B.Sc. Third Year V Semester

ANALYTICAL CHEMISTRY

Paper XIII [Applied Analytical Chemistry-I]

Analysis of Industrial Effluents

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"INDUSTRIAL EFFLUENTS"

* **Definations:** (i) Industrial effluent:
The "Waste water coming out of various industries is called as Industrial effluent"

(ii) Effluent :—
"The waste water coming out of various sources such as sewage, industries etc. is called as effluent"

Introduction:-

The Industrial effluent should not contain heavy metals. If a industrial effluent containing heavy metals like Hg, Cd, Pb, Zn, As and Bi is thrown into the nearby river the rivers water gets not only polluted but also gets poisoned. A small quantity of heavy metal upto 1 mg/L of water makes the water unsuitable for drinking as it is highly toxic and causes dreaded diseases in humans and cattle drinking such water. Such low concentration of heavy metal in river water is fatal (causes death) if used for drinking purpose.

Hence it is necessary to monitor the level of heavy metal in the industrial effluent before it is discharged into the nearby river.

Therefore the analysis of industrial effluent with reference to its heavy metal concentration is a compulsory step before the industrial effluent is discharged into the nearby river.

The level of heavy metals like Hg, Pb, Cd, Cr, Zn, & As in river water should not be above the levels specified by Indian Bearau of Standards.

If the heavy metal concentration in river water is more than the level given by Indian Bearau of Standards then the water is said to be Unfit for drinking purpose.

At the same time if an industry discharges its industrial effluent containing heavy metals into the nearby river, then it is treated as violation of the norms of the Central Pollution control Board (CPCB) or Maharashtra Pollution control Board (MPCB).

* Analysis Of Industrial Effluents

Quantitative Analysis of Toxic metals like Hg, Zn, Cd, Pb, and As in Industrial Effluents:

Before the analysis of Industrial effluent for its heavy metal content is carried out, a preliminary treatment of the sample is done:

PRELIMINARY TREATMENT OF SAMPLE:

The industrial waste water contains organic matter to a greater extent.

Depending upon the amount of organic matter, different reagents are used for digestion of water sample in the preliminary treatment.

Following different digestion reagents are used for the destruction of organic matter in water:

(a) For water with least org. matter	Mixt of HNO_3 & H_2SO_4 is used for digestion
(b) For water with moderate org. matter	Mixt of HNO_3 & $HClO_4$ is used for digestion.
(c) For water with very high org. matter	Only conc. $HClO_4$ is used for digestion.

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The volume of the sample to be digested in preliminary treatment depends on the concentration of toxic heavy metal to be determined. The volume of industrial effluent to be taken for the different concentration of heavy metal content is as follows :-

Volume (ml) of effluent to be Taken	Concentration range of heavy metal present mg/litre
1000 ml	0.01 — 1.0 mg/L
100 ml	1.0 — 10.0 mg/L
10 ml	10.0 — 100.0 mg/L
1 ml	100.0 — 1000.0 mg/L

PRETREATMENT / DIGESTION OF EFFLUENT WITH $HClO_4$

A definite volume of industrial effluent as mentioned above according to conc. of heavy metal is taken in a China dish. Add. Sufficient conc. $HClO_4$ to it. Digest the sample till a clear solution is obtained by heating the sample strongly. The clear solution is diluted to a definite volume in Standard flask :

The diluted solution of industrial waste after prefreament / Digestion is used for estimation Toxic heavy metals in the industrial effluent.

1. Determination of Mercury [Hg] in industrial effluent :-
(Colorimetric method)

PRINCIPLE :- The method is based on the fact that, Mercury-Hg in industrial effluent reacts with the reagent Dithizone in Carbon tetrachloride in presence of a masking agent for interfering ions at pH 2 to form a Red coloured complex of Mercury-Hg-dithizonate as follows :

$$Hg^{++} + (HDz)_n \longrightarrow HgDz_n + nH^+$$

Hg dithizone
Coloured complex
(Red)

The colour Intensity of Red Coloured Hg-Dithizonate complex or O.D of the complex is directly proportional to the concentration of Hg mg/L in industrial effluent.

The coloured complex shows a maximum absorbance at 520-540 nm wavelength.

Hence, by measuring the absorbance / O.D. of the Red coloured complex at 520 nm in a colorimeter against the Standard coloured complex with known concentration of Hg mg/L The concentration of Hg in the industrial effluent can be determined.

The interfering metal ions must be removed. Before the estimation.

Method :-

Part I :- Preparation of Calibration Curve / Standard Curve :

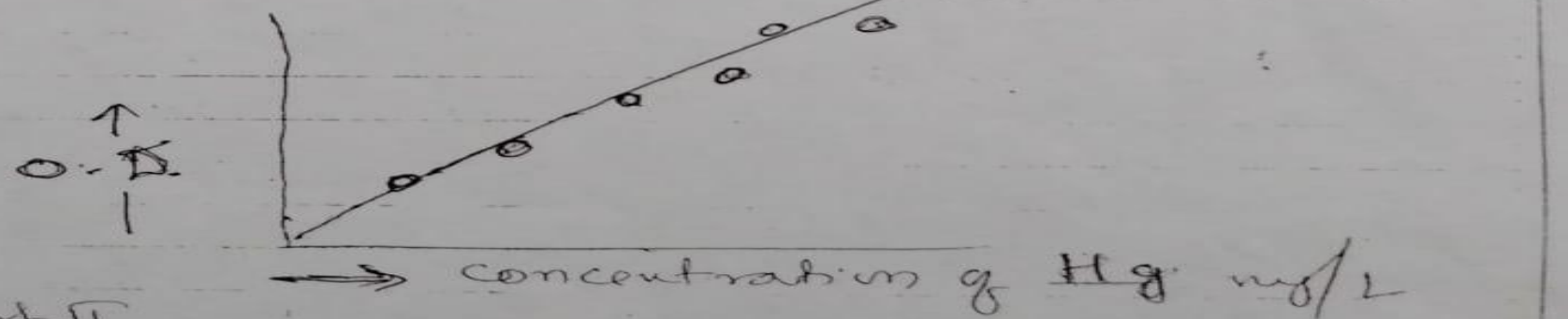
A series of Standard Solutions of Hg-salt with varying and known concentration of Hg in mg/L or µg/L are prepared. The pH of each solution is adjusted by to 2 either by adding Tartaric Acid or by adding Amm. Hydroxide/NH_4OH. Then add Thiocyanate / Thiosulphate / EDTA to each Standard Solution. Extract Hg in each standard

Saturation into CCl_4 layer is the form of Red colour Hg-dithizonate complex by shaking each standard solution of Hg salt with reagent "dithizone in CCl_4".

Measure the O.D. of all Red coloured Hg-Dithizonate complexes at 520 nm nm W.L. in a colorimeter.

Then prepare a calibration curve by plotting O.D. of series of Standard solutions of Hg V/s concentration of Hg in series of Standard Solutions.

A straight line curve known as Calibration curve is obtained.



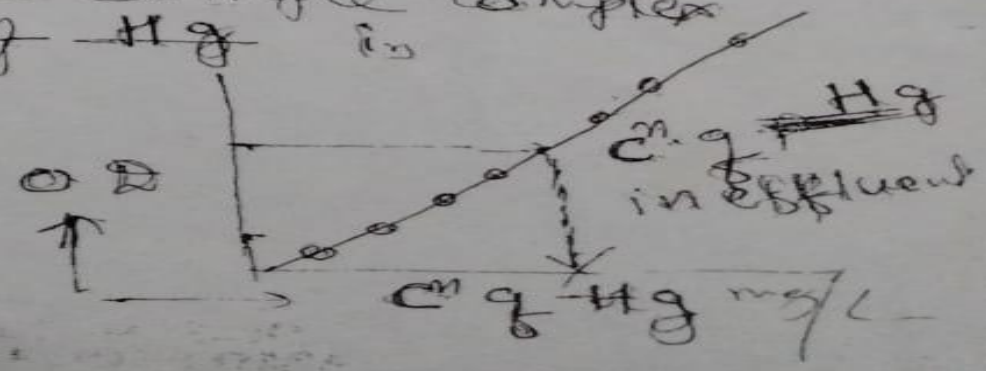
Part II

Preparation of Sample complex:

A definite aliquot of the diluted

Solution of industrial effluent containing Hg after digestion with $HClO_4$ is taken in a standard flask.
The pH of the solution is adjusted to 2 either by adding Tartaric Acid or NH_4OH i.e by adding Ammi Hydroxide
Then add thiocyanate / thiosulphate / EDTA to the sample water solution.
Extract Hg in the sample solution into CCl_4 layer in the form of Red coloured Hg-diphenylthiocarbazonate complex, by shaking the sample solution with the reagent diphenylthiocarbazone in CCl_4

Measure the O.D. of sample coloured complex at 520 nm in a colorimeter.
Finally from the calibration curve, and O.D. of sample complex the concentration of Hg in industrial effluent is determined by extrapolation of the calibration curve.



* 1. Determination of Zinc [Zn] in industrial effluent :-
(Colorimetric method).

PRINCIPLE :- The method is based on the fact that, Zinc - Zn in industrial effluent reacts with the reagent Dithiozone in Carbon tetrachloride in presence of bis-(2-hydroxy ethyl) Dithiocarbamate CN at pH 7 to form a Red pink coloured complex of Zinc - Zn - di thiozonate as follows :

$$Zn^{+2} + (H\,Dz)_n \longrightarrow Zn\,Dzn + n\,H^+$$

Zn - dithiozone
coloured complex.
Pink coloured

The colour intensity of Zn - Dithiozonate Pink Red complex or O.D. of the complex is directly proportional to the concentration of Zn^{+2} in industrial effluent.

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The coloured complex shows a maximum absorbance at 540 nm wavelength.

Hence by measuring the absorbance / O.D of the pink coloured complex at 535 nm in a colorimeter against the Standard coloured complex with known concentration of Zn mg/L the concentration of Zn in the industrial effluent can be determined.

The interfering metal ions must be removed before the estimation.

Method:—

Part I: Preparation of Calibration Curve / Standard Curve:

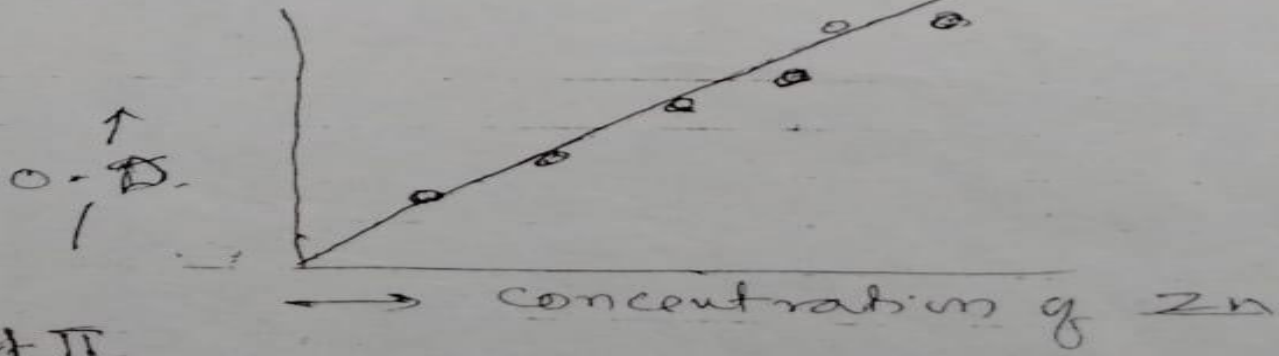
A series of Standard solutions of Zn salt with varying and known concentration of Zn in mg/L or µg/L are prepared. The pH of each solution is adjusted to 7 (neutral) either by adding Tartaric Acid or by adding NH_4OH. Then add bis(2-hydroxy ethyl) dithiocarbamate to each Standard solution. Extract Zn in each standard

Solution into CCl_4 layer in the form of Pink Red coloured dithizonate complex by shaking each Standard solution of Zn salt with reagent "dithizone in CCl_4".

Measure the O.D. of all pink coloured Zn dithizonate complexes at 535 nm nm W.L in a colorimeter.

Then prepare a calibration curve by plotting O.D. of series of Standard solutions of Zn V/s concentration of Zn in series of Standard solutions.

A straight line curve known as calibration curve is obtained.



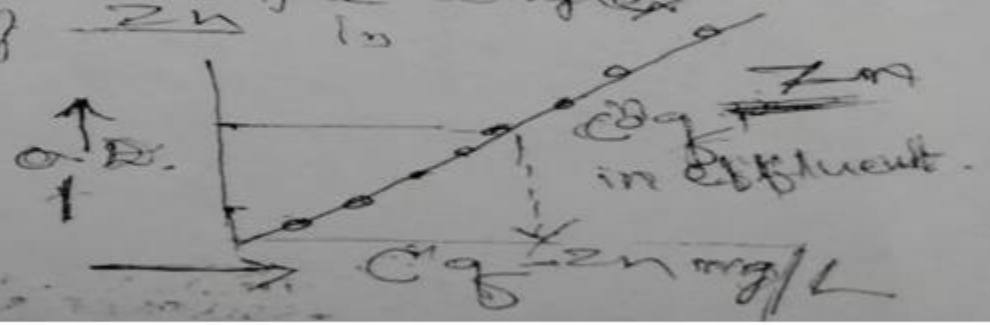
Part II

Preparation of Sample complex:

A definite aliquot of the diluted

Solution of industrial effluent containing Zn after digestion with $HClO_4$ is taken in a Standard flask.
The pH of the solution is adjusted to 7 (neutral) either by adding Tartaric acid or ____ by adding NH_4OH.
Then add bis-(2-hydroxy ethyl) dithiocarbamyl & CA to the sample water solution.
Extract Zn in the sample solution into CCl_4 layer in the form of pink coloured Zn dithizonate complex, by shaking the sample solution with the Reagent Dithizone in CCl_4.

Measure the O.D. of sample coloured complex at 535 nm in a colorimeter.
Finally from the calibration curve, and O.D. of sample complex the concentration of Zn in industrial effluent is determined by extrapolation of the calibration curve.



.....SUGGESTIONS ?

Notes prepared by, Dr. Subhash Lonkar

Thank you.....