Estimation of Iron in Haematite

Aim

To estimate the percentage of iron in haematite solution containing 1.5g of haematite/litre by redox titration

Apparatus

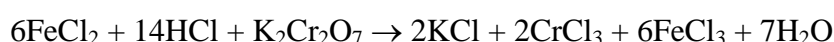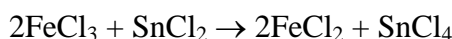Burette, pipette, beaker, glass rod,

Chemicals

Haematite solution, Stannous chloride, Mercuric chloride, potassium dichromate, diphenylamine, phosphoric acid, hydrochloric acid, sulfuric acid

Principle

Haematite is an oxide ore of iron, existing as ferric oxide (Fe_2O_3). On treating with Conc. HCl, ferric oxide dissolves in the acid as ferric chloride

$$Fe_2O_3 + 6HCl \rightarrow 2FeCl_3 + 3H_2O$$
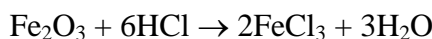
Since iron in $FeCl_3$ is already in the highest possible stable oxidation state (Fe^{3+}), it cannot be oxidized further to a stable higher oxidation state. Therefore, it is first reduced to ferrous state and then titrated with potassium dichromate solution, which is an oxidizing agent. Ferric chloride is reduced by stannous chloride in presence of HCl

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

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

A slight excess of $SnCl_2$ is added to ensure complete reduction of $FeCl_3$ to $FeCl_2$. Since $SnCl_2$ can reduce potassium dichromate, the excess of $SnCl_2$ added to heamatite solution is to be destroyed by adding strong solution of mercuric chloride

$$SnCl_2 + 2HgCl_2 \rightarrow SnCl_4 + Hg_2Cl_2\downarrow$$
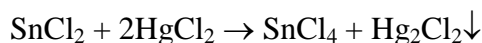
A silky white precipitate of mercurous chloride should be obtained. A black precipitate of finely divided mercury may be produced if large excess of $SnCl_2$ is present or mercuric chloride solution is added slowly. This finely divided mercury will reduce the potassium dichromate and also reduces ferric ion to ferrous ion slowly in due course.

$$Hg_2Cl_2 + SnCl_2 \rightarrow SnCl_4 + 2Hg\downarrow$$
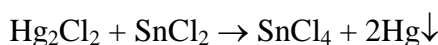
The trial must be discarded if a black precipitate is produced. The trial should also be discarded if no precipitate is obtained on the addition of mercuric chloride because the complete reduction of ferric ion to ferrous ion is not ensured.

As the titration involves oxidation reduction reaction, it is known as oxidation reduction titration. The indicator used for the detection of the endpoint is an oxidation reduction indicator called diphenylamine. In

presence of strong oxidizing agent, it first undergoes irreversible chemical oxidation to give a colorless diphenylbenzidine, which is the real indicator and is reversibly further oxidized to diphenylbenzidine violet.

Diphenylamine   Diphenylbenzidine   Diphenylbenzidine violet
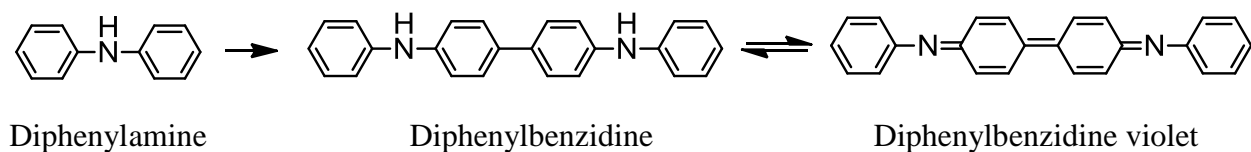
When ferrous ions are exhausted in the solution at the end point, addition of further quantity of dichromate oxidizes colorless diphenylbenzidine to diphenylbenzidine violet. Therefore the endpoint is indicated by the color change from green (due to the Cr^{3+} ion) to greenish violet. But the color change is sharp and detectable only when the formal potential of the indicator is intermediate of the solution titrated (Fe^{3+}/Fe^{2+}) and that of the titrant (Cr^{6+}/Cr^{3+}) and should differ by about at least 0.15 volts from the other two. The formal potentials of Cr^{6+}/Cr^{3+}, Fe^{3+}/Fe^{2+} and indicator are 1.10, 0.77 and 0.76 volts respectively in sulfuric acid medium. Therefore, the potential of the Fe^{3+}/Fe^{2+} system is lowered to a value of 0.6 volts by the addition of sulfuric acid and phosphoric acid mixture.

Procedure

1. A known concentration of potassium dichromate solution is filled in the burette

2. Pipette out 25ml of haematite solution into a clean conical flask

3. Add about 3ml of Conc. HCl and heat it to just boil
   NOTE: Conc. HCl is extremely corrosive, use with caution. Wash any spills immediately with cold water

4. To the hot mixture, add $SnCl_2$ solution drop by drop until the yellow color is discharged completely. Add one or two drops in excess to ensure the complete reduction of iron
   NOTE: Addition of too much $SnCl_2$ should be avoided as the precipitate formed in the next step will interfere in identifying the end point sharply.

5. Cool the product and add 5 ml of $HgCl_2$ solution at a stretch, now one should see a silky white precipitate.
   NOTE: if black precipitate is observed/no silky white precipitate not observed discard the product

6. Add 100 ml of 1.5 M sulphuric acid, 5 ml of phosphoric acid and 3 drops of diphenylamine to the mixture

7. Now titrate it against potassium dichromate solution taken in the burette till a greenish violet color persists.

8. Repeat the titration for concordant values

Observation and calculation

Burette level	Trial-I	Trial-II	Trial-III
Final reading			
Initial reading			
Vol of $K_2Cr_2O_7$ (ml)			

Volume of $K_2Cr_2O_7$ (V_1) = …………………………………… ml

Normality of $K_2Cr_2O_7$ (N_1) = …………………………………………….N

Volume of Heamatite solution (V_2) = 25 ml

Normality of Haematite solution (N_2) = $\dfrac{V1 \ x \ N1}{25}$

Weight of iron per liter of solution (W) = N_2 x Eq. wt of Iron

$$= N2 \ x \ 55.85 = \text{……………………}.g$$

Percentage of iron in haematite $= \dfrac{W \ x \ 100}{Wt.of \ Haematite}$

$$= \dfrac{W \ x \ 100}{1.5} = \text{………………………}.\%$$

Result
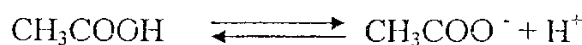
Percentage of Iron in given haematite sample = ……………………%

10. DETERMINATION OF pK$_a$ AND K$_a$ VALUES OF A WEAK ACID

AIM:

To determine the pK$_a$ and K$_a$ values of a weak acid by pH titration method.

PRINCIPLE:

Weak acid is a weak electrolyte and undergoes only partial dissociation in the solution. For example, acetic acid when dissolved in water,

$$CH_3COOH \rightleftharpoons CH_3COO^- + H^+$$

$$\text{The dissociation constant, } K_a = \frac{[CH_3COO^-] \times [H^+]}{[CH_3COOH]}$$

$$pK_a = -\log K_a$$

Higher the value of pK$_a$, lower is the K$_a$ value of the acid and therefore weaker is the acid. pK$_a$ value of a weak acid can be determined by pH titration method. In this method a known volume of the weak acid is titrated with a strong base, and pH of the solution is measured after each small addition of the base. During the titration, the pH of the solution increases gradually till very near to equivalence point and there is a sharp increase in pH at the equivalence point. After the equivalence point the increase in pH is again very gradual. Therefore the equivalence point is indicated by the sharp increase in pH. The equivalence point is determined by plotting volume of the base against $\frac{\Delta pH}{\Delta V}$, as indicated by the volume corresponding to the maximum of the curve.

Once the titration is started, the solution contains weak acid and also its salt with strong base. Thus the mixture is a buffer mixture. The pH of such a mixture is give by Hendersen's equation;

$$pH = pK_a + \log \frac{[salt]}{[acid]}$$

At half equivalence point 50% of the acid is converted in to salt, and therefore,

$$[acid] = [salt]. \text{ Then, } pH = pK_a.$$

Therefore, pH at half equivalence point gives the pK$_a$ value of the weak acid.

PROCEDURE:

Make up the given acid solution in a 250 ml standard flask to the mark and shake well. Pipette out 25 ml of the given weak acid in to a (plastic) beaker. Dip the glass electrode – calomel electrode assembly into the acid and connect the cell to a pH meter. Measure the pH of the solution. Add 1 ml of NaOH at a time into the beaker stir the solution and measure the pH of the solution. Continue the process till a large increase in pH is observed at the end points for the addition of 1 ml of NaOH solution and take four more readings after that. Use the readings to plot a graph of volume of base (x-axis) against pH (y-axis), as shown in the following figures.
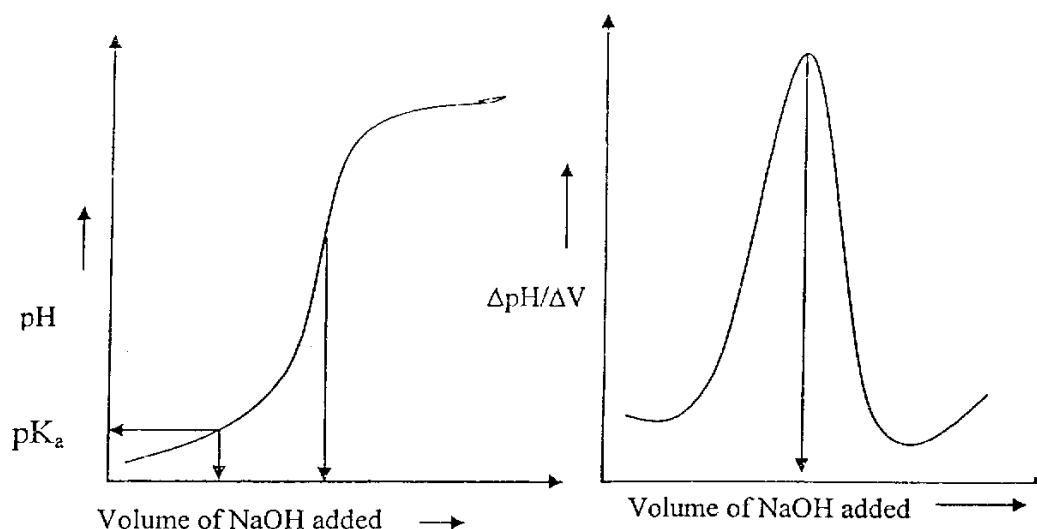
Now clean the beaker well and again pipette out 25 ml of the weak acid. Add NaOH solution, 0.5 ml less than the lower range of the end point directly and then 0.1 ml of NaOH at a time. After each addition, stir the solution and measure the pH of the solution. Continue the titration till a sharp increase in pH is observed for the addition 0.1 ml of NaOH at the end point. Continue the titration further to note down at least four more readings. Plot volume of NaOH against $\frac{\Delta pH}{\Delta V}$ and determine the equivalence point. From the graph of volume of NaOH against pH, determine the value of pK_a as pH at half equivalence point.

OBSERVATIONS AND CALCULATION:

I Trial

Vol. of NaOH added in ml.	pH	Vol. of NaOH added in ml.	pH
1		6	
2		7	
3		8	
4		9	
5		10	

II Trial

Vol of NaOH added in ml.	pH	ΔV	ΔpH	$\frac{\Delta pH}{\Delta V}$	Vol.of NaOH added in ml.	pH	ΔV	ΔpH	$\frac{\Delta pH}{\Delta V}$

RESULT:

From the graph,

i. Equivalence point of the titration = ...ml

ii. pH at half equivalence point = ...

iii. pK_a value of the given weak acid = ...

iv. K_a of the given weak acid = ...

Signature of the staff member

6. ESTIMATION OF CHLORIDE CONTENT IN WATER

AIM:

To estimate the chloride content of the given sample of water, being provided with approximately N/40 silver nitrate solution and pure crystals of potassium chloride.

PRINCIPLE:

Chloride content of water is determined by titrating a known volume of water with silver nitrate solution using potassium chromate as indicator.

Silver nitrate reacts quantitatively with chloride ions present in the water to from a curdy white precipitate of silver chloride.

$$Cl^- + AgNO_3 \longrightarrow AgCl \downarrow + NO_3^-$$
(curdy white)
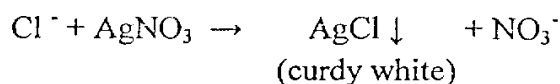
When chromate ions of the indicator are also present in the solution, the added quantity of $AgNO_3$ during titration, preferentially reacts with chloride ions and precipitates them as AgCl. At the end point when chloride ions are completely precipitated as AgCl, further addition of $AgNO_3$ reacts with chromate ions forming brick red colored silver chromate.

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 \downarrow + 2KNO_3$$
(Brick red)
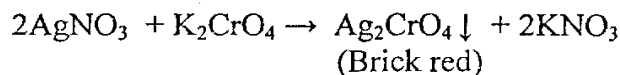
Thus the end point is indicated by the appearance of red tinge in the solution.

The given silver nitrate solution is standardized by preparing a standard solution of potassium chloride and titrating a known volume of it with $AgNO_3$ solution.

$$KCl + AgNO_3 \longrightarrow AgCl \downarrow + KNO_3$$
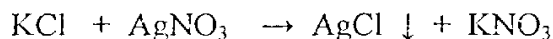
Equivalent wt. of KCl = Mol. wt. of KCl = 74.5

Equivalent wt. of Cl^- = Atomic wt. = 35.5

If the water sample is acidic, it has to be neutralized by the addition of $CaCO_3$ otherwise, the presence of acid converts chromate ion of the indicator to dichromate ion.

$$2CrO_4^{2-} + 2H^+ \longrightarrow Cr_2O_7^{2-} + H_2O$$
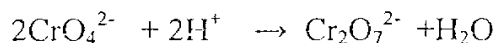
In such cases a blank titration should be carried out with 25ml of distilled water and $CaCO_3$ (same quantity as added into the sample). The blank titre value is subtracted from the estimation titre value.

PROCEDURE:

Preparation of standard potassium chloride solution:

Weigh accurately about 0.5g of KCl crystals and transfer into a 250ml standard flask. Dissolve it in distilled water. Make up the solution to the mark and shake well for uniform concentration.

Standardization of silver nitrate solution

Pipette out 25ml of KCl solution into a clean conical flask. Add 5-6 drops of 5% K_2CrO_4 solution as indicator. Titrate against silver nitrate solution taken in the burette, until a brick-red tinge persists. Repeat the titration for concordant values.

Estimation of chloride content:

Make up the given water sample in a 250 ml standard flask to the mark. Shake well for uniform concentration. Pipette out 25ml of this water sample into a clean conical flask and add 5-6 drops of K_2CrO_4 Indicator. Titrate against silver nitrate solution taken in the burette as before.

OBSERVATIONS AND CALCULATION:

Preparation of standard KCl solution:

Wt. of weighing bottle + KCl   = ...g

Wt. of weighing bottle alone   = ...g

Wt. of KCl transferred   = ...(W) g

$$\text{Normality of KCl, } N_1 = \frac{\text{Weight / litre}}{\text{Eq.wt.}} = \frac{W \times 4}{74.5} = \ldots\ldots\ldots\ldots$$

Standardization of AgNO₃ solution:

In burette	: Silver nitrate solution
In conical flask	: 25ml KCl solution
Indicator	: K_2CrO_4 solution, 5-6 drops
Color change	: Yellow to brick red tinge

Burette level	I	II	III
Final			
Initial	.		
Vol. of AgNO₃,ml			

Agreeing value, V_1 = ...ml

V_1ml of silver nitrate solution reacts completely with 25 ml of .... N KCl solution

$\therefore$ Normality of AgNO₃ solution, $N_2 = \dfrac{V_{KCl} \times N_{KCl}}{V_{AgNO_3}}$

$$= \frac{25 \times N_1}{V_1} = \ldots\ldots\ldots\ldots N$$

Estimation of chloride content:

In burette	: Silver nitrate solution
In conical flask	: 25 ml water sample
Indicator	: K_2CrO_4, 5-6 drops
Color change	: Yellow to brick red tinge

Burette level	I	II	II
Final			
Initial			
Vol.of AgNO₃,ml			

Agreeing value, V_2=....... ml

V_2 ml of AgNO$_3$ of .... N reacts with 25ml of water sample completely.

∴ Normality of water sample w.r.t. Cl⁻, N_3 = $\dfrac{V_{AgNO_3} \times N_{AgNO_3}}{V_{watersample}}$

$= \dfrac{V_2 \times N_2}{25}$ = ...

Weight of chloride in a litre of the solution = N_3 x Eq. wt. of Cl⁻.

= ....x 35.5 = ...g

∴ Chloride content of the solution = ...g/l = ...mg/l

RESULT:

Chloride content in the given sample of water = ...mg/l

Signature of the staff member.

(Note: After completion of the experiment, wash the apparatus with little bench NH$_4$OH soln. followed by water)

Title: Verification of Nernst Equation

Aim: To Verify Nernst equation and determine the standard electrode potential of Copper

Competency: Setting up of Electrochemical cell to determine Electrode Potential

Practical Skills: i) Handling of Potentiometer

ii) Setting up of Electrochemical cell and determination of electrode potential

Practical Significance: Electrode potential values of any metal are directly linked to the reactivity of the metal. Hence whenever a new metal is found it is important to find its electrode potential in order to ascertain its relative reactivity among the metals.

Theory: Every cell is composed of two electrodes. Thus in order to find the potential of a particular electrode under consideration we combine it with another electrode whose potential is known e.g. Calomel Electrode. The EMF (Electromotive Force) can be determined by combining these two electrodes using a Potentiometer.

Potentiometry deals with the measurement of difference in potential between two electrodes which have been combined to form electrochemical cells.

In Potentiometer the principle generally used is that of poggendorff compensation method. This consists in balancing the unknown EMF against a known one in potentiometric circuit. In Otherwards we measure the external potential difference required to stop the passage of current in the cell.

The EMF is related to the Electrode Potential of any Element by the following equation:

$E_{cell} = E_{cathode} - E_{Anode}$ $E_{cell} = EMF$ $E =$ Electrode Potential.

In principle all Standard Electrodes are to be measured using a Primary standard electrode system such as the Hydrogen Electrode. But at the same time we know that it is difficult to build a Primary Hydrogen Electrode in every Laboratory and hence certain Secondary electrode systems have been developed such as the Calomel electrode.

The Standard Electrode potential (E^0) is related to the Electrode potential by the following equation :

$$E = E^0 - \frac{2.303RT}{nF} \log \frac{[\text{Product } (m \times \gamma)]}{[\text{Reactant}(m \times \gamma)]}$$

The above equation is known as the Nernst equation using which the Standard electrode can be found.

The validity of the Nernst equation in finding the Standard Electrode Potential can be verified by using different concentration of the salt solution of the element to be tested.

Requirements: Potentiometer, Calomel Electrode, Copper Electrode, Copper Sulphate, Distilled Water, Volumetric Flasks, Graduated pipette and Beakers.

Precautions: **Handle the Calomel Electrode cautiously.**

Procedure: 1) You are given a stock solution of 1 M $CuSO_4$ solution. Using this solution prepare 0.1, 0.01, 0.05 and 0.005 M solutions using the volumetric flasks with distilled water as solvent provided to you.

2) Place the electrodes in a beaker and pour the most dilute solution first i.e. 0.005 M in this case. The solution should occupy half the capacity of the beaker.

3) Connect the Electrodes to the Potentiometer by following the color codes. Put on the Instrument. Allow the stabilization time of 10 min with proper mixing (by swirling the contents of the flask) of the solution. Read and note the EMF value in the observation table.

4) Transfer back the solution in the respective volumetric flask and repeat step 3 for the remaining solutions.

5) After taking all the readings wash the electrodes with distilled water and place the electrodes in a empty beaker. Put off the instrument.

Observations

Molarilty of $CuSO_4$ soln. (m)	EMF (E_{cell}) in V	Activity Coefficient(γ)	Activity $m \times \gamma$	Log a	Ecu	E^0
0.1		0.15				
0.01		0.41				
0.05		0.21				
0.005		0.48				

Calculations:

$E_{cell} = E_{cathode} - E_{Anode}$

$$E = E^0 - \frac{2.303RT}{nF} \log \frac{[\text{Product } (m \times \gamma)]}{[\text{Reactant}(m \times \gamma)]}$$

Calculate E^0 by simple calculations and graphical method (Graph of Ecell vs Log a)

Result:

Preparation of alkyl chloride from alcohol

Aim:

To convert t-butanol into t-butyl chloride by reacting it with hydrochloric acid and to test the presence of the t-butyl chloride using the silver nitrate test.

Apparatus:

Separatory funnel, measuring cylinder, beaker, distillation setup

Chemicals:

t-butyl alcohol, Conc. hydrochloric acid, 10% sodium bicarbonate solution, anhydrous $CaCl_2$, $AgNO_3$ Solution

Theory:

The hydroxyl group in tertiary alcohols is most readily replaced by simply allowing the alcohol to react with concentrated HCl at room temperature. The reaction is a nucleophilic displacement of the S_N1 type involving the formation of a relatively stable carbocation intermediate.

Substitution reactions are usually favoured under cool conditions and reactant reactivity of $3° > 2° > 1°$. 2o and 1o alcohol require more vigorous conditions to effect the substitution reaction, which is usually achieved by heating the reaction mixture with a catalyst. In this experiment, the tertiary alcohol, t-butanol, acts as the nucleophile which attacks the proton of hydrochloric acid to form a very stable tertiary carbocation and water. Here, the reaction can go two ways: one, the addition of the chlorine atom can form the t-butyl chloride; and two, methylpropene side-product can form under heat and basic conditions. The purpose of this lab is to obtain the substitution product (t-butyl chloride) by using hydrochloric acid. Fortunately, methylpropene will evaporate from the reaction mixture as a gas under reaction conditions.

2-methylprop-1-ene
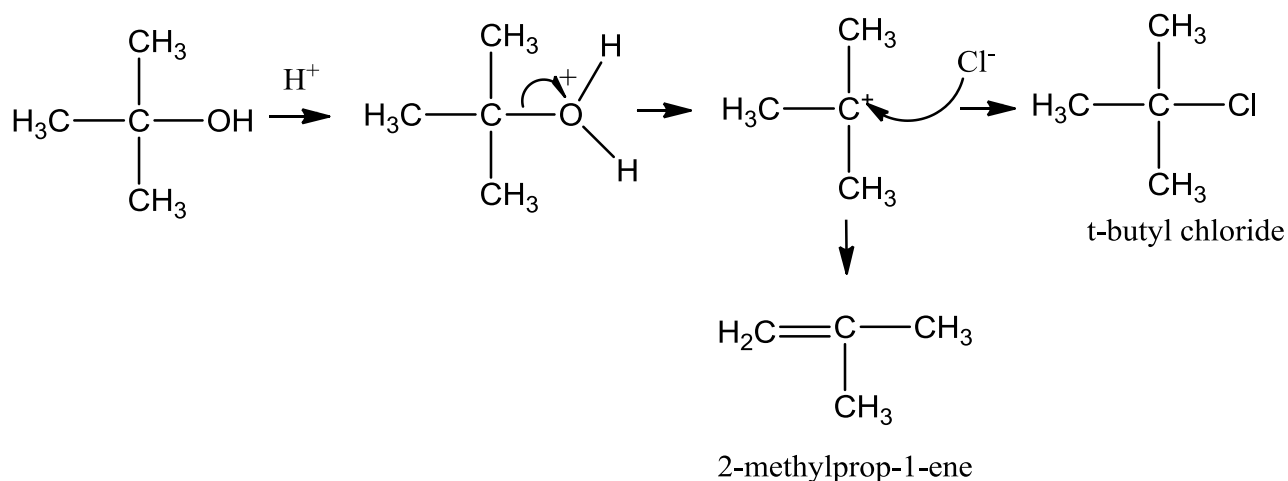
Using extraction or the partitioning of chemicals between different solvents, t-butyl chloride, an organic layer, can be easily isolated from the aqueous water/HCl layer since it is less dense than water and it will "float" on top of the aqueous layer. The common instrument used for extraction is the separatory funnel. The silver nitrate test can be used to confirm the presence of t-butyl chloride due to its rapid reaction with silver nitrate via SN1 mechanism to produce a whitish precipitate, silver chloride.

Procedure:

1. Obtain 30 mL of conc. 12M HCl in a graduated cylinder and transfer to 250mL separatory funnel placed on a ring clamp attached to burette stand
   NOTE: Conc. HCl is extremely corrosive, use with caution. Wash any spills immediately with cold water

2. Add 20 mL of t-butanol into unstoppered separatory funnel slowly about 5 ml at a time and swirl for 5 min after each addition.
   NOTE: Methylpropene gas may be produced. It is highly flammable so no heat allowed!

3. Cloudy aqueous phase and separation of layers should be observed.

4. Shake the funnel with frequent venting to complete reaction in 20 – 30 min
   NOTE: Pressure has to be released very frequently

5. Drain the aqueous layer into waste beaker

6. Wash organic layer with 20 mL of water for 3 times

7. Wash organic layer with 20mL of 10% sodium bicarbonate with careful, frequent venting for 2 times

8. Pour t-butyl chloride layer into clean, dry 250mL Erlenmeyer flask

9. Add anhydrous calcium chloride to cover bottom of flask, stopper flask, and swirl until a clump forms at the bottom of the flask. Allow the mixture to stand for 15 minutes

10. Decant the t-butyl chloride in 100 ml round bottom flask, and add 2-3 chips of porcelain

11. Setup the distillation apparatus, and distil the factions at 49-51° C

12. For silver nitrate test, place few drops of product in a small test tube. 2 drops of silver nitrate solution was added and mix. Presence of white precipitate indicates presence of 3° halide.

Result:

Report with the confirmation test to the instructor

CONDUCTOMETRIC   TITRATION

AIM:

To estimate the concentration of the given strong acid, HCL by conductometric titration with strong base, NaOH.

PRINCIPLE:

In conductometric titrations, the conductance of the solution is measured during titration, before and after the equivalence point, and the variation of conductance of the solution during the titration is utilized to locate the end point.

The strong acid, HCl, undergoes almost complete dissociation in solution and the conductance of the solution is due to H^+ and Cl^- ions. When the strong base, NaOH (which exists as Na^+ and OH^- ions in solution) is added, it neutralizes HCl to form NaCl and weakly dissociating water. NaCl, being a strong electrolyte remains in the solution as Na^+ and CL^- ions. The reaction can be represented as follows:

$$H^+ \ Cl^- + Na^+ \ OH^- \rightarrow Na^+ \ Cl^- + H_2O$$
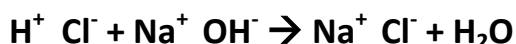
During the titration, the net change is highly conducting H^+ ions in the solution are substituted with less mobile Na^+ ions. Therefore, there is a decrease in conductance of the solution as more and more NaOH is added to neutralize HCl. This continues till all the acid are neutralize by the base. After the end point, the added quantity of NaOH remains as excess, furnishing more of Na^+ ions and OH^- ions. As the quantity of NaOH added increases, the concentration of Na^+ and OH^- increases, thereby increasing the conductance of the solution. Therefore, during the titration the conductance decreases sharply till the end point and increases thereafter. The conductance of the solution is plotted against volume of the base added and the equivalence point corresponds to the point of intersection between two straight line portions.

The solution taken in the burette should be at least ten times more concentrated than the analyte solution to avoid the effect of dilution on the variation of conductance.

PROCEDURE:

Make up the given HCl solution in a 250 ml standard flask to the mark and shake well. Pipette out 25 ml of its solution into a 100ml beaker, inserted with a glass rod. Clean the conductance cell with distilled water, dry it with filter paper strips and dip it in the acid solution. Connect the conductivity cell to the conductometer and select a suitable range for

conductance, so that, maximum number of digits is displayed on the display of the conductometer. Fill a burette with the given strong base, NaOH. Add 0.5ml of the strong base into the beaker at a time, stir well and note the conductance of the solution after each addition. The titration should be continued till the decreasing trend of conductance changes to increasing trend and at least 5-6 reading should be taken after that. Then plot the conductance values along the y-axis and volume of the base added along the x-axis. The point of intersection between the two lines corresponds to the equivalence point.

OBSERVATIONS AND CALCULATION:

Volume of base added	Conductance	Volume of base added	Conductance	Volume of base added	Conductance
0.5ml		3.5ml		6.5ml	
1.0ml		4.0ml		7.0ml	
1.5ml		4.5ml		7.5ml	
2.0ml		5.0ml		8.0ml	
2.5ml		5.5ml		8.5ml	
3.0ml		6.0ml		9.0ml	

Strength of the given base, N_{base} = ……………….N (given)

Volume of the acid pipetted out, V_{acid} = 25 ml

From the graph, equivalence point, V_{base} = ……………..ml

$$V_{base} \times N_{base} = V_{acid} \times N_{acid}$$

$$V_{base} \times N_{base} = 25 \times N_{acid}$$

Strength of the acid, $N_{acid} = \frac{V_{base} \times N_{base}}{25}$

RESULT:

Concentration of the given strong acid= …………………..N

Determination of molecular weight of high polymer

Aim To determine the molecular weight of high polymer by viscosity measurement method

Apparatus/Chemicals Ostwald's Viscometer, pipette, stopwatch and polymer solution

Theory The average molecular weight of a polymer can be determined by simple viscosity measurement. The viscosity of a high polymer solution depends on size and shape of the molecule in solution. Hence the measurement of viscosity is a useful method for study of polymer configuration. The method involves the preparation of a series of polymer solutions of different concentration in a suitable solvent and its viscosity is measured by using ostwald's viscometer. If the absolute viscosity of the solution is 'n' and that of the solvent is 'n_o', the relative viscosity is given by n/n_o . If flow time for solution and solvent are 't' & 't_o' then n_r, relative viscosity = t/t_o = n/n_o provided that the densities of the solution and solvent are very close to each other and hence it is necessary to work with very less density solutions. The specific viscosity is given as $n_{sp} = n_r - 1$ and the limiting or intrinsic viscosity $\eta = n_{sp}/C$. When n_{sp}/C is plotted against a concentration C a straight line should be obtainted which on extrapolation gives the limiting viscosity η For linear polymers , the intrinsic viscosity is related to the molecular weight   by Mark-kuhn-Houwink equation:

$$[\eta] = KM^a$$

M is the average molecular weight of the polymer; K and a are constants foa a given polymer-solvent system. For Polyvinyl alcohol-water system k= 2×10^{-4} a = 0.76

Procedure:
1) Preparation of solutions: varying concentration solutions are prepared e.g. 0.1, 0.2, 0.3 etc in percentage weight ratios by dissolving the polymer in hot water.
2) Viscosity is measured by using the Ostwald's Viscometer for each of the solutions

Observations:

Concentration	Time of flow 't'	Relative viscosity $n_r = t/t_o$	Specific viscosity n_{sp} = $n_r - 1$	Limiting/intrinsic viscosity η = n_{sp}/C

Result: The molecular weight of the given high polymer is found to be ……………………

11. REFRACTOMETRY

AIM:

To determine the percentage composition of the constituents in a binary solution using Abbe's refractometer.

PRINCIPLE:

Refractive index of a binary mixture approximately depends on the composition of the mixture. Therefore this refractive index measurements can be used to determine the percentage composition of a binary mixture in solution. Standard mixtures are prepared with the two constituents present in the given unknown mixture. The refractive index of the standard mixtures and that of the unknown mixture are determined using Abbe's refractometer. A graph is obtained by plotting refractive index against percentage composition of one of the constituents. Knowing the refractive index of the unknown mixture, the percentage composition can be obtained from the graph. The principle of Abbe's refractometer is based on total internal reflection.

PROCEDURE:

Prepare the mixtures of different compositions of constituents A and B by pipetting out different volumes of A and B. Place the refractometer on the table in such a way that light from sky is reflected by the mirror into the split prism. Adjust the mirror to get maximum illumination. Open the prism box and clean the diagonal surface on the prism with acetone using a cotton swab.

When the prism surface is dried, place a drop of the liquid to form thin film upon the ground surface of the lower prism. Close the prism box immediately. Adjust the mirror to get good illumination. Turn the prism gently forward and backward until a colored band appears in the field of view. By turning the milled ring at the base of the telescope a position is found at which the colored band is eliminated and field of view being partly light and partly dark with a sharp dividing line. Then adjust eyepiece to the position where the line of demarcation coincides at the center of the cross wire. At this position read the refractive index of the sample through another eyepiece. Similarly find out the refractive index values for other standard solutions and given unknown sample also. Plot a graph of refractive index (y-axis) against % composition of either A or B (x-axis) to get a straight line. From the graph the composition of the unknown solution can be obtained.

OBSERVATIONS AND CALCULATION:

S.No.	Volume of A (water) in ml	Volume of B (alcohol in ml	% compo- sition of A (water)	% compo- sition of B (alcohol)	Refractive Index, η
1.	9.5	0.5	95	05	
2.	9.0	1.0	90	10	
3.	8.5	1.5	85	15	
4.	8.0	2.0	80	20	
5.	7.5	2.5	75	25	
6.	7.0	3.0	70	30	
7.	6.5	3.5	65	35	
8.	6.0	4.0	60	40	
9.	5.5	4.5	55	45	
10.	UNKNOWN				

RESULT: Percentage composition of the given solution = ... of A / B.

Signature of the staff member

* * * * *

Chemical Oxygen Demand (COD)

Aim:
To determine the COD of given water sample by open reflux method.
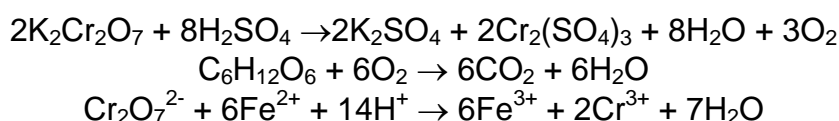
Chemicals:
Potassium dichromate, Sulfuric acid, Ferrous ammonium sulfate, silver sulfate, Mercury Sulfate, Ferroin indicator.

Introduction:
Chemical Oxygen Demand (COD) test determines the oxygen requirement equivalent of organic matter that is susceptible to oxidation with the help of a strong chemical oxidant. It is an important, rapidly measured parameters as a means of measuring organic strength for streams and polluted water bodies. The test can be related empirically to BOD, organic carbon or organic matter in samples from a specific source taking into account its limitations. The test is useful in studying performance evaluation of wastewater treatment plants and monitoring relatively polluted water bodies. COD determination has advantage over BOD determination. COD results can be obtained in 3-4 hrs as compared to 3-5days required for BOD test. Further, the test is relatively easy, precise, and is unaffected by interferences as in the BOD test. The intrinsic limitation of the test lies in its inability to differentiate between the biologically oxidisable and biologically inert material and to find out the system rate constant of aerobic biological stabilization.

Principle
The open reflux method is suitable for a wide range of wastes with a large sample size. The dichromate reflux method is preferred over procedures using other oxidants (e.g. potassium permanganate) because of its superior oxidizing ability, applicability to a wide variety of samples and ease of manipulation. Oxidation of most organic compounds is up to 95-100% of the theoretical value. The organic matter gets oxidised completely by potassium dichromate ($K_2Cr_2O_7$) with silver sulphate as catalyst in the presence of concentrated H_2SO_4 to produce CO_2 and H_2O. The excess $K_2Cr_2O_7$ remaining after the reaction is titrated with ferrous ammonium sulphate [Fe(NH4)2(SO4)2]. The dichromate consumed gives the oxygen (O_2) required for oxidation of the organic matter. The chemical reactions involved in the method are as under:

$$2K_2Cr_2O_7 + 8H_2SO_4 \rightarrow 2K_2SO_4 + 2Cr_2(SO_4)_3 + 8H_2O + 3O_2$$
$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O$$
$$Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

Procedure:

1. Take three conical flask, pipette out 25 ml of sewage/waste water in two of the conical flask and 25 ml of distilled water for blank titration.
2. Add 10 ml of $K_2Cr_2O_7$-$HgSO_4$ solution to each of the three conical flask
3. Add 10 ml of H_2SO_4-Ag_2SO_4 solution through the side of the conical flask (Caution: Now conical flask will be very hot)
4. Transfer the solution to a round bottom flask. Add 10 ml of distilled water to rinse the conical flask and transfer the water to round bottom flask.
5. Reflex the sample for 1.5 hrs, meanwhile fill the burette with FAS solution.
6. Transfer the solution from round bottom flask to the conical flask completely. (You can add 10 ml of distilled water to rinse the RBF and transfer it to conical flask)
7. Add about 5-6 drops of Ferroin indicator, the solution should be greenish blue by now.
8. Titrate the content against FAS, the end point is appearance of reddish brown color.

Calculations

Sl. No	Sample Name	Volume of Sample	Burette reading		Volume of FAS
			Initial	Final	

COD (mg/L) = (a –b) x N x 8000 / Volume of H_2O sample
Where, a = Volume FAS used for blank
b = Volume FAS used for sample

N = normality of FAS

Result:

The COD of waste water is found to be _____