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, ferroin 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 hydrolysis and precipitation of basic salts.

Ceric ammonium sulphate a salt of suitable solubility for the preparation of the standard soln has the approximate formula $Ce(SO_4)_2 \cdot 2(NH_4)_2 SO_4 \cdot 2H_2O$, 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 $Ce(SO_4)_2$ SOL^N: 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 30ml of dil. H_2SO_4, 0.15 ml of osmic acid soln, 0.1 ml of ferroin sulphate soln & slowly titrate with $Ce(SO_4)_2$ 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 $Ce(SO_4)_2$ is equivalent to
≡ ~~0.05159~~ 0.01519 gm of Fe_2SO_4

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

REPORT: The percent purity of ferrous sulphate is 100.254 %

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PRINCIPLE :

$$2\,Ce(SO_4)_2 + 2(FeSO_4) \longrightarrow Ce_2(SO_4)_3 + Fe_2(SO_4)_3$$

CALCULATIONS :

$$\% \text{ purity of } FeSO_4 = \frac{\text{Vol. of } Ce(SO_4)_2 \times \text{IP Factor} \times 100 \times M \text{ (actual)}}{\text{wt. of } FeSO_4 \times M \text{ (expected)}}$$

$$= \frac{0.2 \times 0.01519 \times 100 \times 0.09}{0.5 \times 0.1}$$

$$= \frac{5.0127}{0.05} = 100.254\ \%$$

TITRATION OF 0.1M $Ce(SO_4)_2$ WITH H_2SO_4 SOLN:

S.NO	VOL OF H_2SO_4 SOLN	BURETTE READING		VOL. OF $Ce(SO_4)_2$ RUNDOWN (ml)
		INITIAL	FINAL	
1	30	0	27	27
2	30	0	28	28

$$M \text{ of } Ce(SO_4)_2 = \frac{\text{wt. taken} \times \text{expected M}}{\text{Titration vol.} \times \text{IP Factor}}$$

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

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

$$= 0.142 \text{ M}$$