Expt. No. 3

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Object :- Determine the strength of a ~~Unknown~~ ferrous ammonium sulphate (FAS) solution by titrating it with Pot. Dichromate solution using Potassium ferricyanide as an external indicator.

Apparatus / Reagents required :- Burette (50 ml), pipette (10 ml), Conical flask (100 ml), FAS solution, known solution of $K_2Cr_2O_7$, potassium ferricyanide indicator, 10% Sulphuric Acid.

Theory :- Potassium dichromate is good oxidising reagent and in presence of dilute H_2SO_4 it ~~seems~~ forms chromic sulphate as shown by following equation.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$$

The nascent oxygen so liberated oxidized Fe^{+2} ion of FAS into ferric Fe^{+3} ion. The main reaction is

$$6[FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O] + K_2Cr_2O_7 + 4H_2SO_4$$

$$3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 6(NH_4)_2SO_4 + 43H_2O$$

The external indicator potassium ferricyanide give greenish blue colour with Fe^{+2} ion.

$$3Fe + 2[Fe(CN)_6]^{-3} \longrightarrow Fe_3[Fe(CN)_6]_2$$

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Procedure :-

1. Prepare standard solution FAS of given stength by dissolving the required amount of FAS in distilled water in 100 ml volumentric flask.
2. Fill the burette with known $K_2Cr_2O_7$ solution.
3. Take 10ml of standard (FAS) solution & 10ml Sulphuric acid in a conical flask.
4. Start adding some amount of $K_2Cr_2O_7$ from burette to conical flask.
5. Take a white tile & arrange few drops of external indicator on it.
6. Take a drop of analyte from flask & mixed with a drop of external indicator on white tile.
7. If blue colour appears then it means end point is not come yet, add more amount of $K_2Cr_2O_7$ from burette.
8. Repeat the procedure & check the analyst after addition of every drops of $K_2Cr_2O_7$.
9. If no blue coloration is observed on white tile then it shows that end point comes.
10. Repeat the titration till we get three concordant reading.
11. Follow the titration procedure for the Unknown solution & obtain the burette reading.

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Calculation :-

For standard FAS solution with $K_2Cr_2O_7$

$N_1V_1 = N_2V_2$

$N_1 \times 11 = N/30 \times 10$

$N_1 = \frac{N}{11 \times 3}$

here
N_1 = Normality of $K_2Cr_2O_7$
N_2 = Normality of known FAS = $N/30$
V_1 = Volume of $K_2Cr_2O_7$
V_2 = 10 ml

Now for unknown FAS solution with same $K_2Cr_2O_7$

$N_3V_3 = N_4V_4$

$\because N_1 = N_3$

$\frac{N}{11 \times 3} \times 6.9 = N_4 \times 10$

$N_4 = \frac{6.9}{11 \times 3 \times 10} N$

$N_4 = 0.0209\ N$

Strength of unknown FAS solution
= Normality of FAS × eq weight of FAS
= 0.0209 × 392.16
= 8.196 gm/lit

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Observation :-

Titration between standard FAS solution & intermediate $K_2Cr_2O_7$

S.No	Volume of FAS Solution (ml)	Volume of $K_2Cr_2O_7$ Initial (ml)	Final (ml)	Difference (ml)	Concordant reading (ml)
1.	10 ml	0.0	11.0	11.0	11.0
2.	10 ml	11.0	22.0	11.0	

Titration between Unknown FAS solution and $K_2Cr_2O_7$

S. No.	Volume of FAS Solution (ml)	Volume of $K_2Cr_2O_7$ Initial (ml)	Final (ml)	Difference (ml)	Concordant reading (ml)
1.	10 ml	0.0	6.9	6.9	6.9
2.	10 ml	6.9	13.8	6.9	

Calculation :-

Strength of unknown FAS solution :
= Normality of FAS × eq weight of FAS
= 8.196 gm/lit.

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Result: The strength of unknown FAS solution is 8.196 gm/lit

Precautions:-

(i) All the apparatus should be washed properly.
(ii) $K_2Cr_2O_7$ solution should be added drop wise.
(iii) End point should be observed carefully.

Industrial Application:-

(a) It is used to determine the strength of unknown solution.
(b) Titration is used to analyse acid rain.
(c) Titrations are used to determine the concentration in surface water.
(d) It is also used in food & beverage industry to analyse the standard conc. of specific additives.
(e) It is used in wine industry where SO_2 is used to control microbial growth. Too much / too little however, can be detrimental to the equality of final product. Titration enabled wireless to determine the conc. of SO_2 during the process.
(f) FAS is used in the Fricke's dose meter to measure high doses of gamma rays.

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Viva Questions

Q1 Why dil. H_2SO_4 is added in the preparation of FAS solution?

Ans Dil. H_2SO_4 is essential in FAS preparation to maintain the integrity & effectiveness of the ferrous ammonium sulfate solution.

Q2 What do you mean by Volumetric analysis?

Ans Volumetric analysis is a quantitive chemical analysis method used to determine the concentration of an unknown solution by measuring its volume against a solution of known concentration (standard solution). It is commonly performed through titration.

Q3 What do you understand by concordant reading?

Ans A concordant reading refers to a set of consistent and closely agreeing values obtained in a titration experiment. These reading help ensure accuracy & reliability in volumetric analysis.

Q4 Explain the type of indicator?

Ans Indicator are substance that show a visible change in color or physical property to signal the completion of a reaction, particularly in titration experiment.

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It has different types -

(A) Acid-Base indicator - are used for pH dependent titration.

ex Phenolphthalein, Methyl orange.

(B) Redox indicator - are used in oxidation-reduction reaction

ex. $KMnO_4$, Starch.

(C) Complexometric indicator - are used in metal ion titration.

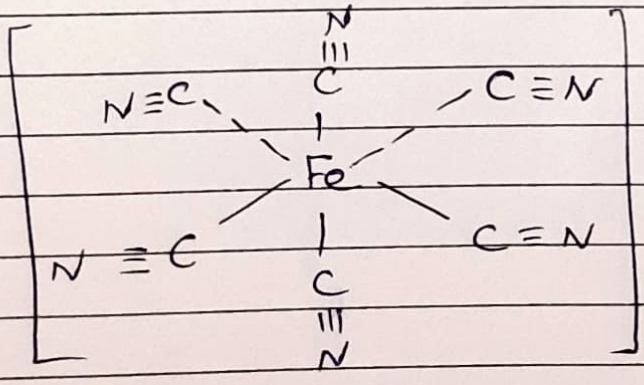
ex. EBT

(D) Precipitation indicator - helps ~~dect~~ detect precipitate formation.

ex fluorescein

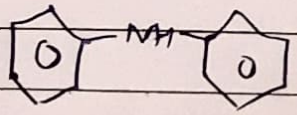
Q5 Which indicator is used in this titration? write its structure.

Ans Potassium ferricyanide, external indicator is used in this titration.



~~dyp~~
~~diphy.~~
diphenylemine

$C_6H_5-NH-C_6H_5$



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Q6 Explain the principle involved in this titration.

Ans It is based on an oxidation - reduction reaction between Fe^{+2} (ferrous ion) in FAS solution & MnO_4^- (permanganate ions) in acidic medium, or $Cr_2O_7^{-2}$ (cromate ion). An external indicator like diphenylamine is required, which change color at end point of titration. (Colorless to blue)

Q7 How will you prepare 1 lit 0.1N $K_2Cr_2O_7$ solution.

Ans Formula used :-

$$\text{weight (g)} = \text{Normality} \times \text{eq weight} \times \text{Volume}$$

$$= 0.1 \times \frac{294.2}{6} \times 1 = 4.903\,g \text{ is used.}$$

step 1 :- weigh 4.903 g of $K_2Cr_2O_7$, dissolve in 500 ml of distilled water in a 1l volumetric flask.

→ Add dil H_2SO_4, make up to 1000 ml with distilled water.

Mix well & store in bottle.

Q8 Why distilled water is used to prepare std solution?

Ans Because it doesn't contain impurities like dissolved ions.

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