**Exp 2:**

**Aim: To determine the unknown concentration of given acids using titrimetric method**

I. **Ostwald Theory:** A hydrogen ion indicator is a weak organic acid or base. The undissociated molecule will have one colour and the ion formed by its dissociation will have a different colour.

II. **Acidimetry:** The estimation of an alkali solution using a standard acid solution is called acidimetry

III. **Alkalimetry:** The estimation of an acid solution using a standard alkali solution is called alkalimetry.

|  |  |  |  |
| --- | --- | --- | --- |
| **Indicator** | **Colour on Acidic Side** | **Range of Colour Change** | **Colour on Basic Side** |
| Methyl Orange | Red | 3.1 - 4.4 | Yellow |
| Methyl Red | Red | 4.4 - 6.2 | Yellow |
| Phenolphthalein | Colourless | 8.3 - 10.0 | Pink |

i.e., at pH value below 5, litmus is red; above 8 it is blue. Between these values, it is a mixture of two colours.

IV. **Indicators Used for Various Titration:**

A. **Strong Acid against a Strong Base:** methyl orange, methyl red and phenolphthalein (pH range 4 to 10)

B. **Weak Acid against Strong Base:** Phenolphthalein(suitable, pH range 8-9.8). Methyl Red (Not Suitable, pH range 3.1-4.5)

C. **Strong Acid against Weak Base:** Methyl Red (Suitable, pH range 3.1-4.5). Phenolphthalein(Not Suitable, pH range 8-9.8).

V. **Procedure:**

* Choose the titrant.
* Choose the titrate.
* Select the normality of the titrate.
* Choose the volume of the liquid to be pipetted out.
* Select the indicator.
* Start titration.
* End point is noted at the colour change of the solution.
* From the final reading the normality of titrant can be calculated by the equation: N1V1 = N2V2

**Viva Questions:**

1. **What is Normality?**

Normality is defined as the number of gram or mole equivalents of solute present in 1 litre of solution

1. **What is Titration?**

The process of adding one solution from the burette to another in the titration flask in order to complete the chemical reaction involved, is known as titration.

1. **What are weak acids and bases?**

Weak acids are acids that partially dissociates into its constituent ions in an aqueous solution. A weak base is the conjugate of weak acids.

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**Exp 3:**

**Aim: To determine the various types of hardness of different water samples**

Hardness in water is that characteristic, which “prevents the lathering of soap”. This is due to presence in water of certain salts of calcium, magnesium and other heavy metals dissolved in it. A sample of hard water, when treated with soap does not produce lather, but on other hand forms a white scum or precipitate. This precipitate is formed, due to the formation of insoluble soaps of calcium and magnesium

In a hard water sample, the total hardness can be determined by titrating the Ca2+ and Mg2+ present in an aliquot of the sample with Na2EDTA solution, using NH4Cl-NH4OH buffer solution of pH 10 and Eriochrome Black-T as the metal indicator.

Na2H2Y (Disodium EDTA solution) → 2Na+ + H2Y-

Mg2+ + HD2- (blue) → MgD (wine red) + H+

D (metal-indicator complex, wine red colour) + H2Y- →Y- (metal EDTA complex colourless) + HD- (blue colour) + H+

**Procedure:**

**Determination of Hardness of Water Sample**

* Select the titrant(EDTA).
* Adjust the speed of the drops from the burette.
* Adjust the molarity of titrant.
* Select a definite volume of water sample.
* Choose the indicator & start the titration.
* When colour changes from wine red to blue click the "stop" button & note the volume of EDTA used.

**Formula for Calculation:**

= \_\_\_\_\_\_ppm

**Hardness Comparison: Tap Water < Well water < Sea Water**

**Viva Questions:**

1. **How can temporary hardness be removed?**

Temporary hardness is caused by carbonate, bicarbonate and hydroxide of calcium and magnesium ions in the water. It can be removed by boiling water or by the addition of lime [Ca(OH)2].

Boiling promotes the formation of carbonate from the bicarbonate and precipitates calcium carbonate out of solution which are deposited as a crust at the bottom of vessel, leaving water that is softer upon coving

1. **What is EDTA?**

Ethylenediamine Tetra-acetic acid is a chemical that binds and holds onto (chelates) minerals and metals. It is added as a titrant.

1. **How is Hardness detected?**

When the water is treated with soap solution, if it prevents lathering and forms white scum, the water contains hardness. Water containing hardness, gives wine red colour with Eriochrome Black –T indicator.

1. **What causes Temporary and Permanent Hardness?**

Temporary or carbonate hardness: It is caused by the presence of dissolved bicarbonates of calcium, magnesium and other heavy metals and the carbonate of iron.

Permanent or non-carbonate hardness: It is due to the presence of chlorides and sulphates of calcium, magnesium, iron, and other heavy metals. Unlike temporary hardness, permanent hardness is not destroyed on boiling.

1. **How can permanent hardness be removed?**

Permanent hardness can be removed using water-softener or ion-exchange column, where the calcium and magnesium ions are exchanged with the sodium ions in the column. It can be removed by Lime – Soda process or Zeolite process.

1. **What is ppm or mq/L?**

Parts per million (ppm): It is defined as the number of parts of CaCO3 equivalent hardness per 1000000 parts of water.

Milligrams per litre (mg/lit): It is defined as the number of milligrams of CaCO3 equivalent hardness per 1 litre of water.

1. **What is Clarke’s and French degree?**

Clarke’s degree (oCl): It is defined as the number of parts of CaCO3 equivalent hardness per 70,000 parts of water.

French degree (oFr): It is defined as the number of parts of CaCO3 equivalent hardness per 105 parts of water.

**Relationship between these units is:**

1ppm = 1 mg/lit = 0.10 oFr = 0.070 oCl

1. **Why is titration involving EDTA carried out slowly towards the end?**

Titration is carried out slowly (drop by drop) at the end to ensure a precise measurement of the acid (or any substance) used to neutralized the solution.

1. **Why does EBT show a Wine Red colour at the beginning and a Blue colour at the end?**

If EDTA is added as a titrant, the calcium and magnesium will be complexed. After sufficient EDTA has been added to complex all the magnesium and calcium, the solution will turn from wine red to blue

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**Exp 4:**

**Aim: To determine chemical oxygen demand (COD) of water samples**

COD is used as a measure of oxygen equivalent to organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific source, COD can be related empirically to BOD. COD determination has advantage over BOD determination in that the result can be obtained in about 5 hours as compared to 5 days required for BOD test.

The organic matter gets oxidized completely by K2Cr2O7 in the presence of H2SO4 to produce CO2 and H2O. The excess of K2Cr2O7 remained after the reaction is titrated with ferrous ammonium sulphate. The dichromate consumed gives the O2 required for oxidation of organic matter.

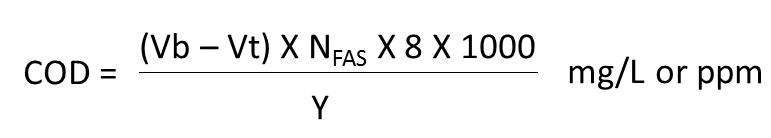
Ferrous Ammonium Sulphate : FAS or Mohr’s Salt (NH₄)₂Fe(SO₄)₂.6(H₂O)

**Procedure:**

* Select the water sample.
* To reflux the contents in the RB flask click the "switch on mantle" button.
* Click "start titration" to titrate the contents.
* Select the normality of ferrous ammonium sulphate (FAS).
* Start titration & note the volume of titrant consumed when colour changes from bluish green to wine red. (Let the volume of titrant be V2mL).
* Repeat the same with the blank (Let the volume of the titrant be V1mL).
* COD calculated using the equation.

**Formula for Calculation:**

COD(mg/L) = where Vol. of FAS = Vblank –Vsample



**COD Comparison: Tap Water < Well Water < Domestic Waste Water**

**Viva Questions**

1. **What is COD**?

Chemical Oxygen Demand or COD is a measure of amount of oxygen required for the chemical oxidation of organic matter when refluxed in acidified potassium dichromate in the presence of Ag2SO4 or HgSO4 catalyst for 3 hours. Chemical Oxygen Demand (COD) refers to the amount of chemically oxidizable matter present in the water. It is ALWAYS greater than BOD.

1. **What Indicator is used for this experiment?**

Ferroin is used as an indicator. Ferroin is added during this titration as it changes its colour from red to pale blue when oxidized.

1. **State Significance of COD**

* COD gives the extent of pollution caused by bio-degradable and non bio-degradable pollutants in wastewater sample
* It helps in rapid determination of pollutants level in water compared to BOD
* It is taken as a basis for calculation of efficiency and designing of water treatment plants.

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**Exp 5**

**Aim: Determine the viscosity average molecular weight of a polymer.**

Consider two liquids are passing through a capillary of same viscometer. Then the coefficient of viscosity of liquid (η2) is given by equation

https://vlab.amrita.edu/userfiles/1/image002%286%29.png

Here*t1* and*t2* are the time of flow of the liquids and*ρ1* and *ρ2* are the respective densities. And *η1* is the coefficient of viscosity of water.

For a given liquid *η* has a specific value at the same temperature.

Various mixtures of two non-interacting liquids viscosities will lie among the viscosities of those pure components.

The time of flow of liquid depends on the viscosity and composition. In this method the flow times are measured for different known compositions and a graph is plot for time of flow and compositions. The unknown composition can be determined by plotting a graph for the time of flow and compositions.

**Procedure:**

* Select the Polymer.
* Select the Solvent.
* Determine the Time of flow of the solvent (t0).
* Determine the time of flow of polymer-solvent system at different concentrations.
* From the concentration and time of flow, the inherent viscosity and reduced viscosity are calculated using the equations;
* A graph is drawn by plotting reduced viscosity against concentration and inherent viscosity against concentration.
* Intrinsic viscosity can be obtained by extrapolating the graph to zero concentration.
* From the value of intrinsic viscosity, the viscosity average molecular weight of the polymer can be calculated by using the equation.

**Formula for Calculation:**

Mark-Houwink Equation:

Intrinsic Viscosity : [𝜼] = KMα where M is Molecular Weight and K and α are constants.

Inherent Viscosity : = 𝜼inh where r =

Reduced Viscosity: = 𝜼red (dl/g)where sp = r  - 1

Relative Viscosity**= «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mfrac»«mi»§#951;«/mi»«msub»«mi»§#951;«/mi»«mn»0«/mn»«/msub»«/mfrac»«mo»=«/mo»«mfrac»«mi»t«/mi»«msub»«mi»t«/mi»«mn»0«/mn»«/msub»«/mfrac»«mo»=«/mo»«msub»«mi»§#951;«/mi»«mi»r«/mi»«/msub»«/math»**

Specific Viscosity **=«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mfrac»«mrow»«mi»§#951;«/mi»«mo»-«/mo»«msub»«mi»§#951;«/mi»«mn»0«/mn»«/msub»«/mrow»«msub»«mi»§#951;«/mi»«mn»0«/mn»«/msub»«/mfrac»«mo»=«/mo»«mfrac»«mrow»«mi»t«/mi»«mo»-«/mo»«msub»«mi»t«/mi»«mn»0«/mn»«/msub»«/mrow»«msub»«mi»t«/mi»«mn»0«/mn»«/msub»«/mfrac»«mo»=«/mo»«msub»«mi»§#951;«/mi»«mi»r«/mi»«/msub»«mo»-«/mo»«mn»1«/mn»«mo»=«/mo»«msub»«mi»§#951;«/mi»«mrow»«mi»s«/mi»«mi»p«/mi»«/mrow»«/msub»«/math»**

Reduced Viscosity =**«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mfrac»«msub»«mi»§#951;«/mi»«mrow»«mi»s«/mi»«mi»p«/mi»«/mrow»«/msub»«mi»C«/mi»«/mfrac»«mo»=«/mo»«msub»«mi»§#951;«/mi»«mrow»«mi»r«/mi»«mi»e«/mi»«mi»d«/mi»«/mrow»«/msub»«/math»**

Inherent Viscosity =**«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mfrac»«mrow»«mi mathvariant=¨normal¨»ln«/mi»«mo»§nbsp;«/mo»«msub»«mi»§#951;«/mi»«mi»r«/mi»«/msub»«/mrow»«mi»C«/mi»«/mfrac»«mo»=«/mo»«msub»«mi»§#951;«/mi»«mrow»«mi»i«/mi»«mi»n«/mi»«mi»h«/mi»«/mrow»«/msub»«/math»**

Intrinsic Viscosity**= «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msub»«mfenced»«mfrac»«msub»«mi»§#951;«/mi»«mrow»«mi»s«/mi»«mi»p«/mi»«/mrow»«/msub»«mi»C«/mi»«/mfrac»«/mfenced»«mrow»«mi»C«/mi»«mo»§#8594;«/mo»«mn»0«/mn»«/mrow»«/msub»«mo»=«/mo»«msub»«mfenced»«mfrac»«mrow»«mi mathvariant=¨normal¨»ln«/mi»«mo»§nbsp;«/mo»«msub»«mi»§#951;«/mi»«mi»r«/mi»«/msub»«/mrow»«mi»C«/mi»«/mfrac»«/mfenced»«mrow»«mi»C«/mi»«mo»§#8594;«/mo»«mn»0«/mn»«/mrow»«/msub»«mo»=«/mo»«mfenced close=¨]¨ open=¨[¨»«mi»§#951;«/mi»«/mfenced»«/math»**

**Flow Time Increases as Concentration of Polymer-Solvent System is increased.**

**Viva Questions**

1. **What is Viscosity Average Molecular Weight?**

The molecular weight of the polymer is measured by using viscometer and the molecular weight obtained by this technique is called viscosity average molecular weight.

1. What is Inherent Viscosity?

Inherent viscosity is the ratio of the natural logarithm of the relative viscosity to the mass concentration of the polymer.

1. **What is Viscosity?**

Viscosity is the resistance of a fluid (liquid or gas) to a change in shape or movement of neighbouring portions relative to one another. Viscosity denotes opposition to flow.

1. **What apparatus is used in this experiment?**

A viscometer is used in this experiment to find out the time of flow for Polymer-Solvent system.

1. **Which method is used to find the flow time in this experiment?**

A simple process called **Ostwalds’s Process** is used in this experiment. In this method viscosity of liquid is measured by comparing the flow times of two liquids of equal volumes using a viscometer.

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**Exp 6**

**Aim: To study the construction and working of compression molding.**

Compression molding is a process that involves the exposure of thermosetting materials to a particular temperature to soften it which results in high strength articles. It can be employed to develop a variety of thermoplastic, thermosetting, rubber and composite products such as bottle caps, sheets, gaskets, O-rings, seals, electrical components, automotive components etc. It uses compressive force and heat to shape a raw material by using a shaped metal mold.

**Procedure:**

* **Opening of mold and application of mold releasing agent:** This step is required to free the mold from any impurity or contamination adsorbed inside of the mold cavity to achieve good surface finished product .The mold releasing agents are used to kick out the contamination generated during the cycle. The releasing agents creates invisible layer over the mold surface and reduces friction in the contact of polymer with mold surface which gives the easy removal of finished product.
* **Loading raw material in the mold cavity:**

The next step is to load the charge or raw material in the mold cavity for molding. Plastics are generally heat–insulators in nature which creates a problem of uneven heat distribution. The raw material is in direct contact with mold first and then the heat is transferred through the other section by conduction. Due to the heat-insulating nature, the curing reaction will start at the surface of the material which is in direct contact with mold. However, the lack of temperature in the bulk material results in restriction of the curing. Preheating of raw material is done which results in the reduction of the curing time of the material and hence this increases frequency of molding. The preheating also removes the moisture or other volatile components prior to molding and reduces mold shrinkage. Though preheating is beneficial in many ways, care must be taken as excessive preheating can cure the material before it reaches the mold cavities.

* **Closing of Mold:** After loading of the raw material or charge the mold is closed by using the hydraulic compression system. To reduce wear between meeting surfaces, the mold is initially closed rapidly till both halves of mold touches each other and the speed is reduced.
* **Breathing cycle:** The evolution of volatile gases during crosslinking reaction happening in the closed mold is important and has to be considered while compression molding operation. These gases must be removed from the closed mold to avoid any defects in the molded article. The trapping of these gases in the product reduces the cross linking density and can cause blisters or ruptures. To avoid this, a breathing cycle has to be given during mold closing to allow easy escape of volatile gases from the molding compound as the raw material experiences heating from the mold and from the exothermic reaction of the resin and catalyst during cure. Hence, the breathing time is the time interval between the opening and closing of mold for removal of volatile gases.
* **Compression Cycle:** After the completion of breathing cycle, the material is held under desired temperature and pressure for certain time to complete the molding/curing (thermosetting) process which is called dwell time for curing. The curing time can be decided based on the materials used and their composition and characteristics to evaluate the curing time required for the material.
* **Opening of mold and ejection of article:** After the completion of curing step, the mold opens up and the finished article is ejected out from the mold either by automatic ejection system or by manual intervention. In some cases, if the product achieved desired stiffness for the ejection, product/s can be removed from the mold before the completion of curing reaction. The curing will continue for the next several minutes as its temperature gradually returns to ambient temperature (cooling). After the completion of this cycle the mold is again filled with a fresh set of raw material and the molding process repeated.

**Formula for Calculation**

1. **Charge of Raw material in the cavity (grams)** = Volume of cavity (cm3) \* Bulk density of polymer raw material (gm/cm3)
2. **Clamping pressure requirement for compression molding (Tonnes)** = Projected area of article (inch2) X y (tonnes/inch2), Where, y is in the range of 1.78-2.67 Tonnes/in2.

**Viva Questions**

1. **What are Thermoplastics and give examples?**

A thermoplastic is a polymeric material that can be remoulded upon application of heat. Common examples of thermoplastics include acrylic, polyester, polypropylene, polystyrene, nylon and Teflon.

1. **Fill in the Blanks:**

\_\_**Printed Circuit Boards**\_\_ are made using Phenol Formaldehyde.

Electrical junction boxes are made using \_\_**BMC/DMC**\_\_.

Polypropylene and Polystyrene are examples of \_\_**Thermoplastics**\_\_.

\_\_**Hydraulic Pump**\_\_ is the equipment part responsible to provide compressive pressure to mold platen.

The step which deals with giving the final shape to the article is \_\_**Compression**\_\_.

1. **Match the following:**

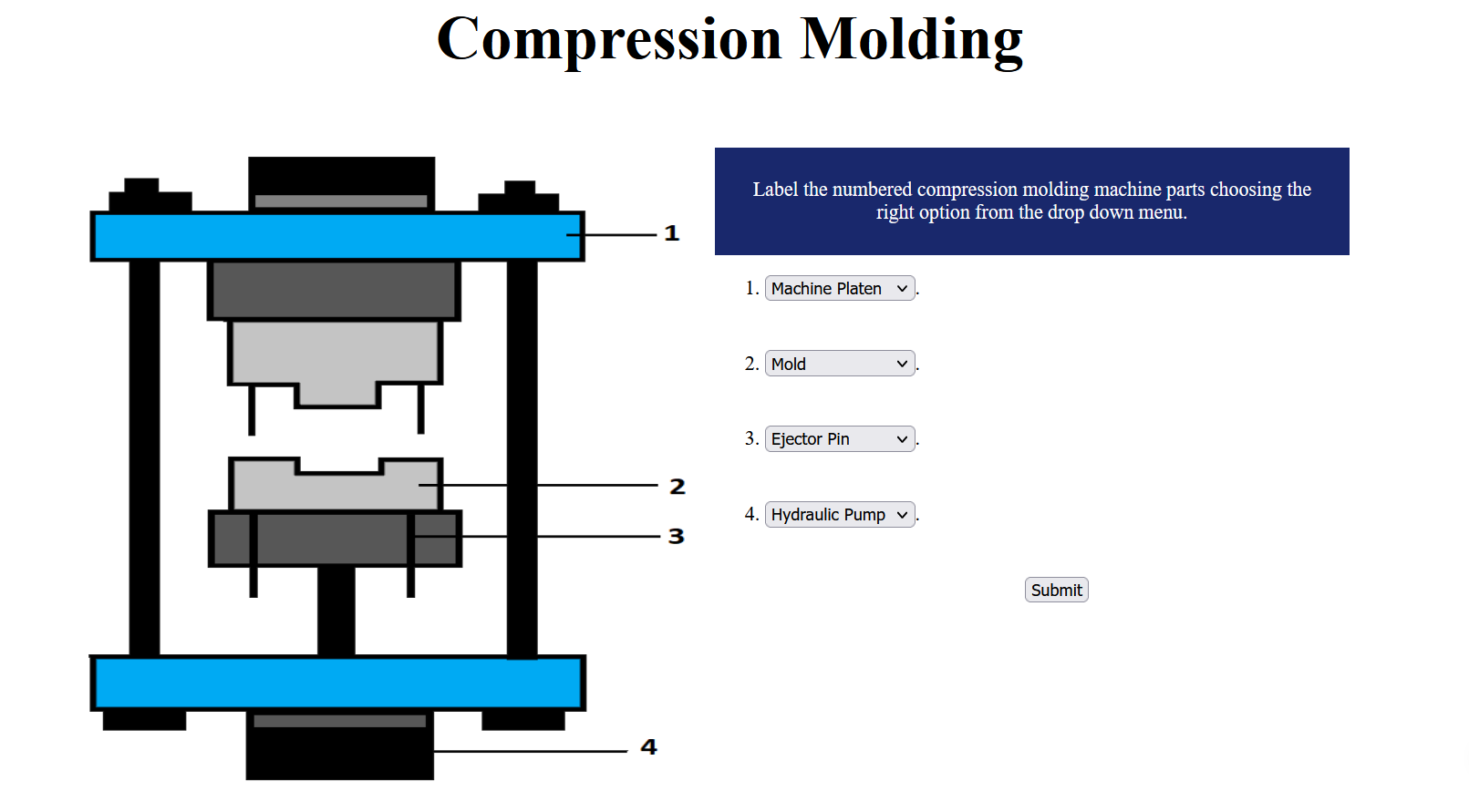
Scorching(Degradation) – High Temperature and Pressure

Excess Flash – OK Temperature and High Pressure.

Article Not Formed – Low Temperature and Pressure.

Improper Shape of Article – Low Temperature and High Pressure.

1. **Label the Machine Parts**



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**Exp 7**

**Aim:** To measure the absorbance of the sample at different wavelengths and to find out the unknown concentration of the sample.

**Different Types of Spectrophotometers:**

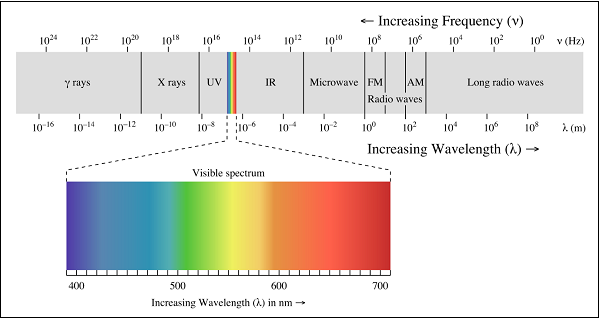
**A. Single Beam:**In this type, all the light passes through the sample .To measure the intensity of the incident light the sample must be removed so that all the light can pass through. This type of spectrometer is usually less expensive and less complicated.

**B. Double Beam:** In this type, before it reaches the sample, the light source is split into two separate beams. From these one passes through the sample and second one is used for reference. This gives an advantage because the reference reading and sample reading can take place at the same time..

**C. Visible Light (**400-700 nm)**:**Visible spectrophotometers can use incandescent, halogen, LED, or a combination of these sources and these spectrophotometers vary in accuracy. Plastic and glass cuvettes can  be used for visible light spectroscopy.

**D. Ultraviolet Light:**UV spectroscopy is used for fluids, and even solids. Cuvettes, only made of quartz, are used for placing the samples.

**E. Infrared Light:**IR spectroscopy helps to study different structures of molecules and their vibrations. Different chemical structures vibrate in different ways due to variation of energy associated with each wave length. For example, mid-range and near infrared (higher energy) infrared tends to cause rotational vibrations and harmonic vibrations respectively.



**Procedure:**

**Determination of Molar Absorption Coefficient:**

1. Select a blank cuvette and place it in the spectrophotometer. Close the lid.
2. Click on 0 ABS 100%T button, the instrument now reads 0.00000 A.
3. Choose a solution with known concentration and measure the absorbance between the wavelengths 350 nm to 700 nm.
4. Record the wavelength at the maximum absorbance value.
5. Calculate the value of molar absorption coefficient, using the equation «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»§#949;«/mi»«mo»=«/mo»«mi»A«/mi»«mo»/«/mo»«mi»c«/mi»«mi»l«/mi»«/math».

**Determination of Unknown Concentration:**

1. Set the wavelength to the value corresponding to maximum absorbance (recorded above).
2. Place the cuvette with same solution but at an unknown concentration.
3. Read the absorbance for this wavelength.
4. Calculate the concentration with the help of the equation, molarity «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mo»=«/mo»«mi»A«/mi»«mo»/«/mo»«mi»§#949;«/mi»«mi»l«/mi»«/math»
5. Enter the calculated concentration value in the given box. (Note : Should enter the value correct to four decimal places)
6. Repeat the same procedure for a second solution.

**Viva Questions:**

1. **What is a Spectrophotometer?**

A spectrophotometer is a photometer that can measure the intensity of light as a function of its wavelength. Single beam and double beam are the two major classes of spectrophotometers. Linear range of absorption and spectral bandwidth measurement are the important features of spectrophotometers.

1. **What is Beer Lambert’s Law?**

Beer-Lambert’s law is the linear relationship between the absorbance and concentration of the absorbing sample, i.e. a logarithmic relation exist between the transmission of light through a substance (T) and the product of absorption coefficient of a substance (α) and  distance travelled by the light through the material(path length l) The absorption coefficient is the product of molar absorptivity, the concentration the material, or an absorption cross section, [σ](https://wumbo.net/symbol/sigma/), and the (number) density.

A = E . c . l

**Lambert’s Law:** Equal Fractions of incident radiation are absorbed by successive layers of equal thickness containing the same number of absorbing species.

**Beer’s Law:** Equal Fractions of incident radiation are absorbed by successive layers of the medium containing equal concentration of absorbing species provided all the layers have same thickness.

1. **What is the nature of the graph?**

According to the Beer-Lambert Law, absorbance is proportional to concentration, so that at dilute solutions a plot of concentration vs. absorbance would be straight line, but the Law breaks down for solutions of higher concentration, and so you might get a curve under those circumstances.

1. **State a few applications of Spectrophotometer**

* It is directly used to measure light intensity at different wavelengths.
* It is used to determine the unknown concentration of solution.
* Spectrometers can be used to determine the equilibrium constant of a reaction involving ions.

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**Exp 8**

**Aim:** To determine the available nitrogen in the soil sample by Kjeldahl Method

The Kjeldahl method permits the available nitrogen to be precisely determined in the plant and in the soil. The method of determination involves three successive phases which are,

* Digestion of the organic material to convert nitrogen into HNO3.
* Distillation of the released Ammonia into an absorbing surface or medium.
* Volumetric analysis of the Ammonia formed during the digestion process.

Digestion:

Digestion of the organic material is carried out by digesting the sample with Con. H2SO4 in the presence of CuSO4.H2O as a catalyst and K2SO4 which raise the digestion temperature. The organic material decomposes into several components i.e.,

C → CO2, O → H2O and N → NH3

In the organic matter, some nitrates are present, most of which are lost during the digestion. The loss may be disregarded for most soils. Since the amount of NO3- - N is far lesser than the Organic Nitrogen.

Distillation:

The Ammonia content of the digest is determined by distillation with excess NaOH and absorption of the evolved NH3 is in standard HCl.

(NH4)2SO4 + 2 NaOH → Na2SO4 + 2 NH3 + 2 H2O

NH3 +HCl → NH4Cl

Volumetric Analysis:

The excess of standard HCl is titrated against standard NaOH using Methyl Red as an indicator. The decrease in the multi equivalence of acid as determined by acid-base titration, which gives a measure of the N content of the sample. The end point is determined by a change of colour from pink to yellow.

2 HCl + 2 NaOH → 2 NaCl + H2O

**Procedure**:

* Weigh 50 g of processed soil sample in 500 mL Kjeldahl flask.
* Add 1 g CuSO4, 10 g K2SO4 and 30 mL Con. H2SO4.
* Shake the contents of the flask until through mixing and allowing to stand for at least 30 minutes with frequent shaking or until complete solution results.
* Digest the content until greenish colour appears. K2SO4 raises the boiling point of the acid. So that the loss of acid volatile solution is prevented. CuSO4 5H2O is digestion accelerator which catalyses the speed of digestion process.
* The reagents sometimes contain impurities so run a blank with the same quantities of reagents and subtract the blank value from the value of the soil digest.
* Digestion is effected on the Kjeldahl digestion rack with low flame for the first 10 – 30 min until the frothing stops and then gradually more strongly until the sample is completely charred. The heat is gradually raised until the acid reaches approximately one third the way up the digestion-flask. The flame is not allowed to touch the flask above the part occurred by the liquid. Excessive boiling may cause volatilization of the acid before the organic matter is oxidized.
* Cool the content and dilute to about 100 mL with distilled water. Swirl the flask for about 2 minutes and transfer the fluid part to a 1000 mL distillation flask.
* Wash the residue left in the Kjeldahl flask with 4 or 5 lots of 50 – 60 mL distilled water, decanting the washings into the distillation flask.
* Add a few, glass bead to prevent bumping.
* Fit the flask with two neck joints to one neck dropping funnel is connected for adding 40 % NaOH while to the other neck Kjeldahl trap, which is used to trap the NaOH coming with the distillate. The trap is connected to the condenser with a delivery tube which dips into 50 mL of 0.1 N HCl contained in a conical flask, with one or two drops of methyl red indicator.
* Add about 125 mL (or 100 ml if bumping is a problem) of 40 % NaOH solution till the content are alkaline in reaction (about 5 times the volume of Con. H2SO4 used during the digestion). Heat the RB flask.
* Allow the ammonia formed to be absorbed in standard HCl. Wash down the end of the tube. 150 mL distilled water is added to the conical flask. When no more ammonia is received (test with a red litmus paper turning blue) stop the distillation.
* Titrate the excess of the acid with 0.1 N NaOH solution till the pink colour changes to yellow.
* From the titre value calculate the multi equivalence of the acid participating in the process of ammonia absorbing during digestion.

Formula for Calculation:

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Where ‘W’ is the weight of Sample taken in gm

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**Exp 9**

**Aim:**

* To find EMF of the cell.
* To calculate the Gibbs free energy change of the cell reaction.
* To calculate the Equilibrium constant.
* To predict the spontaneity of the cell reaction.

Electrode at which oxidation takes place is anode and the electrode at which reduction takes place is cathode. When a metal is in contact with its own ion solution it develops a potential with respect to the electrolyte. The potential difference developed at the anode - electrolyte interface is called oxidation potential and the potential difference developed at the cathode -electrolyte interface is called reduction potential. The potential difference between the anode and cathode is called the EMF of the cell. The potential difference measured at standard conditions (1 atm pressure, 273K) is called standard electrode potential. Standard electrode potential gives the tendency of the electrode to get oxidized or reduced. If the electrolytes are different the two compartments are joined by a salt bridge, which is a tube containing a concentrated electrolyte solution in agar jelly that completes the electrical circuit and enables the cell to function.

Standard EMF of the cell:

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«mo»=«/mo»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»a«/mi»«mi»t«/mi»«mi»h«/mi»«mi»o«/mi»«mi»d«/mi»«mi»e«/mi»«/mrow»«mn»0«/mn»«/msubsup»«mo»-«/mo»«msubsup»«mi»E«/mi»«mrow»«mi»a«/mi»«mi»n«/mi»«mi»o«/mi»«mi»d«/mi»«mi»e«/mi»«/mrow»«mn»0«/mn»«/msubsup»«/math»

Electrode potential at any concentration can be calculated using Nernst equation. For the reaction,

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»a«/mi»«mi»A«/mi»«mo»§nbsp;«/mo»«mo»+«/mo»«mo»§nbsp;«/mo»«mi»c«/mi»«msup»«mi»C«/mi»«mrow»«mi»a«/mi»«mo»+«/mo»«/mrow»«/msup»«mo»§nbsp;«/mo»«mo»§#8594;«/mo»«mo»§nbsp;«/mo»«mi»c«/mi»«mi»C«/mi»«mo»+«/mo»«mo»§nbsp;«/mo»«mi»a«/mi»«msup»«mi»A«/mi»«mrow»«mi»c«/mi»«mo»+«/mo»«/mrow»«/msup»«/math»

Nernst Equation,

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msub»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«/msub»«mo»=«/mo»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«mfrac»«mrow»«mn»2«/mn»«mo».«/mo»«mn»303«/mn»«mi»R«/mi»«mi»T«/mi»«/mrow»«mrow»«mi»n«/mi»«mi»F«/mi»«/mrow»«/mfrac»«mo»-«/mo»«mfrac»«mrow»«mi mathvariant=¨normal¨»log«/mi»«mo»[«/mo»«msup»«mi»C«/mi»«mrow»«mi»a«/mi»«mo»+«/mo»«/mrow»«/msup»«msup»«mo»]«/mo»«mi»c«/mi»«/msup»«/mrow»«msup»«mfenced close=¨]¨ open=¨[¨»«msup»«mi»A«/mi»«mrow»«mi»c«/mi»«mo»+«/mo»«/mrow»«/msup»«/mfenced»«mi»a«/mi»«/msup»«/mfrac»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«mo»=«/mo»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»a«/mi»«mi»t«/mi»«mi»h«/mi»«mi»o«/mi»«mi»d«/mi»«mi»e«/mi»«/mrow»«mn»0«/mn»«/msubsup»«mo»-«/mo»«mo»§nbsp;«/mo»«msubsup»«mi»E«/mi»«mrow»«mi»a«/mi»«mi»n«/mi»«mi»o«/mi»«mi»d«/mi»«mi»e«/mi»«/mrow»«mn»0«/mn»«/msubsup»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»n«/mi»«mo»=«/mo»«mi»c«/mi»«/math»  if  «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»c«/mi»«mo»=«/mo»«mi»a«/mi»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»n«/mi»«mo»=«/mo»«mi»c«/mi»«mo»§#215;«/mo»«mi»a«/mi»«/math»  if  «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»c«/mi»«mo»§#8800;«/mo»«mi»a«/mi»«/math»

 Where;

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»n«/mi»«/math» = number of electrons.

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msup»«mi»E«/mi»«mn»0«/mn»«/msup»«/math» = electrode potential of cell at standard conditions.

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»T«/mi»«/math» = temperature.

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»R«/mi»«/math» = universal gas constant.

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»F«/mi»«/math» = Faraday constant.

When a cell reaction takes place electrical energy is produced which results in decrease in the free energy of the system.

Electrical work = Decrease in free energy

In an electro chemical cell,

Electric work done = Quantity of electric charge produced x E.M.F of the cell

For one mole of electrons quantity of electric charge is 1«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»F«/mi»«/math» (96500 coulomb)

Therefore, for «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»n«/mi»«/math» moles it is «math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»n«/mi»«mi»F«/mi»«/math».

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»E«/mi»«mi»l«/mi»«mi»e«/mi»«mi»c«/mi»«mi»t«/mi»«mi»r«/mi»«mi»i«/mi»«mi»c«/mi»«mo»§nbsp;«/mo»«mi»w«/mi»«mi»o«/mi»«mi»r«/mi»«mi»k«/mi»«mo»§nbsp;«/mo»«mi»d«/mi»«mi»o«/mi»«mi»n«/mi»«mi»e«/mi»«mo»=«/mo»«mi»n«/mi»«mi»F«/mi»«msub»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«/msub»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mo»-«/mo»«mo»§#9651;«/mo»«mi»G«/mi»«mo»=«/mo»«mi»n«/mi»«mi»F«/mi»«msub»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«/msub»«/math»

For a standard cell,

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mo»-«/mo»«mo»§#9651;«/mo»«msup»«mi»G«/mi»«mn»0«/mn»«/msup»«mo»=«/mo»«mi»n«/mi»«mi»F«/mi»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«/math»

By van 't Hoff relation,

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mo»§#9651;«/mo»«msup»«mi»G«/mi»«mn»0«/mn»«/msup»«mo»=«/mo»«mo»§nbsp;«/mo»«mo»-«/mo»«mi»R«/mi»«mi»T«/mi»«mi mathvariant=¨normal¨»ln«/mi»«mi»K«/mi»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«mo»=«/mo»«mfrac»«mrow»«mi»R«/mi»«mi»T«/mi»«/mrow»«mrow»«mi»n«/mi»«mi»F«/mi»«/mrow»«/mfrac»«mi mathvariant=¨normal¨»ln«/mi»«mi»K«/mi»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi mathvariant=¨normal¨»ln«/mi»«mi»K«/mi»«mo»=«/mo»«mfrac»«mrow»«mi»n«/mi»«mi»F«/mi»«msup»«mi»E«/mi»«mn»0«/mn»«/msup»«/mrow»«mrow»«mi»R«/mi»«mi»T«/mi»«/mrow»«/mfrac»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«/math»

«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»K«/mi»«/math» = equilibrium constant

**Spontaneity or Feasibility of Reaction:**

| **«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mo»§#9651;«/mo»«mi»G«/mi»«/math»** | **«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«mi»K«/mi»«/math»** | **«math xmlns=¨http://www.w3.org/1998/Math/MathML¨»«msubsup»«mi»E«/mi»«mrow»«mi»c«/mi»«mi»e«/mi»«mi»l«/mi»«mi»l«/mi»«/mrow»«mn»0«/mn»«/msubsup»«/math»** | **Reaction** |
| --- | --- | --- | --- |
| Negative | >1 | Positive | Spontaneous |
| Zero | =1 | Zero | Equilibrium |
| Positive | <1 | Negative | Non - spontaneous |