Sample Titration Problems

Problem #1

A 20.00 mL sample of an unknown H_3PO_4 solution is titrated with a 0.100 M NaOH solution. The equivalence point is reached when 18.45 mL of NaOH solution is added. What is the concentration of the original H_3PO_4 solution?

$H_3PO_{4\,(aq)} + 3\ NaOH_{(aq)} \Rightarrow 3\ H_2O_{(l)} + Na_3PO_{4\,(aq)}$

Problem #1

$\dfrac{0.100 \text{ moles NaOH}}{\text{L NaOH solution}} * 0.01845 \text{ L NaOH} = 0.001845 \text{ moles NaOH}$

$0.001845 \text{ moles NaOH} * \dfrac{1 \text{ mol } OH^-}{1 \text{ mol NaOH}} * \dfrac{1 \text{ mol } H^+}{1 \text{ mol } OH^-} = 0.001845 \text{ mol } H^+$

$0.001845 \text{ mol } H^+ * \dfrac{1 \text{ mol } H_3PO_4}{3 \text{ mol } H^+} = 0.0006150 \text{ mol } H_3PO_4$

$\dfrac{0.0006150 \text{ mol } H_3PO_4}{0.020 \text{ L}} = 0.03075 \text{ M } H_3PO_4$

Problem #1

$i_2M_1V_1 = i_1M_2V_2$

$i_{NaOH}M_{H3PO4}V_{H3PO4} = i_{H3PO4}\ M_{NaOH}V_{NaOH}$

$H_3PO_{4\,(aq)} + 3\ NaOH_{(aq)} \rightleftharpoons 3\ H_2O_{(l)} + Na_3PO_{4\,(aq)}$

$(3)\ (M_{H3PO4})\ (20\ mL) = (1)\ (0.100\ M)(18.45\ mL)$

$M_{H3PO4} = 0.03075\ M\ H_3PO_4$

Problem #3

100.00 mL of a wastewater solution is diluted to 250.00 mL and titrated with 0.1106 M NaOH. Equivalence is reached after addition of 9.62 mL of the sodium hydroxide solution. What is the pH of the original wastewater sample?

$H^+ + OH^- \Leftrightarrow H_2O$

$i_2M_1V_1 = i_1M_2V_2$

$i_{OH}M_{H+}V_{H+} = i_{H+}\ M_{OH}V_{OH}$

$(1)\ (M_{H+})\ (100\ mL) = (1)\ (0.1106\ M)(9.62\ mL)$

$M_{H+} = 0.01064\ M\ H^+$

pH = - log [H+] = - log (0.01064 M)

pH = 2.97

Problem #3

100.00 mL of wastewater is titrated with a 0.1062 M solution of $Pb(NO_3)_2$. Lead will react with chloride ion to form lead chloride. If it requires 6.52 mL of lead nitrate to reach equivalence, what was the concentration of chlorie in the original solution?

$Pb(NO_3)_{2\,(aq)} + 2\ Cl^-_{(aq)} \rightleftharpoons PbCl_{2\,(s)} + 2\ NO_3^-{}_{(aq)}$

$Pb^{2+}_{(aq)} + 2\ Cl^-_{(aq)} \rightleftharpoons PbCl_{2\,(s)}$

NO_3^- is a “spectator ion”

$Pb^{2+}_{(aq)} + 2\ Cl^-_{(aq)} \rightleftharpoons PbCl_{2\,(s)}$

$i_2M_1V_1 = i_1M_2V_2$

$i_{Cl-}M_{Pb2+}V_{Pb2+} = i_{Pb+}\ M_{Cl-}V_{Cl-}$

$(2)\ (0.1062\ M)\ (6.52\ mL) = (1)\ (M_{Cl-})(100.00\ mL)$

$M_{Cl-} = 0.01385\ M\ Cl-$

Problem #4

A solution is prepared by mixing 0.10 L of 0.12 M sodium chloride with 0.23 L of a 0.18 M $MgCl_2$ solution. What volume of a 0.20 M $AgNO_3$ solution is required to precipitate all of the chloride ion as silver chloride?

$Cl^-_{(aq)} + Ag^+_{(aq)} \rightarrow AgCl_{(s)}$

$i_2M_1V_1 = i_1M_2V_2$

$i_{Cl-}M_{Ag+}V_{Ag+} = i_{Ag+}\ M_{Cl-}V_{Cl-}$

$(1)\ (0.20\ M)\ (V_{Ag+}) = (1)\ (M_{Cl-})(V_{Cl-})$

What is the volume of the chloride solution?

What is the Molarity of Chloride?

$(1)\ (0.20\ M)\ (V_{Ag+}) = (1)\ (M_{Cl-})(V_{Cl-})$

What is the volume of the chloride solution?

0.10 L + 0.23 L = 0.33 L

What is the Molarity of Chloride?

(.12 M * 0.10 L) + (0.18M*0.23L) = 0.0534 mol Cl-

$\frac{0.0534 \text{ mol } Cl^-}{0.33 \text{ L}} = 0.1618 \text{ M } Cl^-$

$(1)\ (0.20\ M)\ (V_{Ag+}) = (1)\ (M_{Cl-})(V_{Cl-})$

$(1)\ (0.20\ M)\ (V_{Ag+}) = (1)\ (0.1618\ M_{-})(0.33\ L)$

$V_{Ag+} = 0.2670\ L$

Might have been easier just to go with the moles from beginning.

Total moles of Cl^- = 0.0534 mol Cl-

[(.12 M * 0.10 L) + (0.18M*0.23L) = 0.0534 mol Cl-]

$$0.0534 \text{ mol } Cl^- * \frac{1 \text{ mol } Ag^+}{1 \text{ mol } Cl^-} = 0.0534 \text{ mol } Ag^+$$

$$0.0534 \text{ mol } Ag^+ * \frac{1 \text{ L Ag solution}}{0.20 \text{ mol Ag solution}} = 0.2670 \text{ L Ag solution}$$

Tutorial 12 - Solutions

Determining the Concentration of a Specific Ion Using Precipitation Titrations.

Answer to **Question 1** on page 12 of Tutorial 12.

1. In order to find the concentration of chloride ion in a sample of pool water, a 50.0 mL sample of the pool water was titrated with 0.500 M $AgNO_3$ solution, using sodium chromate solution ($Na_2CrO_{4\,(aq)}$) as an indicator. At the equivalence point, it was found that 53.4 mL of $AgNO_3$ solution had been added.

   a) Calculate the moles of Ag^+ used.

   moles = M x Litres

   = 0.500 M x 0.0534 L

   = <u>0.0267 moles of Ag^+</u>

   b) Write the balanced net-ionic equation for the titration.

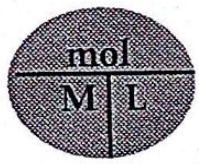
   $$Ag^+_{(aq)} + Cl^-_{(aq)} \rightarrow AgCl_{(s)}$$

   c) Determine the moles of Cl^- ions in the sample.

   From the ratio of coefficients in the balanced net-ionic equation:

   $$\text{moles of } Cl^- = \text{moles of } Ag^+ \times \frac{1 \text{ mol } Cl^-}{1 \text{ mol } Ag^+}$$

   = <u>0.0267 moles of Cl^-</u>



   d) Calculate the $[Cl^-]$ in the sample of pool water.

   $$M = \frac{\text{moles}}{L} = \frac{0.0267 \text{ moles}}{0.05 \text{ L}} = \underline{0.534 \text{ M}}$$

   Since the lowest number of significant digits in the question is 3, the answer would have three significant digits.

   so $[Cl^-]$ = <u>0.534 M</u>

Answer to **Question 2** on page 13 of Tutorial 12.

2. A solution containing silver ions (Ag^+) is titrated with 0.200 M KSCN solution to find the [Ag^+] in the sample. The indicator $Fe(NO_3)_{3\,(aq)}$ is used to signal when the equivalence point is reached. It is found that 15.6 mL of 0.200 M KSCN is needed to titrate a 25.0 mL sample of Ag^+ solution. Determine the [Ag^+] in the sample. Show all steps in a clear concise manner. (Use question 1 as a guide.)

moles of SCN^- used:

moles = M x L

= 0.200 M x 0.0156 L

= 0.00312 moles of SCN^-

Balanced Net-Ionic Equation:

$$Ag^+_{(aq)} + SCN^-_{(aq)} \rightarrow AgSCN_{(s)}$$

moles of Ag^+ in sample:

Since the ratio of coefficients is 1:1,

moles of Ag^+ = moles of SCN^- x $\frac{1 \text{ mol } Ag^+}{1 \text{ mol } SCN^-}$

= 0.00312 moles of Ag^+

Molar concentration of Ag^+ ([Ag^+])

$$M = \frac{moles}{L} = \frac{0.00312 \text{ moles}}{0.025 \text{ L}} = 0.1248 \text{ M}$$

The answer would be rounded to 3 significant digits, as this was the lowest number of significant digits in the data.

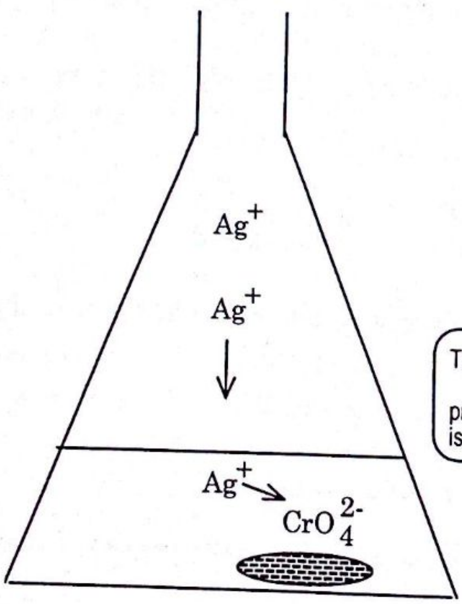
[Ag^+] = 0.125 M

Answer to **Question 3** on page 13 of Tutorial 12.

3. Explain how the indicator Na_2CrO_4 works in titrations for chloride (Cl^-) ion concentration using Ag^+ as a standard solution.

The Ag^+ ions will keep bonding with the Cl^- ions forming the white precipitate AgCl ***as long as there are Cl^- ions present.***

As soon as all the Cl^- ions are used up, the Ag^+ will then start precipitating with the CrO_4^{2-} ions, forming the precipitate Ag_2CrO_4. But recall from the last page that the colour of Ag_2CrO_4 is ***brick red***. Thus, as you can see, ***as soon as all the Cl^- ions are used up, the next drop of Ag^+ solution will turn the solution red.***



The Cl^- ions are now gone, so the only thing Ag^+ ions can precipitate with are the CrO_4^{2-} ions. The precipitate that forms is a brick red colour.

So, as soon as all the Cl^- is consumed, and a small amount of Ag_2CrO_4 forms, a faint brick red colour will be noticed. At this point, we would STOP the titration.

****

Answer to **Question 4** on Page 13 of Tutorial 12.

4. Explain how the indicator $Fe(NO_3)_3$ works in titrations for silver (Ag^+) ion concentration using SCN^- as a standard solution.

The main reaction for the titration is a precipitation of Ag^+ and SCN^- ions to form a precipitate of $AgSCN_{(s)}$:

$$\underset{\textit{colourless}}{Ag^+_{(aq)}} + \underset{\textit{colourless}}{SCN^-_{(aq)}} \rightarrow \underset{\textit{white precipitate}}{AgSCN_{(s)}}$$

Once just enough SCN^- solution has been added to react with all the Ag^+ ions, (the ***equivalence point***), any excess SCN^- ions added will react with the indicator, Fe^{3+} ions and form a complex ion (a larger ion made up of smaller ones) called $FeSCN^{2+}$. This ion, called the ferrothiocyanate ion, is NOT a precipitate, BUT is IS a very intense red colour. You may recall seeing it when you did Experiment 19-A on equilibrium. The reaction is:

$$\underset{\textit{pale rust}}{Fe^{3+}_{(aq)}} + \underset{\textit{colourless}}{SCN^-_{(aq)}} \rightarrow \underset{\textit{dark red}}{FeSCN^{2+}_{(aq)}}$$

A slight permanent red would appear at the *endpoint* of the titration. This would indicate that the *equivalence point* (the point where there is just enough SCN^- to react with all the Ag^+ in the sample) has been reached.

This is the end of Tutorial 12 - Solutions

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GRAVIMETRIC ANALYSIS PROBLEMS - EXERCISES IN STOICHIOMETRY

1. In the analysis of 0.7011 g of an impure chloride containing sample, 0.9805 g of AgCl were precipitated. What is the percentage by mass chloride in the sample?

$$m_{sample} = 0.7011\ g$$

$$m_{AgCl} = 0.9805\ g$$

$$m_{Cl} = \frac{0.9805\ g}{143.32\ g/mol} \times 35.453\ g/mol$$

$$= 0.2425\ g\ Cl$$

$$\%Cl = \frac{0.2425\ g\ Cl}{0.7011\ g} \times 100 = 34.59\%Cl$$

2. A 0.4054 g solid organic sample containing covalently bound bromide and no other halogens was placed in a porcelain crucible with about one gram of fresh sodium metal. In a process known as *sodium fusion*, the mixture was heated in a furnace to 450°C which charred and vaporized the organic portion of the molecule and converted the covalent bromide into sodium bromide. Excess sodium was decomposed by adding small portions of water which also dissolved the sodium bromide. The clear solution was quantitatively transferred to a beaker, acidified with dilute nitric acid, and diluted to about 50 mL. A 6 mL quantity of 0.1 M $AgNO_3$ was added to the solution and the mixture heated to about 60°C for an hour to age and digest the precipitate. After filtering, the mass of the silver bromide produced was determined to be 37.8 mg. What is the percentage by mass bromine in the organic compound?

$$m_{sample} = 0.4054\ g = 405.4\ mg$$

$$m_{AgBr} = 37.8\ mg$$

$$\%Br = \frac{\frac{37.8\ mg}{187.77\ g/mol} \times 79.904\ g/mol}{405.4\ mg} \times 100$$

$$= 3.97\%\ Br$$

3. A 1.1105 g sample of bauxite (the primary ore of aluminum) was analyzed for aluminum. The sample was pulverized and dissolved in concentrated nitric acid. The HNO_3 was removed by evaporation and solids dissolved in hot water with a very small quantity of nitric acid added. Insoluble solids were removed by gravity filtration. The solution was made basic by the slow addition of dilute NH_3 at which point a gelatinous solid precipitated ($Al(OH)_3$ and $Al_2O_3.x\ H_2O$). The precipitate was heated to coagulate it as much as possible then filtered over "fast" filter paper. The paper and solids were placed in a porcelain crucible and the paper ashed and precipitate "ignited" at 600°C to convert the precipitate to pure Al_2O_3. The mass of Al_2O_3 isolated was 0.3605 g. The average mass of ash remaining after charring 10 sheets of the identical filter paper was 0.0006 g. What is the percentage aluminum in the bauxite ore?

$$m_{bauxite} = 1.1105\ \text{g}$$
$$m_{Al_2O_3 w/ash} = 0.3605\ \text{g}$$
$$m_{ash} = 0.0006\ \text{g}$$
$$m_{Al_2O_3 w/o\ ash} = 0.3599\ \text{g}$$
$$\%Al = \frac{\frac{0.3599\ \text{g}}{101.96\ \text{g/mol}} \times \frac{2\ \text{Al}}{1\ \text{Al}_2\text{O}_3} \times 26.9815\ \text{g/mol}}{1.1105\ \text{g Bauxite}}$$
$$= 17.15\%\ \text{Al}$$

4. A 0.8870 g sample containing only NaCl and KCl was treated with $AgNO_3$. The AgCl formed had a mass of 1.913 g. Calculate the %Na and %K in the sample.

K

$$m_{sample} = 0.8870\ \text{g KCl / NaCl}$$
$$m_{AgCl} = 1.913\ \text{g}$$
$$M_{AgCl} = 143.32\ \text{g/mol} \quad M_{KCl} = 74.55\ \text{g/mol} \quad M_{NaCl} = 58.44\ \text{g/mol}$$
$$n_{AgCl} = 0.01335\ \text{mol}$$
$$m_{sample} = m_{KCl} + m_{NaCl}$$
$$m_{sample} = n_{KCl} \times M_{KCl} + n_{NaCl} \times M_{NaCl}$$
$$0.01335\ \text{mol} = n_{KCl} + n_{NaCl}$$
$$0.8870\ \text{g} = 74.55\ \text{g/mol} \cdot n_{KCl} + 58.44\ \text{g/mol} \cdot n_{NaCl}$$
$$0.8870\ \text{g} = 74.55\ \text{g/mol} \cdot n_{KCl} + 58.44\ \text{g/mol} \cdot (0.01335\ \text{mol} - n_{KCl})$$
$$0.8870\ \text{g} = 74.55\ \text{g/mol} \cdot n_{KCl} + 0.7802\ \text{g} - 58.44\ \text{g/mol} \cdot n_{KCl}$$
$$0.1068\ \text{g} = 16.11\ \text{g/mol} \cdot n_{KCl}$$
$$6.629 \times 10^{-3}\ \text{mol} = n_{KCl} = n_K$$
$$m_K = 6.629 \times 10^{-3}\ \text{mol} \times 39.098\ \text{g/mol} = 0.2592\ \text{g K}$$
$$\%K = \frac{0.2592\ \text{g K}}{0.8870\ \text{g}} \times 100 = 29.22\%\ \text{K}$$

Calculate the %Na in a similar fashion.

Problem Set 2a Precipitation Titrations and Gravimetric Analysis

1] What is pAg when 25.00-mL of 1.00e-2 M $AgNO_3$ is added to 25.00-mL of 1.00e-2M NaCl? K_{sp} (AgCl) = 1.8e-10 [1]

2] A solution of 0.100 M XNO_3 is used to titrate a 100.00 mL solution of 0.100 M KCl. The K_{sp} of XCl is 1.8e-11

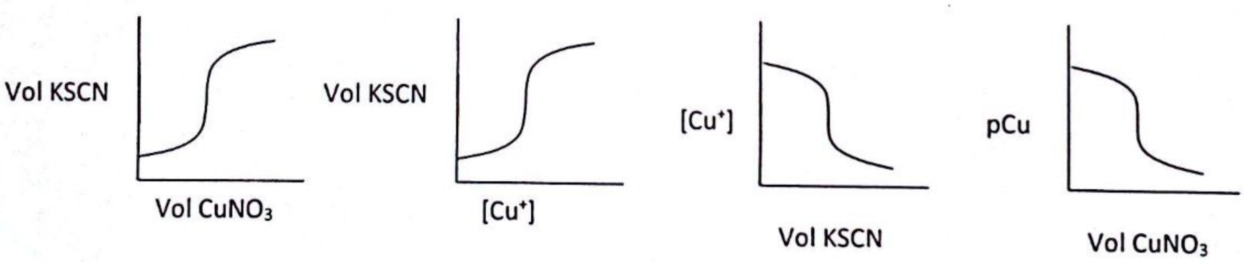
a) What is pX if 50.00 mL of the titrant is added to the KCl solution?

b) What is pX if 100.00 mL of the titrant is added?

c) What is pX if 150.00 mL of the titrant is added? [2]

3] A 0.9961 g silver ore sample was treated with HNO_3 and then with excess NaCl(aq). A precipitate was dried and weighed 0.0711 g. What is percent silver in the ore? AW: Ag 107.9, H 1.008, O 16.00, N 14.01, Cl 35.45 [3]

4] Which diagram best describes the curve for the titration of 50.0-mL of 0.100 M KSCN with 0.0500 M $CuNO_3$? The K_{sp} of CuSCN is 4.8e-15 [4]



I II III IV

5] A mixture of AgCl (MW 143.35, K_{sp}=1.8e-10) and AgBr (MW 187.9, K_{sp}=5.0e-13) weighs 2.000 g. This mixture is reduced to silver metal (AW 107.9), which weighs 1.300 g. Calculate the mass of AgCl in the original sample. [5]

6] What is the concentration Cl^- required to remove 99% of Ag^+ in a solution of 0.100 F $AgNO_3$? [6]

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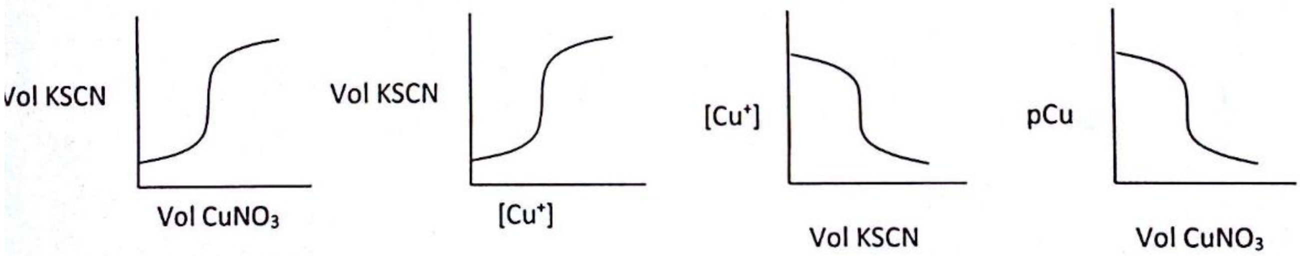

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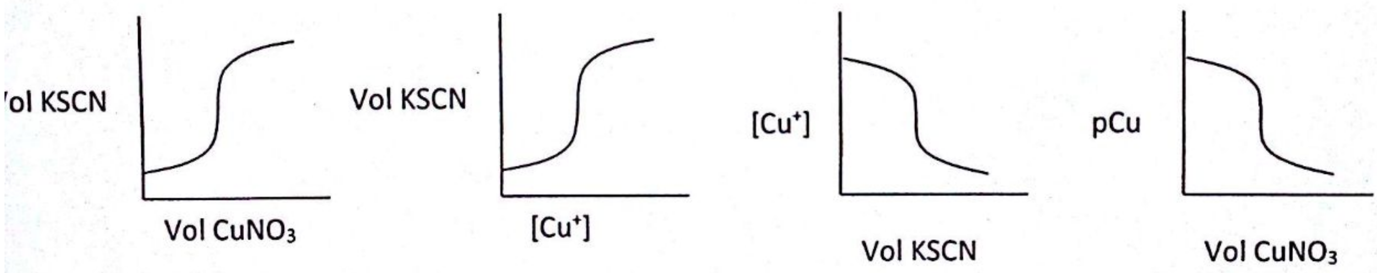

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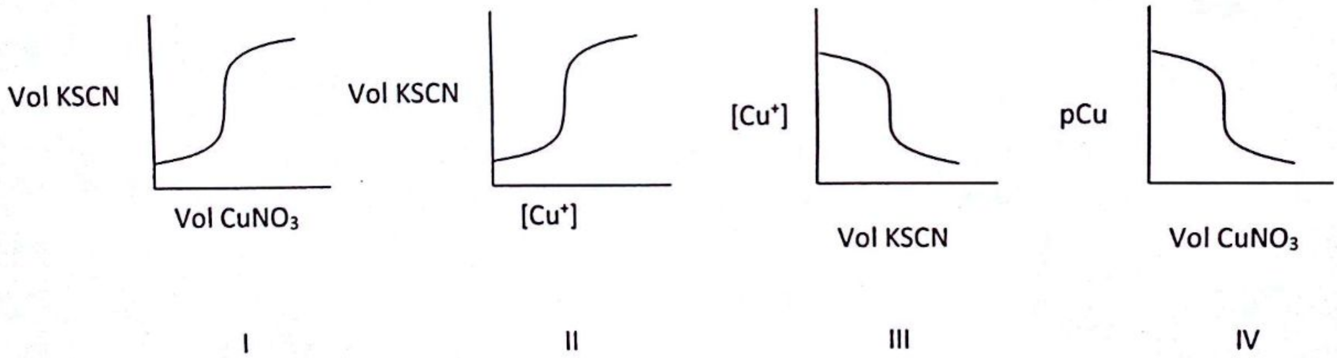

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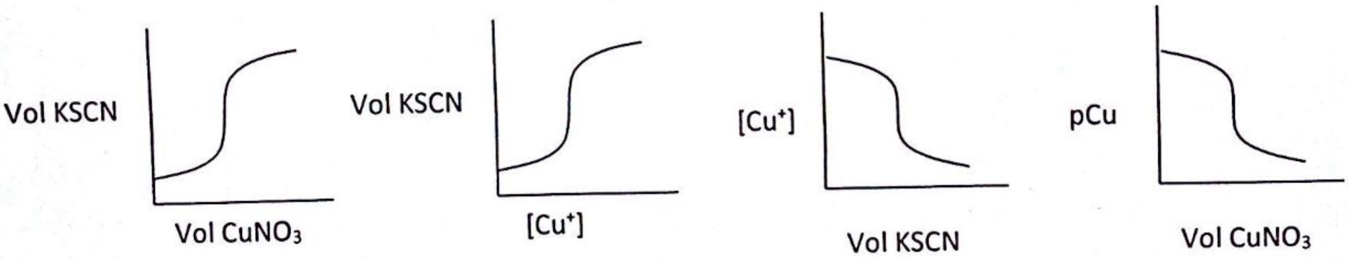

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---

x g AgCl*(mol AgCl/143.35 g)*(mol Ag/mol AgCl)*(107.9 g/mol) = 0.7527x g Ag

y g AgCl*(mol AgBr/187.9 g)*(mol Ag/mol AgBr)*(107.9 g/mol) = 0.5742y g Ag

0.7527x g Ag + 0.5742y g Ag = 1.300 g

y = 2.000 – x   sub into above

0.7527x + 0.5742(2.000 – x) = 1.300 g

mass Ag = 0.849 g

[6] $[Ag^+]$ = (1-0.99) 0.100 F = 1.00e-3 M $K_{sp} = [Ag^+][Cl^-]$ = 1.8e-10 = 1.00e-3 M*$[Cl^-]$ → $[Cl^-]$ = 1.80e-7 M

********** 2^ND SECTION (PART II) **********

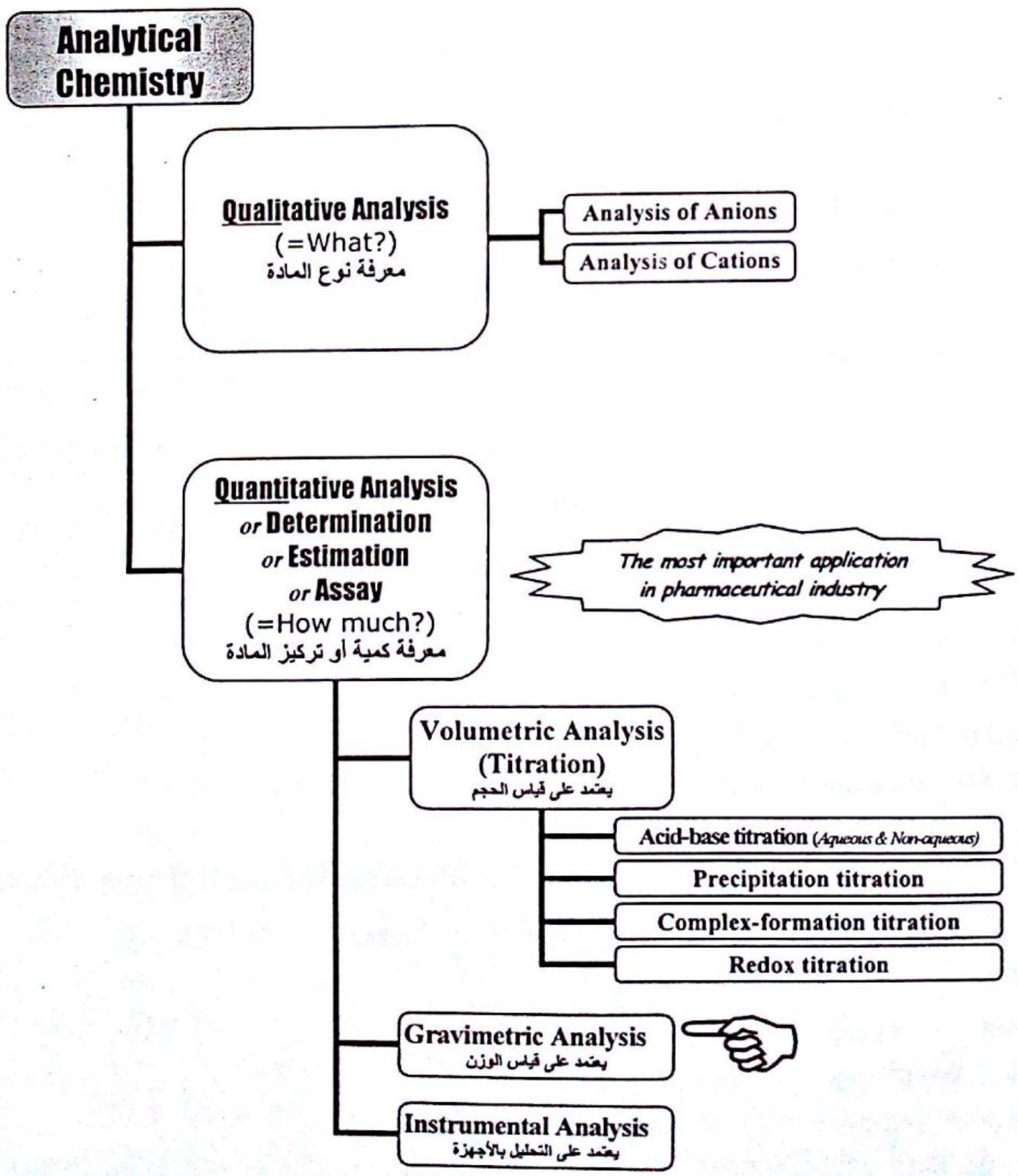
Gravimetric Analysis

Definition of Gravimetric Analysis:

It is a type of quantitative analysis that involves weighing of the constituent under determination. IN OTHER WORDS, It is the process of isolating and weighing an element or compound in a pure form.

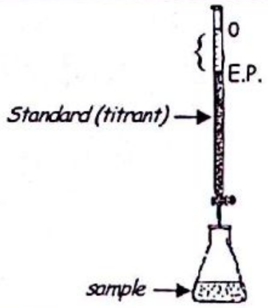
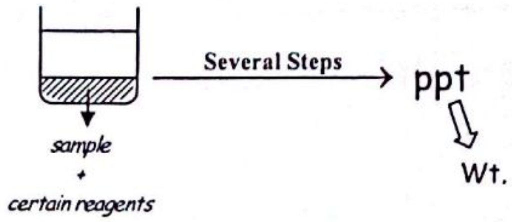
⇒ ***Remember:***

Classification of Analytical Chemistry



Comparison between

Volumetric Analysis (Titration) & Gravimetric Analysis

	Volumetric Analysis (Titration)	Gravimetric Analysis
Diagram:		
Sample:	Liquid or Solid	Liquid or Solid
Result obtained:	E.P. (Vol. consumed from titrant) <u>Ex</u>: 4 ml	Weight of the ppt <u>Ex</u>: 4 g
Required:	Concentration of the sample	Concentration of the sample
Steps of Calculations:	$\boxed{1^{st}}$: Calc. of **equivalence** factor (F). $\boxed{2^{nd}}$: Calc. of Concentration.	$\boxed{1^{st}}$: Calc. of **gravimetric** factor (F). $\boxed{2^{nd}}$: Calc. of Concentration.

Examples of gravimetric determinations :

1) Gravimetric determination of NaCl:

$$NaCl + AgNO_3 \longrightarrow \underset{\boxed{ppt}}{AgCl\downarrow} + NaNO_3$$

filtered, dried and weighed and then from the weight of the ppt, the concentration of NaCl sample can be calculated.

2) Gravimetric determination of Potash alum [$KAl(SO_4)_2.12H_2O$]:

$$2\ KAl(SO_4)_2.12H_2O \longrightarrow \underset{\boxed{gelatinous\ ppt}}{2\ Al(OH)_3\downarrow} \xrightarrow{ignition} \underset{\boxed{ppt}}{Al_2O_3\downarrow}$$

$2\ Al(OH)_3$: Not suitable for weighing.

Al_2O_3: More suitable for weighing & from its weight, the concentration of Potash alum sample can be calculated.

Calculations for Gravimetric Analysis { 2 STEPS }:

1st step: Calculation of the Gravimetric Factor OR Conversion Factor (F):

Gravimetric Factor is the weight of the sample equivalent 1 g of the ppt.

$$F = \frac{m \times (\text{M.W. of substance } \overbrace{\text{sought}}^{\text{sample}})}{n \times (\text{M.W. of substance } \underbrace{\text{weighed}}_{\text{final ppt}})}$$

No Unit

m & n are integers that make the molecular weights in the numerator (البسط) & denominator (المقام) chemically equivalent.

N.B. Gravimetric factor (F) is completely different from Equivalence factor (F).

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2nd step: Calculation of the Concentration:

If the sample is liquid (solution):

$$\text{Concn.} = \frac{\text{Wt. of the ppt} \times F \times 1000}{\text{Volume taken from the sample}} = \text{.......... g/L}$$

$$= \frac{\text{Wt. of the ppt} \times F \times 100}{\text{Volume taken from the sample}} = \text{.......... g\% (i.e. g/100 ml)}$$

If the sample is solid (powder):

$$\text{Concn. or Purity} = \frac{\text{Wt. of the ppt} \times F \times 100}{\text{Wt. taken from the sample}} = \text{.......... g\% (i.e. g/100 g)}$$

Example: *In the determination of $MgSO_4.7H_2O$:*

The reaction can be summarized as follows:

$$\underset{\boxed{sample}}{2\ MgSO_4.7H_2O} \longrightarrow \underset{\boxed{Mag.\ amm.\ phosphate}}{2\ MgNH_4PO_4.6H_2O \downarrow} \xrightarrow{ignition} \underset{\boxed{Mag.pyrophosphate}}{\overset{\underline{final\ ppt}}{Mg_2P_2O_7 \downarrow}}$$

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$$\underset{m}{2}\ \underset{sample}{(MgSO_4.7H_2O)} \equiv \underset{n}{1}\ \underset{final\ ppt}{(Mg_2P_2O_7)}$$

$$F = \frac{2 \times (M.W.\ of\ MgSO_4.7H_2O)}{1 \times\ (M.W.\ of\ Mg_2P_2O_7)}$$

Then the concentration can be calculated from one of the above mentioned equations:

$$Concn. = \frac{Wt.\ of\ the\ ppt \times F \times 1000}{Volume\ taken\ from\ the\ sample} = \text{..........}\ g/L$$

Derivation of Gravimetric Factor (F):

Example: *In the determination of $MgSO_4.7H_2O$:*

$$\underset{sample}{2\ (MgSO_4.7H_2O)} \longrightarrow \underset{final\ ppt}{1\ (Mg_2P_2O_7)}$$

The Gravimetric Factor (F) means:

the weight of the sample ($MgSO_4.7H_2O$) equivalent $\boxed{1\ g}$ of the ppt ($Mg_2P_2O_7$).

$$2\ moles\ of\ MgSO_4.7H_2O \longrightarrow 1\ mole\ of\ Mg_2P_2O_7$$

i.e. $2 \times (M.W.)_g$ of $MgSO_4.7H_2O \longrightarrow 1 \times (M.W.)_g$ of $Mg_2P_2O_7$

$$\therefore\ ?\ g\ of\ MgSO_4.7H_2O \longrightarrow \boxed{1\ g}\ of\ Mg_2P_2O_7$$

$$\therefore\ F = 1 \times \frac{2 \times (M.W.\ of\ MgSO_4.7H_2O)}{1 \times\ (M.W.\ of\ Mg_2P_2O_7)}$$

$$\therefore\ F = \frac{2 \times (M.W.\ of\ MgSO_4.7H_2O) \longrightarrow \text{sample}}{1 \times\ (M.W.\ of\ Mg_2P_2O_7) \longrightarrow \text{final ppt}}$$

Basic Steps for Any Gravimetric Analysis

1. Sampling & Preparation for precipitation.

2. Precipitation.

3. Digestion (i.e. Leaving the ppt in contact with the soln. for a considerable time).

4. Filtration & Washing.

5. Drying OR Ignition.

6. Weighing & Calculations.

Required Glassware for Gravimetric Analysis (الأدوات المطلوبة):

- Beaker 250 ml.
- 10-ml bulb pipette.
- 10-ml graduated pipette.
- 10-ml measuring cylinder.
- Filter papers (Whatmann no.1).
- Funnel.
- Flask.
- Watch glass.
- Test tube.

Exp.(8): Gravimetric Determination of $Na_2SO_4.10\ H_2O$ (USP 2007)

Principle :

It depends on the precipitation of SO_4^{--} as $BaSO_4$ by adding $BaCl_2$ to a hot solution of SO_4^{--} slightly acidified with HCl.

$$\underset{sample}{SO_4^{--}} + \underset{BaCl_2\ (ppting\ agent)}{Ba^{2+}} \longrightarrow \underset{white\ ppt}{BaSO_4 \downarrow}$$

Procedure :

Sampling & Preparation for Precipitation

1- Transfer 10 ml of the sample of sodium sulphate into a 250 ml beaker, Dilute to about 150 ml with water.

2- Add about 10 ml dilute HCl, and heat to boiling.

Precipitation

3- Remove the flame, and add slowly with constant stirring (*by glass rod*), a hot 2 % $BaCl_2$ solution in slight excess (*about 10 ml heated in a test tube)*.

4- **Test for complete precipitation** by leaving the precipitate to settle, and then adding to the clear supernatant liquid, drops of the precipitating agent (2% $BaCl_2$), noting if any turbidity appears. *If turbidity appears* → add more drops of $BaCl_2$ soln. and repeat the test.

If no turbidity → complete the next steps.

Digestion

5- Cover the beaker with a watch glass, and place it in a water bath almost at the boiling point for about one hour.

Filtration & Washing

6- Decant the supernatant liquid in a suitable ashless filter paper and wash the precipitate in the beaker by decantation two times with hot water.

Decantation means: *Pouring off the clear upper portion (supernatant) of a fluid leaving a sediment or a ppt at the bottom WHILE* ***Washing by Decantation means:*** *mixing some of the washing solution with the ppt, then allow the ppt to settle down again and then the clear supernatant is decanted.*

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Repeat washing & filtration until all the ppt is filtered

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Drying → **Weighing & Calculations**

Calculations :

$$\underset{\text{sample}}{\underset{m}{1}\,(Na_2SO_4.10H_2O)} \equiv \underset{\text{ppt}}{\underset{n}{1}\,(BaSO_4)}$$

$$F = \frac{1 \times (\text{M.W. of } Na_2SO_4.10H_2O)}{1 \times (BaSO_4)}$$

$$\text{Concn.} = \frac{\text{Wt. of the ppt} \times F \times 1000}{\text{Volume taken from the sample}} = \text{.........} \text{ g/L}$$

***** >> Best wishes >> *****