



DEPARTMENT OF CHEMISTRY

Laboratory Experiments

Experiment No	Experiment
1	Estimation of copper from e-waste/Brass
2	Estimation of iron in haematite ore/Rust
3	Colorimetric estimation of copper
4	Flame photometric estimation of sodium
5	Determination pKa of weak acid
6	Determination of viscosity of a given liquid
7	Estimations of acids by conductometric method
8	Potentiometric estimation of iron

Experiment No. 1: Estimation of copper from e-waste/Brass

Principle

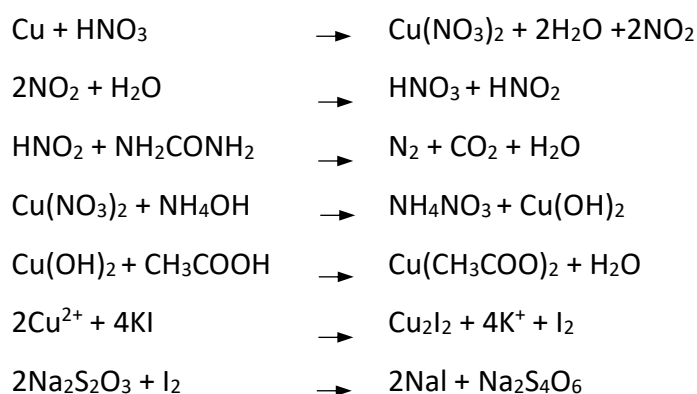
Copper is estimated by iodometric titration. Cu^{+2} ions oxidise KI to I_2 in acidic condition. The amount of I_2 liberated is estimated by titrating against sodium thiosulphate using starch as the indicator.

Theory

Copper solution is prepared by dissolving e-waste sample in nitric acid. Oxides of nitrogen are destroyed by boiling with urea. Excess acid is neutralized by adding ammonia. The solution is changed to weak acidic medium by adding acetic acid. To this solution potassium iodide is added. Iodine is liberated by the cupric ions. Then the solution is titrated against sodium thiosulphate solution using starch as an indicator. The amount of sodium thiosulphate consumed is a measure of the amount of copper present in the solution. The principle of this experiment is iodometric titration.

If it is brass sample, a brass piece is weighed and dissolved in minimum amount of concentrated nitric acid.

Following are the reactions occurring while estimating the amount of copper in the given solution;



Procedure

Part-A: Preparation of standard sodium thiosulphate solution

Weigh accurately the given $\text{Na}_2\text{S}_2\text{O}_3$ and transfer it into a 100 cm^3 standard flask using a funnel. Add small amount of distilled water to dissolve the salt and make it up to the mark by adding some more distilled water. Shake it well to get uniform concentration. Rinse the burette and fill the burette with the prepared standard sodium thiosulphate solution.

Normality of $\text{Na}_2\text{S}_2\text{O}_3 = (\text{weight of } \text{Na}_2\text{S}_2\text{O}_3) / \text{gram equivalent weight of } \text{Na}_2\text{S}_2\text{O}_3 (248.17)$

Part-B: Preparation of copper solution and estimation of copper

Take 25 cm^3 of the prepared e-waste solution. Add one spatula urea and 1 tt of distilled water to the solution and heat it to boiling and cool it. Add ammonium hydroxide drop by drop until a pale blue precipitate is obtained. Dissolve the precipitate by adding dil. acetic acid drop by drop, followed by $\frac{1}{4}$ tt of acetic acid and 1 tt of 10% KI solution. Add a test tube of distilled water. Swirl the conical flask thoroughly for about 30 seconds. Titrate the liberated iodine against standard sodium thiosulphate solution taken in the burette until the solution becomes pale yellow or cream colored. Add about 2 cm^3 of freshly prepared starch solution as an indicator. Continue the titration by adding sodium thiosulphate solution strictly drop by drop until the dark blue coloration disappears, leaving behind a white ppt.

Repeat Part- B two more times. Calculate the percentage of copper present in the Brass samples by taking one titer value.

Part-C: Calculation

Weight of PCB sample taken = 'W' g (will be given)

Normality of sodium thiosulphate = 'Y'

Volume of sodium thiosulphate = 'X' cm^3 (from titration)

1000 cm^3 1N sodium thiosulphate = 63.54g of copper

Therefore 'X' cm^3 of 'Y' N sodium thiosulphate = $\frac{63.54 \times X \times Y}{1000}$ g of copper

= 'A' g of Cu

250 cm^3 of prepared PCB solution contains = '10A' g of Cu

Experiment No. 2: Estimation of iron in haematite ore/Rust

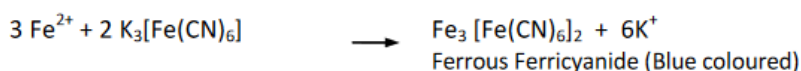
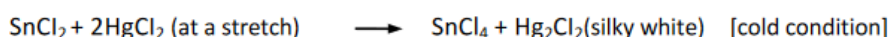
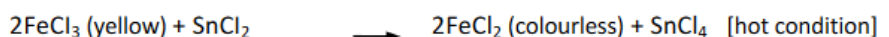
Principle

Iron in haematite ore can be quantitatively estimated using external indicator method based on the complex formation of lower valent ferrous (outer coordination sphere) with higher valent ferric ions.

Theory

The reduction of Fe^{3+} ions using SnCl_2 reductant under acidic medium in the hot conditions provides an environment of selective estimation of iron in the given ore. Unlike the conventional redox titration, the analyte forms complex with external indicator without changing their oxidation state. As the external indicator is not added to the conical flask, the disadvantage of using excess indicator in the solution medium can be avoided.

Procedure: A known quantity of $\text{K}_2\text{Cr}_2\text{O}_7$ is dissolved in water and transferred to a standard flask and diluted with water up to the mark. This standard solution is used as titrant and taken in the burette. A given solution of haematite is acidified and heated to boiling followed by the addition of SnCl_2 to trigger the reduction of Fe^{3+} to Fe^{2+} ions, which is accompanied by the oxidation of Sn^{2+} to Sn^{4+} ions. After cooling, the HgCl_2 is added to ensure the complete oxidation of Sn^{2+} ions and reduced Hg_2Cl_2 silky white precipitate is formed. This solution is titrated against standard $\text{K}_2\text{Cr}_2\text{O}_7$ using $\text{K}_3[\text{Fe}(\text{CN})_6]$ as external indicator (potassium ferricyanide) which is placed on a paraffin paper. A drop of reacted mixture is mixed with external indicator which results in deep blue/green color due to the formation of $\text{Fe}_3[\text{Fe}(\text{CN})_6]_3$ complex. The failure of reactant mixture to induce any color to the indicator reflects the end point of the reaction. The trials are repeated to get the similar values (Y).



Calculations

Part – A: Calculation of normality of $K_2Cr_2O_7$

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \text{ crystals dissolved in}}{\text{Gram equivalent weight of } K_2Cr_2O_7 (49.03)}$$

Part-B: Estimating the amount of iron

$$1000 \text{ cm}^3 \text{ of } 1N \text{ } K_2Cr_2O_7 = 1 \text{ equivalent of iron} \\ = 55.85 \text{ g of Iron}$$

$$\text{Therefore 'Y' cm}^3 \text{ of 'x' N } K_2Cr_2O_7 = \frac{55.85 \times x \times Y}{1000} \text{ g of iron} = \text{'Z' g of iron.}$$

$$25\text{cm}^3 \text{ of Haematite ore solution contains 'Z' g of iron}$$

$$250\text{cm}^3 \text{ of Haematite ore solution contains '10xZ' g of iron}$$

$$\text{Weight of Haematite ore dissolved in } 250\text{cm}^3 \text{ of the solution} = \text{'W'g}$$

$$\text{'W' g of haematite ore contains '10 xZ' g of iron.}$$

$$\text{Therefore, 100g of haematite ore contains} = \frac{10Z \times 100}{W} \text{ g of iron} = \text{'B' g of iron}$$

Experiment No. 3: Colorimetric estimation of copper

Principle: The colored compounds can absorb a certain wavelength of light when monochromatic light is passed through them. The magnitude of light absorption is critically dependent on the intensity of the color. The colorimeter works on the principle of Beer-Lambert's law.

Theory

When a monochromatic light is passed through a colored solution, part of it will be absorbed (I_a), transmitted (I_t) and reflected (I_r). Thus,

$$I_0 = I_a + I_t + I_r \quad (1)$$

The ' I_r ' is negligible if the light propagates through glass medium.

$$I_0 = I_a + I_t \quad (2)$$

The ratio ' I_t/I_0 ' is referred to as 'transmittance' and logarithmic derivative ' $\log(I_t/I_0)$ ' is regarded as optical density (O.D) or absorbance (A). The optical density and the concentration of colored solution is related by Beer-Lambert's law, which is expressed by the equation:

$$A = \epsilon \cdot c \cdot l \quad (3)$$

Where ' c ' refers to the concentration of the solution and ' l ' indicates the path length of the light or thickness of the medium. The ' ϵ ' is a dimensionless parameter which is referred to as molar extinction coefficient.

Thus, absorption of any colored solution is directly proportional to concentration of the colored solution at constant ' l '.

Instrumentation

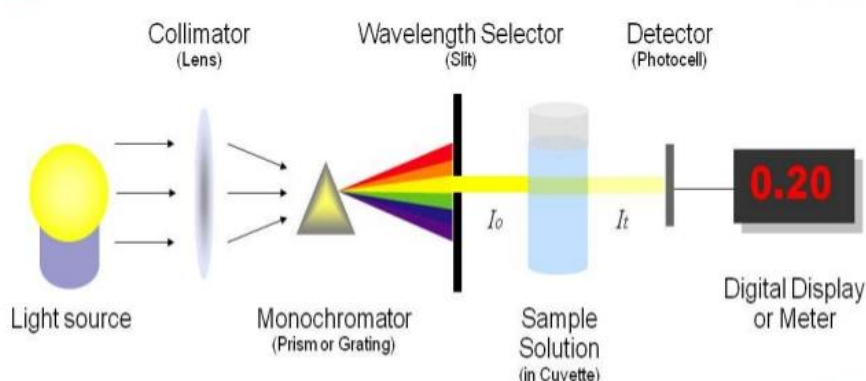


Figure 1.1: Components in Colorimeter.

Light source: A low intense diffuse light (LED source or W filament) which provides constant intensity for long-term operations is used.

Monochromators: It transforms the polychromatic light to monochromatic and the slit enables the light to pass through the solution in a unidirectional way.

Sample solution: Light passes through the colored solution and amount of transmitted light reaches the detector, which is read as 'Optical Density'

Experimental Procedure

- (1) A known volume of CuSO_4 solution (2, 4, 6, 8 and 10 ml) is transferred to series of 25 ml volumetric flasks, followed by the addition of definite volume of aqueous NH_3 to form $[\text{Cu}(\text{NH}_3)_4]^{2+}\text{SO}_4$ solution. The formation of this complex is evidenced by intensification of blue color after the addition of NH_3 .
- (2) The complex solution is diluted with water up to the mark using distilled water.
- (3) The blank solution is prepared using NH_3 and H_2O without adding CuSO_4 solution.
- (4) The colorimeter is calibrated using the blank solution via setting the absorbance to zero at wavelength of 620 nm.
- (5) Absorbance of prepared solution is measured accordingly without any further adjustments. A calibration curve is drawn by plotting the values of absorbance against the volume of copper ion solution, and straight line is drawn from the origin. Followed by this, volume of unknown solution is calculated by knowing its absorbance value.

Model graphs

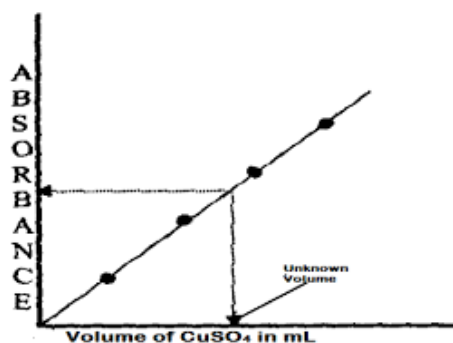


Figure 1.2: Plot of absorbance vs volume of CuSO_4

Calculations

Weight of copper sulphate pentahydrate present in 100 cm^3 of the given solution = 'X' mg

Weight of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in 1 cm^3 of its solution = $X/100 \text{ mg} = \dots\dots\dots = \text{'Y' mg}$

Weight of copper present in 1 cm^3 of its solution 'Y' $\times 63.54 / 249.54$

Weight of copper present in 'Z' cm^3 of its solution

Experiment No. 4: Flame photometric estimation of sodium

Principle: In a certain concentration range, the intensity of the emitted light is directly proportional to the number of excited atoms returning to the ground state. The emission wavelength depends on the energy gap between the lower and higher energy states.

Theory: When an alkali/alkali-earth metal ion salt solution is exposed to flame, a sequence of reactions proceeds producing an intense colour for the flame.

- (i) Desolvation: Metal salt solution is dehydrated via removal of solvent molecules by the flame.
- (ii) Vaporization: Metal particles are also subjected to dehydration.
- (iii) Atomization: Metal ions are reduced to metal atoms by the flame.
- (iv) Excitation: Metal atoms absorb heat and thermally excited to higher energy states.
- (v) Emission: Excited states are highly unstable and therefore, excited atoms return to ground state by emitting the absorbed energy (radiative emission), which imparts color to the flame.

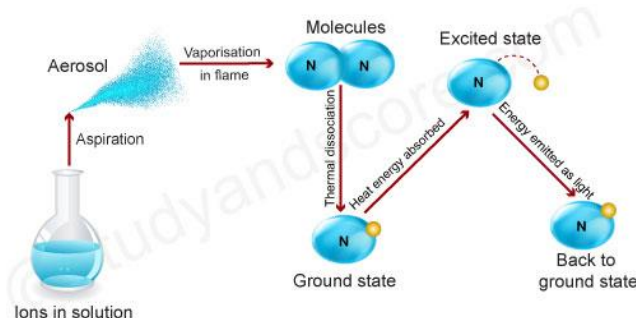


Figure 2.1: Process in flame photometer measurements

Instrumentation

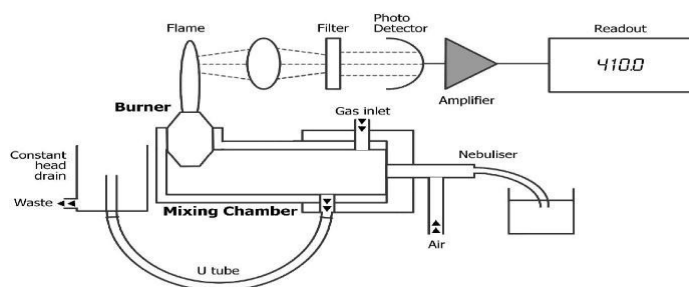


Figure 2.2: Components in Flame photometer

Burner: It maintains the constant temperature and intensity of the flame.

Nebulizer: It regulates the homogeneous flow of solution into the flame.

Optical System: Convex mirror transmits the light energy emitted from the atoms, and convex lens facilitates the light to pass through the slit.

Filters: It isolates the emission wavelength to be measured specific to the metal atoms.

Photodetectors: It measures the intensity of radiation emitted by the flame and converts to electrical signal.

Amplifiers: It is used to amplify the electrical signal and provides a suitable output.

Experimental Procedure

(1) A known volume of NaCl solution (2, 4, 6, 8 and 10 ml) is transferred to series of 25 ml volumetric flasks and diluted with water up to the mark using distilled water.

(2) Flame is calibrated by adjusting the air and gas and readings are set to zero by spraying the distilled water into the flame.

(5) Emission intensity of the prepared solution is measured accordingly without any further adjustments. A calibration curve is drawn by plotting the values of emission intensity against the volume of NaCl solution. Followed by this, volume of unknown solution is calculated by recording the emission intensity values.

Model graph

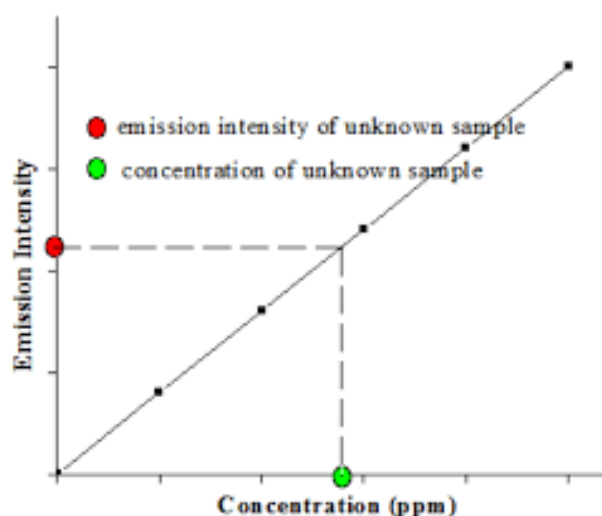


Figure 2.3: Plot of emission intensity vs concentration of NaCl solution

Calculations

Weight of NaCl present in 100 cm³ of the given solution = 'm' g

58.5 g of NaCl contains 23 g of Na.

Therefore, 1 cm³ of the given stock solution contains = 'A'g of Na

'Z' cm³ of the given stock solution contains $w \cdot 23 / (58.5 \cdot 100) = (A \times Z)$ g of Na

Experiment No. 5: Determination pKa of weak acid

Principle: pKa of the solution is equivalent to the pH of the solution at its half equivalence point (i.e., at the stage where concentration of salt is equal to concentration of acid).

Theory: The Henderson-Hasselbalch equations relates both 'pH' and 'pKa' by the equation:

$$\left| \text{pH} = \text{pKa} + \log_{10} \frac{[\text{Salt}]}{[\text{Acid}]} \right| \quad \left| \text{CH}_3\text{COOH} + \text{NaOH} \longrightarrow \text{CH}_3\text{COONa} + \text{H}_2\text{O} \right|$$

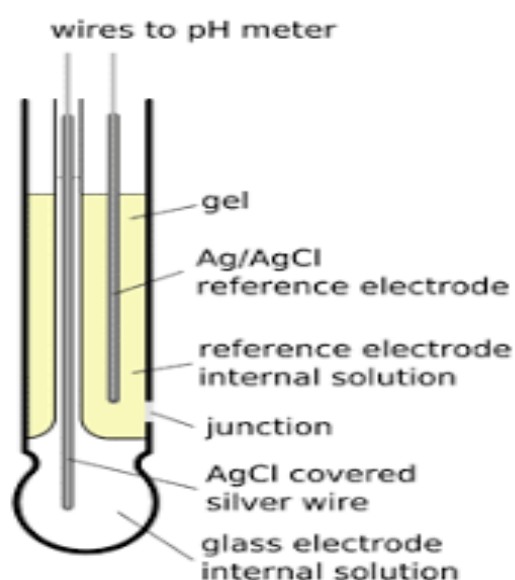
At equivalent point, all the acid will be neutralized upon the addition of base. However, ratio of [salt]/[acid] approaches unity at half-equivalence point and equation reduces to $\text{pH} = \text{pKa}$. Thus, pKa is measured at half-equivalence point.

Instrumentation:

(i) Combined pH electrodes constitute Ag/AgCl as reference electrode immersed in saturated KCl solution. Another Ag/AgCl immersed in buffer solution serves as internal reference electrode. The electrodes are enclosed in a glass case without any connectivity among them.

(ii) When the electrode is dipped in the solution, protons tend to adsorb on the outer surface via formation of gel-layer. If the concentration of protons on the outer and inner gel layer remains same, then the solution is regarded as neutral. Alternatively, if the concentration of protons on the outer layer is less than that of inner layer, the solution is basic and vice-versa.

Figure 3.1: Components in pH sensor



Experimental Procedure: Pipette out 50 cm³ of the given soft drinks (weak acid) into a clean 250 cm³ beaker. Insert glass electrode - calomel electrode assembly into it and connect it to pH meter. Measure the pH of the acid. Fill the burette with the given sodium hydroxide. In the beginning add 0.2 cm³ of NaOH at a time and stir the mixture. Measure the pH. Continue adding 0.2 cm³ of NaOH & measure the pH regularly after each addition until the increase in pH is comparatively large. Take a few more reading by adding 0.2 cm³ of NaOH.

Determine the pKa of the given weak acid from the two graphs:

(i) $\Delta \text{pH}/\Delta V$ against volume of NaOH added.

(ii) pH against volume of NaOH added

Model graph:

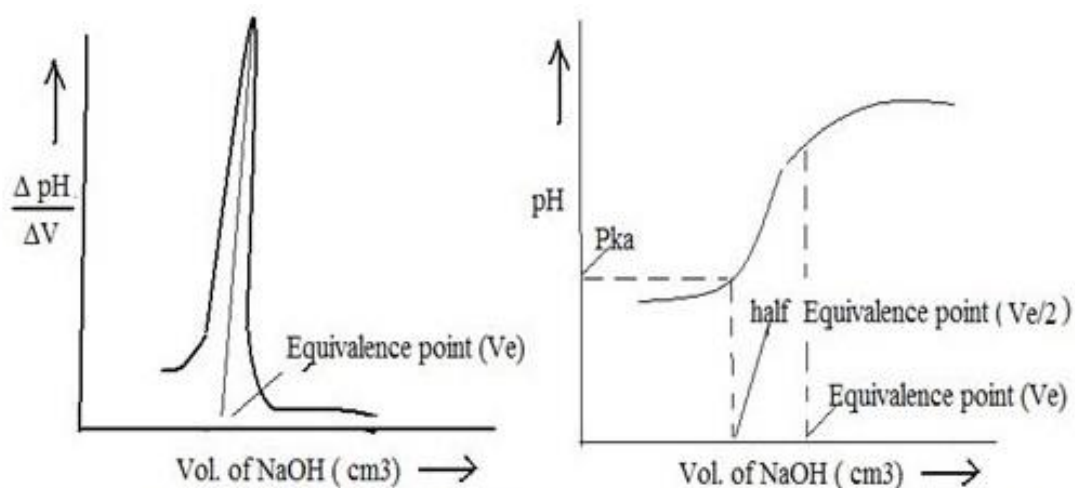


Figure 3.2: Plot of pH vs NaOH (right side) and the corresponding first-derivate (left side).

Calculations

Table:

Volume of NaOH	pH	ΔpH	$\Delta \text{pH}/\Delta V$
0			
0.2			

Experiment No. 6: Determination of viscosity of a given liquid.

Principle: The resistance of a liquid to flow is referred to as viscosity and arises due to the contact liquid layers with each other. It is a measure of fluid's internal resistance to flow and may be regarded as measurement of fluid friction.

Theory: Viscosity arises due to friction between moving layers of a liquid. A liquid flowing through a cylindrical tube of uniform diameter is expected to move in the form of molecular layers. Layers close to the surface are almost stationary while that at the axis of the tube moves faster than any other intermediate layer. A slow-moving layer exerts a drag or frictional force on its nearest moving layer, backwards. This property of the liquid which retards or opposes the motion between the layers is called viscosity. The coefficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradient between the two successive layers of the liquid situated at unit distance apart. The viscosity is influenced by the parameters like temperature, pressure, density, and type of flow of the liquids.

Instrumentation

Ostwald viscometer or viscometer is an instrument that works on the principle of Poiseuille's law. As per Poiseuille's law, in the case of the laminar flow of the fluids, the flow rate of the liquid is defined as the ratio of pressure difference with respect to the viscous resistance.

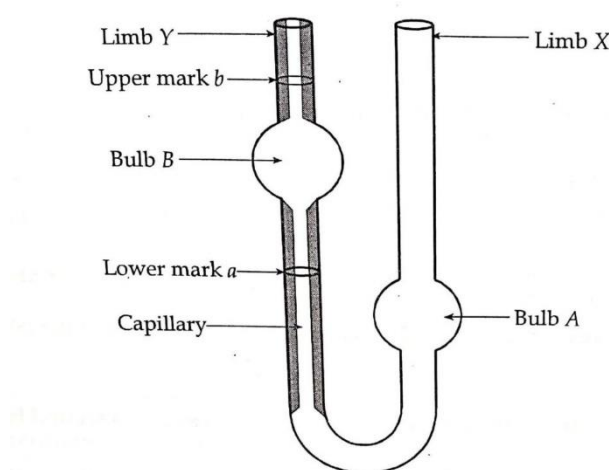


Figure 4.1: Components in viscometer

An Ostwald viscometer is made up of two bulbs that are attached to each other with the help of a U-shaped tube. One of the bulbs is connected to the wider limb on one side and to the

U-shaped tube on the other side. The second bulb is connected to the capillary tube on one side and to the rubber tube on the other side. The second bulb consists of two markings on either side. The main purpose of the two etched marks is to track the limit of the flow of the liquids through the capillary tube.

$$Q = \frac{\pi P r^4}{8 \eta l}$$

Q	Flow rate
P	Pressure
r	Radius
η	Fluid viscosity
l	Length of tubing

Procedure

Take a clean and dry viscometer. Fix it vertically to a stand. Using graduated pipette, transfer known volume (say 10 cm³) of the given liquid into the viscometer. Then it is placed in a water bath such that its big bulb is completely immersed in water. Suck the liquid above the upper mark of the small bulb of the viscometer. Allow it to flow through the capillary. When the liquid crosses the upper mark of the small bulb, start a stopwatch and when the liquid crosses the lower mark, stop the watch. Note down the time of flow in seconds.

Repeat the experiment 3-4 times and calculate the average time of flow.

Remove the liquid from the viscometer, clean well with acetone and dry it in oven. Cool the viscometer to room temperature. Similarly, measure the time of flow of water by taking the same volume. Calculate average time of flow of water. Note down the room temperature. Knowing the density, viscosity of water and density of the given liquid, viscosity of the liquid can be calculated.

Calculations

The corresponding density and viscosity of the water as well as the density of the solvent is noted at a particular temperature. Accordingly, viscosity of the given liquid is calculated using the following equation.

$\eta_y = \eta_w \frac{d_y t_y}{d_w t_w}$	η_w : viscosity of water η_y : viscosity of tested liquid d_w : density of water d_y : density of tested liquid t_w : timing of runoff of water t_y : timing of runoff of tested liquid
---	--

S. I. unit of viscosity is kg/m/s or Pa.s (pascal.seconds)

C. G. S unit of viscosity is 'Poise'.

Experiment No. 7: Estimations of acids by conductometric method

Principle

The conductivity of the solution is related to the mobility of ions which in turn is related with the size of the ions.

Theory

Conductance of any solution (G) is the ease with which current flows through it. It is the reciprocal of resistance (R) offered by the solution.

$$G = 1/R$$

Specific Conductivity

The extent to which a given sample of electrolytic solution can conduct an electric current is called its conductance. Conversely, the extent to which the given sample of electrolytic solution can resist the flow of electric current is called its resistance. Of course, the conductance is the reciprocal of the resistance of that particular electrolytic solution. The SI unit of conductance is S (Siemens).

The conductivity or specific conductivity is the measure of the ability of an electrolytic solution to conduct electricity. The SI unit of specific conductivity is Siemens per meter (S/m).

$$R = \rho l/A$$
$$\Rightarrow \rho = AR/l$$

According to the definition of conductivity

$$\kappa = 1/\rho$$
$$\Rightarrow \kappa = l/AR$$
$$\Rightarrow \kappa = l/A \times 1/R$$

Here, l/A is a constant value called the cell constant. And $1/R$ gives the conductance of the electrolytic solution, therefore,

$$\kappa = \text{Cell constant} \times \text{Conductance (G)} \text{ Siemens/metre}$$

The following factors affect the conductivity of electrolytic solutions;

- The nature and the concentration of the electrolyte added
- The size of the ions produced and their solvation.
- Solvent nature and viscosity.
- Temperature.

Instrumentation

Conductivity Cell

it consists of two platinum electrodes coated with platinum black (finely divided metallic Pt is deposited on the electrodes electrochemically) assembled inside the glass container.

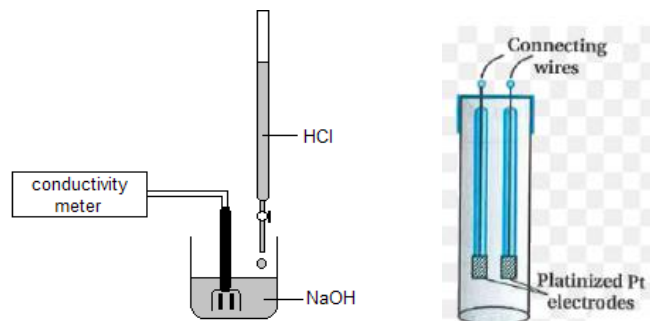


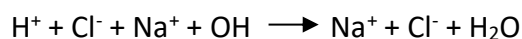
Figure 6.1: Schematic representation of conductometry

Procedure

Pipette out 50 cm³ of acid into a clean 200 cm³ beaker. Dip the conductivity cell in the acid, such that the two platinum electrodes are immersed completely. The cell is connected to the conductivity bridge. Measure the conductance. Take the given NaOH solution in a burette rinsed with the same solution. Rundown 0.2 cm³ of NaOH solution into the beaker and shake well. Note down the conductance. Note down the conductance by adding 0.2 cm³ of NaOH each time to the beaker. Conductance decreases in the beginning and finally starts increasing by large value. Take around 8-10 readings on the increasing side. Draw a graph of conductance against volume of NaOH added. The equivalence point in the graph corresponds to the neutralization of HCl. Calculate the normality and amount of HCl present in 1000 cm³ of its solution.

1. Titration of strong acid (HCl) with strong base (NaOH)

Before NaOH is added, the conductance is high due to the presence of highly mobile hydrogen ions. When the base is added, the conductance falls due to the replacement of hydrogen ions by the added cation as H⁺ ions react with OH⁻ ions to form undissociated water. This decrease in the conductance continues till the equivalence point. At the equivalence point, the solution contains only NaCl. After the equivalence point, the conductance increases due to the large conductivity of OH⁻ ions



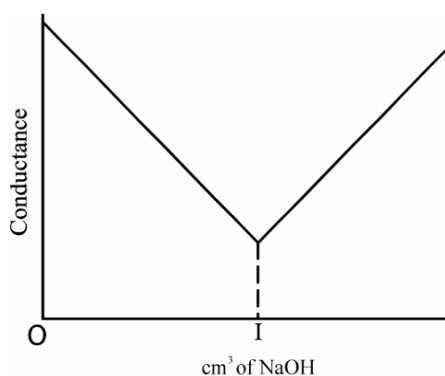


Figure 6.2: Titration curve for strong acid vs strong base.

2. Titration of mixture of acids with strong base (NaOH)

When the strong acid has been completely neutralized only then the weak acid starts neutralizing. The second break point corresponds to the neutralization of weak acid and after that the conductance increases due to the excess of OH⁻ ions in case of a strong base as the titrant. However, when the titrant is a weak base, it remains almost constant after the end point like Initially the conductance is low due to the feeble ionization of acetic acid.

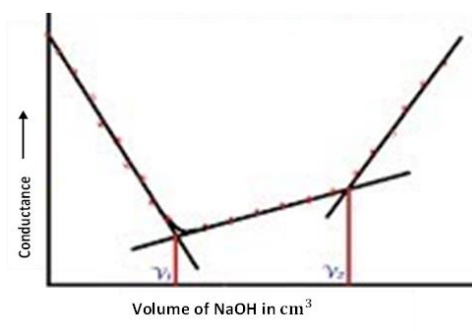
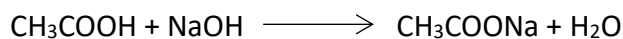


Figure 6.3: Titration curve for mixture of acids vs strong base.

On the addition of base, there is decrease in conductance not only due to the replacement of H⁺ by Na⁺ but also suppresses the dissociation of acetic acid due to common ion acetate. But very soon, the conductance increases on adding NaOH as NaOH neutralizes the un-dissociated CH₃COOH to CH₃COONa which is the strong electrolyte. This increase in conductance continues raise up to the equivalence point. The graph near the equivalence point is curved due the hydrolysis of salt

CH₃COONa. Beyond the equivalence point, conductance increases more rapidly with the addition of NaOH due to the highly conducting OH⁻ ions.

Calculation:

$$(NV)_{\text{acid}} = (NV)_{\text{NaOH}}$$

$$N_{\text{acid}} = \frac{(NV)_{\text{NaOH}}}{V_{\text{acid}}}$$

Amount of HCl present in 1000 cm³ of its solution = N_{acid} x gram equivalent weight of HCl

Amount of CH₃COOH present in 1000 cm³ of its solution = N_{acid} x gram equivalent weight of CH₃COOH

Experiment No. 8: Potentiometric estimation of iron

Principle

Formation of redox electrodes in the solution during titration and switching over of iron electrode to chromium electrode at equivalence point with significant increase in the potential value.

Theory:

Nernst equation gives the relation between the potential at an electrode and the concentration of the active species in the solution.

For the reaction,



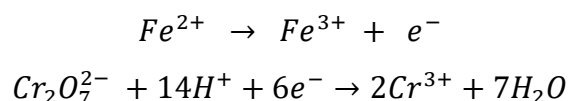
Nernst equation can be written as

$$E = E^\circ + \frac{0.0591}{n} \log \frac{[\text{oxidized form}]}{[\text{reduced form}]}$$

Where E° is the standard electrode potential and the terms within the brackets represent their concentrations.

It is evident from the equation that the potential depends upon the concentrations of the oxidised and the reduced species in the solution. When a reducing agent in solution is oxidized using an oxidizing agent, the concentrations and hence the potential changes. Potentiometry essentially involves the measurement of change in potential as and when a species in solution is oxidized or reduced. When a titration of a reducing agent is carried out against an oxidising agent, the potential gradually changes in the beginning but changes rapidly near the end point. A plot of change in potential against volume reveals a sudden change in potential at the equivalence point. This sudden change in the present experiment can be explained as follows.

The reactions that take place when dichromate is added to ferrous solution are:



The potential of the system before the equivalence point is given by

$$E = E^\circ + \frac{0.0591}{1} \log \frac{[Fe^{3+}]}{[Fe^{2+}]}$$

i.e., the potential is dependent on the concentration of Fe^{2+} and Fe^{3+} ions. The potential of the solution will be around 0.75V. At the equivalence point the potential is decided by the

concentrations of Fe^{2+} , Fe^{3+} , $\text{Cr}_2\text{O}_7^{2-}$ and Cr^{3+} ions. The potential at equivalence point is given by

$$E = \frac{E_{\text{Fe}^{3+}/\text{Fe}^{2+}}^0 + E_{\text{Cr}^{6+}/\text{Cr}^{3+}}^0}{2}$$

$$= \frac{0.75\text{V} + 1.33\text{V}}{2} = 1.04\text{V}$$

Beyond the equivalence point as no ferrous ions exist, the potential is determined by the concentration of $\text{Cr}_2\text{O}_7^{2-}$ and Cr^{3+} ions and it is given by

$$E = E^0 + \frac{0.0591}{6} \log [\text{Cr}^{6+}] / [\text{Cr}^{3+}]$$

$$= 1.33\text{V} + \frac{0.0591}{6} \log [\text{Cr}^{6+}] / [\text{Cr}^{3+}]$$

therefore, an abrupt increase in the potential is observed at the equivalence point. This increase marks the end point of the titration. Potential change at an electrode cannot be directly measured. The electrode at which the potential changes with concentration (indicator electrode) is connected to another electrode whose potential remains constant [ex:-calomel electrode (Standard reference electrode)], to form a cell.

$$\text{EMF} = E_{\text{cathode}} - E_{\text{anode}}$$

As the potential of the indicator electrode changes, the EMF of the cell also changes. It is the change in EMF that is measured during a potentiometric titration.

Procedure

Take 25 cm³ of rust solution into a clean beaker using the dispenser. Add 1 test tube of dil. Sulphuric acid. Dip the electrode assembly into the solution and connect to a potentiometer. Measure the potential. Add 0.2 cm³ of Potassium dichromate from a burette. Stir the solution well and measure the potential. Continue the process till the potential shows a tendency to increase rapidly. Now add dichromate in increments of 0.2 cm³ and measure the potential after each addition. Plot a graph of $\Delta E / \Delta V$ against volume of dichromate added as shown in the figure and find out the end point. Calculate the normality of the ferrous solution and determine the amount of iron in the given volume.

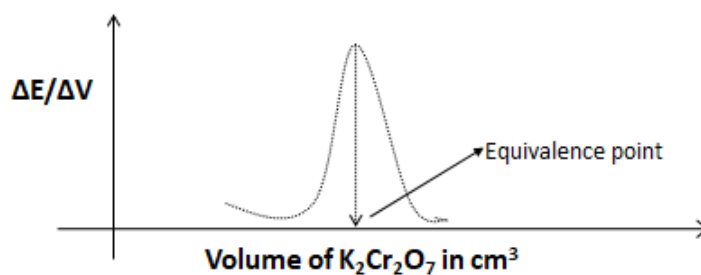


Figure 7.1: Titration curve for potentiometric titration (Derivative graph).

Calculation:

Volume of $K_2Cr_2O_7$ required for the reaction = V_{cm^3} (From graph),

$$N_{iron} = \frac{NV_{K_2Cr_2O_7}}{V_{iron}}$$

Amount of iron presented in 1000 cm^3 of its solution = $N_{iron} \times \text{gram equivalent weight of Iron}$
 = g