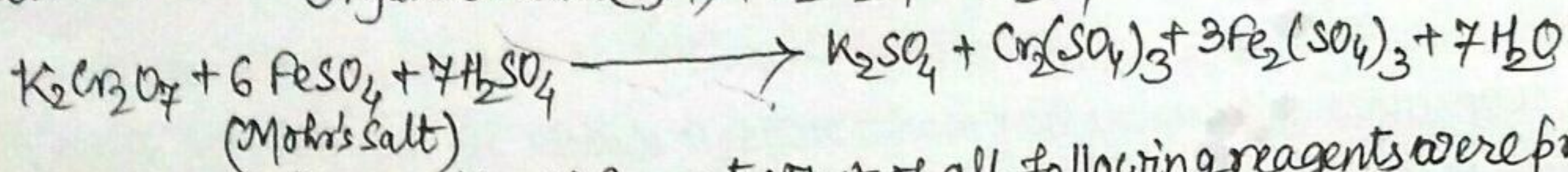


→ What is COD?

COD is Chemical oxygen demand. It is indirectly measure of the amount of organic matter present in waste water, which has adverse effect (decrease concentration of oxygen) on aquatic system. The amount of oxygen required to completely oxidize the organic matter present in the water sample is called chemical oxygen demand (COD).

Determination of COD of given water sample:

Principle: The oxygen matter present in water sample get oxidized by a strong oxidizing agent ( $K_2Cr_2O_7$ ) in presence of Conc  $H_2SO_4$ ,  $HgSO_4$  &  $Ag_2SO_4$  to  $CO_2$  &  $H_2O$ . The water sample is refluxed with a known amount of  $K_2Cr_2O_7$  in acidic medium ( $H_2SO_4$ ). The excess (unreacted)  $K_2Cr_2O_7$  is determined by titration against standard Mohr's salt (Ferrous ammonium sulphate) solution using ferroin as an indicator. The  $K_2Cr_2O_7$  consumed by the water sample is equivalent to the amount of  $O_2$  required to oxidise the organic matter, gives COD value.



Procedure: 1. Preparation of Reagents: First of all following reagents were prepared:

- (i)  $N/4$   $K_2Cr_2O_7$  solution: 9.065 gm potassium dichromate ( $K_2Cr_2O_7$ ) was accurately weighed and transferred into a 250 ml measuring flask containing 100 ml distilled water. The flask was stoppered and shaken thoroughly to dissolve  $K_2Cr_2O_7$ . The solution made upto 250 ml mark by adding more distilled water.
- (ii)  $N/10$  ferrous ammonium sulphate (Mohr's salt) solution: 9.802 gm of Mohr's salt was weighed accurately and transferred into a 250 ml measuring flask containing 100 ml distilled water and 20 ml dilute  $H_2SO_4$ . The flask was stopped and shaken thoroughly to dissolve it. The solution made upto 250 ml flask by adding more distilled water <sup>mark in</sup>.
- (iii) Ferroin indicator: 1.485 gm of 1,10-phenanthroline and 0.695 gm of ferrous sulphate ( $FeSO_4$ ) were dissolved in 100 ml of distilled water.

2. 50 ml water sample was taken in a 500 ml round bottom flask fitted with a reflux condenser. About 1 gm  $HgSO_4$  was added into it and mixture was shaken thoroughly. Now, 25 ml  $N/4$   $K_2Cr_2O_7$  solution and 75 ml  $H_2SO_4$  (conc.) were added to it. A small piece of porcelain chip was added in the flask and the mixture was refluxed for two hours.

3. The refluxed mass (reaction mixture) was cooled and inside portion of condenser was washed with distilled water. It was diluted by adding 150 ml distilled water.

4. Standard Mohr's salt solution was filled in a clean and rinsed burette, and initial burette reading noted.

5. 25 ml of diluted reaction mixture was pipetted out in a conical flask. 2-3 drops of ferroin indicator were added into it.

6. Now, Mohr's salt solution was added slowly from burette into conical flask till blue-green colour changed to reddish brown. The final burette reading was noted.

7. The titration process was repeated in order to get two concordant readings.

8. The blank titration was performed similarly using 25 ml distilled water.

Experimental results & Calculation: Let volume of Mohr's salt solution used with residual  $K_2Cr_2O_7$  solution after oxidising organic matter =  $V_1$ ; volume of Mohr's salt solution used with total  $K_2Cr_2O_7$  solution (Blank titration) =  $V_2$ , volume of  $K_2Cr_2O_7$  solution used in Oxidation of organic matter, i.e., satisfying COD =  $(V_2 - V_1)$  ml.

(2)

us, Volume of  $N/10$  Mohr's salt solution  $\equiv$  Volume of  $N/2$   $K_2Cr_2O_7$  solution used in satisfying COD  $= (V_2 - V_1)$  ml

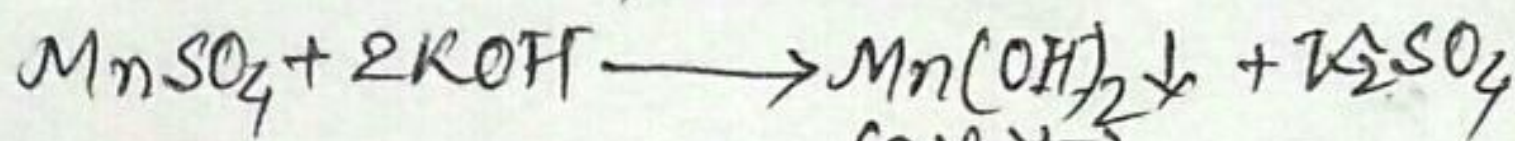
Normality of water sample  $= \frac{(V_2 - V_1)}{25 \times 10}$

$\therefore$  COD of water sample  $= \frac{(V_2 - V_1) \times 8}{25 \times 10}$  gm/litre  $= \frac{(V_2 - V_1) \times 8 \times 1000}{25 \times 10} = (V_2 - V_1) \times 32$  mg/litre

What is DO? DO is dissolved oxygen in a water sample. It is one of the essential requirement for the survival of aquatic life (animal or plant). During pollution of water, the DO concentration falls, which is quite harmful for the aquatic life.

Determination of DO in a water sample:

Principle: DO (dissolved oxygen) can be estimated by Winkler method easily. It is based on the reaction of manganous sulphate ( $MnSO_4$ ) with alkaline KI in water sample to produce white precipitate of  $Mn(OH)_2$ . This indicates that there is no dissolved oxygen in the water.

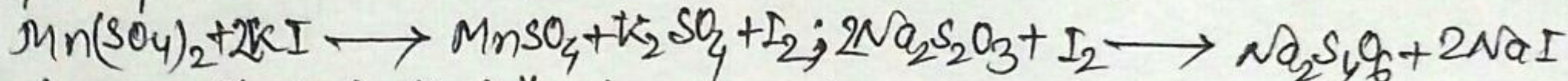


If dissolved oxygen in water, it reacts with  $Mn(OH)_2$  to give brown of manganic basic oxide.  $Mn(OH)_2 + O_2 \longrightarrow 2MnO(OH) \downarrow$

$Conc. H_2SO_4$  is added to dissolve brown precipitate.  $MnO(OH)_2 + H_2SO_4 \longrightarrow Mn(SO_4)_2 + 3H_2O$

Manganic sulphate immediately reacts with KI to liberate  $I_2$ . (Manganic sulphate)

The amount of  $I_2$  liberated is equivalent to the amount of oxygen present in the water sample. The amount of  $I_2$  is estimated by titrating with standard  $N/20$   $Na_2S_2O_3$  sol.



Procedure: 1. First of all following reagents were prepared:

(i) Manganous sulphate solution: 12 gm of manganous sulphate crystals ( $MnSO_4 \cdot 4H_2O$ ) were dissolved in 250 ml of distilled water.

(ii) Alkaline KI solution: 125 gm of KOH and 37.5 gm of KI were dissolved in 250 ml of distilled water. sodium thio sulphate crystals

(iii)  $N/40$   $Na_2S_2O_3$  solution: 6.2 gm of  $Na_2S_2O_3 \cdot 5H_2O$  weighed and then dissolved in one litre of distilled water. The solution was kept overnight to make homogeneous. Then, this solution was standardized by titrating against standard  $K_2Cr_2O_7$  solution.

2. 150 ml water sample was taken in a 250 ml conical flask. 1 ml manganous sulphate solution was added to it. Now, 1 ml alkaline KI solution was added. The flask was stoppered and shaken thoroughly. It was kept aside for few minutes and precipitate was allowed to settle. Now, 1-2 ml conc  $H_2SO_4$  was added into the flask, stoppered and then shaken thoroughly to dissolve the precipitate. The flask was kept at room temp. for five minutes.

3. 50 ml of the reaction mixture was transferred in a 250 ml conical flask and titrated against standard  $Na_2S_2O_3$  solution. When the colour of the solution became slightly yellow, 1 ml of freshly prepared starch solution was added. The solution turned blue. The titration process continued until the blue colour became colourless. The titration process repeated in order to get at least two concordant readings.

Experimental results & Calculation: Volume of water sample taken ( $V$ ) = 150 ml  
Volume of water sample taken for titration ( $V_1$ ) = 50 ml

(3)

Volume of  $MnSO_4$  solution & KI (alkaline) solution added ( $V_2$ ) = 1+1 = 2 ml

Strength of  $Na_2S_2O_3$  solution ( $N_1$ ) =  $N/40$  (f = 1.02)

Volume of  $Na_2S_2O_3$  solution consumed when whole water sample <sup>( $V$  ml)</sup> is used =  $V_3$  ml (let)

" " " part of water sample <sup>( $V_1$  ml)</sup> =  $V_4$  ml

(I) When whole water sample is used, DO (dissolved oxygen) =  $\frac{V_3 \times N_1}{V \times V_2} \times 8$  gm/litre

$$= \frac{V_3 \times \frac{1}{40} \times 1.02 \times 8}{150 \times 2} \text{ gm/litre} = \frac{V_3 \times 1.02}{1500} \text{ gm/litre} = V_3 \times 0.9 \text{ mg/litre}$$

(II) When part of water sample used;

$$\text{Dissolved oxygen (DO)} = \frac{V_4 \times N_1}{V_1 \times (V - V_2)} \times 8 \text{ gm/litre}$$

$$= \frac{V_4 \times \frac{1}{40} \times 1.02 \times 8}{50 \times (150 - 2)} \text{ gm/litre} = \frac{306 \times V_4}{74} \text{ mg/litre}$$

⇒ What is BOD?

All naturally occurring organic compounds/matter can be degraded by microbial activity in the presence of oxygen ( $O_2$ ). Biochemical oxygen demand (BOD) is measure of the oxygen required by microbes to oxidize the organic matter/compound present in water sample. Thus, the <sup>amount of</sup> oxygen required by microbes to oxidize organic matter/compound present in a sample of water is called BOD.

Determination of BOD in a water sample: Principle: BOD (Biochemical oxygen demand) is the amount of oxygen required by microbes to oxidize organic compounds present in a water sample. The <sup>amount of</sup>  $O_2$  required to convert the given quantity of biologically oxidizable organic matter to  $CO_2$  &  $H_2O$  is fixed. So the BOD value can interpret concentration of organic matter/organic consuming tendency. BOD value can be determined by knowing DO (dissolved oxygen) values in different conditions.

Procedure: 1. 150 ml of the water sample was taken in a 250 ml conical flask and its DO (dissolved oxygen) was determined by usual method (discussed earlier)

2. Another 150 ml of the water sample was taken and it was incubated at 20°C for 5 days. Dissolved oxygen ( $DO_2$ ) of incubated water sample was determined by suitable <sup>usual</sup> method.

3. From the experimental results, BOD was calculated by using formula given below:

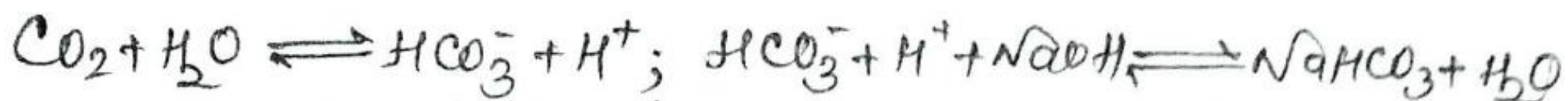
$$BOD = \frac{(DO_1 - DO_2) \times V_1}{V_2}$$

[Where  $DO_1$  = initial dissolved oxygen of the water sample,  $DO_2$  = dissolved oxygen of the water sample after 5 days incubation,  $V_1$  = volume of the water sample,  $V_2$  = volume of the water sample after dilution].

⇒ What is  $DO_2$ ?  $DO_2$  means free dissolved  $CO_2$  in water sample. This is responsible for corrosion.  $CO_2$  is a normal component of natural water. It is formed by the bio-oxidation of organic matter (C, H) present in the water. The aquatic plants use this dissolved  $CO_2$  gas as a raw material for the production of organic matter (e.g. carbohydrates) by means of photosynthesis.

Determination of  $DO_2$  in a water sample:  $DO_2$  in the water sample can be estimated by titration method. Principle: A standard <sup>(of 10)</sup> solution of NaOH reacts with the dissolved <sup>present</sup>  $CO_2$  in the water to form sodium bicarbonate ( $NaHCO_3$ )

(4)



Free dissolved  $\text{CO}_2$  ( $\text{Dco}_2$ ) can be calculated as:

Amount of free dissolved  $\text{CO}_2$  (in ppm) = [Volume of alkali used for total acidity (in ml) - Volume of alkali used by free mineral acid (blank) (in ml)]  $\times$  Normality  $\times 0.22 \times 10^6$  / volume of the water sample.

Procedure: 1. Preparation of solutions: First of all following solutions were prepared:

(i) N/50 NaOH solution: About 0.8 gm NaOH was weighed accurately, dissolved in 100 ml  $\text{CO}_2$  free distilled water and diluted to one litre in a graduated flask. Then solution was standardized with <sup>Standard</sup> (N/10) HCl solution.

(ii) Phenolphthalein indicator: 0.5 gm of phenolphthalein (powder) was dissolved in a mixture of 50 ml ethanol and 50 ml of distilled water. 0.02N NaOH solution added drop wise till a faint pink colour appeared.

2. Determination of total acidity: <sup>Standard</sup> NaOH solution was filled in a clean & rinsed burette and initial reading was noted. 50 ml of water sample was pipetted into a clean 250 ml conical flask. 2-3 drops of phenolphthalein indicator were added to it, and then colourless solution was titrated against standard (N/50) NaOH solution till a permanent faint pink colour appeared. Final burette reading noted. The titration process repeated to obtain at least two concordant readings.

3. Blank titration: Similarly, free mineral acid titrated against N/50 NaOH solution (Blank titration). Knowing the volume  $V_2$  of standard NaOH solution used in above two titrations,  $\text{Dco}_2$  calculated.

Note: If the colour of water sample turns pink by adding phenolphthalein indicator, shows free dissolved  $\text{CO}_2$  is absent.]

Calculation: Volume of water sample = 50 ml, volume of alkali (NaOH) used for total acidity =  $V_1$  ml; strength of alkali (NaOH) solution =  $\frac{N}{50}$  ( $f=1.02$ ),

Volume of alkali used by free mineral acid (Blank titration) =  $V_2$  ml.

We have, Amount of free  $\text{CO}_2$  in the water sample (in ppm) =

$$(V_1 - V_2) \times \text{strength of alkali solution} \times 0.022 \times 10^6 / \text{volume of water sample}$$

$$= \frac{(V_1 - V_2) \times \frac{1}{50} \times 1.02 \times 0.022 \times 10^6}{1} = 8.976 \times (V_1 - V_2)$$

⇒ Determination of Chloride ( $\text{Cl}^-$ ) in a water sample:

Water from any source contains dissolved chloride salts. Polluted or hard water contains high  $\text{Cl}^-$  concentration. Chloride ( $\text{Cl}^-$ ) is estimated/determined by titration method.

Principle: In all natural waters, chloride ( $\text{Cl}^-$ ) concentrations widely vary. Chlorides reach natural waters in many ways: water dissolves salts ( $\text{Cl}^-$ ) from the surface of the soil, spray from oceans on nearby soil, human excreta, industrial waste etc. Chlorides in reasonable conc<sup>n</sup> (250 mg/L) are not harmful. The sudden increase of chlorides in water of low chloride value indicates organic pollution in water. The water sample is titrated against standard (N/355)  $\text{AgNO}_3$  solution using an indicator ( $\text{K}_2\text{CrO}_4$ ), and chlorides estimated by using formula:

(5)

Amount of  $Cl^-$  (in mg/L) =  $\frac{[\text{Volume of } AgNO_3 \text{ solution used for water sample (in ml)} - \text{volume of } AgNO_3 \text{ solution used for blank titration (in ml)}] \times 1000}{\text{volume of water sample taken (in ml)}}$  Slightly differ with the

Procedure: The procedure of determination of chloride ( $Cl^-$ ) slightly differ with the type of water sample used.

1. Preparation of solutions: Following solutions were prepared:

(i) Standard (N/35.5)  $AgNO_3$  solution: 1.196 gm Silver nitrate (AR grade) was dissolved in 250 ml, chloride free distilled water.

(ii)  $Al(OH)_3$  suspension: 31.25 gm of Potash alum was dissolved in 250 ml of distilled water. 5N  $NH_4OH$  solution was added to precipitated  $Al(OH)_3$ . The precipitate was washed with water until free from  $Cl^-$  ions.

(iii)  $K_2CrO_4$  indicator: 1.94 gm of  $K_2CrO_4$  was dissolved in 100 ml of distilled water.

(iv) Phenolphthalein indicator: 0.5 gm of phenolphthalein powder was dissolved in a mixture of 50 ml ethanol & 50 ml of distilled water. 0.02N  $NaOH$  solution was added dropwise to it till a faint pink colour appeared.

(v) 1N  $NaOH$  solution: 4 gm of  $NaOH$  was in 250 ml of distilled water.

(vi) 1N  $H_2SO_4$  solution: 7 ml of conc.  $H_2SO_4$  (sp. gr. 1.84, 98%) was dissolved in water and diluted to 250 ml.

Type (A) for water sample free from colour,  $SO_3^{2-}$ ,  $S^{2-}$  &  $S_2O_3^{2-}$ : 2. Standard (N/35.5)  $AgNO_3$  solution was filled in a clean and rinsed burette and initial reading was noted. 100 ml of the water sample was taken in a 250 ml conical flask. 1-2 drops of  $K_2CrO_4$  indicator were added to it, and standard  $AgNO_3$  solution runned from burette dropwise with constant stirring until permanent faint reddish-brown colour appeared. final burette reading was noted. The titration process repeated to get at least two concordant readings.

3. Blank titration: Now, 100 ml of chloride free distilled water was taken in a conical flask, 2 drops of  $K_2CrO_4$  indicator added and then titrated with standard  $AgNO_3$  solution.

4. Knowing the amounts of standard  $AgNO_3$  used in two cases,  $Cl^-$  estimated by calculation.

Type (B) for coloured water sample: 2. 100 ml of the water sample was taken in a 250 ml conical flask. 3 ml of  $Al(OH)_3$  suspension was added to it, mixed thoroughly and allowed to settle. The mixture was filtered, washed and filtrate together with washing were collected.

3. Now, proceeded as in Type (A).

Type (C) for water sample containing  $S^{2-}$ ,  $SO_3^{2-}$  &  $S_2O_3^{2-}$ : 2. 100 ml of the water sample was taken in 250 ml conical flask. This was made alkaline to phenolphthalein by adding dropwise 1N  $NaOH$  solution. 1 ml of  $H_2O_2$  also added to it. The whole mixture was stirred and neutralised with equal amount of 1N  $H_2SO_4$  solution.

3. Now, proceeded as in Type (A).

Experimental results & Calculation: Volume of water sample taken (V) = 25 ml

Volume of standard  $AgNO_3$  solution used for water sample =  $V_1$  ml

" " " distilled water =  $V_2$  ml

We have, Amount of  $Cl^-$  (in mg/L) =  $\frac{(V_1 - V_2) \times 1000}{25} = 40 \times (V_1 - V_2)$ .

⇒ Determination of Fluoride ( $F^-$ ) in the water sample (Volhard method): Discussion or

Principle: This method is based on the precipitation of lead chlorofluoride, in which the Chlorine is determined by Volhard's method, and from this result the fluorine content can be calculated. The advantages of the method are the precipitate is granular, settles & filtered easily. The process carried out at pH 3.6-5.6, so substances which might be co-precipitated such as  $PO_4^{3-}$ ,  $SO_4^{2-}$ ,  $CrO_4^{2-}$ ,  $CO_3^{2-}$  do not interfere. In the presence of about 0.1 gm of  $F^-$ , very small quantities of  $Al^{3+}$ ,  $Mg^{2+}$ ,  $Na^+$  or  $K^+$  cause low results.

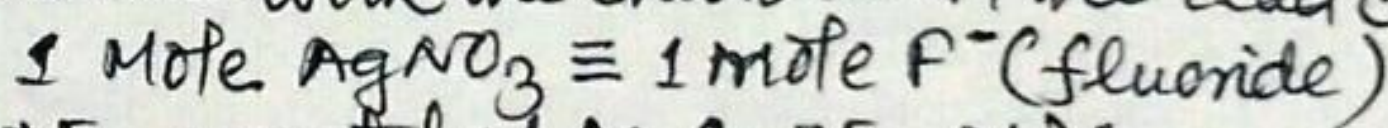
(-6)

Procedure: 1. 25 ml of water sample pipetted into a 250 ml conical flask, two drops of bromophenol blue indicator, 3 ml of 10% NaCl solution were added to it, and the mixture was diluted to 250 ml. Dilute  $\text{HNO}_3$  was added until the colour just changes to yellow, and then dilute NaOH solution was added until the colour just changes to blue. It was treated with 1 ml conc. HCl, then with 5 gm of lead nitrate, and heated on a water bath. The mixture was stirred gently till lead nitrate had dissolved & then 5 gm of crystallized sodium ethanoate added immediately with vigorous stirring. The contents were digested on water bath for 30 minutes with occasional stirring, and allowed to stand overnight.

2. A washing solution of lead chlorofluoride was prepared as follows: A solution of 10 gm lead nitrate in 200 ml of distilled water was added to 100 ml of a solution containing 1 gm of NaF and 2 ml of conc. HCl, and mixed thoroughly. The precipitate was allowed to settle. The supernatant liquid was decanted, precipitate washed by decantation with five portions (each of 200 ml) of water. Finally, 1 litre of water was added to the precipitate, the mixture shaken at intervals of an hour, allowed to settle and then filtered.

3. The original precipitate (1) was separated by decantation through a Whatman no. 542 or 42 paper. The precipitate was transferred to the filter, washed once with cold water, 4-5 times with the saturated solution of lead chlorofluoride, and finally once more with cold water. The precipitate and filter paper were transferred in a beaker containing 100 ml of 5%  $\text{HNO}_3$ , stirred well and heated on water bath until the precipitate had dissolved. A slight excess of standard ( $M/10$ )  $\text{AgNO}_3$  solution was added, digested on the water bath for a 30 minutes, and allowed to cool to room temp. The precipitate of AgCl was filtered through a sintered glass crucible, washed with a little cold water and the residual  $\text{AgNO}_3$  was titrated with standard ( $M/10$ ) thiocyanate solution.

4. The amount of silver found in the filtrate was subtracted from the amount originally added. The difference represents, the amount of silver that was required to combine with the chlorine in the lead chlorofluoride precipitate.



### ⇒ Determination of Heavy metals (As, Cr, Hg, Pb):

1. Determination of Arsenic (As): A number of methods available for determination of minute amounts of arsenic. The 'Molybdenum blue method' is most precise & sensitive one. It is readily applied colorimetrically or spectrophotometrically.

Principle: When sample containing arsenic as arsenate is treated with ammonium molybdate solution, heteropolymolybdoarsenate (arsenomolybdate) is formed. On reduction with hydrazinium sulphate or with  $\text{SnCl}_2$  it converted into blue soluble complex 'Molybdenum blue'. The stable blue colour has a maximum absorption at about 840 nm and shows no appreciable change in 24 hours.

Both macro and micro quantities of arsenic may be isolated by distillation of arsenic(III) chloride from HCl solution in all glass apparatus in a stream of  $\text{CO}_2$  or  $\text{N}_2$ ; a reducing agent, such as hydrazinium sulphate, is used to reduce  $\text{As(V)}$  to  $\text{As(III)}$ .

Procedure: 1. Preparation of Reagents: (i) KI solution: 15 gm of KI crystals was dissolved in 100 ml of distilled water.

(ii)  $\text{SnCl}_2$  solution: 40 gm of hydrated tin(II) chloride was dissolved in 100 ml conc. HCl.

(iii) Amm. molybdate-hydrazinium sulphate solution: 10 gm amm. molybdate was dissolved in 10 ml distilled water and then 90 ml of  $\text{H}_2\text{SO}_4$  added (solution A). 0.15 gm hydrazinium sulphate

(4)

100 ml distilled water (sol. B). The two solutions A & B were mixed before use.  
(i) Standard arsenic solution: 1.320 gm of  $As_2O_3$  (arsenic oxide) was dissolved in the minimum volume of 1M NaOH solution. A solution containing 0.001 mg/ml 'As' was prepared by dilution.

(ii) Iodine-KI solution: 0.25 gm  $I_2$  was dissolved in a small volume of water containing 0.4 gm KI, and diluted to 100 ml.

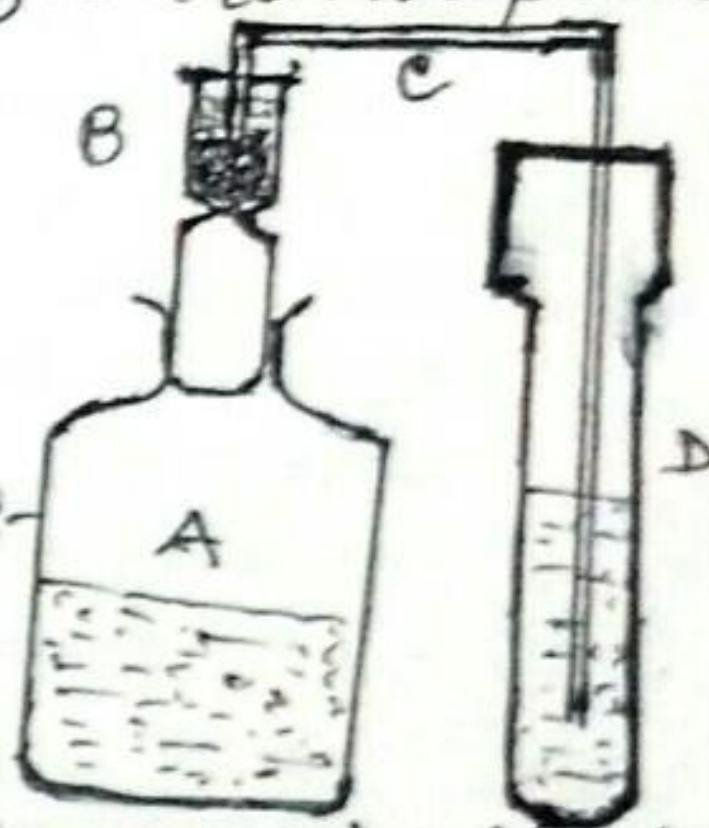
(iii) Sodium disulphite or Sodium metabisulphite solution: 0.5 gm of the solid sodium metabisulphite ( $Na_2S_2O_5$ ) was dissolved in 10 ml of distilled water. This solution/reagent prepared in fresh.

(vii) Sodium bicarbonate ( $NaHCO_3$ ) solution: 4.2 gm of the solid  $NaHCO_3$  was dissolved in 100 ml of distilled water.

2. The arsenate, obtained by the wet oxidation of a sample, was reduced by  $KI$  &  $SnCl_2$  in conc. HCl. 25 ml of the 'arsenate' solution transferred to the 50 ml Pyrex evolution vessel (A), 5-6 ml conc. HCl was added to it followed by 2 ml of KI solution and 0.5 ml of  $SnCl_2$ . The mixture was stand at room temp. for 20-30 min to permit the complete reduction.

3. The tube (B) was loosely packed with cotton wool soaked in lead ethanoate solution (to remove  $H_2S$  & acid spray). It connected with narrow absorption tube (D) through a capillary tube (C). 1 ml  $I_2$ -KI solution & 0.2 ml of  $NaHCO_3$  solution was placed in tube (A) and mixed with the end of the delivery tube.

4. 2.0 gm of Zn was rapidly added to vessel (A), immediately the stopper inserted and allowed the gases to bubble through the solution for 30 min. The delivery tube (C) disconnected and left it in the absorption tube. 5.0 ml of amm. molybdate-hydrazine reagent and 1-2 drop of  $Na_2S_2O_5$  solution were added. The resulting colourless solution was heated in a water bath at 95-100°C, cooled and then transferred to a 10 ml measuring flask, and made up to mark.



5. The absorbance of the solution was measured at 840 nm. The reference cell was charged with a solution obtained by taking the  $I_2$ - $I_2$ - $HCO_3$  mixture and treating it with molybdate-hydrazinium sulphate-disulphite as in the actual procedure.

6. The calibration curve was constructed by taking say 0, 2.5, 5.0, 7.5 & 10.0  $\mu g$  As/10 ml mixing with  $I_2$ - $I_2$ - $HCO_3$  solution, adding molybdate-hydrazinium sulphate-disulphite and heating to 95-100°C.

⇒ 2. Determination of Chromium (Cr): Principle: Small amounts of Chromium (upto 0.5%) may be determined colorimetrically in alkaline solution as Chromate. The absorbance of the solution is measured at 365-370 nm. The standard solution used for the preparation of the reference curve should have the same alkalinity as the sample solution, and should preferably have the same concentration of foreign salts. Standard may be prepared from analytical grade  $K_2CrO_4$ . A more sensitive method is to employ 1,5-diphenylcarbazide,  $[C_6H_5NHC(=O)NH_2]$ , in acid solution (~0.2M), Chromates give a soluble violet compound with this reagent.

Procedure: A 0.25% solution of diphenylcarbazide was prepared by dissolving 0.25 gm of the compound in 100 ml of 5% acetone. The test may contain 0.2-0.5 ppm Chromate. To about 15 ml of this solution, sufficient amount of 3M  $H_2SO_4$  added to make the concentration ~0.1M when subsequently diluted to 25 ml. 1 ml of the diphenylcarbazide reagent was added and made up to 25 ml with distilled water. The colour produced against standards prepared from 0.00002M  $K_2CrO_4$  solution. A green filter transmission maximum at about 540 nm might be used.