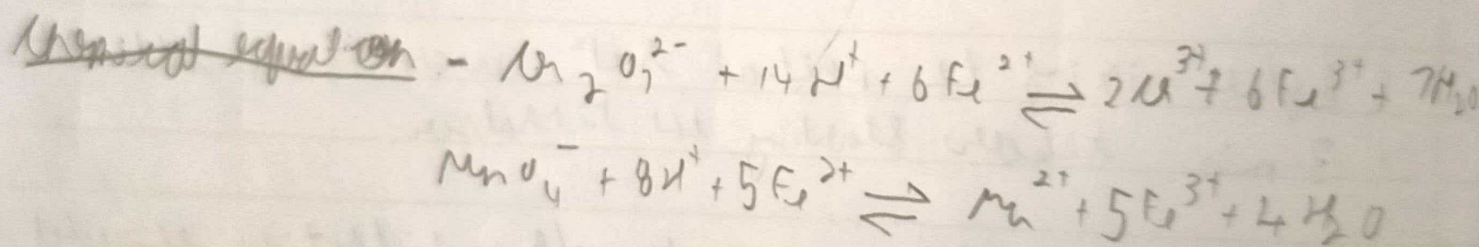


Experiment - 4

Experiment - to determine hexavalent chromium content of a water sample by back titration method.

Apparatus - Pipette, burette, beakers, conical flask, funnel, burette stand and clamp.

Chemicals - $K_2Cr_2O_7$ of unknown strength, H_2SO_4 , Molar $FeSO_4 \cdot (NH_4)_2 \cdot SO_4 \cdot 6H_2O$, $KMnO_4$.



Observation - (i) Standardization of $KMnO_4$ solⁿ volume of 0.1 N FAS (N₂) solⁿ taken for titration = 10 ml

S No	Initial reading (ml)	Final reading (ml)	Vol of $KMnO_4$ used
1.	0	10.2	10.2
2.	0	10.2	10.2
3.	0	10.2	10.2

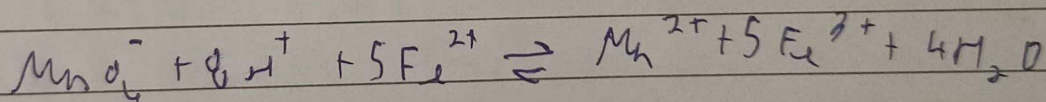
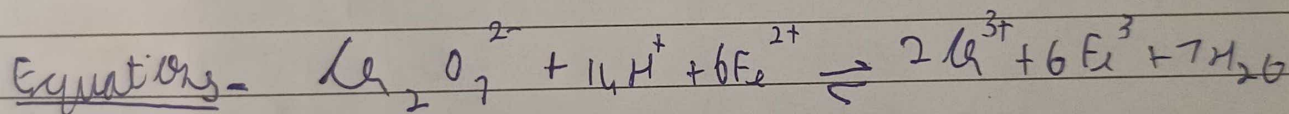
Mean value of $KMnO_4$ used (V_1) = 10.2 ml

Experiment-4

Experiment - To determine hexavalent chromium content of a water sample by back titration method

Apparatus - Pipette, burette, beakers, conical flask, funnel, burette stand and clamp

Chemicals - Potassium dichromate solution ($K_2Cr_2O_7$) of unknown strength, sulphuric acid (H_2SO_4), Mohr's salt solⁿ (Ferrous ammonium sulphate, $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$) and $KMnO_4$



Theory - In an acidic solⁿ of Fe^{2+} , Cr^{6+} oxidizes Fe^{2+} to Fe^{3+} while it self getting reduced to trivalent chromium. If to a certain volume of the sample solution (containing Cr^{6+} ions) is added a known amount of the Mohr salt solⁿ (known concentration) in presence of sulphuric acid, Cr^{6+} is reduced to Cr^{3+} , while the Fe^{2+} is oxidised to Fe^{3+} . The ~~un-oxidised~~ un-oxidised Fe^{2+} can then be determined by titration against a standard $KMnO_4$ solⁿ. From the volume of ferrous ion consumed, the amount of hexavalent chromium can be determined.

(b) Determination of Cr^{6+} solⁿ by back titration method.

Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ sample solⁿ taken for the titration = 10 ml

Volume of Mohr salt (0.1 M) solⁿ added = 10 ml

S No	Initial Reading (ml)	Final Reading (ml)	Volume of KMnO_4 used
1	0	4.7	4.7
2	0	4.7	4.7
3	0	4.7	4.7

Mean volume of KMnO_4 used (V_2) = 4.7 ml

Calculation - (a) Normality of KMnO_4 solⁿ

$$N_0 V_0 \text{ (Given FAS)} = N_1 V_1 \text{ (KMnO}_4\text{)}$$

Calculate N_1 = normality of KMnO_4 being used -

$$N_1 = \frac{N_0 V_0}{V_1} = \frac{0.1 \times 10}{10.2} = 0.0980$$

(b) Determination of Cr^{6+} content

Total Fe^{2+} added to Cr^{6+} solution = $10 \times 0.1 = 1$

Volume of N_1 KMnO_4 solⁿ used is in titration
= V_2 ml = 4.7 ml

The more salt solution (Fe^{2+}) added to the dichromate is in excess so that complete reduction of Cr^{6+} ions to Cr^{3+} ions takes place. This method of volumetric analysis is known as back-titration method.

Procedure

Procedure - (a) Standardization of KMnO_4 :-

Transfer 10 ml of the standard 0.1 FAS solⁿ in a clean conical flask using a pipette following by addition of 5 ml of 4 N sulphuric acid. Titrate the solⁿ against KMnO_4 solⁿ taken in a burette.

Note the volume of solⁿ being used when colour of the solⁿ in conical flask changes from colourless to pink. Repeat the titration minimum 3 times and take mean of the closely related readings.

Insert this as volume V_1 .

(b) Determination of Cr^{6+} contents:-

Transfer 10 ml of Cr^{6+} solⁿ and 10 ml of the FAS solⁿ with a pipette add 5 ml of 4 N sulphuric acid with a graduated cylinder. Titrate the solⁿ against the standardization KMnO_4 solution. Note the volume of solⁿ used when colour of the solⁿ in conical flask changes from green to pink.

Fe^{2+} left in solution after reaction of Ce^{6+} to Ce^{3+}

$$= V_2 N_1 \text{ meq} = 4.7 \times 0.0980 = 0.46078$$

$$\text{Amount of } \text{Fe}^{2+} \text{ reacted with } \text{Ce}^{6+} = [10 \times 0.1 N(\text{FAS}) - V_2 N_1]$$

$$= 0.53922$$

This is equal to amount of Ce^{6+} present in 10 ml of the solution.

$$\text{Equivalent weight of cerium} = \frac{52}{3} = 17.33 \text{ g/m}$$

Thus amount of Ce^{6+} present in solution

$$= \frac{[10 \times 0.1 N(\text{FAS}) - V_2 N_1] \times 17.33}{10} \text{ gm}$$

$$= 0.93446$$

Result - Amount of Ce^{6+} present in the given sample solution was found to be 0.93446 g/L

Repeat the titration minimum three times & take mean of the closely related readings. Record this as volume V_2 .

Calculations:- i. Normality of KMnO_4 solⁿ = 0.1 N

$$N_0 V_0 \text{ (given FAS)} = N_1 V_1 \text{ (KMnO}_4\text{)}$$

Total Fe^{2+} added to Cr^{6+} solⁿ = $10 \text{ ml} \times 0.1 \text{ N}$ eq

Volume of N, KMnO_4 solⁿ used in the titration = $V_2 \text{ ml}$.

Fe^{2+} left in solⁿ after conversion of Cr^{6+} to Cr^{3+} = $V_2 N_1 \text{ meq}$

Amount of Fe^{2+} reacted with Cr^{6+} = $[10 \times 0.1 \text{ N} - V_2 N_1] \text{ meq}$

This is equal to amount of Cr^{6+} present in 10 ml of the solⁿ

Equivalent weight of Chromium = $52/3 = 17.33 \text{ g/L}$

Thus, amount of Cr^{6+} present in solⁿ

$$= [10 \times 0.1 \text{ N (FAS)} - V_2 N_1 \times 17.33] / 10 \text{ g/L} = 0.93446$$

Result - Amount of Cr^{6+} present in the given sample solⁿ was found to be 0.93446 g/L