**EXPERIMENT: 1**

**AIM:** Determination of iron content in the given sample using K3[Fe(CN)6] as an external indicator.

**APPARATUS:**

Burette, burette stand, conical flask, white glazed tile, beaker, glass rod, measuring cylinder, funnel.

**CHEMICAL USED:**

K2Cr2O7, [K3Fe(CN)6], H2SO4, FeSO4

**THEORY:**

This estimation is based on the principle that the solution containing ferrous ammonium sulphate can be quantitatively titrated against standard K2Cr2O7 solution in the presence of H2SO4 using potassium ferricyanide as an external indicator.

Oxides of ferrous sulphate present in Mohr’s salt into ferric sulphate in the presence of dilute H2SO4. K3Fe(CN)6 is used as an external indicator gives a greenish blue colour due to formation of ferro-ferricyanide complex.

**OBSERVATION TABLE:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S. No.** | **Volume of sample used** | **Burette**  **reading**  **(initial) (mL)** | **Burette**  **reading**  **(final) (mL)** | **Vol. of titrant**  **(K2Cr2O7)**  **Used (ml)**  **(Final-Initial)** |
| **1** | **10** | **0** | **R1** | **V1** |
| **2** | **10** | **0** | **R2** | **V2** |
| **3** | **10** | **0** | **R3** | **V3** |

**CALCULATIONS:**

Applying law of chemical equivalence,

**N1V0 = N2Vcr**

N1: Normality of Fe2+ ion

V0: Volume of sample used

N2: Normality of K2Cr2O7 (N/50)

Vcr: Volume of concurrent burette reading

Content of Fe2+ in the given solution = N1 x eq. wt. of Iron.

**RESULT:** The Iron content in given sample is………..g/L

**EXPERIMENT: 2**

**AIM:** Determination of temporary and permanent hardness in water sample using EDTA as standard solution.

**APPARATUS:**

Burette, Pipette, Measuring Cylinders, Measuring Flask, Conical Flask.

**CHEMICALS:**

Standard (0.01M) EDTA solution water sample, standard hard water, buffer solution (NH4OH-NH4Cl) pH 9-10. Erichrome Black-T as an internal indicator.

**THEORY:**

The hardness of water is due to Ca2+ & Mg2+ ions. It can be determined by complexometric titration by using EDTA as titrant whose strength is known and Erichrome Black-T act as an internal indicator.

Ca2+ and Mg2+ ions from stable complex with EDTA. EDTA is generally used in its disodium or tetrasodium salt as these are more soluble in aqueous medium.

When 2-3 drops of Erichrome black-T indicator is added to hard water solution at pH 9-10, it gives blue colour to the solution.

**OBSERVATION TABLE:**

**TABLE:** Reading of EDTA Vs Hard Water Sample

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S. No.** | **Volume of sample used** | **Burette**  **reading**  **(initial) (mL)** | **Burette**  **reading**  **(final) (mL)** | **Vol. of titrant**  **(EDTA)**  **Used (ml)**  **(Final-Initial)** |
| **1** | **10** | **0** | **R1** | **V1** |
| **2** | **10** | **0** | **R2** | **V2** |
| **3** | **10** | **0** | **R3** | **V3** |

**CALCULATIONS:**

Applying law of chemical equivalence,

**N1V0 = N2Vcr**

N1: Normality of Hard water

V0: Volume of sample used

N2: Normality of EDTA (N/50)

Vcr: Volume of concurrent burette reading

Total hardness (in ppm of CaCO3) = N1 x 50,000 ppm

**RESULT:** Total hardness of water is ……………..ppm.

**EXPERIMENT: 3**

**AIM:** Determination of alkalinity in the given water sample.

**APPARATUS:** Burette, pipette, conical flask, beaker, measuring flask.

**CHEMICALS:** Water sample, N/50 HCl, phenolphthalein and methyl orange

indicator.

**THEORY:** Alkalinity of water is mainly due to the presence of the following.

(i) Hydroxides only

(ii) Carbonates only

(iii) Bicarbonates only

(iv) Hydroxides and carbonates

(v) Carbonates & bicarbonates

Since OH- & HCO3- ion cannot co-exist because both combine to form carbonates.

The extent of alkanity present in a water sample is determined by titrating the water sample (titrate) with a standard acid (titrant) using phenolphthalein indicator and alkalinity if found out in terms of CaCO3 equivalent by using normality equation. This is called phenolphthalein alkalinity (P). At this point, complete neutralization of hydroxide and conversion carbonate to bicarbonate takes place.

**INDICATORS:** Phenol phtalien and methyl orange.

**END POINTS:** Pink to colourless (phenolphthalein), Yellow to red (methyl orange).

**OBSERVATIONS TABLE:**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **S. No.** | **Volume of sample used** | **Burette**  **reading**  **(initial) (mL)** | **Burette**  **reading**  **(final) (mL)** | | **Vol. of titrant**  **(EDTA)**  **Used (ml)**  **(Final-Initial)** | |
| **P** | **M** | **2P** | **M-2P** |
| **1** | **10** | **0** | **P1** | **M1** | **X1** | **Y1** |
| **2** | **10** | **0** | **P2** | **M2** | **X2** | **Y2** |
| **3** | **10** | **0** | **P3** | **M3** | **X3** | **Y3** |

**CALCULATIONS:**

1. Phenolphthalein alkalinity in terms of CaCO3 equivalent.

Applying law of chemical equivalence,

**N1V0 = N2Vcr**

N1: Normality of Carbonate ion

V0: Volume of sample used

N2: Normality of HCl (N/50)

Vr: Volume of concurrent burette reading

Alkalinity due to CO3 ion = N1 x 50,000 ppm

1. Methyl orange alkalinity in terms of CaCO3 equivalent.

Applying law of chemical equivalence,

**N1V0 = N2Vcr**

N3: Normality of Bicarbonate ion

V0: Volume of sample used

N2: Normality of HCl (N/50)

Vr: Volume of concurrent burette reading

Alkalinity due to HCO3 ion = N3 x 50,000 ppm

Total alkalinity = Alkalinity due to CO32- +Alkalinity due to HCO3-

**RESULT:**

Total alkalinity of given sample of water is …………. Ppm.

**EXPERIMENT: 4**

**AIM:** Determination of chloride content in the given water sample by Mohr’s method.

**APPARATUS:** Burette, pipette, conical flask, beaker, measuring flask.

**CHEMICALS:** Water sample, N/50 HCl, phenolphthalein and methyl orange

indicator.

**Theory:**

This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium chromate, to form a red-brown precipitate of silver chromate.

This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water. Seawater is used as the example here.

The pH of the sample solutions should be between 6.5 and 10. If the solutions are acidic, the gravimetric method or Volhard’s method should be used.

**OBSERVATION TABLE:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S. No.** | **Volume of sample used** | **Burette**  **reading**  **(initial) (mL)** | **Burette**  **reading**  **(final) (mL)** | **Vol. of titrant**  **(K2CrO4)**  **Used (mL)**  **(Final-Initial)** |
| 1 | 10 | 0 | R1 | V1 |
| 2 | 10 | 0 | R2 | V2 |
| 3 | 10 | 0 | R3 | V3 |

**CALCULATIONS:**

Applying law of chemical equivalence,

**N1V0 = N2Vcr**

N1: Normality of Cl- ion

V0: Volume of sample used

N2: Normality of K2CrO4 (N/50)

Vcr: Volume of concurrent burette reading

Content of Cl- in the given solution = N1 x eq. wt. of Chlorine.

**RESULT:** The Chlorine content in given sample is………..g/L

**EXPERIMENT 5:**

**AIM:** Determination of percentage of available chlorine in bleaching powder sample.

**APPARATUS:** Burette, Pipette, Measuring Flask, Conical Flask, Funnel and Beaker.

**CHEMICALS:**

Potassium iodide, glacial acetic acid, sodium thiosulphate, distilled water and freshly prepared starch sol as indicator.

**THEORY:**

The residual chlorine is obtained by oxidation of KI consuming residual chlorine by volumetric titration.

If water sample containing residual Cl2 is treated with KI it acidizes KI and liberates I2 in equivalent amount.

Cl2 may be present in water as HOCL, OCl, NHCL2, these salts also reacts with KI and liberates Iodine.

The liberated I2 is titrated against hypo solution using starch as indicator.

The end point is disappearance of blue colour.

**OBSERVATION TABLE:**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **S. No.** | **Volume of sample used** | **Burette**  **reading**  **(initial) (mL)** | **Burette**  **reading**  **(final) (mL)** | **Vol. of titrant**  **(Hypo Solution)**  **Used (mL)**  **(Final-Initial)** |
| 1 | 10 | 0 | R1 | V1 |
| 2 | 10 | 0 | R2 | V2 |
| 3 | 10 | 0 | R3 | V3 |

**CALCULATIONS:**

Applying law of chemical equivalence,

**N1V0 = N2Vcr**

N1: Normality of Cl- ion

V0: Volume of sample used

N2: Normality of Hypo Solution (N/50)

Vcr: Volume of concurrent burette reading

Content of Cl- in the given solution = N1 x eq. wt. of Chlorine.

% of available Chlorine

**RESULT:** % of available Chlorine in given sample is………..%

**EXPERIMENT 6:**

**AIM:** pH-metric titration between strong acid and strong base.

**APPARATUS:**

Standard flask Beaker PH Meter PH Paper Glass Electrode etc.

**CHEMICALS:** Buffer solution, hydrochloric acid, sodium hydroxide etc.

**THEORY:** The pH value of solution is defined as negative logarithm of hydrogen ion concentration. It is expressed in gram/ion lit.

pH = -Log [H+] = Log10 1/[H+]

AT 250C the ionic product of water

Kw = [H+] [OH-] =10-14

Also in pure water [H+] = [OH-]

In neutral solution [H+] = [OH-]

[H+] [OH-] = (1×10-7) (1×10-7)

pH of neutral solution = -Log10 10-7 = 7

Consequently, pH of neutral solution is 7; in acidic solution PH<7, since [H+]>10-7 on the other hand pH

of the alkaline soln. is >7, Since [H+] <10-7

pH value of distilled water = 7

pH of the soln. is tasted by using pH paper. This gives different colour with different values.

**PROCEDURE:**

1) Take the standard solution in 100mL Beaker

2) Deep the pH electrode in the Beaker & note down the pH.

3) Then take another solution in a beaker deep the electrode & note pH of solution also deep the pH paper & observe the colour Change& note down the nature of solution.

4) Take the fourdifferent reading of four different solutions & Write the result either the solution are acidic, basic or neutral.

**OBSERVATION TABLE:**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Sr. No. | Solution | pH paper | pH of  solution | Change in  Colour | Type of  solution |
| 1. | Unknown Solution-1 | Neutral pH  paper |  |  |  |
| 2. | Unknown Solution-2 | Neutral pH  paper |  |  |  |
| 3. | Unknown Solution-3 | Neutral pH  paper |  |  |  |

**RESULT:**

1) pH of first unknown solution is ------ hence the nature of solution is --------

2) pH of second unknown solution is ------ hence the nature of solution is --------

3) pH of third unknown solution is ------ hence the nature of solution is --------

**EXPERIMENT 7:**

**AIM:** Viscosity of a polymer like polystyrene by Viscometric method.

**APPARATUS:** Ubbeholde Viscometer, beaker, measuring cylinder, thermostat, stop watch.

**THEORY:**

Viscosity is an internal property of a fluid that offers resistance to flow. It is due to the internal friction of molecules and mainly depends on the nature & temperature of the liquid.

Many methods are available for measuring viscosity of polymer solution. The Ubbeholde method is a simple method for the measurement of viscosity, in which viscosity of liquid is measured by comparing the viscosity of an unknown liquid with that of liquid whose viscosity is known. In this method viscosity of liquid is measured by comparing the flow times of two liquids of equal volumes using same viscometer.

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

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 η2 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. 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. The molecular weight of the polymer solution is very high so the viscosity of polymer solution is very high compared to that of pure solvent.

From the Mark-Houwink equation the relationship among the molecular weight and viscosity are given below

Where is the intrinsic viscosity, is Molecular weight, and are constants for a particular polymer solvent system.

If we know the and values for a given polymer solution the intrinsic viscosity and molecular weight can be calculate using the above equation.

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No.** | **Polymer-solvent system** | **K x 103mL/g** | **α** |
| 1. | PMMA-Acetone | 7.70 | 0.70 |
| 2. | PMMA-Benzene | 5.20 | 0.76 |
| 3. | PMMA-Toluene | 7.0 | 0.71 |
| 4. | Poly vinyl acetate-Acetone | 10.2 | 0.72 |
| 5. | Poly vinyl acetate-Benzene | 56.3 | 0.62 |
| 6. | Poly vinyl acetate-Acetonitrile | 41.5 | 0.62 |
| 7. | Poly vinyl alcohol-Water | 45.3 | 0.64 |
| 8. | Poly Styrene-Benzene | 10.6 | 0.735 |
| 9. | Poly styrene-Toluene | 11.0 | 0.725 |

Terms related to viscosity measurement.

For measuring intrinsic viscosity of polymer sample, solutions of known concentrations are prepared, the flow times of solvent (t0) and the solutions (t) are measured using viscometer. Double extrapolation plots of reduced viscosity against concentration and inherent viscosity against concentration is plotted by calculating the corresponding reduced viscosity and inherent viscosity. The intrinsic viscosity is given by the common ordinate intercept of these graphs.

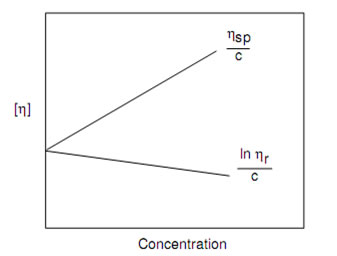


Figure – 1

**CALCULATION:**

**RESULTS:**

The resultant Mol. Wt. of given sample is……………..g/mol

**EXPERIMENT 8:**

**AIM:** Functional group identification in organic compounds.

**THEORY:**

The purpose of this experiment is to observe chemical reactions characteristic of common organic functional groups. The observations will be used to identify a set of unknown compounds

Most organic and biochemical compounds are composed of carbon, hydrogen, and a few other elements (such as oxygen, nitrogen, and sulfur). These compounds participate in a wide variety of reactions generally related to molecular structure. Distinctive arrangements of atoms and bonds, known as functional groups, are responsible for the reactivity of families of organic compounds.

Sodium Bicarbonate Test for Carboxylic Acids

When sodium bicarbonate reacts with carboxylic acids it produces bubbles of carbon dioxide. The bubbles are only present with a carboxylic acid because they are the only family that has an acidic hydrogen.

Fehling’s Test for Aldehyde

In this test the presence of aldehydes but not ketones is detected by reduction of the deep blue solution of copper(II) to a red precipitate of insoluble copper oxide. The test is commonly used for reducing sugars but is known to be NOT specific for aldehydes. For example, fructose gives a positive test with Fehling's solution.

Fehling Solution = Fehling A + Fehling B

Fehling A = Aqueous solution of Copper Sulphate (CuSO4.6H2O)

Fehling B = Na, K- Tartarate 

Copper(II) Complex: 



**OBSERVATION TABLE:**

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No.** | **Observation** | **Inference** | **Result** |
| 1. | Action of NaHCO3    To a few mL of the saturated NaHCO3 solution taken in a test tube, a little of the substance is added. | Brisk effervescence with the liberation of CO2. | Presence of Carboxylic acids. |
| 2. | Add fehling’s solution mixture to given sample and heat up to 50-60oC | Brick red precipitate is observed | Presence of aldehyde group |

**RESULT:**

The given sample A and B contains functional group is ………….and …………respectively.

**EXPERIMENT 9:**

Preparation of a polymer like Bakelite.

**APPARATUS:** Beaker, Conical flask, glass rod, measuring cylinder, fractional weight box etc.

**CHEMICALS:** Glacial acetic acid, 40 % formaldehyde solution, phenol, conc. HCl, Dist. water etc.

**THEORY:**

Phenol resins are condensation polymerization product of phenolic derivative with aldehyde (like formaldehyde, furfural) It is prepared by condensing phenol with formaldehyde in presence of acid or alkaline catalyst.

**Step I:** Formation of ortho and para hydroxy benzyl alcohols from phenol and formaldehyde.



**Step II:** Formation of Bakelite.



**PROCEDURE:**

1) Place 5 ml of glacial acetic acid and 2.5 ml of formaldehyde solution in 500 ml beaker.

2) Add 2 gr of phenol and 1 ml of conc. HCl solution in it.

3) Heat the solution slowly with constant stirring for 5 minutes.

4) A large mass of pink Plastic is formed.

5) The residue obtained is washed several times with distilled water.

6) Dry the product and calculate the yield accurately.

**RESULT:** The weight of obtained Bakelite is ………….. grams.

**EXPERIMENT 10:**

**AIM:** Preparation of Sodium Cobaltinitrite salt.

**CHEMICALS:** Hexaqua dinitro cobalt salt, sodium nitrite

The compound is prepared by oxidation of cobalt(II) salts in the presence of sodium nitrite.

4 [Co(H2O)6](NO3)2 + O2 + 24 NaNO2 → 4 Na3[Co(NO2)6] + 8 NaNO3 + 4 NaOH + 22 H2O

**PROCEDURE:**

1. A solution of 150 g. of NaNO3 in 150 ml. of H3O is cooled to 50-60°C; some of the NaNO3 is thus reprecipitated.
2. Then 50 g of Co(NO3)3 • 6H2O is added, followed by 50 ml. of 50% CH3COOH in small portions (agitation). Then a fast stream of air is bubbled through for one half hour. After standing for two hours, the brown precipitate is filtered off. The filtrate must be clear at this point. The precipitate is stirred with 5 0 ml. of H2O at 70-80°C.
3. The solution is separated from undissolved K3[Co(NO3)6] on a small filter and combined with the above-mentioned clear filtrate. The combined solution (about 300 mL) is treated with 250 mL of 96% alcohol.
4. The resulting precipitate can settle for about two hours, then filtered, suction-dried, washed four times with 25 ml. of alcohol, then twice with ether, and dried in air. Yield: 50-53 g (75%).
5. Reprecipitation with alcohol is desirable. The pure preparation must give a perfectly clear solution in H2O. To precipitate the salt, the alcohol is added from a wash bottle; during the addition, the flask is vigorously shaken to ensure that the particle size of the precipitate will not be too small.

**RESULT:** The yellow crystalline powder was observed.