**EXPERIMENT NO. 03**

**Determination of Chemical Oxygen Demand [COD]**

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| **CO - 4** | Analyze the quality of water and suggest suitable methods of treatment. |

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| **AIM** | **:** | To determine the **Chemical Oxygen Demand (COD)** in a waste water sample. |
| **PRINCIPLE** | **:** | ***Chemical Oxygen Demand (COD),*** is a measurable parameter to determine the total amount of organic matter in streams, industrial wastes and sewage.  COD is an important and quickly measured parameter for stream, industrial waste studies and control of waste treatment plants. It is an index to the water-oxygen demand of substances in water.  The chemical oxygen required for determining the amount of organic matter in water is derived from K2Cr2O7. Potassium dichromate is a strong chemical oxidising agent capable of oxidising all the organic matter present in water by chemical oxidation.  COD is defined as ***the amount of oxygen required by organic matter in a sample of water for its oxidation by a strong oxidising agent like K2Cr2O7 in the presence of catalysts like HgSO4 or Ag2SO4.***  It is expressed as the ppm (mg/litre) of oxygen taken from a K2Cr2O7 solution in two hours. The basic reactions that occur during the COD determination can be represented by the chemical equations shown on the opposite page.  The COD of a system varies with the water composition, temperature and contact time. Boiling in a mixture of chromic acid and sulphuric acid can destroy most types of organic matter. |
| **REACTIONS** | **:** | CxHyOz + (x + y/4 - z/2)O2 🡪 CO2 + y/2H2O  Cr2O72- + 14 H+ + 6e- 🡪 2Cr3+ + 7H2O  6Fe2+ 🡪 6Fe3+ + 6e-  Cr2O72- + 14 H+ + 6Fe2+ 🡪 2Cr3+ + 7H2O + 6Fe3+ |
| **REAGENTS**  **REQUIRED** | **:** | 1. Standard 0.1N K2Cr2O7 solution 2. Standard 0.1N Fe(NH4)2(SO4).6H2O solution (FAS) 3. Ferroin indicator (1,10-phenanthroline ferrous iron complex indicator) 4. Sulphuric acid-Silver sulphate (H2SO4-Ag2SO4) solution 5. HgSO4 (AR) Grade |
| **APPARATUS REQUIRED** | **:** | Burette, flat bottom flask (250 ml), burette stand, water bath, Measuring cylinder (25 ml) |
| **PROCEDURE** | **:** | **Part I: Determination of Blank Titration**  Pipette out 10 ml of 0.1N K2Cr2O7 solution, add 15 ml H2SO4 & titrate against 0.1N ferrous ammonium sulphate (FAS) solution using ferroin indicator. Note the blank reading **(‘a’ ml)**  **Part II: Determination of COD**   1. Place 0.4 g of HgSO4 and 20 ml of water sample into a flat bottom flask with a reflux condenser. 2. Add 10 ml of 0.1N K2Cr2O7 solution and drop some pumice stones. 3. Slowly add 15 ml of sulphuric acid-silver sulphate (H2SO4-Ag2SO4) solution with shaking. 4. Connect the reflux condenser and reflux the same to get 10 ml of concentrate. 5. Take 10 ml of concentrated sample in a conical flask. Shake the flask well several times to make the solution homogeneous. 6. Add few drops of ferroin indicator. 7. Titrate the unreacted K2Cr2O7 with 0.1N FAS solution in the conical flask changes from ***green to wine red.*** 8. Repeat the titration till you get two constant burette readings. 9. Note down this burette reading as **(‘b’ ml)** i.e. the amount of K2Cr2O7 not consumed. 10. **[a-b]** ml gives the amount of K2Cr2O7 consumed by the 20 ml of water sample. |
| **OBSERVATION TABLE** | **:** | **Part I: Determination of Blank Titration**   |  |  | | --- | --- | | **Burette :**  0.1N Ferrous  Ammonium sulphate solution | **Conical flask :** 10 ml of 0.1N  K2Cr2O7 solution +  15 ml of H2SO4 | | **Indicator :** Ferroin | **End point :** Blue green to wine red |   **Part II: Determination of COD**   |  |  | | --- | --- | | **Burette :** 0.1N Ferrous  Ammonium sulphate solution | **Conical flask :** 10 ml of  Concentrated solution +  15 ml of H2SO4 | | **Indicator :** Ferroin | **End point :** Blue green to wine red | |
| **CALCULATIONS** | **:** | 1. Normality of ferrous ammonium sulphate = N = 0.1N 2. Volume of original sample taken = 20 ml   [a-b] X N X 8 X 1000   1. mg/litre of COD = --------------------------------------------   Volume of original sample taken (ml)   1. The amount of K2Cr2O7 consumed by the sample = [a-b] = ----ml |
| **RESULT** | **:** | The COD of the given sample = -------------------mg/litre. |



**EXPERIMENT NO. :**  \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ **DATE :**  \_\_\_\_\_\_\_\_\_\_\_\_\_

**DIVISION/ BATCH :**  \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ **SAP ID :** \_\_\_\_\_\_\_\_\_\_\_\_

**COURSE :\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ COURSE CODE :\_\_\_\_\_\_\_\_\_\_**

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**COURSE OUTCOME : \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

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**AIM :** To determine the chemical oxygen demand (COD) in a waste water sample.

**REAGENTS :** \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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**REACTIONS :** \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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**PROCEDURE :** \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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**OBSERVATIONS :**

**TABLE 1 : DETERMINATION OF BLANK TITRATION**

|  |  |
| --- | --- |
| **Burette :**  0.1N Ferrous  Ammonium sulphate solution | **Conical flask :** 10 ml of 0.1N  K2Cr2O7 solution + 15 ml of H2SO4 |
| **Indicator :** Ferroin | **End point :** Blue green to wine red |

|  |  |  |  |
| --- | --- | --- | --- |
| Reading (ml) | Trial  1 | Trial  2 | Trial  3 |
| Initial  Reading |  |  |  |
| Final  Reading |  |  |  |
| Volume of \_\_\_\_\_\_\_\_\_\_  Solution |  |  |  |

**CBR 1 (a) = \_\_\_\_\_\_\_\_\_\_ml**

**TABLE 2 : DETERMINATION OF COD**

|  |  |
| --- | --- |
| **Burette :**  0.1N Ferrous  Ammonium sulphate solution | **Conical flask :** 10 ml of Concentrated solution + 15 ml of H2SO4 |
| **Indicator :** Ferroin | **End point :** Blue green to wine red |

|  |  |  |  |
| --- | --- | --- | --- |
| Reading (ml) | Trial  1 | Trial  2 | Trial  3 |
| Initial  Reading |  |  |  |
| Final  Reading |  |  |  |
| Volume of \_\_\_\_\_\_\_\_\_\_  Solution |  |  |  |

**CBR 1 (b) = \_\_\_\_\_\_\_\_\_\_ml**

**CALCULATIONS :** \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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**RESULT :** The COD of the given sample = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_mg/L

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| **D.J.S.C.E. (Chemistry)** | | |
| **Journal** | | |
| **(Lab Ethics)** | **5** |  |
| **(Performance)** | **5** |  |
| **(Documentation)** | **5** |  |
| **(Knowledge)** | **5** |  |
| **(Punctuality)** | **5** |  |
| **Total** | **25** |  |

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