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Expt. 4 → Estimation of Chemical Oxygen Demand as an Indicator of Organic Pollutants.

Aim

To estimate the amount of organic content present in a given effluent sample.

Principle.

Chemical oxygen demand (COD) is defined as the amount of a specified oxidant that reacts with the sample under controlled conditions. The quantity of oxidant consumed is expressed in terms of its oxygen equivalence. COD is a defined test; the extent of sample oxidation can be affected by digestion time, reagent strength, and sample COD concentration. COD often is used as a measurement of pollutants in wastewater and natural waters. The organic matter present in sample gets oxidized completely by potassium dichromate in presence of sulphuric acid, silver sulphate & mercury sulphate to produce carbon dioxide & water. The sample is refluxed with a known amount of potassium dichromate in the sulphuric acid medium and the excess potassium dichromate is determined by titration against ferrous

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ammonium sulphate, using ferroin as an indicator. The dichromate consumed by the sample is equivalent to the amount of oxygen required to oxidize the organic matter.

$$\text{COD as mg } O_2/L = \frac{(A-B) \times M \times 8000}{\text{mL sample}}$$

where,

A = mL FAS used for blank
B = mL FAS used for sample,
M = molarity of FAS, and
8000 = milli equivalent weight of oxygen x 1000 mL/L

Apparatus and Reagents

Round bottom flask, water condenser, burette, pipette, heating mattle.

- 0.25N $K_2Cr_2O_7$ Reagent: Dissolve 12.259 g $K_2Cr_2O_7$, primary standard grade, previously dried at 150°C for 2h, in distilled water and dilute to 1000 mL.

- Sulfuric acid reagent: Add Ag_2SO_4, reagent or technical grade, crystals or powder, to conc H_2SO_4 at the rate of 5.5 g Ag_2SO_4 / kg H_2SO_4. Let stand 1 to 2 d to dissolve. Mix.

- Ferroin indicator solution: Dissolve 1.485 g 1, 10- phenanthroline monohydrate and 695 mg ~~FeSAH~~ $FeSO_4 \cdot 7H_2O$ in distilled water & dilute to 100 mL.

- Standard ferrous ammonium sulfate (FAS) titrant, 0.25 M: Dissolve 98 g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water. Add 20 mL conc. H_2SO_4, cool, and dilute to 1000 mL.

Methodology

1. Take two COD digestor vials (one for the sample and one for the blank).

2. Add 1 mL of the sample (domestic wastewater or industrial effluent) to the sample vial and make up to 20 mL with distilled water.

3. Add 20 mL of distilled water to the blank vial.

4. Add 10mL of potassium dichromate reagent - digestion solution to each of the two vials.

5. Carefully add 30 mL of sulfuric acid reagent - catalyst solution in the same manner.

6. Place the COD vials into a COD digestor & the samples were allowed for digestion at 150°C for 2 hours.

7. After 2 hours, remove the vials & allow it to cool to the room temperature.

8. Fill the burette with the ferrous ammonium sulphate solution (0.1N), adjust to zero and fix the burette to the stand.

9. Transfer the contents of the blank vial to conical flask, add 2-3 drops of ferroin indicator. The solution becomes bluish green in colour.

10. Titrate it with the ferrous ammonium sulphate taken in the burette till the appearance of the reddish brown color (brick red). Repeat the same titration for sample & note down the

Calculations

Volume of FAS consumed for blank = 22.4

Volume of FAS consumed for sample = 21.2

$$COD = \frac{(\text{Blank} - \text{sample}) \times \text{Molarity of FAS} \times 8 \times 1000}{\text{Volume of sample (mL)}}$$

Here,

Molarity of FAS = 0.25 of FAS

Volume of sample (mL) = 1 mL

Therefore,

$$COD = \frac{(22.4 - 21.2) \times 0.25 \text{ of FAS} \times 8 \times 1000}{1 \text{ mL}}$$

$$\boxed{COD = 960 \text{ mg/L}}$$

end point value.

Result and Interpretation

$$COD = \frac{(22.4 - 21.2) \times 0.25 \text{ of FAS} \times 8 \times 1000}{1 \text{ mL}}$$

$$COD = 960 \text{ mg/L}$$

The chemical oxygen demand of the given sample is 960 mg/L. It indicates the level of organic pollutants in the given wastewater.