Experiment No. : 7

Determination of Biochemical Oxygen Demand (BOD) of given water sample.

Apparatus required :

1) Incubator 2) Burette 3) Pipette

4) Conical flask 5) BOD bottles,

Chemical required:

1) Manganese (II) sulphate
2) Sodium azide
3) Potassium iodide
4) Sodium hydroxide
5) Starch, potassium fluoride
6) Sodium thiosulphate

Theory :

BOD is the amount of oxygen utilized by microorganisms in biological process that break down organic matter in water. It is a measure of organic pollutant load. Greater the oxidizable organic matter in water, the greater will be the BOD, Thus, the strength of waste water is expressed in terms of BOD. BOD, is important to know the amount of organic matter present in the waste and that the quantity of oxygen required for its stabilization. The BOD values are thus very useful in process designed to measure treatment plant efficiency and operation.

The origin of organic compounds are human excreta, vegetables, animal waste and industrial waste. The amount of organic matter present in water shows the amount of biological oxygen demand. Large amount of sewage introduced into water body do not get diluted sufficiently. Microorganisms use up all the oxygen in the water to oxidize organic matter as a result of which water becames useless for drinking and for other purposes.

The complete degradation of organic matter may take 20 to 30 days. Simple organic compounds like glucose are almost completely oxidized in 5 days while domestic sewage is oxidized to 65% in this period. Complex organic compounds get oxidized only upto 40% in the same period. The 20 to 30 days period is of least significance in practice. Therefore, the BOD test has been developed for 5 days at $20^{\circ}C$. BOD in general gives a qualitative index of organic substance which are degraded quickly in a short period of time.

The BOD value is measured by measuring the difference of dissolved oxygen (DO) of the same water sample in 5 days. DO present in water is calculated by using the formula.

$$\text{Dissolved Oxygen (mg/l)} = \frac{A.N.8}{S - C} \times 1000$$

Where, A = ml of 0.025 N thiosulphate solution consumed

N = Normality of standard thiosulphate solution

C = Total volume of potassium Manganese (II) sulphate, alkali iodide azide, potassium fluoride

S = Volume of water sample

Procedure:

1) Water sample was collected in two BOD bottles.

2) One BOD bottle was incubated in BOD incubator for 5 days at $20^{\circ}C$ while DO of another BOD was determined on the 1st day.

3) After 5 days, another BOD bottle was removed from the incubator and DO was determined.

4) Difference in DO was calculated, which gave the measure of BOD.

Observation Table

S. No.	Volume of water Sample (ml), S	Volume of thiosulphate solution (ml), A	Total volume of $MgSO_4$, KF and alkali iodide azide solution (ml), C
1.			
2.			
3.			

Calculation:

BOD of water is calculated by using the formula,

$$\text{BOD (mg/l)} = \frac{DO_1 - DO_5}{P}$$

Where, DO_1 = DO in water sample in 1st day.

DO_5 = DO in water sample after 5 days.

P = decimal volumetric fraction

$$\text{Dissolved Oxygen (mg/l)} = \frac{A.N.8}{S - C} \times 1000$$

Where, A = ml of 0.025 N thiosulphate solution consumed

N = Normality of standard thiosulphate solution

C = Total volume of potassium manganese (II) sulphate solution, alkali iodide azide solution, potassium fluoride solution

S = Volume of water sample

Results:

Experiment No. : 8

Determination of Chemical Oxygen Demand (COD) of given water sample.

Apparatus required:

1) Reflux condenser 2) Conical flask 3) 4) Pipette

5) Burette 6) Round bottom flask

Chemical required:

1) Silver sulphate
2) Conc. sulphuric acid
3) Potassium dichromate
4) Ammonium iron (II) sulphate (Mohr's salt),
5) Ferroin indicator
6) Mercury (II) sulphate.

Theory:

The amount of oxygen required to oxidize all the organic material in a sample of water is known as Chemical Oxygen Demand (C.O.D.) and is expressed in terms of milligrams of oxygen required per litre of water, mg/l. In COD test, a strong chemical oxidising agent is used to oxidise the organics rather than relying on micro-organisms to do the job as in BOD. The COD test is much quicker than BOD test, taking only a matter of hours. However, it does not distinguist between the oxygen demand that will actually be felt in natural environment due to biodegradation, and chemical oxidation of inorganic matter.

Many types of organic matters are oxidized by boiling them with a mixture of potassium dichromate and sulfuric acid solution. Hence, a sample is refluxed in strong acid solution with a known excess of potassium dichromate.

$$Cr_2O_7^{-} + 8H^{+} \rightarrow 2Cr^{+++} + 4H_2O + 3[O]$$
$$n(CH_2O) + n[O] \rightarrow n(CO_2) + n(H_2O)$$

After digestion, the remaining unreacted (unreduced) potassium dichromate is titrated with ferrous ammonium sulphate to determine the amount of potassium dichromate consumed. The oxidizable organic matter is calculated in terms of oxygen equivalent. The analysis of the water sample is carried out parallelly with a blank determination on distilled water.

Procedure:

Transfer 50 ml water sample in 500 ml round bottom flask that can be fitted with a water condenser for refluxing. Add 1g of mercury sulphate*, followed by 80 ml of silver sulphate-sulphuric acid solution[1]. Then add 10 ml of standard potassium dichromate solution[2], the flask with the reflux condenser and boil the mixture for 15 minutes. On cooling, rinse the inside of condenser with 50 ml of water. Add ferrion indicator and tritrate with 0.025 M Mohr's salt (ammonium iron (II) sulphate solution)[3]. Ferrion gives a colour change from blue-green to red brown at the end point. Call this titration A ml. Repeat the titration for the blank, B ml.

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* *This method gives high results with the samples possessing a high chloride content due to the reaction between the mercury (II) sulphate and chloride ions. In these cases the problem can be overcome by using chromium (III) potassium sulphate. Cr(III)K(SO4)2.12H2O*

[1] 5g of silver sulphate in 500mL of concentrate sulphuric acid.

[2] 1.225g of potassium dichromate in 500ml. of deionized water.

Observation Table

S. No.	Vol. of water sample (ml)	Vol. of 0.025M ammonium iron (II) sulphate for water sample, A (ml.)	Vol. of pure water (ml)	Vol. of 0.025M ammonium iron (II) sulphate for pure water, B (ml.)

Calculation:

The difference between two values (i.e., A and B) is the amount of potassium dichromate used up in the oxidation. The C. O. D. is calculated from the relationship

$$\text{C. O. D.} = (\text{B} \quad \text{A}) \times 0.025 \times 8 \times 20 \text{ mg / l}$$

Results :

[3]Dissolve 3.92g of ammonium iron(II) sulphate hexahydrate in 150mL of water and add 2.5mL of concentrated sulphuric acid. Dilute the solution to 500mL in a volumetric flask.