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**Experiment No. ……**

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| Object |
| Determination of pH of a solution using a pH meter and titration of such a solution pHmetrically. |

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| **Reagents/Chemicals** |
| Standard NaOH solution…………, unknown acid solution, distilled water, buffer solution of pH 4 and 9. |

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| **Apparatus** |
| pH meter, glass electrode, reference electrode, beaker (400 ml), burette, stirrer. |

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| **Theory**  |
| pH = -log [H+]When an alkali is added to an acid, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very fast. The equivalence point can be observed from sharp break in curve. From the equivalence point, the strength of the solution can be calculated by normality equation. |

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| **Method** |
| First the pH meter was standardized against the buffer of pH 4 and pH 9. Then the glass electrode was washed with distilled water and dried. Then 100 ml of HCl solution was taken in a beaker and the electrode was dipped in HCl solution and pH of the pure acid solution was noted down. Standard NaOH solution (1ml) from burette was added into the beaker, stirred well and noted the pH of solution. Similarly 9-10 ml of NaOH was added into acid solution gradually and noted the pH after each addition of NaOH solution. At equivalence point there was a sharp change in the pH. Now alkali solution was added in fractions (0.2 ml). A graph was plotted which gave the actual volume of NaOH at neutralization. |

**Observations**

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Volume of alkali added (ml)** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| **pH** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

|  |  |  |
| --- | --- | --- |
| **Result** | : | The pH of given solution is \_\_\_\_\_\_\_\_\_\_ |
|  | : | The strength of the given acid solution is \_\_\_\_\_\_\_\_\_\_ g/l. |

**Precautions**

|  |  |
| --- | --- |
| 1. | The electrode is washed thoroughly and dried before dipping into the solution. |
| 2. | After each addition of alkali, the solution is stirred thoroughly. |
| 3. | The knob of pH meter is on standby position at the time of adding alkali in the acid solution. |

|  |
| --- |
| Calculations |

|  |  |  |
| --- | --- | --- |
| Volume of alkali used at the end point | = | V2 ml |
| N1V1 | =  | N2V2 |
| (Acid) |  | (Alkali) |
| N1 x 50 | = |  |
| N1 | = |  |

**Experiment No. …….**

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| --- |
| Object |
| Determination of constituents and amount of Alkalinity of the supplied water sample. |

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| --- |
| **Reagents/Chemicals** |
| Water sample, Standard HCl............, Phenolphthalein and Methyl Orange Indicator |

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| --- |
| **Apparatus** |
| Burette, pipette, conical flask, beaker, measuring flask |

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| --- |
| **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 and Bicarbonates, since OH- and HCO3- ion cannot coexist because both combine together to form carbonates  |
| OH- + HCO3- |  | CO3-2 + H2O |
| The extent of alkalinity present in a water sample is determined by titrating the water sample with a standard acid using phenolphthalein end point (P). At this point complete neutralization of hydroxide and conversion carbonate to bicarbonate takes place |
| OH- + H+ |  | H2O |
| CO3-2 + H+  |  | HCO3- |
| Now, methyl orange is added in the solution and further continue the titration till the colour of the solution changes from yellow to brick red. It is methyl orange end point (M) |
| HCO3-+ H+ |  | H2O + CO2 |
| We are able to calculate the magnitude of various forms of alkalinity by knowing the values of (P) ml and (M) ml |
| (i) | When only OH- are present – In this case only phenolphthalein indicator is used, therefore volume of HCl used is (P) ml |
| (ii)  | When only HCO3-are present – In this case only methyl orange indicator is used therefore volume of acid used is (M) ml |
| (iii) | When only CO3-2 are present - In this case both phenolphthalein and methyl orange indicator are used. The volume of HCl used is 2M ml. |
| (iv) | Both OH- and CO3-2 are present - In this case both phenolphthalein and methyl orange indicator are used. The volume of HCl for OH- is (P-M) ml and for CO3-2 is 2M ml |
| (v) | Both CO3-2 and HCO3- are present - In this case both phenolphthalein and methyl orange indicator are used. The volume of HCl for CO3-2 ions is 2P ml and for HCO3- is (M-P) ml |

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| --- |
| **Method** |
| First the burette was rinsed with standard HCl solution and then filled with standard HCl solution. Then …………. ml of water sample was taken out in a conical flask and 2-3 drops of phenolphthalein indicator was added which may give pink colour. Now the solution was titrated with standard solution of HCl till the pink colour changes to colourless. Disappearance of pink colour indicates the end point. It gives the value of phenolphthalein end point (P). Now 2-3 drops of methyl orange was added in the same solution which may give yellow colour and the solution was further titrated till the colour changes to brick red colour. This gives the value of methyl orange end point (M). The process was repeated till two concordant readings were obtained.  |

**Observations**

|  |  |  |
| --- | --- | --- |
| **S. No** | **Volume of Water Sample (ml)** | **Volume of HCl used (ml)** |
|  | **Initial Reading**  | **Phenolphthalein end point (P)** | **Methyl Orange end point (M)** |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

|  |  |  |
| --- | --- | --- |
| **Result** | : | The alkalinity due to CO3-2 is\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ppm |
|  |  | The alkalinity due to HCO3- is\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ppm |
|  |  | The alkalinity due to OH- is\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ppm |

**Precautions**

|  |  |
| --- | --- |
| 1. | Phenolphthalein indicator was added first and then methyl orange |
| 2. | The volume of indicator was same in all the titrations |
| 3. | The reaction mixture was shaken properly |

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| **Method**First the burette was rinsed and filled with standard EDTA solution. Then………ml of hard water sample was taken out in a conical flask, and 2 ml of buffer solution and 2 drops of Eriochrome black-T indicator were added to it. |
| The solution was titrated against standard EDTA solution till the wine red colour changes into blue colour. That was the end point. The process was repeated till two concordant readings were obtained. Thereafter hard water sample……… ml was taken out in a beaker and boiled gently for about half an hour and then filtered. The filtrate was titrated in the same manner as described above. The volume of EDTA used corresponds to the permanent hardness. Now the same procedure was repeated for the standard hard water sample.

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| **Calculations** |

|  |  |  |
| --- | --- | --- |
| 1 ml of standard solution (EDTA) | = | 1 mg of CaCO3 |
| V1 ml of EDTA | =  | V ml of standard solution |
| 1 ml of EDTA | = |  V V1 | mg of CaCO3 |
| V ml of hard water solution | = | V2 ml of EDTA |
|  | = |  V2  | x | VV1 | mg of CaCO3 |
| 1 ml of hard water solution | = | V2V1 | x | VV | mg of CaCO3 |
| 1000 ml of hard water solution | = | V2V1 | x | 1000 | mg of CaCO3 |
| Total hardness | = | V2V1 | x | 1000 | ppm |
| V ml of boiled water | = | V3 ml of EDTA |
| Permanent hardness | = | V3V1 | x | 1000 | mg of CaCO3 |
|  | = | V3V1 | x | 1000 | ppm |
| Temporary hardness | = |  Total hardness – Permanent hardness |

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**Experiment No. …….**

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| --- |
| Object |
| Determination of temporary, permanent and total hardness of water by complexometric titration method. |

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| **Reagents/Chemicals** |
| Standard EDTA solution............, water sample, standard hard water, buffer solution, Eriochrome black-T as an internal indicator |

|  |
| --- |
| **Apparatus** |
| Burette, pipette, measuring flask, conical flask. |

|  |
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| **Theory** |
| The hardness of water can be determined by complexometric titration. EDTA is used as complexing reagent. The Ca++ and Mg++ present in water are titrated with EDTA using Eriochrome black-T as an indicator.When Eriochrome black –T indicator is added to hard water solution at pH 9 to 10, it gives wine red coloured unstable complex with Ca++ and Mg++ ions of the water sample. As this solution (wine red colour complex) is titrated against EDTA the free Ca++ and Mg++ ions in water form stable metal ion EDTA complex with the result , the indicator is set free, which gives blue colour to the solution. |
| Related image**Structure of Eriochrome black-T** |

## Observations

**Reading with standard hard water**

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No** | **Volume of water sample V (ml)** | **Burette Reading (ml)** | **Volume of EDTA used (ml)****(V1)** |
|  | **Initial** | **Final** |  |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

##### Reading with hard water sample

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No** | **Volume of water sample V (ml)** | **Burette Reading (ml)** | **Volume of EDTA used (ml)****(V2)** |
|  | **Initial** | **Final** |  |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

**Reading with boiled Water**

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No** | **Volume of water sample V (ml)** | **Burette Reading (ml)** | **Volume of EDTA used (ml)****(V3)** |
|  | **Initial** | **Final** |  |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

|  |  |  |
| --- | --- | --- |
| **Result** | : | The temporary hardness of the given water sample is \_\_\_\_\_\_\_\_\_ ppm  |
|  | : | The permanent hardness of the given water sample is \_\_\_\_\_\_\_\_\_\_ ppm |
|  | : | The total hardness of the given water sample is \_\_\_\_\_\_\_\_\_\_ ppm |

**Precautions**

|  |  |
| --- | --- |
| 1. | The amount of indicator was same in all the titrations. |
| 2. | The pH of the solution was maintained carefully to precipitate the complex.  |
| 3. | The apparatus was rinsed with distilled water.  |

### Experiment No. ……

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| Object |
| Determination of the percentage of available chlorine in a given sample of Bleaching Powder (Iodometrically). |

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| **Reagents/Chemicals** |
| Bleaching powder or solution, potassium iodide, dilute acetic acid (~5%) sodium thiosulphate solution….................distilled water and freshly prepared starch solution as indicator.  |

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| --- |
| **Apparatus** |
| Burette, pipette, measuring flask, conical flask, funnel, pastel and mortar. |

|  |
| --- |
| Theory |
| The amount of chlorine liberated by the action of dilute acids on hypochlorite is termed as “the available chlorine”. It is generally expressed as the percentage by weight of bleaching powder. In practice the available chlorine is determined by treating it with sodium thiosulphate solution in presence of acetic acid, the liberated chlorine is immediately treated with potassium iodide (KI) to give free iodine, which actually reacts with standard sodium thiosulphate solution, hence termed as Iodometric titration.  |
| CaOCl2 + 2CH3COOH(Bleaching Powder)  |  | (CH3COO)2Ca + H2O + Cl2 ↓(available chlorine) |
| 2KI + Cl2 |  | 2KCl + I2 |
| 2Na2S2O3 + I2 |  | 2NaI + Na2S4O6(sodium tetrathionate) |

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| CaOCl2 | = | 2Cl | = | Na2S4O6(sodium tetrathionate) |

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Na2S4O6(1 gm equivalent) | = | Cl(1 gm equivalent) | = | I(1 gm equivalent) |

|  |
| --- |
| **Method** |
| …………gm sample of bleaching powder was weighed out accurately and made a paste with little distilled water in a mortar. This mixture was transferred to a 250 ml measuring flask. The solution was made up to 250 ml with distilled water to obtain a homogeneous suspension.………….. ml of this suspension was taken in an appropriate conical flask and 1-2 gm of potassium iodide and 10-20ml of dilute acetic acid was added to this solution. The liberated iodine was immediately titrated with standard sodium thiosulphate solution till colour turns light yellow. A few drops of freshly prepared starch solution were added to the light yellow solution, which makes it dark. It was further titrated with thiosulphate solution till the colour disappeared and the burette reading noted. The same titration was repeated until at least two concordant readings were obtained. |

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| **Observations** |
| Weight of empty weighing tube = ………………. (a) gm |
| Weight of weighing tube + bleaching powder = …………..(b) gm |
| Weight of bleaching powder = …………………. (b-a) gm |
| Normality of hypo solution (Na2S2O3) = …………….  |

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No** | **Volume of bleaching powder**  | **Burette Reading (ml)** | **Volume of Na2S2O3 (ml)** |
|  | **Initial** | **Final** |  |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

|  |  |  |
| --- | --- | --- |
| **Result** | : | The percentage of available chlorine present in given sample of bleaching powder is\_\_\_\_\_\_\_\_\_\_% |

**Precautions**

|  |  |
| --- | --- |
| 1. | All the apparatus were cleaned thoroughly. |
| 2. | A uniform paste of bleaching powder was prepared.  |
| 3. | The amount of starch solution and acetic acid remained constant in all the titration. |

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| **Calculations** |
| N1V1 | =  | N2V2 |
| (Bleaching Powder) |  | (Sodium Thiosulphate) |
| Normality of available chlorine (N1) | = | N2 x V2V1 |
| Amount of chlorine per litre of solution  | = | N1 x 35.51000 |
| % of available Chlorine  | = |  N1 x 35.5 x 250 X 1001000 (b-a) |

**Experiment No. …..**

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| Object |
| Determination of the Chloride Content in the supplied water sample using Mohr’s Method |

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| **Reagents/Chemicals** |
| Water Sample, Standard AgNO3 solution……………, K2CrO4 indicator |

|  |
| --- |
| **Apparatus** |
| Burette, pipette, conical flasks, beakers, measuring flask |

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| --- |
| Theory |
| A slightly alkaline solution (pH 7-8) of the water sample is titrated against AgNO3 solution using K2CrO4 as an internal indicator. The chloride ions present in the water sample reacts with silver ions forming insoluble white precipitate of silver chloride (AgCl). When all the chloride ions forms silver chloride then the extra drop of AgNO3 reacts with chromate ions (CrO4-2) forming reddish brown precipitate of silver chromate thus indicating the end point  |
| AgNO3 + NaCl |  | AgCl ↓ + NaNO3 |
| 2Ag+ + CrO4-2 |  | Ag2CrO4 |
| Ag2CrO4 + 2 Cl- |  | AgCl ↓ + CrO4-2 |
| 2AgNO3 + K2CrO4 |  | Ag2CrO4 + KNO3 (Reddish Brown) |

|  |
| --- |
| Method |
| The burette was rinsed with standard AgNO3 solution and filled it with AgNO3 solution. Now………. ml of water sample was taken in a conical flask and 2-3 drops of K2CrO4 indicator were added. The solution was titrated with the standard solution of AgNO3 taken in the burette till reddish brown colour persisted. That was the end point. The process was repeated till two concordant readings were obtained. Now the same procedure was also followed with distilled water to obtain the blank correction. The blank correction reading was deducted from the titre value.  |

**Observations**

##### Reading with Water Sample

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No** | **Volume of Water Sample (ml)** | **Burette Reading (ml)** | **Volume of AgNO3 used** |
|  | **Initial** | **Final** |  |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

**Reading with Distilled Water**

|  |  |  |  |
| --- | --- | --- | --- |
| **S. No** | **Volume of Distilled water (ml)** | **Burette Reading (ml)** | **Volume of AgNO3 used** |
|  | **Initial** | **Final** |  |
| **1.** |  |  |  |  |
| **2.** |  |  |  |  |
| **3.** |  |  |  |  |

|  |  |  |
| --- | --- | --- |
| **Result** | : | The chloride ion concentration in the given water sample is \_\_\_\_\_\_\_\_\_\_ ppm  |

**Precautions**

|  |  |
| --- | --- |
| 1. | The same volume of water sample and distilled water for blank correction was taken. |
| 2. | The same drops of indicator were taken in all experiments. |
| 3. | The pH of solution was maintained between 7-8. |

|  |
| --- |
| **Calculations** |

|  |  |  |
| --- | --- | --- |
| Actual Volume of AgNO3 used | = | Volume of AgNO3 – Blank Correction |
| N1V1 | =  | N2V2 |
| (AgNO3) |  | (Water Sample) |
| N2 | = | N1V1V2 |
| Strength of Cl- ion | = | N2 x 35.5 x 1000 ppm |

**EXPERIMENT No - TOTAL SOLIDS**

**AIM:** To determine the total solids present in water sample.

**GENERAL:** The term solid refers to the matter either filterable or in filterable that remains as residueafter evaporation and subsequent drying at a defined temperature. Different forms of solids are defined on the basis of method applied for their determination.

Residue after the evaporation and subsequent drying in oven at specific temperature, 103-105ºC of a known volume of a sample are total solids. The loss in weight on ignition of the same sample at 550°C (in which organic matter is converted to CO2 and H2 O) gives organic solids present in the sample.

**PROCEDURE:**

1. Take empty weight of beaker. (WI)
2. Take a known volume of a well mixed sample in the above beaker.
3. Evaporate the sample to dryness at 103°C for 24 hours.
4. Cool and weigh and record the reading (W2)

5. Keep the beaker for 15-20 min. in a muffle furnace maintained at 550 + 50°C.

6. Cool the beaker and record the final weight (W3).

**CALCULATIONS**:Total solids mg/L=(W2– W1) x 1000ml of sample Organic solids mg/L. = (W2 – W3) x 1000ml sample

**OBSERVATIONS:**

|  |  |  |
| --- | --- | --- |
| Weight of empty beaker | W1= ------------ | mg |
| Weight of beaker after evaporation at 103°C | W2 =------------ | mg |
| Weight of beaker after evaporation in | W3 = ------------ | mg |
| muffle furnace |  |  |

**RESULT:**

Total Solids = mg/l

 **EXPERIMENT-**

**CHEMICAL OXYGEN DEMAND**

**AIM:** To determine the chemical oxygen demand of given sample.

**THEORY:** Chemical oxygen demand (COD) test determines the oxygen required for chemical oxidationof organic matter with the help of strong chemical oxidant. The limitation of COD test is that it cannot differentiate between the biologically oxidizable and biologically inert material. COD determination has an advantage over BOD determination in that the result can be obtained in about 3-4 hours as compared to 5 days required for BOD test.

**PRINCIPLE:** The organic matter gets oxidized completely by K2Cr2O7in the presence of H2SO4toproduce CO2 + H2O. The excess K2Cr2O7 remaining after the reaction is titrated with Fe (NH4) 2 (SO4) 2. The dichromate consumed gives the O2 required for oxidation of organic matter.

**APPARATUS:**

1. Reflux apparatus consisting of a flat bottom 250 to 500 m1 capacity flask
2. Burner or hot plate with temperature regulator.

**REAGENTS**:

* 1. **Standard potassium dichromate 0.250 N**: Dissolve 12.259g by K2Cr2O7dried at

103°C for 24 hours in distilled water and dilute to 1000 ml. Add about 120 mg sulphamic Acid to take care of 6 mg/L NO2-N.

1. **Sulphuric Acid reagent:** Add 10 g of Ag2SO4to 1000 ml cone. H2SO4and keep overnight for dissolution.
2. **Standard ferrous ammonium sulphate 0.1 N**: Dissolve 39 g Fe(NH4.)2(SO4)26H2O inabout 400 ml distilled water. Add 20 ml cone. H2SO4 and dilute to 1000 ml.
3. **Ferroin indicator**: Dissolve 1.485 g of 1, 10 phenanthroline monohydrate and 695 mgFeSO4, 7H2O and dilute to 100 ml with distilled water.
4. **Mercuric Sulphate**: HgS04crystals, analytical grade.

**PROCEDURE:**

* 1. Place 0.4 g HgSO4 in a reflux flask.
1. Add 20 ml sample and mix well.
2. Add pumice stone or glass beads followed by 10 ml of standard K2Cr2O7.
3. Add slowly 30 ml H2SO4 containing Ag2SO4 mixing thoroughly. This slow addition along with swirling prevents fatty acids to escape out due to high temperature.
4. Connect the flask to condenser: Mix the contents before heating.
5. Reflux for a minimum of 2 hours cool and then wash down the condenser with distilled water.
6. Dilute for minimum of 150 ml, cool and titrate excess K2Cr2O7.with 0.1 N Ferry. Amm sulphate using ferroin indicator. Sharp colour change from blue green to wine red indicates end-point.
7. Reflux blank: in the same manner using distilled water instead of sample.
8. Calculate COD from the following formula:

**COD** mg/L =(a-b)x2N8000ml of sample

Where a = ml of Ferr. Amm. Sulphate for Blank, b= ml of Ferr. Amm. Sulphate for sample, N= normality of Ferr. Amm. Sulphate.

**OBSERVATIONS:**

|  |  |  |
| --- | --- | --- |
| S. No. | Burette Reading of Ferr. Amm. Sulphate | Remarks |
|  |  |  |  |  |
|  | Initial | Final | Difference |  |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  | . |
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|  |  |  |  |  |
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|  |  |  |  |  |
|  |  |  |  |  |

**RESULT:**

COD of the given sample = ------------------mg/l.