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EXPERIMENT: To determine hexavalent chromium content of a water sample by back-titration method.

THEORY: In an acidic medium solution of Fe^{2+}, Cr^{6+} oxidizes Fe^{2+} to Fe^{3+} while itself getting reduced to the trivalent chromium. If to a certain volume of the sample solution containing Cr^{6+} ions is added a known amount of the Mohr Salt solution (known concentrations), in presence of sulphuric acid, Cr^{6+} is reduced to Cr^{3+}, while the Fe^{2+} is oxidized to Fe^{3+}. The un-reacted Fe^{2+} can then be determined by titration against a standard $KMnO_4$ solution. From the volume of ferrous ions consumed, the amount of hexavalent chromium can be determined. The Mohr Salt solution (Fe^{2+}) added to the dichromate is in excess so that complete reduction of Cr^{6+} ions to Cr^{3+} takes place. This method of volumetric analysis is known as Back-Titration method.

PROCEDURE:

a) Standardization of $KMnO_4$

Transfer 10ml of the standard 0.1N Ferrous Ammonium Sulphate (FAS) solution in a clean conical flask using a pipette followed by addition of 5ml of 4N sulphuric acid. Titrate the solution against $KMnO_4$ solution taken in a burette. Note the volume of solution used when color of the solution changes from colorless to pink. Repeat the titration minimum 3 times and take mean of the closely related readings. Treat this as volume V_1.

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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 solution (Ferrous Ammonium Sulphate; $FeSO_4(NH_4)_2SO_4.6H_2O$) and Potassium permanganate ($KMnO_4$).

CHEMICAL EQUATIONS:

$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightleftharpoons 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

$$MnO_4^- + 8H^+ + 5Fe^{2+} \rightleftharpoons Mn^{2+} + 5Fe^{3+} + 4H_2O$$

INDICATOR: $KMnO_4$ (acts as a self-indicator)

OBSERVATIONS:

a) Standardization of $KMnO_4$ solution

Volume of 0.1N FAS (N_0) solution taken for titration = 10 mL (V_0)

Sr. No.	Initial Reading (mL)	Final Reading (mL)	Volume of $KMnO_4$ Used (mL)
1.	0.0	10.4	10.4
2.	0.0	10.4	10.4
3.	0.0	10.4	10.4

Mean of the volume used (V_1) = 10.4 mL

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b) Determination of Cr^{6+} Content

Transfer 10ml of Cr^{6+} content and 10ml of the FAS solution with a pipette. Add 5ml of 4N sulphuric acid with a graduated cylinder. Titrate the solution against the standardized $KMnO_4$ solution. Note the volume of solution used when color of the solution changes from green to pink. Repeat the titration minimum three times and take mean of the closely related readings. Treat this as volume V_2.

RESULT: Amount of Cr^{6+} present in the given sample solution was found to be 0.917 g/L

PRECAUTIONS: 1. Lower meniscus to be read for colorless solution.
2. Upper meniscus to be read for colored solution.
3. Continue the titration until a permanent pink color appears in the conical flask.

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b) Determination of Cr^{6+} solution by back-titration method

Volume of $K_2Cr_2O_4$ sample solution taken for the titration = 10 mL

Volume of Mohr Salt (0.1M) solution added = 10 mL

Sn. No.	Initial Reading (mL)	Final Reading (mL)	Volume of $KMnO_4$ used (mL)
1.	0.0	4.9	4.9
2.	0.0	4.9	4.9
3.	0.0	4.9	4.9

Mean of the volume used (V_2) = 4.9 mL

CALCULATIONS:

a. N_0V_0 (given FAS) = N_1V_1 ($KMnO_4$)

$0.1 \times 10 = N_1 \times 10.4$

$N_1 = 0.096\ N$

b. Total Fe^{2+} added to Cr^{6+} solution = 10×0.1 meq = 1 meq

Volume of N_1 $KMnO_4$ solution used in the titration = 4.9 mL

Fe^{2+} left in solution after conversion of Cr^{6+} to Cr^{3+} = 4.9×0.096

= 0.4704 meq

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

= [1 - 0.4704]

= 0.5296 meq

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

Equivalent weight of chromium = 52/3 = 17.33 g/L

Thus, amount of Cr^{6+} present in solution = $\frac{0.5296 \times 17.33}{10}$ = 0.917 g/L

RESULT: Amount of Cr^{6+} present in the given sample solution was found to be 0.917 g/L.

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