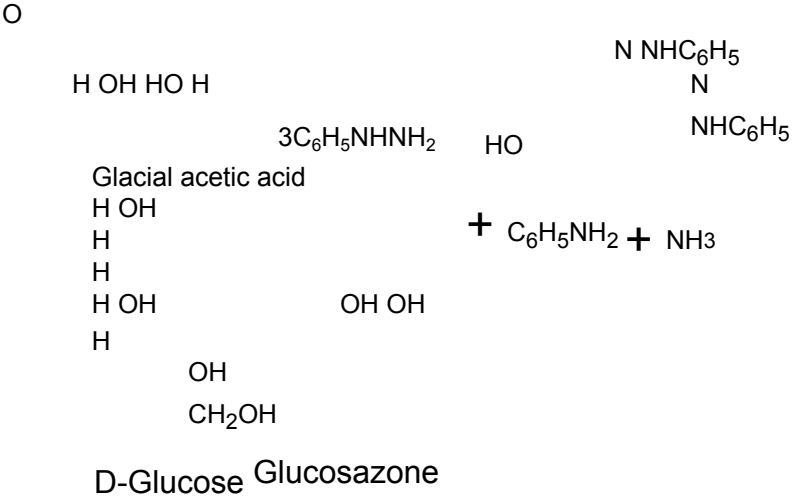
Short Organic Preparations and Their Purification

II. Osazone of Glucose

Aim: To prepare Glucosazone from glucose.

Theory: Glucose reacts with excess of phenyl hydrazine in glacial acetic acid and forms glucosazone. Glucose and fructose form same osazone which indicates the configuration at C-3, C-4 and C-5 is same.

Reaction:



Procedure:

(1) Dissolve 1 g or supplied quantity of glucose in a 5 cm^3 of distilled water in a 100 cm^3 conical flask.

(2) Add 2 cm^3 of phenyl hydrazine and 6 cm^3 of glacial acetic acid to it.

(3) Mix well and heat it on a boiling water bath. Shake the flask from time to time. (4) The yellow product starts separating in about 15 minutes. The heating is continued with occasional stirring for 45 minutes.

(5) Cool the flask, filter the yellow solid product and wash it with cold water.

(6) Recrystallize the product from alcohol and dry it.

(7) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

180 g of glucose = 358 g of glucosazone

1 g of glucose $= \frac{358 * 1}{180} = 1.98$ g.

(II) Percentage yield:

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

Short Organic Preparations and Their Purification

Results:

(1) Melting point of the product: _____°C (Expected melting point = 204°C with

decomposition)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

[***Note***: Osazone of fructose is prepared in the same way]

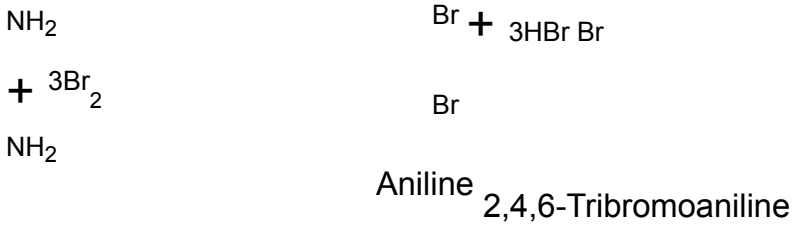
****

III. Bromination of Aniline

Aim: To prepare 2, 4, 6 - tribromoaniline from aniline.

Theory: Aniline is very reactive due to the presence of electron donating -NH_2 group. It readily undergoes electrophilic substitution reaction like bromination to give 2, 4, 6-tribromoaniline at room temperature.

Reaction:



Procedure:

(1) Take 1 cm^3 or supplied quantity of aniline and 5 cm^3 of distilled water in a 100 cm^3 conical flask and shake well.

(2) Add 20% bromine water solution dropwise with constant stirring till permanent yellow colour is obtained. Keep it at room temperature for 15 minutes.

(3) Filter the product under suction. Wash it with cold water to remove all the traces of bromine.

(4) Recrystallize it from aqueous alcohol and dry it.

(5) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

Density of aniline = 1.02 g/cm^3.

1 cm^3x 1.02 g of aniline $= \frac{330 * 1.02 * 1}{93} = 3.6$ g.

93 g of aniline = 330 g of 2, 4, 6 - tribromoaniline

(II) Percentage yield:

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

Results:

(1) Melting point of the product: _____°C (Expected melting point = 118°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

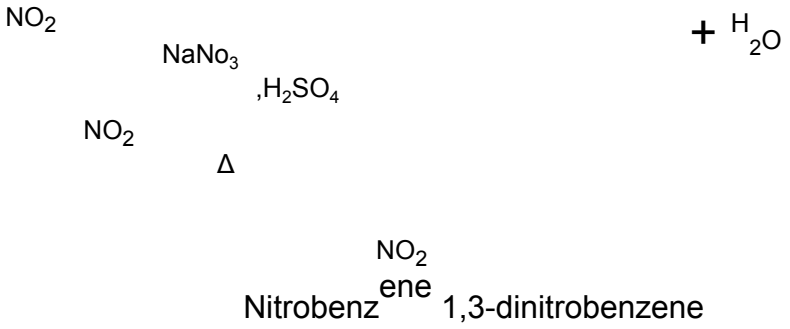
****

IV. Nitration of Nitrobenzene

Aim: To prepare *m*-dinitrobenzene from nitrobenzene.

Theory: Nitration is an electrophilic aromatic substitution; the electrophile is a nitronium ion, which displaces a hydrogen ion from the benzene ring. It is generated from a Lewis base, nitric acid, in the presence of a Lewis acid catalyst, sulfuric acid. When the nitration of nitrobenzene is performed, nitro group deactivates the benzene ring and directs the incoming nitro group to the meta position.

Reaction:



Procedure:

(1) Take 3 g of sodium nitrate and 5 cm³ of Conc. Sulphuric acid in a dry 100 cm³ conical flask and warm it with constant swirling the contents of the flask.

(2) Add 1 cm³ or supplied quantity of nitrobenzene drop by drop to it with gentle shaking of the flask.

(3) After the complete addition, heat the reaction mixture on a sand bath for 30 minutes with occasional shaking.

(4) Allow the mixture to cool and pour it slowly in about 50 cm³ of ice-cold water taken in a 150 cm³ beaker to get the product.

(5) Filter the product under suction and wash it with cold distilled water.

(6) Recrystallize it from aqueous alcohol and dry it.

(7) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____ °C.

Calculations:

(1) Theoretical yield:

Density of nitrobenzene = 1.204 g/cm^3.

1 cm^{3}x 1.204 g of nitrobenzene $= \frac{168 * 1.204 * 1}{123} = 1.6$ g.

123 g of nitrobenzene = 168 g of *m*-dinitrobenzene

(II) Percentage yield:

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

= _____%

Results:

(1) Melting point of the product: _____°C (Expected melting point = 90°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

****

V. Anhydride of Dicarboxylic acid

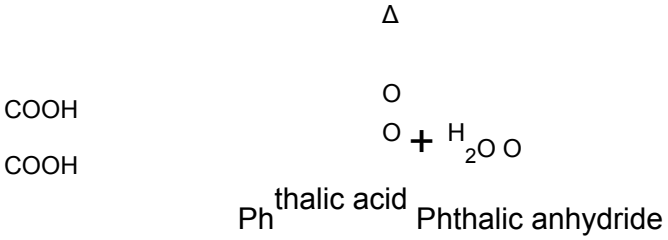
Aim: To prepare phthalic anhydride from phthalic acid.

Theory: Phthalic anhydride is prepared by sublimation. Sublimation is the process of transition of a substance from the solid phase to the gas phase without passing through an intermediate liquid phase.

At normal pressures, most chemical compounds possess three different states at different temperatures. In these cases, the transition from the solid to the gaseous state requires an intermediate liquid state. Sublimation is a technique to purify compounds or prepare anhydride from dicarboxylic acid. A solid is typically placed in a sublimation apparatus and heated. The solid volatalies and condenses as a purified compound on a cooled surface, leaving a non-volatile residue of impurities behind. The purified compound may be collected from the cooling surface.

Short Organic Preparations and Their Purification

Reaction:



Procedure:

(1) Place 1 g or supplied quantity of phthalic acid in a porcelain dish and a filter paper having holes is placed on the dish.

(2) Keep an inverted funnel on the filter paper. Plug the stern of the funnel with cotton. (3) Heat the dish on a sand bath on a low flame. The anhydride formed rises through holes in the filter paper and condenses in the hollow of the funnel and on the filter paper. (4) Collect the crystals of phthalic anhydride.

(5) Find the mass and melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

166 g of phthalic acid = 148 g of phthalic anhydride

$$1 \text{ g of phthalic acid} = \frac{148 * 1}{166} = 0.89 \text{ g.}$$

(II) Percentage yield:

$$= \frac{\text{practical yield}}{\text{theoretical yield}} \times 100 = \_\_\_\_\_ \%$$

Results:

(1) Melting point of the product: _____°C (Expected melting point = 132°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

****

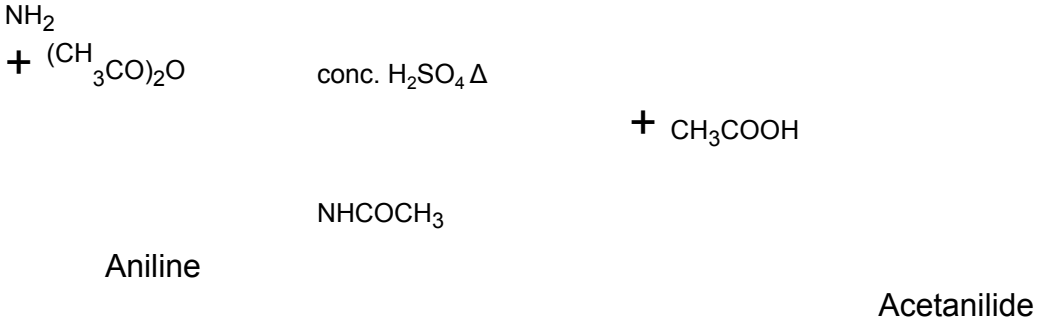
Short Organic Preparations and Their Purification

VI. Acetylation of Aromatic Primary Amine

Aim: To prepare Acetanilide from Aniline.

Theory: Replacement of active hydrogen of -NH_2 group by an acyl group (-COR) is called N-acetylation of primary amine. Aniline on acetylation with acetic anhydride in the presence of Conc. H_2SO_4 gives acetanilide.

Reaction:



Procedure:

(1) Place 1 cm^3or supplied quantity of aniline in a 100 cm^3dry conical flask. (2) Add 4 cm^3 of acetic anhydride to it carefully with constant shaking of the flask. (3) Now add 2 drops of conc. Sulphuric acid and swirl the contents in order to ensure thorough mixing.

(4) Heat it on a boiling water bath for 20 minutes while stirring mixture with a glass rod. (5) Pour the hot reaction mixture to a 100 cm^3 beaker containing 50 cm^3 of cold distilled water with constant stirring.

(6) Filter the solid and wash it with cold distilled water.

(7) Recrystallize the product from hot water and dry it.

(8) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

Density of aniline = 1.02 g/cm^3.

93 g of aniline = 135 g of acetanilide

$2\ cm^3 * 1.02g\ of\ aniline = \frac{135 * 1.02 * 2}{93} = 2.96\ g.$

(II) Percentage yield:

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

Results:

(1) Melting point of the product: _____°C (Expected melting point = 114°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

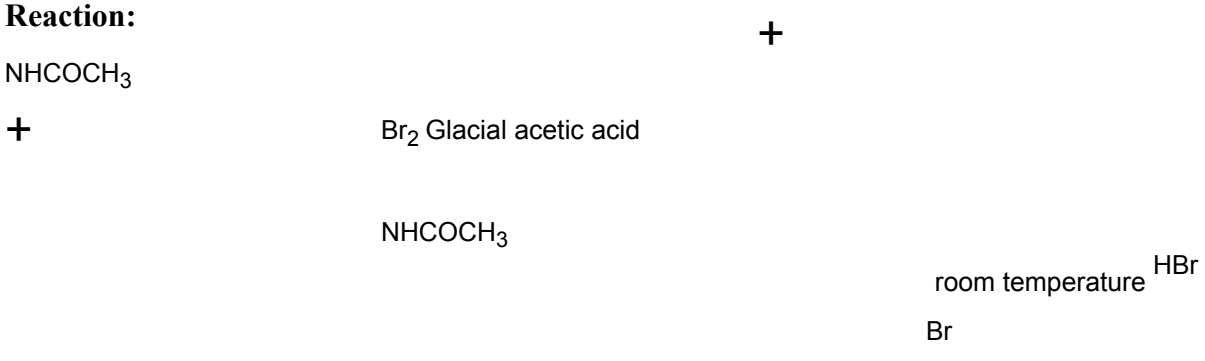
****

VII. Bromination of Acetanilide

Aim: To prepare *p*-bromoacetanilide from acetanilide.

Theory: Bromination of acetanilide is an electrophilic aromatic substitution reaction. The anilide group activates the ring; therefore, bromination occurs at room temperature. Anilide group is ortho and para directing; the anilide group provides steric hindrance at the ortho position. Therefore, bromination of acetanilide occurs mainly at the para position.

Reaction:



Acetanilide p-Bromoacetanilide

Procedure:

(1) Dissolve 1 g or supplied quantity of acetanilide in 5 cm³ of glacial acetic acid in a dry 100 cm³ conical flask.

(2) Add 5 cm³ of 15% solution of bromine in glacial acetic acid slowly with constant stirring till solution becomes orange colored.

(3) Allow it to stand at room temperature for 15 minutes with occasional shaking. (4) Filter the product under suction. Wash it with cold distilled water to remove all the traces of bromine.

(5) Recrystallize it from aqueous alcohol and dry it.

(6) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

135 g of acetanilide = 214 g of *p*-bromoacetanilide

$$1\text{ g of acetanilide} = \frac{214 * 1}{135} = 1.58\text{ g.}$$

(II) Percentage yield: $= \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100 =$ _____%

Results:

(1) Melting point of the product: _____°C (Expected melting point = 168°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

****

VIII. Iodoform from Acetone

Aim: To prepare iodoform from acetone.

Theory: Iodoform (CHI_3) is a pale yellow crystalline solid having a characteristic odour. It is used as a mild antiseptic disinfectant. Iodoform can be prepared by treating any organic compound containing $CH_3CH(OH)$- group (e.g., ethanol, 2-propanol, and 2-butanol) or CH_3CO-group (e.g. propanone, 2-butanone) with iodine in presence of sodium hydroxide.

Reaction:

Procedure:

(1) Place 1 cm^3 or supplied quantity of acetone and 10 cm^3 of water in a 250 cm^3 conical flask. Add freshly prepared 10 cm^3 of 10% NaOH solution. Stir the mixture thoroughly. (2) To this, add slowly with stirring a solution of iodine (2.5 g of iodine dissolved in a solution of 5 g of KI in 10 cm^3 of distilled water) till the color of iodine persists.

(3) Place this mixture in a warm water bath at a temperature of 60 - 70°C for about 15 minutes. If iodine color disappears, add more iodine solution.

(4) Allow the solution to cool to obtain yellow crystals of iodoform.

(5) Filter the product and wash it with cold distilled water.

(6) Recrystallize it from alcohol and dry it.

(7) Weight the mass and note the melting point of the purified product.

Alternate procedure:

(1) Dissolve 1 g of iodine in 1 cm^3 or supplied quantity of acetone in a 100 cm^3 conical flask.

(2) Add freshly prepared 5% sodium hydroxide solution slowly with shaking until the color of iodine is discharged.

(3) Allow contents of flask to stand for 10-15 minutes.

(4) Filter the yellow precipitate of iodoform through Buchner funnel.

(5) Wash the precipitate with cold distilled water.

(6) Dry the precipitate between filter paper.

(7) Weight the mass and note the melting point of the purified product.

Observations:

(1) Weight of the product: _____ g.

(2) Melting point of the product: _____°C.

Calculations:

(1) Theoretical yield:

Density of acetone = 0.791 g/cm^3.

58 g of acetone = 394 g of iodoform

1 cm^3x 0.7901 g of acetone $= \frac{394 * 1 * 0.791}{58} = 5.37$ g.

(2) Percentage yield:

$$= \frac{\text{practical yield}}{\text{theoretical yield}} \times 100 = \_\_\_\_\_\%$$

Results:

(1) Melting point of the product: _____°C (Expected melting point = 119°C)

(2) Practical yield: _____ g.

(3) Theoretical yield: _____ g.

(4) Percentage yield: _____ %

****

Semester – IV

Chemistry Practical (Major)

(24_USCH404)

Potentiometry

Experiment No. 1

Aim: To determine standard emf and standard free energy change of Daniel Cell.

Theory: A chemical cell is formed by the combination of two single electrodes. The cell potential gives the difference of the potential of the two electrodes.

Daniel cell is constructed by combination of zinc electrode and copper electrode. The reaction taking place in Daniel cell can be represented as: $Zn + Cu^{2+} \leftrightarrow Zn^{2+} + Cu$

Hence, the cell can be represented as:

$$^{-}Zn\,(s)\,|Zn^{2+}{}_{(aq)}\,(m_1\gamma_1)\,||Cu^{2+}{}_{(aq)}\,(m_2\gamma_2)\,|Cu(s)^{+}$$

Where m_1 and γ_1 are molarity and activity coefficient of Zn^{2+} ions while m_2 and γ_2 denotes molarity and activity coefficient of Cu^{2+} ions.

The cell potential of the above can be given by Nernst equation (for cell):

$$2\text{[illegible]}_{10}[\text{[illegible]}^{+2}][\text{[illegible]}]$$

$$^{0}\_0.0592$$

$$\text{[illegible]} = \text{[illegible]}_{\text{[illegible]}} \qquad \text{[illegible]}_{\text{[illegible]}}$$

by convention [Zn] = [Cu] = 1

$$(\text{[illegible]}_2\gamma_2)$$

while $[Zn^{2+}] = m_1\gamma_1$ and $[Cu^{2+}$

$$^{0} = \text{[illegible]} + 0.0296\ (\text{[illegible]}_1\gamma_1)$$

] = $m_2\gamma_2$

The standard cell potential is given by: $\text{[illegible]}_{\text{[illegible]}}$

$$^{0} - 0.0296\ (\text{[illegible]}_1\gamma_1) \qquad (\text{[illegible]}_2\gamma_2)$$

$$[\text{[illegible]}][\text{[illegible]}^{+2}]$$

Hence, we have, $\text{[illegible]} =$

Measuring the cell potential under experimental condition and knowing the values of m and γ for the particular solution, standard cell potential can be calculated.

Requirements: 0.1M $ZnSO_4$, and 0.1M $CuSO_4$ solutions, copper and zine electrodes (or wires), salt bridge, 100 cm^3standard measuring flasks, beakers, pipettes etc.

Procedure:

Part I: Preparation of solutions:

(1) Prepare 0.05M and 0.01M $ZnSO_4$, solution in two $100cm^3$standard measuring flasks from 0.1M $ZnSO_4$, solution as follows:

Concentration of $ZnSO_4$	0.05M	0.01M
Volume of 0.1M $ZnSO_4$ solution in cm^3	50.0	10.0

Dilute the above volumes of $ZnSO_4$ solution in $100cm^3$standard measuring flask (add two drops of dilute H_2SO_4 in each flask to avoid hydrolysis). Dilute solutions to $100cm^3$ with distilled water. Shake each solution thoroughly well

(2) Prepare 0.05M and 0.01M $CuSO_4$ solutions in the same manner using 0.1M $CuSO_4$ solution.

Potentiometry

Part II: **Standardisation of potentiometer:**

Standardise the potentiometer as per instructions given by instructor and keep it on till the end of experiment.

Part III: **Determination of emf of the cell:**

(1) In a clean dry 100 cm^3 beaker, pipette out about $50cm^3$of 0.1M $ZnSO_4$, solution. Dip a clean, polished zinc rod (electrode) in it. Connect it to the negative terminal of the potentiometer.

(2) In another clean dry $100cm^3$beaker, pipette out about $50cm^3$of 0.1M $CuSO_4$, solution. Dip a clean, polished copper rod (electrode) in it. Connect it to the positive terminal of the potentiometer.

(3) Place a KCl or KNO_3 salt bridge between the two solutions to make electrical contact. (4)

The cell set up is represented as follows: $Zn(s) \mid Zn^{2+}_{(aq)}(m_1\gamma_1) \| Cu^{2+}_{(aq)}(m_2\gamma_2) \mid Cu(s)$ …. (I)

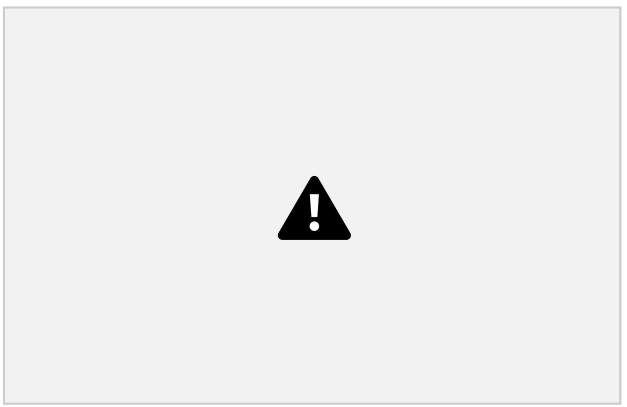


Figure 1. Schematic Presentation of Danniel Cell

(5) Note the potentiometer reading. This is the emf of the cell in volts (E_{cell}).

(6) Repeat the above procedure using prepared different concentrations of $ZnSO_4$ and $CuSO_4$ solutions.

(7) Record your observations as follows:

Obs. No.	$ZnSO_4$ Solution		$CuSO_4$ Solution		emf of the cell (E_{cell})	Standard emf of cell (E^0)
	Molar ity (M_1)	Activity Coefficient (γ_1)	Molarity (M_2)	Activity Coefficient (γ_2)		
1	0.10	0.148	0.10	0.733		
2	0.05	0.202	0.05	0.812		
3	0.01	0.387	0.01	0.892		
				Mean (E^0)		

Potentiometry

Calculations:

Determination of E^0 using Nearnst equation:

$$Zn + Cu^{2+} \leftrightarrow Zn^{2+} + Cu$$

The cell potential is given by the expression:

$$E = E^0 - \frac{0.0592}{2} \log_{10} \frac{[Zn^{+2}]}{[Cu^{+2}]}$$

$E = E^0$ $[Zn^{+2}]$ $(\because [Cu] = [Zn] = 1)$

$$E = E^0 + 0.0296 \log \frac{(M_1\gamma_1)}{(M_2\gamma_2)}$$

Substitute the values of E_{cell} from the observation table at corresponding values of molarities and activity coefficients of Zn^{2+} and Cu^{2+} ion solutions to get the value of standard cell potential.

E^0 is related to standard free energy change, $\Delta G°$ by the relation,

$$\Delta G° = - n F E^0 = - 2 * 96500 * E^0$$

^{0}(∵ n = 2).

^{0}it is possible to obtain the value of ΔG°.

Hence, knowing the value of ��$_{\square\square\square\square\square\square\square\square}$,

Results:

^{0} = _____volts.

(1) The standard cell potential, ��$_{\square\square\square\square\square\square\square\square}$

(2) The standard free energy change for the cell, ΔG° for the reaction = _____ Joule / mol. **********

Potentiometry

Experiment No. 2

Aim: To determine the amount of a given strong acid (HCl) by potentiometric titration using quinhydrone electrode.

Theory: In this method, cell is set up which consists of two half cells, one called the *indicator electrode* and the other *reference electrode*. The latter is a saturated calomel electrode. The indicator electrode in present case is quinhydrone electrode which is represented below (platinum wire is used for electrical contact only) contains equimolar mixture of H_2Q and Q.Pt| H_2Q: Q

where, H_2Q is $C_6H_6O_2$ - Hydroquinone, and Q is $C_6H_4O_2^-$ Quinone. The reduction reaction

$C_6H_4O_2 + 2H^+ + 2e^- \rightarrow C_6H_6O_2$

During the titration the change in electrode potential depends on the quantity of hydrogen ion removed by neutralisation. In initial stage of the titration, emf changes slowly, but towards the equivalence point is comparatively rapid and sharp. Further addition of alkali shows, again a small change in emf. The volume corresponding to the point of inflection of the graph (emf against volume of alkali added), is the volume of alkali required for neutralisation.

Requirements: 0.1N NaOH solution, HCl solution, Sat. Calomel electrode, Platinum electrode, Quinhydrone powder, Saturated KCl solution, Salt bridge etc.

Procedure:

(1) Fill the burette with the supplied 0.1N NaOH solution.

(2) Dilute the given solution of HCl to 100 cm^3 with distilled water in a standard measuring flask and pipette out 10 cm^3(or any other specified volume) of it in a 100 cm^3beaker. Add 25 $cm^3$of distilled water to it.

(3) Add a small quantity (pinch) of quinhydrone powder to the acid solution and stir thoroughly. (Solution should be saturated with quinhydrone).

(4) Insert a platinum electrode in the solution (add if necessary, a small quantity of distilled water) and connect it to positive terminal of the potentiometer.

(5) Take 40 – 50 cm^3saturated KCl in another 100 cm^3beaker solution and place a saturated calomel electrode in it. Connect calomel electrode to negative terminal of the potentiometer. (6) Put KCl salt bridge connecting the two beakers, as shown in Figure 2. The cell may by represented as follows:

$$^{-}\text{Saturated Calomel Electrode} \parallel H^{+} H_2Q : Q \mid Pt^{+}$$

(7) Standardise the potentiometer and measure the emf of the cell. This will be emf for zero volume of 0.1N NaOH solution.

(8) Add 0.5 $cm^3$of NaOH solution at a time from the burette. Stir the solution. Determine the emf of the solution after each addition, till the emf of the cell shows a negative value (i.e., approximately 15- 20 cm^3 0.1N NaOH solution). This gives an idea about the approximate equivalence point. This reading can be taken as a pilot reading.

(9) For the main titration, pipette out the acid solution as in step No. 2. Add quinhydrone, insert a platinum electrode and connect it to positive terminal of the potentiometer. Complete the cell. (10) Repeat the experiment by adding 1 $cm^3$of 0.1N NaOH solution at a time initially and then 0.2 $cm^3$of NaOH solution towards the neutralisation point. Stir the solution thoroughly. Wait for a minute or so (to attain equilibrium between quinone and hydroquinone) before determining the emf of the cell. Record the readings. Add 0.5 cm^3 of 0.1N NaOH solution throughout the experiment (As Per given instruction).

(11) After the cell attains negative voltage, take four to five readings more (At this stage, the colour of the solution becomes very dark).

Potentiometry

(12) Plot the graphs of (Figure 3a) E_{cell} against volume of NaOH added, (Figure 3b) ($\Delta E/\Delta V$) against mean value V_m i.e., $V_m = (V_1 + V_2) / 2$ and (Figure 3c) *p*H against mean volume to determine the volume of alkali required for neutralisation and calculate the strength of HCl solution.

Observations and Calculations:

Volume of acid pipetted out for titration (V_1) = _____ cm^3

Normality of NaOH solution used = 0.1N.

Obs. Nos.	Volume of NaOH solution added in	emf of the cell E in *volts*	Difference in Volume (ΔV)	Difference in emf (ΔE)	($\Delta E/\Delta V$) *mV/cm³*	$\Delta^2 E$	ΔV^2	($\Delta^2 E/\Delta V^2$) *mV/cm³*	*p*H = $\frac{0.4574 - ▯▯▯▯▯▯}{▯▯▯ 0.0592}$

	cm^3*(V)*								
1									
2									
3									
4									
5									
6									
7									

ΔV = Volume added - Previous Volume = $V_2 - V_1$ & ΔE = Previous emf - Obtained emf = $E_1 - E_2$

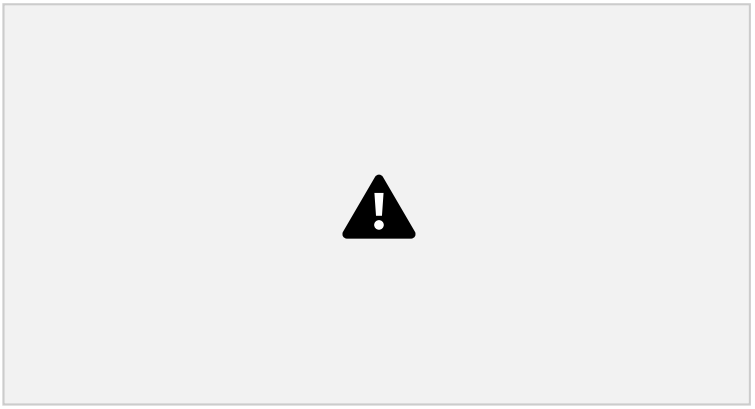


Figure 2. Potentiometric Cell Set-up

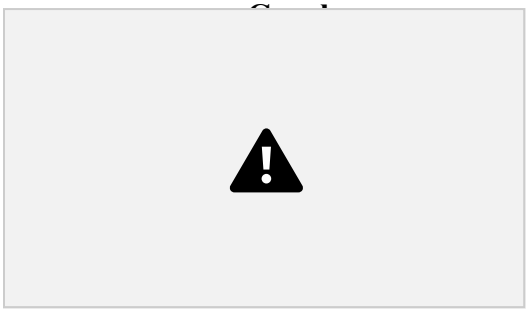
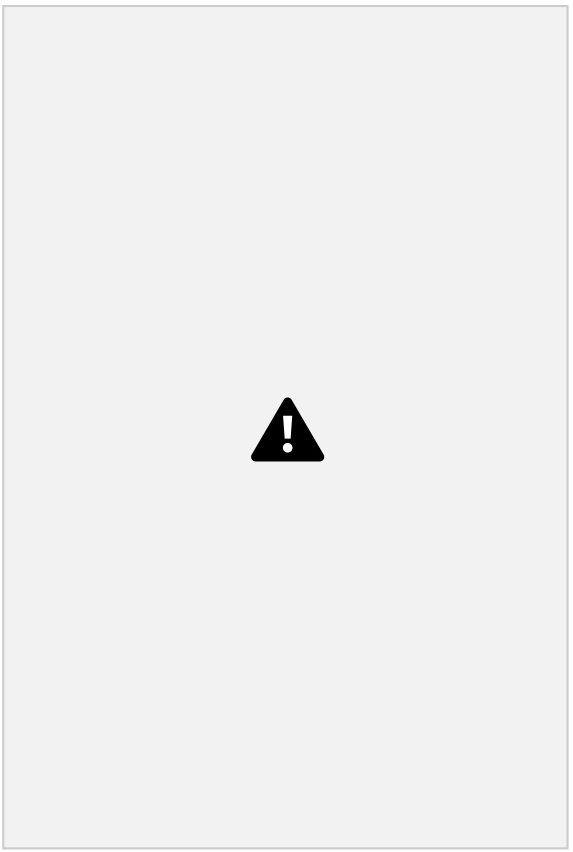


Fig. 3a graph of emf vs volume of NaOH



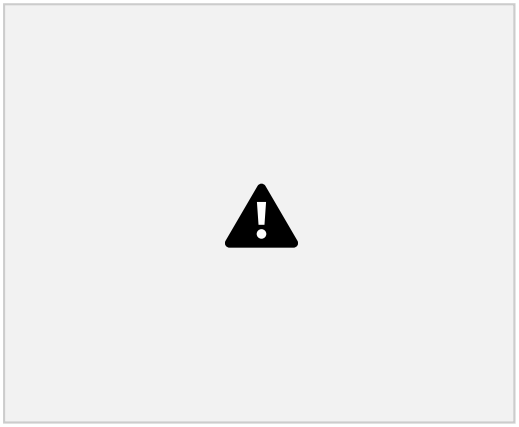


Fig. 3c graph of *p*H vs volume of NaOH **Fig. 3b graph of I st & II nd derivative**

Calculations:

(1) Calculate *p*H of the solution after every addition using the E_{cell} observed at each step using the following equation: $pH = \frac{0.4574 - ••}{0.0592}$

(2) **From the graph of**:

(a) E_{cell} against volume of NaOH solution and (b) $(\Delta^2E/\Delta V^2)$ against volume of 0. 1N NaOH solution required to neutralise 10 cm^3 of given strong acid (HCl) $V_x = \frac{••_1 + ••_2}{2}$

∵10cm^3 of HCl = V_x cm^3 of 0.1N NaOH

∴ Normality of HCl = N_{HCl} * 10 = V_x * 0.1

∴ N HCl = (V_x * 0.1) / 10 = *A*N

Knowing normality of the supplied HCl after dilution we have,

1000cm^3 of 1N = 36.5 g of HCl

∴ 100 cm^3 of *A*N HCI = (100*A*36.5) / 1000 =_____g of HCl.

Results:

(1) The equivalence point from the graph:

(i) $\Delta E/\Delta V$ against volume V = _____ cm^3

(ii) *p*H against volume V = _____ cm^3

(2) Volume of 0.1N NaOH solution required to neutralise 10 $cm^3$of the diluted HCl, V_x (mean)= _____ cm^3

(3) Amount of HCl in the given solution = _____ g

****

Chemical Kinetics

Experiment No. 1

Aim: Compare the strengths of HCl and H_2SO_4 by studying kinetics of acid hydrolysis of methyl acetate.

Theory: The rate of hydrolysis of an ester (i.e. methyl acetate) is found to be directly proportional to the concentration of H^+ions of the acid used as a *catalyst*.

Thus the specific reaction rate of acid catalysed hydrolysis of an ester can be used to compare concentrations of hydrogen ions in equinormal solutions of the two acids.

If equal volumes of the two acids HCI and H_2SO_4 of same normalities are used for catalysing the hydrolysis of an ester and if K_1 and K_2 are specific reaction rates of with HCI and H_2SO_4 respectively, then the ratio of strengths (*S*) is given by:

$$\frac{K_{H_2SO_4}}{K_{HCl}} = \frac{K_1}{K_2}$$

Requirements: Glass stoppered bottles, distilled water, burette, 100cm^3conical flask, 5cm^3pipette, 0.5N HCl solution, 0.5N H_2SO_4 solution, 0.25N NaOH solution, methyl acetate, ice, 1% phenolphthalein indicator etc.

Procedure:

(1) The two Sets of the experiment can be represented as:

Set I: 5 $cm^3$of methyl acetate + 100 $cm^3$of 0.5N HCl

Set II: 5 $cm^3$of methyl acetate + 100 cm^3 of 0.5N H_2SO_4

(2) Carry out the above two sets by titrating 5 $cm^3$of reaction mixture at 0, 5, 10, 15, 20, 25 and 30 minutes using 1% phenolphthalein indicator and 2-3 pieces of ice against 0. 25N NaOH solution from the burette. Record the readings. (T_t)

(3) Using the supplied T_∞ values, calculate the values of specific reaction rate for ***Set I*** and ***Set II.***

(4) Determine the values of specific reaction rate for ***Set I*** and ***Set II*** by plotting graph of $\log_{10}(a-x)$ vs t (min).

Observations:

Set I : 5 $cm^3$of methyl acetate + 100 $cm^3$of 0.5N HCl

At room temperature T_1 = _____°C , T_∞ = _____ cm^3 (Given), x = T_t - T_o cm^3, a = ($V_\infty - V_0$)

Time (t) min.	Titre/Burette Reading T_t (cm^3)	$(a-x) = ●●_{\infty} - ●●_{●●}$ (cm^3)	$\log_{10}(a-x)$	$●● / ●● - ●●$	$\log ●●_{●●} / ●● - ●●$	$●● = 2.303 / ●● \log ●● / ●● - ●●$
00	T_0					
05						
10						
15						
20						
25						
30						
					Mean k_1	min^{-1}

Similarly, prepare the observation Table for Set II and calculate mean K_2 value with H_2SO_4.

Chemical Kinetics

●●log ●● / ●● − ●●

●● = 2.303

Result and Interpretation: 1. Values of *K* in min^{-1}:

●●[log ●● − log(●● − ●●)]

∴ log (a-x) = log a = (kt / 2.303)

Thus Plot log (a-x) against time t for both the Sets and obtain values of *K* from the slope

*K = - slope * 2.303*

●●●●●●●●●●●●● ●●●●●● ●●●●●●●●●●●●●●●●●●●●●●●●●●●●:

●● = 2.303

Method	***Set I***	***Set II***
By Calculation	k_1 =	k_2 =

By Graph	k_1=	k_2 =

2. Relative strength of acids:

$$\frac{k_{H_2SO_4}}{k_{HCl}} = \frac{k_1}{k_2} = \frac{\text{Slope of } H_2SO_4}{\text{Slope of HCl}}$$

****

Paper Chromatography

Experiment No. 1

Separation of Ni^{+2}, Cu^{+2} and Fe^{+3} metal ions.

Aim: To separate metal ions from a given mixture by *ascending paper*

chromatography. **Requirements**:

(A) Materials:

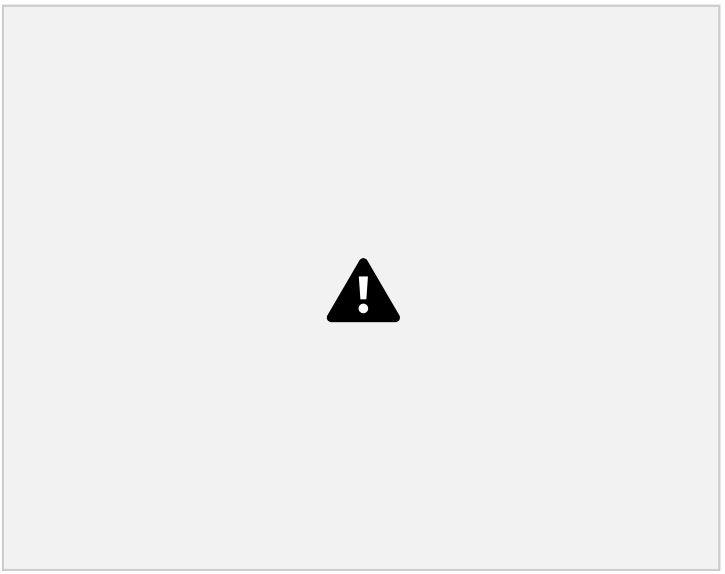
Whatman filter paper No.1 strip (about 25 cm * 8 cm), paper chromatographic jar having 30 cm height and 10 cm diameter, capillary tubes etc.

(B) Chemicals:

(i) ***Salt solution***: 1% acidified aqueous solution of nitrates of three cations.

(ii) Developing solvent: A mixture of 22 $cm^3$of acetone, 105 cm^3 of water and 2 cm^3 of Conc. HCl.

(iii) Spraying reagent: Saturated alcoholic solution of dimethyl glyoxime reagent and saturated ammonia gas.



Procedure:

(1) Fill the jar with a developing solvent so that height of solvent is approximately 2 - 4 cm from the bottom. Cover it with a lid and allow it to stand for some time, so that the jar becomes saturated with the solvent vapours.

(2) Draw a starting line with lead pencil about 2 cm away from one edge of the filter paper strip (base line). Make four cross marks labelled as A, B, C, D and X on this line 2 cm apart from each other as from the edge of the paper. (as shown in Fig.).

(3) Prepare a mixture of metal ions solution (A), (B), (C) and (D) in equal proportions.

(4) Apply by means of a capillary tube, solution of one of the metal ions on cross marked as A taking care that the drop does not spread more than 1 mm.

(5) By means of another capillary tube, apply solution of another metal ion on cross marked as B taking care as in step No.3.

Paper Chromatography

(6) Similarly, by means of another capillary tube, apply solution of another metal ion on cross marked as C & D taking care as in step No. 3.

(7) Now, apply a drop of mixture solution by means of a capillary tube on cross marked as X taking care as described in step No.3.

(8) Allow the spots to dry and fix the other end of the filter paper strip on a glass rod by means of U clip.

(9) Carefully dip the filter paper strip into the jar by taking the following

precautions: (a) It should not touch the walls of the jar.

(b) Developing agent should be below baseline.

(10) Cover the jar with the lid. After the solvent has travelled a distance of about 15 cm, take out the filter paper strip which is now called chromatogram and let it dry in air. Note the distance travelled by the solvent front.

(11) Expose the chromatogram to another jar containing liquor ammonia vapours to neutralise and spray the mentioned reagents for respective ion(s) given in the table

Ions	Spraying reagent	Colour
Copper : Cu^{2+}	Ammonia	Blue
Nickel : Ni^{2+}	Dimethyl glyoxime (dmg)	Pink
Ferric : Fe^{3+}	Ammonia	Rust

(13) Measure the distance travelled by developing solvent (l_{Sol}) and by spots as length: $l_{Fe^{3+}}$, $l_{Cu^{2+}}$ & $l_{Ni^{2+}}$ calculate R*f* values for ion using the given relation,

$$R_f = \frac{\text{Distance travelled by ion from base line}}{\text{Distance travelled by solvent from base line}} = \frac{h}{l_{Sol}}$$

Note: The sequence of R*f* values is as follows: **$Fe^{+3} > Cu^{+2} > Ni^{+2}$**

Expected approximate R*f* values of metal ions are: **$Fe^{+3} = 0.98$, $Cu^{+2} = 0.71$ & $Ni^{+2} = 0.39$**

Result: R*f* values of the ions,

(i) Cu^{2+} ion = _____ (ii) Ni^{2+} ion = _____ (iii) Fe^{3+} ion = _____

****

Solvent Extraction

Experiment No. 1

Aim: To investigate the partition of Ferric ions between aqueous and organic phase (Ethyl acetate) and thereby to determine,

(a) the partition coefficient of Fe(III) between water and ethyl acetate.

(b) the extraction efficiency of ethyl acetate as the solvent of extraction.

Theory: Iron (III) can be separated using 6M HCl when iron (III) gets converted to $[H^+(FeCl_4)^-]$. This ion gets extracted in ethyl acetate. This will separate iron (III) in organic phase whereas, a small portion remains in the aqueous phase. The iron (III) present in organic phase is stripped into the aqueous phase and then can be estimated by titrating with standard Potassium dichromate solution

by using 1% diphenylamine indicator.

Requirements: Ferric alum solution, ethyl acetate (A.R.), 6M HCl, 0.01N $K_2Cr_2O_7$ solution, 1% diphenylamine indicator, 3% $SnCl_2$ solution, 5% $HgCl_2$ solution, acid mixture of H_2SO_4 and H_3PO_4, Conc. HCl, 25 cm^3 pipette, 100 cm^3 flask, 150 or 100 cm^3 separating funnel, 100 cm^3 measuring cylinder, water bath, 6M HCI, distilled water etc.

Procedure:

Part 1:

(1) Take 10 cm^3 or the supplied (A cm^3) of 0.1N ferric ammonium sulphate solution in a 100 cm^3 beaker and add 13 cm^3 of 6M HCl to it. Stir it well.

(2) Transfer this solution to 250 cm^3 standard measuring flask very carefully and dilute it to the mark with distilled water.

(3) Pipette out 25 cm^3 of it in a 250 cm^3 conical flask. Add 5 cm^3 of Conc. HCl to it, shake well and heat to near boiling.

(4) To this hot solution, add dropwise 3% $SnCl_2$ solution from the burette, to reduce Fe^{3+} ions to Fe^{2+} ions. (Solution becomes almost colourless). Add 1- 2 drops of 3% $SnCl_2$ solution in excess to it and cool the solution rapidly under tap water.

(5) Add 5 – 10 cm^3 of 5% $HgCl_2$ solution to it. Silky white precipitates are obtained. (If grey or black precipitates form, then reject the whole solution).

(6) Add 5 – 10 cm^3 of acid mixture of H_2SO_4 and H_3PO_4 to it and shake well. (7) Add 10 cm^3 of distilled water and 2-3 drops of 1% diphenylamine indicator to it. (8) Shake well and titrate it against 0.01N $K_2Cr_2O_7$ solution from the burette. End point will be from green / green floating particulates to violet colour. (X_1 cm^3)

Part II:

(1) Pipette out 25 cm^3 of the diluted Fe^{3+} ions solution in a 150 or 100 cm^3 separating funnel. Add 25 cm^3 of ethyl acetate (A.R.) using a 100 cm^3 measuring cylinder to it.

(2) Cork the separating funnel and shake it cautiously for 5 minutes (vigorous shaking should be avoided, as ethyl acetate is miscible with water).

(3) On standing, two layers separate out. Transfer the lower (aqueous) layer to a 100 cm^3 beaker. (4) Boil it in a water bath to remove traces of ethyl acetate (if any) from it. (5) Cool and transfer it to a 100 cm^3 conical flask.

(6) Follow step Nos. 4 to 9 of Part I. (X_2 cm^3)

Reactions:

(1) $2Fe^{3+} + 6HCl \rightarrow 2FeCl_3 + 3H_2SO_4$

(2) $2FeCl_3 + SnCl_2 \rightarrow 2FeCl_2 + SnCl_4$

(3) $SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + Hg_2Cl_2$ (white precipitate)

(4) $SnCl_4$ (excess) + $Hg_2Cl_2 \rightarrow SnCl_4 + Hg$ (Grey precipitate)

(5) $6FeCl_2 + K_2Cr_2O_7 + 14HCl \rightarrow 6FeCl_3 + 2KCl + 2CrCl_3 + 7H_2O$

Observation and Calculations:

Part I:

25 cm³ of diluted Fe (III)solution = X_1 cm^3 0.01N $K_2Cr_2O_7$ solution.

250 cm³ of diluted Fe (III)solution = 10 X_1 cm^3 0.01N $K_2Cr_2O_7$ solution.

1000 cm³ of 0.01N $K_2Cr_2O_7$ solution = 55.85 g of iron

∴ 10X_1 cm^3 of 0.01N $K_2Cr_2O_7 = \frac{55.85 * 10 * 0.01}{1000}$ = _____ g of iron (w_o)

Part II:

25 cm³ of diluted Fe (III)solution = X_2 cm^3 0.01N $K_2Cr_2O_7$ solution.

250 cm³ of diluted Fe (III)solution = 10 X_2 cm^3 0.01N $K_2Cr_2O_7$ solution.

1000 cm³ of 0.01N $K_2Cr_2O_7$ solution = 55.85 g of iron

∴ 10 X_2 cm^3 of 0.01N $K_2Cr_2O_7 = \frac{55.85 * 10 * 0.01}{1000}$ = _____ w_1 g of iron (w_1)

Partition coefficient : $\frac{(w_o - w_1)}{w_o} * 100$ = _____ %

Results:

(1) Partition coefficient of Fe(III) between water and ethyl acetate =_____. (2) Extraction efficiency (%) of ethyl acetate as the solvent of extraction = _____%.

****

Conductometry

Acid – Base titration

Aim: Estimation of given acid by conductometric titration with strong base and calculation of % error.

Theory: Changes in the conductance during the titration of strong acid (HCl) against a strong base (NaOH) are as follows:

(1) The initial conductance is high due to the complete dissociation of strong acid (HCl) and H^+ions have high conductance value.

(2) The addition of strong base (NaOH) in the acid solution gives the following reaction

$$H^+ + Cl^- + Na^+ + {}^-OH \rightarrow Na^+ + Cl^- + H_2O$$

(3) As NaOH is added, H^+ ions combine with ^-OH ions to form undissociated water molecules and H^+ ions get replaced by Na^+ ions having much lower conductance value. Hence, the conductance of the solution goes on decreasing and reaches a minimum (at equivalence point). After the equivalence point, NaOH added remains unreacted. Being a strong base, it ionises completely producing ^-OH ions of high conductivity. Hence, conductance increases again.

(4) A 'V' shaped curve is obtained. The point of intersection of the two segments in the graph gives the equivalence point of the titration as shown in the Figure 4.

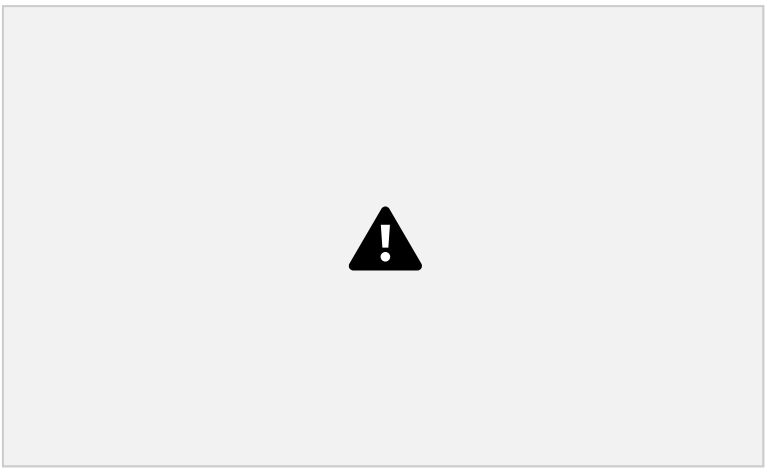


Figure 4. Conductometric titration curve for Strong Acid – Strong Base

Requirements: Conductometer and conductivity cell, burette, 100 cm³ standard measuring flask, beaker, pipette, conductivity water, 0.1N NaOH solution, 1N HCl solution etc. **Procedure**:

(1) Dilute the given acid solution using conductivity water to 100 cm³ in a standard measuring flask. Shake the solution well.

(2) Rinse and fill the burette with supplied 0.1N NaOH solution.

(3) Pipette out 10 cm³ of the diluted solution in a clean 100 cm³ beaker.

(4) Place the conductivity cell in the beaker containing acid solution and add just sufficient quantity of conductivity water to it, so that platinum plates immerse in solution completely. (5) Insert a glass rod and stir the solution well. Do not take it out of beaker till titration is complete. (6) Connect the cell to the conductometer and determine the initial conductance of the solution by selecting the proper range (As instructed).

(7) Add a small volume (say 0.5 cm³) of NaOH solution at a time from the burette, stir the solution well and note the resultant conductance value. Continue addition until the conductance values start

increasing. Take 5 to 6 more readings.

(8) Plot the graph of conductance against the volume of NaOH added. It gives a 'V' shaped curve. (9) The volume corresponding to the intersection of two lines (V_x) gives the equivalence point of titration. Note the volume V_x.

(10) Calculate the normality and strength of the acid solution as given in the calculations.

Observations and Calculations:

(1) Temperature= _____°C (___ *K*).

(2) Normality of NaOH solution = 0.1N.

(3) Volume of the diluted acid pipetted out = 10 cm³.

(4) Equivalent weight of the acid (HCl) = 36.5

Observation Table:

Obs. No.	Volume of NaOH solution added in cm³	Conductance (1/R) mhos [S]

Calculations:

(1) Volume of NaOH solution required for equivalence point (V_x) = _____ cm³ [By graph]

(2) Normality of HCl = $\frac{\text{Volume of NaOH}}{\text{Volume of HCl}} * \frac{\text{Normality of NaOH}}{}$

$= \frac{0.1 * \text{Volume of NaOH}}{\text{Volume of HCl}}$

$10 = $ _____ N

(3) 10 cm³of the diluted acid solution required V_x cm³ of 0.1N NaOH solution. ∴

100 cm³ of the diluted acid solution will require 10 V_x cm³ of 0.1N NaOH solution ∴

1000 cm³ of 1N NaOH = 36.5 g of HCl

∴ 10 V_x cm³ of 0.1N NaOH = (36.5 * 10V_x * 0.1)/1000 g of HCl

= _____g of HCl

(**Note**: While doing calculations, substitute the titration reading obtained in place of V_x).

$$\% \text{ Error} = \left| \frac{\text{Experimental value} - \text{Accepted / Theoretical value}}{\text{Accepted value}} \right| \times 100$$

Conductometry

Results:

(1) Volume of NaOH solution required for the equivalence point (V_x) = _____

cm³ (2) Normality HCI = _____N.

(3) Amount of HCI present in the given solution = _____ g.

Note: For weak acid against strong base, acetic acid can be used as a weak acid and the experiment may be performed similarly as described above. The graph of conductance against volume of titrant (NaOH) will be as follows:

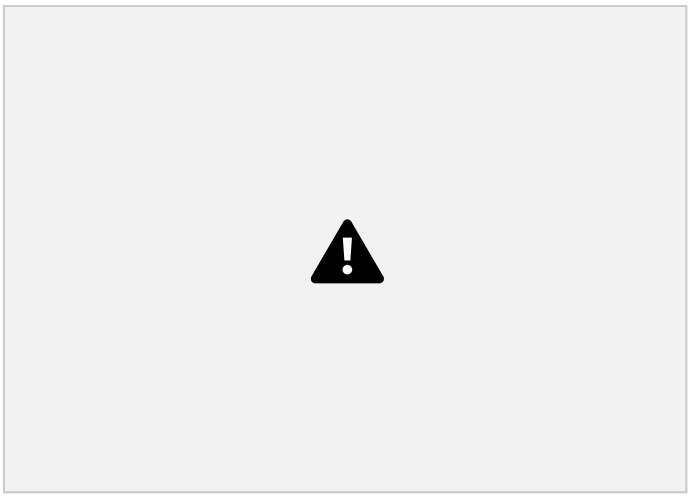


Figure 5 Conductometric titration curve for Weak Acid – Strong Base

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pH Metry

Determination of Buffer Capacity

Aim: Determination of buffer capacity of buffer solutions.

Theory: The buffer solutions are the solutions which resist to change of *p*H on addition of small

amount of acid or base. The capacity of the buffer solution is the amount of acid or base added to change the *p*H value of buffer by 1 *p*H unit.

Buffer capacity = ◆◆◆◆◆◆◆◆◆◆◆◆◆ ◆◆◆◆ ◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆ ◆◆◆◆
◆◆◆◆◆◆◆◆◆/◆◆◆◆l
◆◆◆◆◆◆◆◆◆◆◆◆◆ ◆◆◆◆ ◆◆◆◆◆◆◆◆◆◆◆◆◆ ◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆◆ ◆◆◆◆ ◆◆◆◆^{3}

Requirements: 0.1M sodium acetate solution, 0.1M acetic acid solution, 0.1N HCl solution, 0.1N NaOH solution, *p*H meter, distilled water, glass electrodes, 100 cm^3beakers, 25 $cm^3$and 10 cm^3pipettes, 50 cm^3burette, etc.

Procedure:

(1) Prepare acid buffer by mixing 25 $cm^3$of 0.1M sodium acetate solution and 25 $cm^3$of 0.1M acetic acid solution in a 100 cm^3clean dry beaker.

(2) Note down the *p*H of the above solution.

(3) Take 10 $cm^3$of the acid buffer solution prepared above, in 100 cm^3beaker and add 25 $cm^3$of distilled water to it.

(4) Insert the glass electrode in the above solution and measure the *p*H of the above solution.

(5) Fill the burette with 0.1M HCl solution.

(6) Add 1$cm^3$of 0.1N HCl solution each time from the burette and note the change in *p*H. (7) Add 0.1N HCl solution in the beaker till the *p*H of the buffer solution changes by about 1 *p*H units. (V_1 cm^3).

(8) Repeat the procedure for 0.1 N NaOH solution and note down the change in *p*H by about 1 *p*H unit. (V_2 cm^3).

Observations: ***a. For HCl solution:***

Obs. No.	**Volume of 0.1N HCl solution added in cm^3**	***p*H**
1.		
2.		
3.		
4.		

b. ***For NaOH solution:***

Obs. No.	**Volume of 0.1N NaOH solution added in cm^3**	***p*H**
1.		
2.		
3.		

4.		

pH Metry

Mean volume of NaOH/HCl solution = $\frac{V_1 + V_2}{2}$

Mean volume $\bar{V}$ = _____ cm^3

Mean volume of NaOH/HCl solution in dm^3	Number of equivalents of NaOH/HCl solution mean volume in dm^3 *x* Normality	Buffer capacity
$\frac{\bar{v}}{1000} = x\ dm^3$	x * 0.1 = _____	$\frac{10}{1000} / = \frac{0.1 * V}{0.01} =$ _____ $\frac{}{V * 0.1}$

Results:

Buffer capacity of the given acid buffer = _____

Solution	***Capacity***
acetic acid / sodium acetate, *p*H = 4.75, 0.001M	6.2×10^{-4}
acetic acid / sodium acetate, *p*H = 4.75, 0.01M	0.0058
solution with added ammonia buffer (as used in complexometric Mg^{2+} titration)	0.01
acetic acid / sodium acetate, *p*H = 4.75, 0.1M	0.058
acetic acid/sodium acetate, pH = 4.75, 1M	0.49

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Tools of Analytical Chemistry - II

Filtration Flask, Funnels, Distillation Apparatus, Vaccum Distillation, Centrifuge machine, Electrophoresis, Chromatographic Separation Apparatus, Solvent Extraction, Electrodes and Salt Bridge

1. A filtration flask, also known as a Buchner or vacuum flask, is a robust conical flask featuring a crucial side arm for connection to a vacuum source. This specialized glassware is fundamental for vacuum filtration, a technique that significantly accelerates the separation of solids from liquids. Typically used with a Büchner funnel and filter paper, the vacuum pulls the liquid through, leaving the solid residue behind. Filtration flasks are indispensable in chemistry and biology labs for tasks like collecting precipitates, purifying synthesized compounds, clarifying solutions, and preparing samples for analysis, ensuring efficient and effective separation.

Fig. 8.1 Filtration flasks

2. Vacuum filtration is a rapid technique for separating solids from liquids using a vacuum. A filtration flask connects to a vacuum source, creating a pressure difference that pulls the liquid through a filter paper held in a Büchner funnel atop the flask. This accelerates separation compared to gravity filtration. It's widely used to collect precipitates, purify solids, clarify solutions, and prepare samples in chemistry, biology, and environmental labs, ensuring efficient and thorough separation.

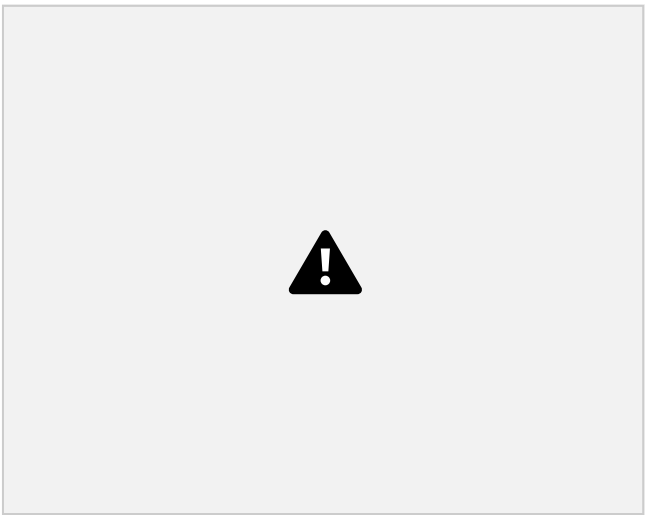
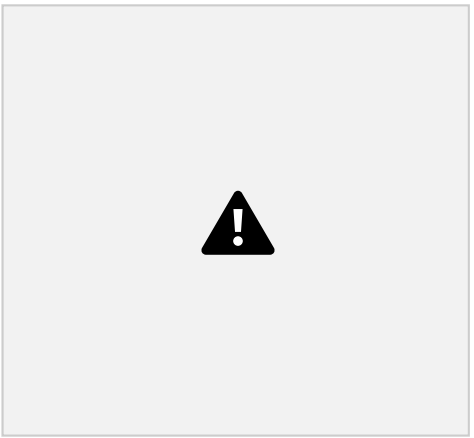


Fig. 8.2 Vaccum Filtration

3. Gravity filtration is a simple separation technique relying on gravity to pull a liquid through a filter medium, leaving solid particles behind. A funnel holds the filter paper, and the mixture is poured in, allowing the liquid (filtrate) to pass into a receiving flask. It's often used to remove solid impurities from a liquid, like drying agents, or when the liquid is the desired product. Though slower than vacuum filtration, it's useful for larger particles and volatile solvents, requiring minimal equipment.

4. A funnel is a cone-shaped utensil with a wide opening at the top and a narrow tube at the bottom, designed to guide liquids or powders into containers with small openings, preventing spills and waste. Historically made from clay, funnels now come in various materials like plastic, glass, and metal, each suited for different uses.

In the lab, funnels are essential for transferring liquids, adding reagents dropwise (dropping funnel), separating immiscible liquids (separatory funnel), and vacuum filtration (Büchner funnel). Powder funnels have a wide bore to prevent clogging when transferring solids. Everyday uses include filling bottles, transferring cooking oil, and even in car maintenance.



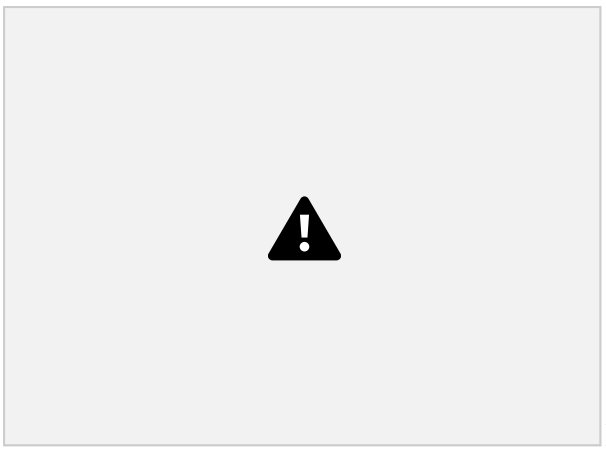


Fig. 8.3 Funnels

5. Distillation is a separation technique that exploits differences in boiling points of liquids in a mixture. The mixture is heated, and the component with the lower boiling point vaporizes first. This vapor is then cooled and condensed back into a liquid [1] (the distillate), which is collected separately. By repeating this process (fractional distillation), components with closer boiling points can be effectively separated. Distillation is crucial for purifying liquids, separating solvents, and in industries like petroleum refining and alcohol production.

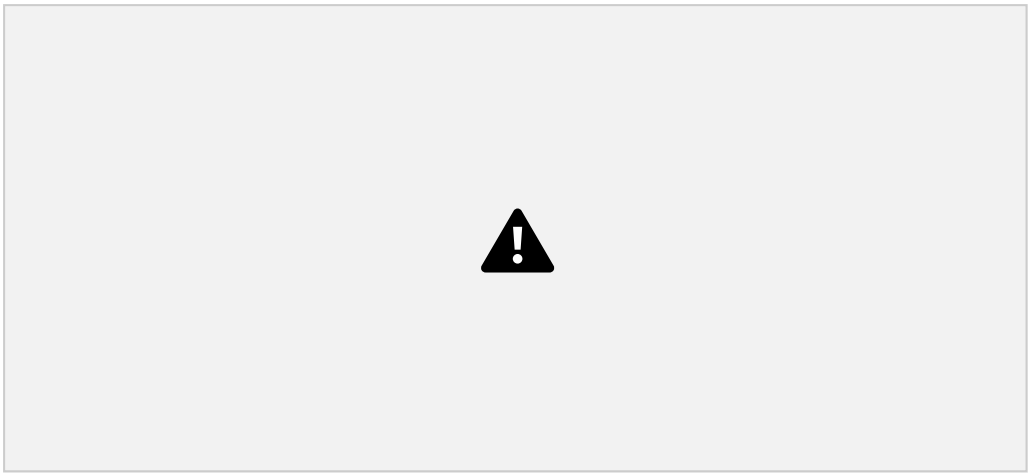


Fig. 8.4 Distillation Unit

6. Vacuum distillation is a technique used to separate liquids with high boiling points or those that decompose at their boiling points. By reducing the pressure in the distillation apparatus, the boiling points of the liquids are lowered, allowing for gentler separation at lower temperatures. This prevents thermal degradation and saves energy.

It's crucial in industries like petroleum refining (separating heavy hydrocarbons), pharmaceuticals (purifying heat-sensitive APIs), and food & beverage (extracting delicate flavors and creating non alcoholic drinks). Vacuum distillation enhances yield, purity, and safety for such applications.

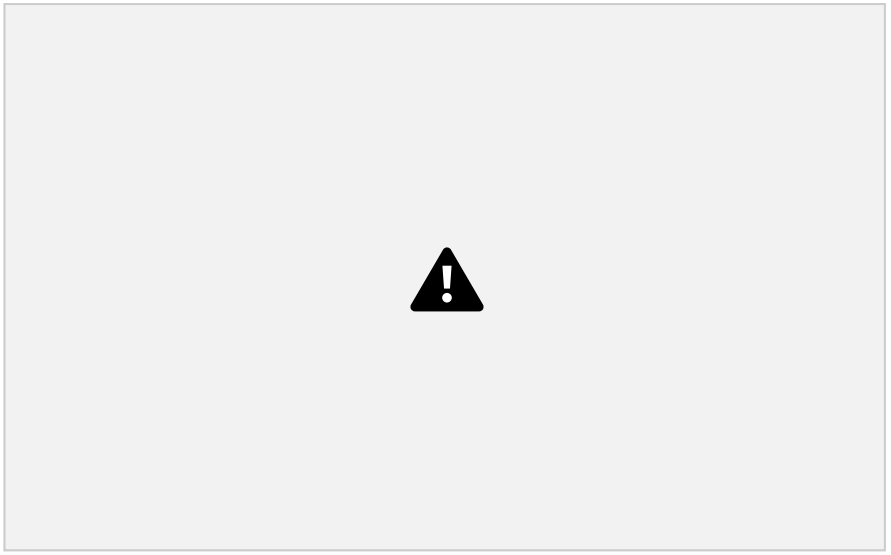


Fig. 8.5 Vacuum distillation

7.A **centrifuge** machine is a vital laboratory instrument that separates components of a liquid or suspension based on their density using centrifugal force. It spins samples at high speeds, causing denser particles to move outward and settle at the bottom, while lighter ones remain near the top. Crucial in various fields like medicine (blood separation), research (DNA/protein isolation), and industry (purification), centrifuges come in different types (benchtop, high-speed, ultracentrifuges) to suit diverse applications and sample volumes.

Fig. 8.6 Centrifuge Machine

8.An electrophoresis apparatus in chemistry is used to **separate charged molecules** based on their size and charge by applying an electric field. While it's a cornerstone technique in biochemistry and molecular biology for separating biomolecules like DNA, RNA, and proteins, it also has significant

applications in pure chemistry and related fields.

In a chemistry lab, an electrophoresis apparatus can be used for:

· Separation and purification of synthesized molecules: Charged organic or inorganic molecules can be separated from reaction mixtures based on their electrophoretic mobility. · Analysis of ionic compounds: Identifying and separating different ions present in a solution.

· Studying the properties of charged polymers and nanoparticles: Analysing their size, charge, and behaviour in an electric field.

· Separation of dyes and pigments: Analyzing the components of a mixture of dyes or pigments.

· Environmental chemistry: Separating and identifying charged pollutants in water or soil samples.

· Food chemistry: Analyzing charged additives or components in food products. · Pharmaceutical chemistry: Determining the purity of charged drug molecules or separating related ionic species.

· Forensic chemistry: Analyzing charged components in samples like inks or explosives. The fundamental components of the apparatus remain the same as in biological applications: a chamber with electrodes, a power supply, a buffer solution to carry the current and maintain pH, and a support medium (which could be a gel, paper, or a capillary). The specific type of electrophoresis (e.g., gel electrophoresis, capillary electrophoresis) and the detection methods used will depend on the nature of the chemical substances being analysed.

Fig. 8.7 Electrophoresis Apparatus

9. A developing chamber in chromatography is a closed container that houses the stationary phase (like a TLC plate or paper) and the mobile phase (solvent or solvent mixture) during the development stage of the separation process.

Key functions and features:

· *Controlled Environment*: It creates a controlled atmosphere saturated with solvent vapors, which ensures even and consistent migration of the mobile phase and thus better separation of the sample components. This saturation minimizes solvent evaporation from the stationary phase, which could lead to inconsistent results.

· *Vapor Saturation*: Often, the chamber contains a piece of filter paper lining the walls that is wetted with the mobile phase. This increases the surface area for evaporation and helps achieve saturation more quickly.

· *Protection*: The closed nature of the chamber protects the developing chromatogram from dust, drafts, and other environmental factors that could interfere with the separation. · *Various Designs*: Developing chambers come in different shapes and sizes depending on the type of chromatography being performed (e.g., TLC tanks, jars for paper chromatography, specialized chambers for horizontal or two-dimensional TLC).

· *Material*: They are typically made of glass or inert plastic, allowing visualization of the solvent front's movement.

In essence, the developing chamber is crucial for obtaining reliable and reproducible results in chromatographic separations by providing a stable and controlled environment for the mobile phase to interact with the stationary phase and the sample.

Fig. 8.8 Development Chamber

10. Solvent extraction in chemistry, also known as liquid-liquid extraction or partitioning, is a separation technique that exploits the **differential solubility** of a compound (solute) between two **immiscible or partially immiscible liquids**. Typically, one solvent is aqueous (polar) and the other is an organic solvent (non-polar or less polar).

The fundamental principle relies on the **distribution law (Nernst distribution law)**, which states that at equilibrium and a constant temperature, the ratio of the concentrations of a solute in two immiscible solvents is constant, provided the solute has the same molecular form in both solvents. This ratio is known as the **distribution coefficient (D)** or partition coefficient:

$$D = C_{(aq)} / C_{(Org.)}$$

where C_{org} is the concentration of the solute in the organic phase, and C_{aq} is the concentration of the same solute in the aqueous phase at equilibrium. This ratio indicates the preference of a solute for one phase over another in a two-phase system.

The process involves:

a. *Contacting* the solution containing the solute with the extracting solvent in a separatory funnel.

b. *Mixing* the two phases thoroughly to allow the solute to distribute between them based on its solubility.

c. *Allowing the layers to separate* due to their immiscibility and different densities. d. *Separating* the two layers, one enriched with the solute (the extract) and the other depleted. e. *Recovering* the solute from the extract by evaporating the solvent.

Key factors influencing solvent extraction include:

- *Choice of solvent:* The extracting solvent should have a high affinity for the target solute, be immiscible with the original solvent, be easily separable, and be non-reactive.
- *Distribution coefficient (D):* A higher D favours the transfer of the solute to the extracting solvent.
- *Number of extractions:* Multiple extractions with smaller volumes of fresh solvent are generally more efficient than a single extraction with a large volume.
- *p*H *(for ionisable compounds):* The pH can significantly affect the solubility and thus the distribution of acidic or basic compounds.
- *Temperature:* Affects solubility and the distribution coefficient.

- *Salting out:* Adding salts to the aqueous phase can decrease the solubility of some organic compounds in water, enhancing their transfer to the organic phase.

Solvent extraction is a versatile technique with **numerous applications** in chemistry, including: • Separation and purification of organic and inorganic compounds.
• Isolation of natural products from plant and animal sources.

• Removal of impurities from reaction mixtures.
• Extraction of metal ions from aqueous solutions (hydrometallurgy).
• Environmental analysis (e.g., extraction of pollutants from water and soil). •
Pharmaceutical industry (e.g., drug purification, extraction of antibiotics). •
Food industry (e.g., extraction of vegetable oils, decaffeination).

It is a relatively simple, cost-effective, and widely used method for separating and purifying substances in various chemical and related fields.

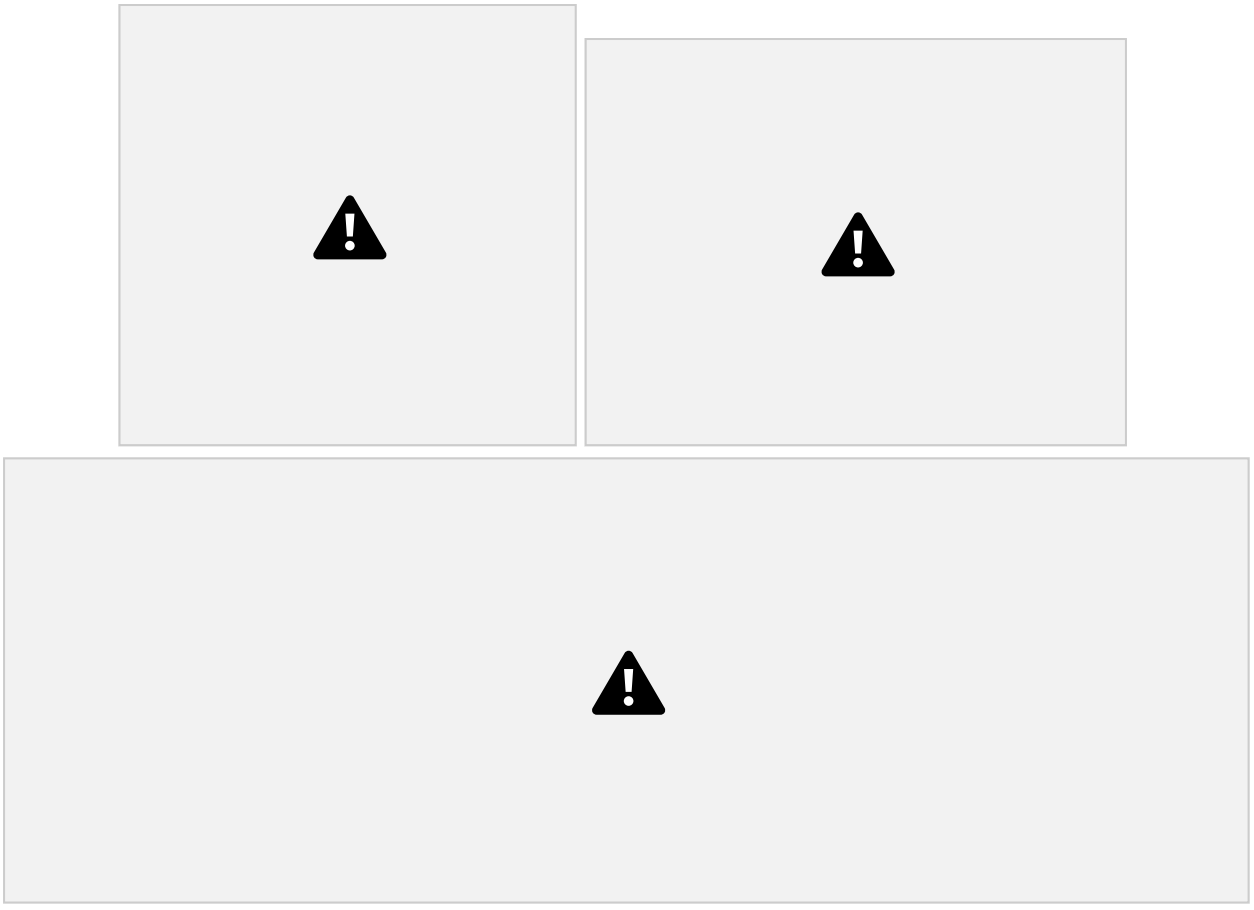


Fig. 8.9 Separating Funnel (Solvent Extraction)

11. Electrodes:

i. The **standard hydrogen electrode (SHE)** is a reference electrode with a defined potential of zero volts at standard conditions (298 K, 1 atm pressure, and 1 M H^+ concentration). It consists of a platinized platinum electrode immersed in an acidic solution with hydrogen gas bubbling through it. The SHE serves as the **primary standard** for measuring the electrode potentials of other half-cells. By constructing a galvanic cell with the SHE and another electrode, the potential difference measured directly corresponds to the standard electrode potential of that electrode. While fundamental, the SHE is not routinely used in labs due to practical challenges in its setup and maintenance.