

31/10/17

ASSAY OF FERROUS SULPHATE BY CERIMETRY

AIM: To perform the assay of given sample of ferrous sulphate.

APPARATUS: Conical flask, volumetric flask, 10 ml pipette, burette, funnel etc.

MATERIALS/CHEMICALS: Fe_2SO_4 , water, 1 M H_2SO_4 , 0.1 M ceric ammonium sulphate, ferric solⁿ as indicator, As_2O_3 , 8% w/v NaOH solⁿ.

PRINCIPLE: Ceric ammonium sulphate in H_2SO_4 medium can function as a strong oxidising agent & have high oxidation potential. Sufficient H_2SO_4 is used to prevent hydrolytic and precipitation of basic salts.

Ceric ammonium sulphate a salt of suitable solubility for the preparation of the standard solⁿ has the approximate formula $\text{Ce}(\text{SO}_4)_2 \cdot 2(\text{NH}_4)_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$, but the solution has to be standardised against a solⁿ of As_2O_3 . Since ceric ammonium sulphate is a strong oxidising agent we can determine various reducing substances by simple titration. In the presence of reducing agent it undergoes reduction to the cerous state.

PROCEDURE:

A) STANDARDISATION OF 0.1M $\text{Ce}(\text{SO}_4)_2$ SOLⁿ: Weigh accurately about 0.2 gm of As_2O_3 , previously dried at 105°C for 1 hour & transfer to a 500 ml conical flask. Wash down the inner walls of flask with 25 ml of 8% w/v solⁿ of NaOH, swirl to dissolve, add 100 ml of water & mix. Add 30 ml of dil. H_2SO_4 , 0.15 ml of osmic acid solⁿ, 0.1 ml of ferroin sulphate solⁿ & slowly titrate with $\text{Ce}(\text{SO}_4)_2$ solⁿ until the pink colour is changed to very pale blue colour.

B) FOR ASSAY: Weigh accurately about 0.5 gm of Fe_2SO_4 , dissolve in a mixture of 30 ml water and 20 ml of ~~0.1M~~ H_2SO_4 and titrate with 0.1 M ceric ammonium sulphate solⁿ using ferroin sulphate solⁿ as indicator.

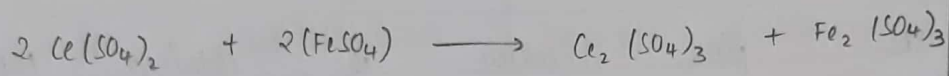
IP FACTOR: Each ml of 0.1M $\text{Ce}(\text{SO}_4)_2$ is equivalent to $\frac{0.051519}{0.01519}$ gm of Fe_2SO_4

REFERENCE: The Indian Pharmacopoeia, vol - II, 2007 edition, pg no : 1125.

REPORT: The percent purity of ferrous sulphate is 100.254 %

~~Value~~

PRINCIPLE :



CALCULATIONS :

$$\begin{aligned} \% \text{ purity of FeSO}_4 &= \frac{\text{Vol. of Ce}(\text{SO}_4)_2 \times \text{IP Factor} \times 100 \times M (\text{actual})}{\text{wt. of FeSO}_4 \times M (\text{expected})} \\ &= \frac{0.2 \times 0.01519 \times 100 \times 0.09}{0.5 \times 0.1} \\ &= \frac{5.0127}{0.05} = \underline{100.254 \%} \end{aligned}$$

TITRATION OF 0.1M $\text{Ce}(\text{SO}_4)_2$ WITH H_2SO_4 SOLN:

| S. NO | VOL OF H_2SO_4 SOL ⁿ | BURETTE READING | | VOL. OF $\text{Ce}(\text{SO}_4)_2$ RUNDOWN (ml) |
|-------|---|-----------------|-------|---|
| | | INITIAL | FINAL | |
| 1 | 30 | 0 | 27 | 27 |
| 2 | 30 | 0 | 28 | 28 |

$$M \text{ of } \text{Ce}(\text{SO}_4)_2 = \frac{\text{wt. taken} \times \text{expected } M}{\text{Titration vol.} \times \text{IF Factor}}$$

$$= \frac{0.2 \times 0.1}{28 \times 0.004946}$$

$$= \frac{0.02}{0.1483}$$

$$= 0.142 \text{ M}$$