

Ques.Assignment - 1

Q1 Define alkalinity. In what terms is alkalinity reported.

Alkalinity is way to measure the acid neutralizing capacity of water or we can say that it is the ability of water to maintain relative constant pH. Alkalinity is due to the presence of sufficient alkaline ions in water.

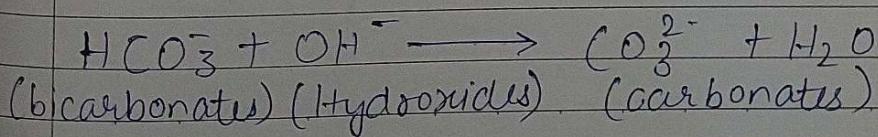
Alkalinity is reported in terms of moles per litre (mol/l)

Q2 Why can natural water be sometimes alkaline?

Ans Sometimes natural water can also be alkaline. This is primarily because of the presence of salts of weak acids and bicarbonates in the water.

Q3 Explain why hydroxides and bicarbonates do not exist together in a given water sample.

Ans In a given sample of water hydroxides (OH^-) and bicarbonates (HCO_3^-) cannot exist together because they combine they combine with each other to form respective carbonates.



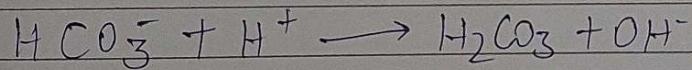
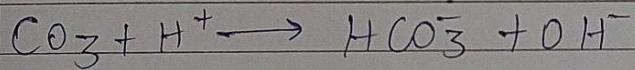


Q4 Write and explain the complete reactions involved in

i) Alkalinity due to carbonate and bicarbonate

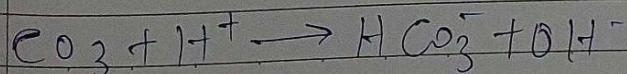
Carbonate ion reacts with the hydronium ion that is formed by the dissociation of water to form bicarbonates and releasing one hydroxide ion.

Further, bicarbonates react again with the hydronium ion by water to form carbonic acid thus releasing one more hydroxide ion which contributes to the alkalinity of the water

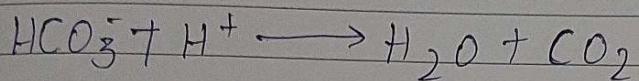


ii) Titration of HCl v/s carbonate in water

Titration of HCl and water sample with carbonate ion is carried out in two steps. First phenolphthalein is added in the water sample and titration is carried out to form bicarbonates and the end point of the titration will be pink to colourless.

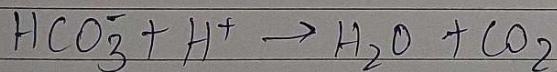


In the later half, we add methyl orange to the water sample to neutralize bicarbonates into water and bicarbonates an carbon dioxide and the end point of the titration will be yellow to bright red.



iii) Titration of HCl v/s bicarbonate in water.

Titration of HCl and water sample with bicarbonate ion is carried out by adding few drops of methyl orange in the water sample and titration is carried out to form water and carbon dioxide and end point of titration will be yellow to bright red

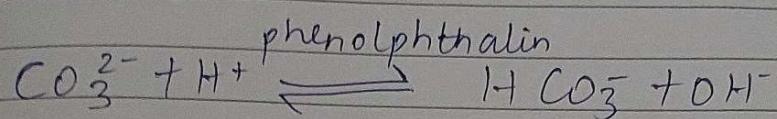


Q5 Why are two indicators and not a single indicator used for determining the alkalinity

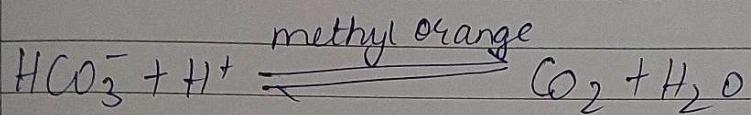
Two indicators are used for determining of the alkalinity instead of one because of the different end parts of the carbonate ion.

In the phase 1 the carbonate ions are titrated to bicarbonate ions and the titration is confirmed

by colour change because of phenolphthalein indicator



In phase II, bicarbonate ions are titrated to carbon dioxide in the presence of methyl orange indicator.



Experiment - I

Aim

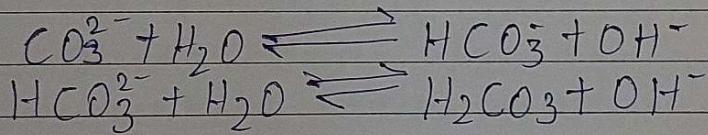
Determine the alkalinity of the given unknown water sample using the double indicator method.

Material Required

Volumetric flasks (100mL), Burette (50 mL), Pipette (10 mL), Weighing bottle, conical flask (100mL), funnel, HCl solutions, sodium carbonate solution, unknown water sample, Phenolphthalein, Methyl orange.

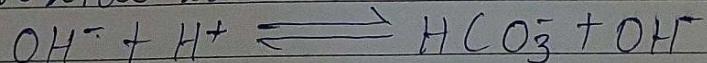
Theory

Alkalinity is a measure of the ability of a water body to neutralize acidity, carbonate and bicarbonate ion cause alkalinity due to hydrolysis as per following equations

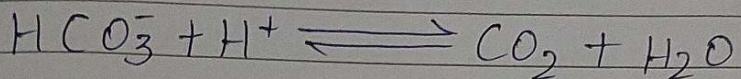
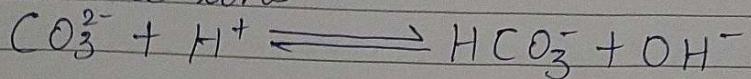


Double indicator method for the determination of the alkalinity of water explains the titration behaviour of the alkaline ions with HCl: Reactions of OH^- and CO_3^{2-} with a monoprotic acid like HCl can be represented as:

hydroxide ions :



Carbonate ions



In a water sample containing carbonate ions titrate as strong base and in the second half of neutralization titrate as weak base.

Procedure

a) Preparation of standard solution of sodium carbonate (100mL)

The required amount of dry anhydrous sodium carbonate was weighed in a weighing bottle. Sodium carbonate was transferred to a clean 100mL volumetric flask through a funnel. The weighing bottle was again weighed after transferring sodium carbonate. The exact mass of sodium carbonate transferred was found by subtracting this mass from the mass of the weighing bottle plus sodium carbonate. The solution was prepared by dissolving sodium carbonate in distilled water in the volumetric flask and made up to the volume mark.

b) Standardisation of hydrochloric acid solution by titration against standard sodium carbonate solution

Fill up the burette with hydrochloric acid solution with the help of a funnel and mount it on the burette stand. Note the reading on the burette as the initial reading. Carefully pipette out 10 mL of the standard sodium carbonate solution and transfer to a clean 100 mL conical flask. Add 1-2 drops of methyl orange indicator. Titrate the yellow coloured solution with constant swirling against a white tile until a red colour is obtained.

Note the burette reading as the final reading. Repeat the titration to get at least two readings.

c) Determination of PE by titration against standardised hydrochloric acid

Pipette 20 mL of the water sample into a conical flask; add 1 drop of phenolphthalein indicator and titrate the sample with the standardised HCl solution till colourless.

Record the initial and final readings. Repeat the titration to get at-least two concordant readings.

d) Determination of ME by titration against standardised hydrochloric acid.

Pipette 20 mL of the water sample into a

conical flask, add 1 drop of phenolphthalein indicator and titrate the sample with the standardized HCl solution till colourless. Now, add 1-2 drops of methyl orange indicator and titrate standardized HCl solution till a persistent red colour is observed. Record the initial and final burette readings. Repeat the titration to get at least two concordant readings.

Observations

i) Preparation of standard solution of sodium carbonate

Mass of weighing bottle + anhydrous sodium carbonate

$$= m_1 g = \text{ } g$$

Mass of weighing bottle (after transferring the salt.)

$$= m_2 g = \text{ } g$$

Amount of sodium carbonate transferred

$$= (m_1 - m_2) g = mg$$

=

Molar mass ($M_{\text{Na}_2\text{CO}_3}$) of sodium carbonate
 $= 106 \text{ g mol}^{-1}$

Volume of sodium carbonate prepared = 100 cm^3

Molarity of sodium carbonate solution =

$$M_{\text{Na}_2\text{CO}_3} = \frac{m}{106} \times \frac{1000}{100} = \frac{10m}{106} = M$$

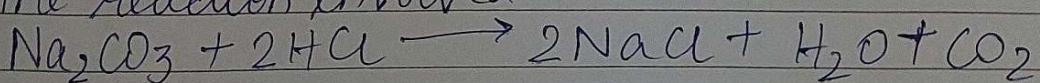
- b) Standardisation of hydrochloric acid solution by titrating against standard sodium carbonate

Volume of standard Na_2CO_3 solution taken in conical flask, V_N = cm solution in the burette : HCl

Indicator: Methyl Orange

Observation

- a) The concentration of the given hydrochloric acid solution can be determined as follows.
- b) The reaction involved.



Molarity eqⁿ: $2M_n V_N = M_H V_H$

$$M_H = \frac{2M_n V_N}{V_H}$$

c) Determination of phenolphthalein end point P_E by titrating against standardized hydrochloric acid.

Volume of given sample of water taken in conical flask, $V_{N^1} = \text{cm}^3$

Solution in the burette: HCl.

Indicator used: Phenolphthalein.

d) Determination of methyl orange end point M_E by titrating against standardised hydrochloric acid.

Volume of given sample of water taken in conical flask, $V_{N^2} = \text{cm}^3$

solution of the burette: HCl

Indicator used: Methyl orange

Calculations

Hydroxide alkalinity

$$M_{\text{hydroxide}} \times V_{\text{water}} = M_{\text{HCl}} \times V_{\text{HCl}}$$

One mole of carbonate ions consume 2 moles of HCl whereas 1 mole of hydroxide ions consume 1 mole of HCl. The carbonate equivalent of hydroxide alkalinity would be 50% of the hydroxide alkalinity.

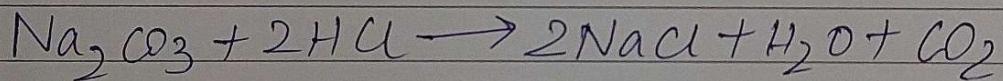
$$M_{\text{carbonate}} = M_{\text{hydroxide}} / 2$$

Hydroxide alkalinity (Equivalent of CaCO_3) -

$$= M_{\text{hydroxide}} / 2 \times 100 (\text{g/mol}) \times 1000 (\text{mg/g}) \\ = \text{mg/dm}^3$$

$$\begin{aligned} \text{Hydroxide alkalinity} &= M_{\text{hydroxide}} \times 50000 \\ &= \text{mg/dm}^3 \\ &= \text{ppm} \cdot \text{CaCO}_3 \end{aligned}$$

Carbonate alkalinity
The reaction involved. :



Molarity equations : $2M_{\text{carbonate}} V_{\text{carbonate}} = M_{\text{HCl}} V_{\text{HCl}}$

Carbonate alkalinity expressed in terms of milligrams of calcium carbonate per litre can be calculated as under:

$$\begin{aligned} \text{Carbonate alkalinity} &= M_{\text{carbonate}} \times 100 (\text{g/mol}) \times \\ &\quad 1000 (\text{mg/g}) \\ &= \text{mg/dm}^3 \end{aligned}$$

$$\begin{aligned}\text{Carbonate alkalinity} &= M_{\text{carbonate}} \times 100,000 \\ &= \text{mg/dm}^3 \\ &= \text{ppm}\end{aligned}$$

Total alkalinity

$$\begin{aligned}\text{Total alkalinity} &= \text{Hydroxide alkalinity} + \\ &\quad \text{Carbonate alkalinity}\end{aligned}$$

$$\begin{aligned}&= [M_{\text{carbonate}} \times 100000) + (M_{\text{hydroxide}} \times 50000)] \\ &= [(\text{ } \times 100000) + (\text{ } \times 50000)] \\ &= \text{ } \text{mg}\end{aligned}$$

Results

- i) Hydroxide alkalinity = mg of CaCO_3 = ppm
- ii) Carbonate alkalinity = mg of CaCO_3 = ppm
- iii) Total alkalinity = mg of CaCO_3 = ppm

Inference

Alkalinity is a measure of the capacity of water to neutralize acids. The predominant chemical system present in natural water is one where carbonates, bicarbonates and hydroxides are present. The bicarbonate

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ion is usually prevalent. However, the ratio of their ions is functions of pH; mineral composition, temperature and ionic strength. Water may have low alkalinity but relatively high pH or vice versa, so alkalinity alone is not a major importance as a measure of water quality. Alkalinity is not considered detrimental to humans but is generally associated with high pH values; hardness and excess dissolved solids. High alkalinity water also have a distinctly flat, unpleasant taste.

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Assignment 2

Q1 What is the denticity of EDTA? In what ratio is the complex EDTA-metall ion complex formed?

Ans Denticity of EDTA is 6 it contains 4 O atoms and 2 N atoms. The EDTA ligand is a hexadentate ligand. This means that there are 6 lone pairs capable of participating in coordinate bonding.

A metal ion/atom (M) capable of forming a 6 valent coordination complex will react with one EDTA molecule

Hence the ratio of moles of metal to EDTA in a complex is 1:1

Q2 What is the colour of doubly ionized Enochrome Black T indicator in slightly basic solution?

Ans Enchrome Black (EBT) T are such indicators that change from blue to pink when they complex with calcium or magnesium. The endpoint of a complexometric EDTA titration using either Calmagite or EBT as the indicator is detected as the colour changes from pink to blue

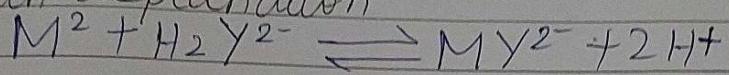
Q3 What is the purpose of adding buffer in complexometric titration.

Ans A buffer solution is used in EDTA titration because it resists the change in pH. This is because all the reactions between the metal ions and EDTA are pH-dependent.

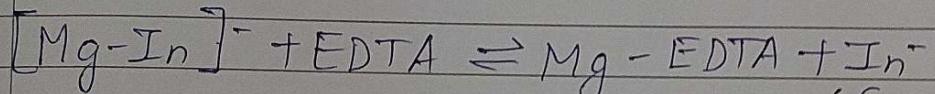
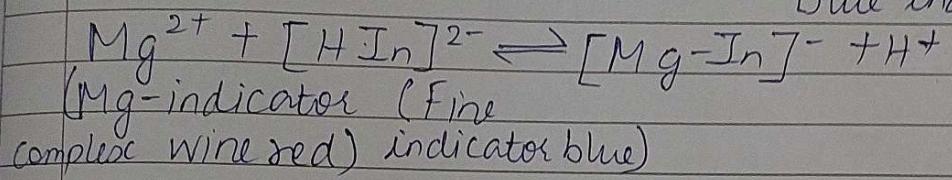
Q4 Is it possible to use the sodium salt of EDTA as a primary standard? Give reasons.

Ans The hydrated salt of sodium of EDTA; $\text{Na}_2\text{H}_2\text{Y} \cdot 2\text{H}_2\text{O}$ can't be used as a primary standard for titrations due to uncertainties in the water content. An alklimetric titration of the homogenized solid in the presence of excess of $\text{BaCl}_2 \cdot \text{H}_2\text{O}$ allows one to titrate quantitatively the released two hydrogen cations with end-point indication by phenolphthalein or potentiometry. This leads one to calculate the molar mass of the reagent and its water content, allowing to use it to prepare EDTA standard solutions. One titrated sample led to the formula $\text{Na}_2\text{H}_2\text{Y} \cdot 1.876 \text{H}_2\text{O}$ and 370.01 g/mol for the average molar mass.

Q5 Write the reaction involved in this titration with explanation

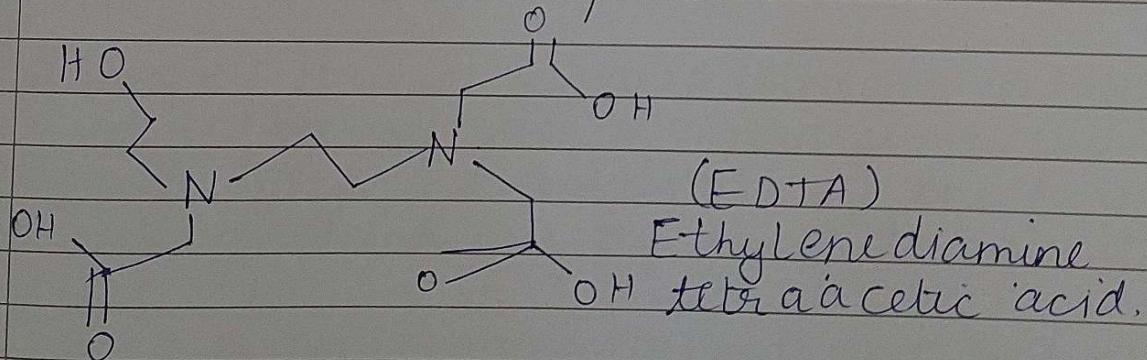


Blue indicator



(Free indicator blue)

Q6 Draw the structure of EDTA and indicate sites at which the metal is present.



Experiment - 2

Aim → Estimation of $MgSO_4 \cdot 7H_2O$ by complexometric titration

Requirements

Ammonia solution, Ammonium chloride, 400 ml beaker, distilled water, $MgSO_4 \cdot 7H_2O$ volumetric flask, funnel, burette, conical flask, weighing bottle, EDTA solution, pipette, measuring cylinder, dropper, unknown solution and Eriochrome Black-T.

Theory

Complexometric titration involves the formation of a stable, soluble and stoichiometric complex between a metal ion and the complexing agent to give a neutral compound.

A complex is usually formed between a central metal cation and anion or a neutral molecule called ligand. The ligand contain at least one lone pair of electron that they can donate to the central metal atom.

The no of coordination bonds and their arrangement in space decides the coordination number of the central metal.

Complexometric titration is a form of volumetric analysis in which the formation of a coloured complex is used to indicate the end point of a titration

A metal ion indicator capable of producing colour change is usually used to detect the end point of the titration.

These titrations are particularly useful for the determination of metal ion or mixture of different metal ions in the solution.

Procedure

Preparation of Buffer solution of $\text{NH}_3\text{-NH}_4\text{Cl}$ ($\text{pH} = 10$)
 It is prepared by adding 142 mL concentrated NH_3 solution to 17.5 g NH_4Cl and diluting to 250 mL using distilled water.

Preparation of standard $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ solution
 The required amount of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ was weighed in a weighing bottle and transferred to a clean 100 mL volumetric flask through a funnel. The weighing bottle was given weight after transferring $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. The exact mass of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ transferred was found by subtracting this mass from the mass of the weighing bottle plus $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$. The solution was prepared by dissolving $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water in the volumetric flask, and made up to the volume mark.

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Standardization of EDTA solution by titrating against standard $MgSO_4 \cdot 7H_2O$ solution

The burette was filled up with EDTA solution with the help of a funnel and mounted on a burette stand. The reading on the burette was noted as the initial reading 10mL of the standard $MgSO_4 \cdot 7H_2O$ solution was pipetted out transferred to a clean 100mL conical flask. 5mL ammonia-ammonium chloride buffer was added, followed by a pinch of EBT indicator. The wine-red colour solution was titrated versus EDTA with constant swirling against a white tile until a blue colour was obtained. The wine red coloured solution was titrated versus EDTA with constant swirling against a white tile until a blue colour was obtained. The final burette reading was noted. The titration was repeated to get at least two concordant readings.

Determination of strength of given unknown $MgSO_4 \cdot 7H_2O$ solution

The above titration was repeated with the given unknown $MgSO_4 \cdot 7H_2O$ solution. The strength was reported in g/L.

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Inference

We estimate presence of $MgSO_4 \cdot 7H_2O$ by complexometric titration

Basic principle of complexometric titration are these reactions where a simple ion is transferred into a complex ion and the equivalence point is determined by using metal ion indicators or electrometrically.

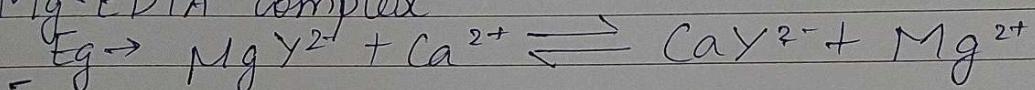
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Assignment - 3

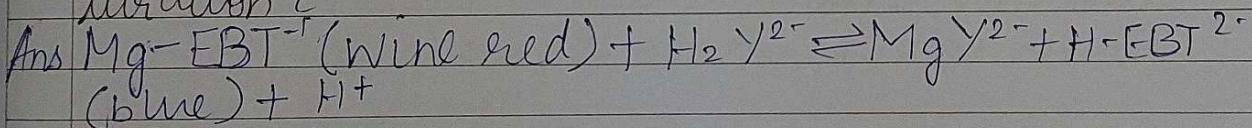
Q1 Define displacement titration with an example.

In displacement titration, an unmeasured excess of a solution containing the magnesium or zinc complex of EDTA is introduced into the analyte solution.

Thus on the addition of Mg-EDTA to Ca(II) solution, the displacement reaction given below at Ca-EDTA complex is more stable than the Mg-EDTA complex



Q2 Write and explain the reaction involved in this titration?



Explanation:

At the end point. When all the Mg(II) ions are displaced from the Mg-EBT complex, the colour of the solution changes from wine red to blue.

Q3 During the Ca(II) vs EDTA titration, what would happen if the following reagents are not added to the Ca(II) solution.

a) Ammonium-ammonium chloride buffer.

If Ammonium chloride buffer is not added to the solution then the buffer does not adjust the pH

value and it doesn't ensure that the reaction is going towards the completion.

b) Mg-EDTA solution
 $\text{Sd}^{\text{r}} \text{ Ca-(EDTA)} > \text{Mg-(EDTA)}$

As a consequence significant conversion of Ca-In (red) to MnIn^{2-} (blue) occurs before equivalence if the colour change occurs prematurely in the titration of Ca^{2+} with EDTA because Ca^{2+} ions have a large tendency to form Ca-EDTA complex. Thus with Ca^{2+} ions alone no sharp end point can be obtained and the transition from red to blue is not observed. Thus, when Ca^{2+} ions are titrated with EDTA a relatively stable calcium complex is formed. Whereas with Mg(II) ions, a somewhat less stable complex is formed.

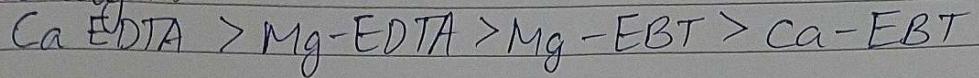
Enichrome Black-T

If EBT is not added to the solution then it doesn't give the blue end-point

Q4 Ca(II) displaces Mg(II) from the Mg-EDTA complex but does not displace Mg(II) from Mg-EBT complex. Why?

Ans The Mg-EBT complex is more-stable than the Ca-EBT complex, while the reverse is true for their EDTA complexes, so the order of

Stability is:



Ca^{2+} ions can be determined by displacement titration. Thus, if $\text{Mg}(\text{II})$ ions are not present in the solution containing $\text{Ca}(\text{II})$ ions they must be added since they are required for the colour change of the indicator.

Q5 Why is standardisation of EDTA important?

Ans EDTA cannot be used as a primary standard, because it is not highly pure and has limited solubility in water. Therefore, the solution of EDTA has to be standardized against some accepted primary standard.

Example - ZnSO_4 , MgSO_4 , MgCl_2 , ZnCl_2 , etc.

Experiment -3

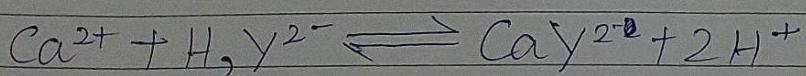
Aim-Determine the strength (in g/L) of CaCO_3 in a given unknown solution using the displacement titration method versus EDTA.

Requirements

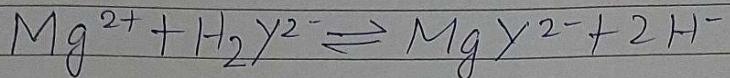
Volumetric flasks (100mL), Burette (50mL), Pipette (10mL), Weighing bottle, Conical flask (100 mL), funnel, EDTA solution, 0.1M Mg-EDTA complex solution, CaCO_3 unknown Solution, ammonia-ammonium chloride buffer ($\text{pH}=10$), Eriochrome Black T (EBT) indicator

Theory

- $\text{Ca-In} < \text{Mg-In}$ (In=indicator)
- $\text{Ca-EDTA} > \text{Mg-EDTA}$
- As a consequence, significant conversion of Ca-In (red) to HIn^{2+} (blue) occurs permanently in the titration of Ca^{2+} with EDTA because Ca^{2+} ions has a large tendency from Ca-EDTA complex. Thus with Ca^{2+} ions alone no sharp point can be obtained and the transition from red to blue is not observed. Thus, when Ca^{2+} ions are titrated with EDTA, a relatively stable calcium complex is formed

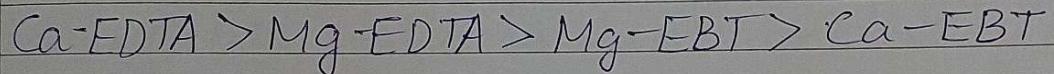


- * Whereas with Mg(II) ions, a somewhat less stable complex is formed.

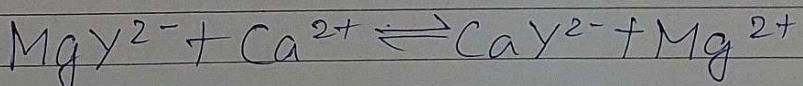


- * The Mg-EBT is more stable than the Ca-EBT complex, while the reverse is the for their EDTA complexes.

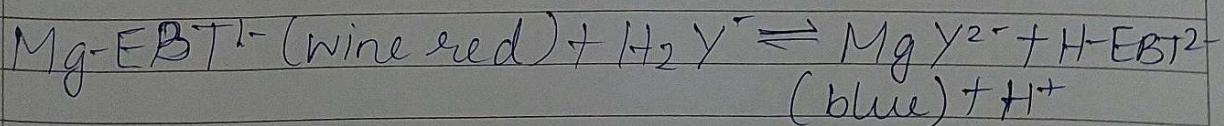
So the order of stability is



- * On addition of Mg-EDTA to Ca(II) solution, the displacement reaction given below occurs because the Ca-EDTA complex is more stable than the Mg-EDTA complex



- * At the end point, when all the Mg(II) ions are displaced from the Mg-EBT complex, the colour of the solution changes from wine red to blue



Procedure

a) Preparation of standard $MgSO_4 \cdot 7H_2O$ solution
 The required amount of $MgSO_4 \cdot 7H_2O$ was weighed in a weighing bottle and transferred to a clean 100mL volumetric flask through a funnel. The weighing bottle was again weighed after transferring $MgSO_4 \cdot 7H_2O$.

The exact mass of $MgSO_4 \cdot 7H_2O$ transferred was found by subtracting this mass from the mass of the weighing bottle plus $MgSO_4 \cdot 7H_2O$.

The solution was prepared by dissolving $MgSO_4 \cdot 7H_2O$ in distilled water in the volumetric flask and made up to the volume mark.

b) Standardization of EDTA solution by titrating agent standard $MgSO_4 \cdot 7H_2O$ solution.

The burette was filled up with EDTA solution with the help of a funnel and mounted on a burette. The initial reading 10mL of the standard $MgSO_4 \cdot 7H_2O$ solution was pipetted out and transferred to a clean 100mL conical flask. 5mL ammonia - ammonium chloride buffer was added, followed by a pinch of EBT indicator. The wine red coloured solution was titrated versus EDTA with constant swirling against a white tile until a blue colour was obtained. The final burette reading was noted. The titration was repeated.

to get at least two concordant readings.

3) Determination of strength of given unknown CaCO_3 solution via displacement titration

10 mL of the given unknown CaCO_3 solution was pipetted out in a conical flask. To this, 2 mL ammonia-ammonium chloride buffer was added. Then, 1 mL of 0.1M Mg-EDTA solution was added, followed by a pinch of EBT indicator to obtain a wine-red coloured solution. The initial burette reading was noted. The solution in the conical flask titrated versus standardized EDTA with constant swirling against a white tile until a blue red point was obtained. The final burette reading was noted. The titration was repeated to get at least two concordant readings. The strength of the CaCO_3 solution was calculated and reported in g/L.

Inference

- We estimate the strength of CaCO_3 by displacement titration.
- Basic principle of displacement titration is the analyte displaces a reagent usually from a complex and the amount of displaced reagent is determined by it.

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Assignment 4

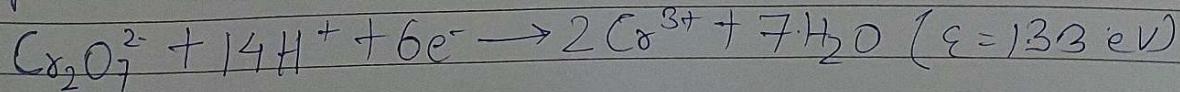
Q1 What is double salt? Give examples.

Double salt is a salt that contains more than one different cation or anion. Many coordination complexes form double salt. It only exist in the solid. When dissolved, it acts as a mixture of the two separate salts. It completely dissociates into simple ions.

Examples: Tutton's salt, potassium sodium tartarate
Mohr's salt, Bromide etc

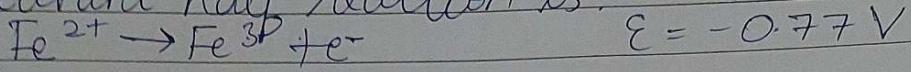
Q2 Write and explain the reactions involved in Fe(II) v/s potassium dichromate titration

Being an oxidant dichromate, has some advantages over permanganate, but as it is less powerful, its use is much more limited. It is obtainable in state of high purity and can be used as primary standard. Solutions of dichromate in water are stable indefinitely. The half reaction for the dichromate system is

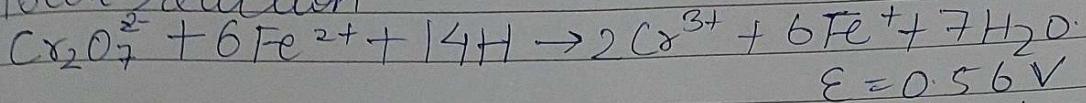


The most important application of dichromate is its reaction with iron (II) in which it is often preferred to potassium permanganate.

The relevant half reaction is:



Total reaction



Q3 In the Mohr's salt v/s dichromate titration what is the role of H_3PO_4 .

The role of H_3PO_4 in the Mohr's salt v/s dichromate titration is to reduce the electrode potential for the

$\text{Fe}^{3+} \rightarrow \text{Fe}^{2+}$ reaction by stabilizing the ferric iron. Because phosphoric acid is a weak acid, it is typically used as a analyte. Phosphoric acid can be weak acid in a weak acid strong base titration reaches the first equivalence, the solution will contain the conjugate base H_2PO_4^- . This will give the solution a pH greater than seven at that equivalence point.

Q4 Why is acidic medium required for redox titration of dichromate?

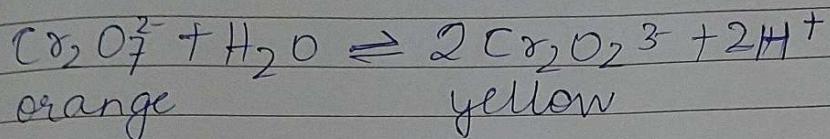
Potassium dichromate acts as oxidizing agent in acidic medium, because the neutral aqueous solution of potassium dichromate is in equilibrium mixture of dichromate

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and chromate, a consequence of hydrolysis of dichromate ions.



Chromate ions are weaker oxidizing agent than dichromate. Thus, oxidizing strength of dichromate is reduced in neutral solution. The above hydrolysis reaction can be reversed by adding acid to solution and explains the necessity of acidic medium for reaction.

Q5 Is potassium dichromate a primary standard? Why not?

Potassium dichromate is a primary standard because its solution has long lasting stability in acid and are stable to light to most of the organic matter and to chloride ion. It is always used in solution of acidic medium. The main disadvantage is that both the reactant $\text{Cr}_2\text{O}_7^{2-}$ & product Cr^{3+} are highly coloured orange and green respectively.

Q6 Why potassium dichromate cannot be used as a self indicator like potassium permanganate

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Potassium dichromate behaves as an oxidizing agent only in acidic medium, therefore it cannot be used self indicator, as its reduction production gets itself blocked in the visual detection at the end of reaction.

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Experiment 4

Aim

To determine the strength (in g/L) of the given unknown Mohr's salt solution by titrating versus standard $K_2Cr_2O_7$ solution

Material Required

Volumetric flask (100mL); Burette (50mL), pipette (10mL), weighing bottle, conical flask (100mL), funnel, test tubes, $K_2Cr_2O_7$, unknown Mohr's salt solution, dil H_2SO_4 (2N) conc H_3PO_4 , diphenylamine (indicator) Mohr's salt (solid); distilled water.

Theory

Redox titration: Titration which involves a redox reaction (transference of electrons) if simultaneous oxidation and reduction is called a redox reaction

The process of releasing electron is oxidation and the process of accepting electrons is called reduction.

Redox indicator are substances which shows different colors in oxidized and reduced forms, eg Diphenylamine, ferroin

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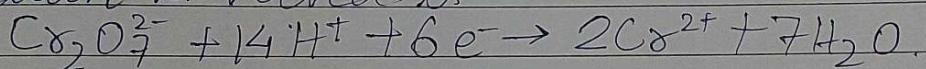
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nitroferroin; $KMnO_4$

Colour change at end point can happen if their reduction potential of reacting agents.

Three important oxidation and reduction reagent - $KMnO_4$, $K_2Cr_2O_7$.

$K_2Cr_2O_7$ is a less powerful oxidizing agent than $KMnO_4$ solution. It act as an oxidizing agent in acidic medium and the equation involved is



Procedure

Preparation of standard Mohr's salt solution:

The required amount of Mohr's salt was weighed in a weighing bottle and transferred to a clean 100mL volumetric flask through a funnel. The weighing bottle was again weighed after transferring Mohr's salt. The exact mass of Mohr's salt transferred was found by subtracting this mass from the mass of the weighing bottle plus Mohr's salt. The salt solution was prepared by dissolving Mohr's salt in dilute sulphuric acid (~10ml) and diluted with distilled water in volume-

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tric flask to make up to the volume mark.

ii) Standardization of $K_2Cr_2O_7$ solution

The burette was filled up with given $K_2Cr_2O_7$ solution with the help of funnel and mounted on a burette was noted as the initial reading. 10 mL of standard Mohr's solution solution was piped out and transferred to a clean 100 mL conical flask. A test tube full of dil H_2SO_4 (~10 mL) was added followed by 2-5 mL of conc H_3PO_4 solution. 3-5 drops of Diphenylamine indicator were then added.

iii) Determination of strength of given unknown Mohr's salt solution by titration v/s standardisation $K_2Cr_2O_7$ solution.

The above titration was repeated with the given unknown Mohr's salt solution solution. The strength was calculated and reported in g/L.

Assignment-5

Q1 Define calorimetric constant.

Ans The amount of heat energy required to raise the temp of Calorimeter by 1°C .

Q2 Define heat of neutralisation.

Ans The enthalpy of heat of neutralisation of an acid can be defined as the enthalpy change associated with the complete neutralisation of its dilute aqueous solution containing one mole of H^+ ions by a dilute solution of a base containing one mole of OH^- ions.

Q3 Explain the significance of positive and negative ΔH values.

Ans The positive value of ΔH indicate that heat released during the reaction (maybe due to bond dissociation) and thus reaction is exothermic

The negative value of ΔH indicate that heat is released during the reaction (due to bond formation) and thus reaction is endothermic

Q4 In comparison to heat evolved in neutralisation reaction between a strong acid and strong base. Why is lesser quantity of heat involved when any one of the acid or base is weak and still less when both are weak?

Ans The heat of neutralisation depends upon the amount of dissociated acid and base into H^+ and OH^- respectively. In the case of weak acid or weak base? The dissociation is incomplete i.e. they do not completely dissociate into H^+ and OH^- ions, hence a low value of heat of neutralisation. Thus the following order arises.

Both acid & $>$ Either of acid or $>$ Weak acid & base are strong base is weak weak base

Q5 Why is ΔH for strong acid strong base neutralisation a constant?

Ans Strong acid and strong base dissociate completely into H^+ and OH^- respectively. Thus concentration is same for all standard solution. They have constant ΔH_{neq} .

Practical - 5

Aim → Determination of the enthalpy of neutralisation of hydrochloric acid with sodium hydroxide

Apparatus → Thermos flask, glass stirrer, thermometer, stop watch, beaker, measuring cylinder (100 mL), sodium hydroxide, hydrochloric acid.

Theory

Generally, chemical reactions involve the breaking and making of bond.

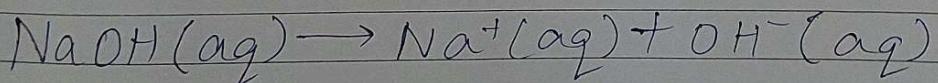
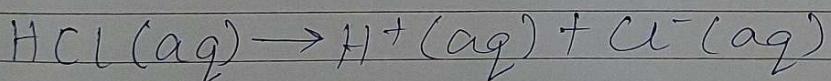
Formation of chemical bond releases energy in the form of heat thus called exothermic reaction.

Reaction accompanied by adsorption of heat are called endothermic reaction.

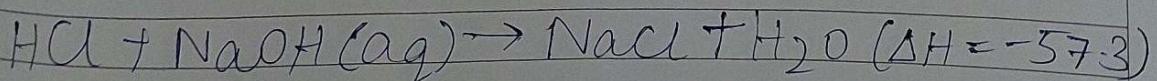
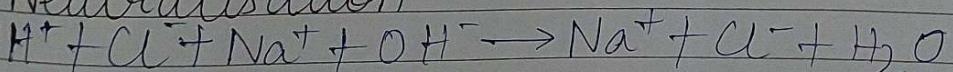
A calorimeter consists of two vessels on outer vessel and an inner vessel. The space between these vessels act as a heat insulator and hence there is very little heat exchange in between inner and outer vessel. A thermometer measures the temp of the liquid in the vessel. The stirrer stirs the liquid to distribute the heat in entire vessel.

The fibre rings in the calorimeter helps to hold the inner vessel hanging in the centre of outer vessel. It also has insulating covers with holes for attaching the stirring rod and the thermometer.

The enthalpy of neutralisation of acid can be defined as the enthalpy change associated with complete neutralisation of its dilute aqueous solution containing 1 mole of OH^- ions. Let us consider the example of neutralisation of hydrochloric acid with sodium hydroxide (HCl - strong acid) (NaOH - strong base). Thus, both completely dissociated in aqueous solution.

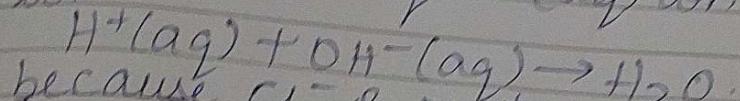


Neutralisation



Thus, neutralisation of a strong acid with strong base can be considered as the

combination of $H^+(aq)$ ion with $OH^-(aq)$ ion



because $C_2O_4^{2-}$ & Na^+ remain unchanged.

Procedure:

I. Determination of Molarities of $H_2C_2O_4$ & $NaOH$

a) Preparation of standard oxalic acid

The required amount of oxalic acid was weighed in a weighing bottle. Oxalic acid was transferred to a clean 100mL volumetric flask through a funnel. The weighing bottle was again weighed after transferring oxalic acid. The exact mass of oxalic acid transferred was found by subtracting the mass from the mass of weighing bottle plus oxalic acid. The solution prepared by dissolving oxalic acid in distilled water in a volumetric flask and made up to the volume mark.

b) Standardization of $NaOH$ v/s standard oxalic acid.

Fill up the burette with $NaOH$ with funnel mark it. Note initial reading. Pipette

out 10mL of fresh oxalic acid and transfer to clean 100mL conical flask. Add 1-2 drops of phenolphthalein indicator. Titrate the colourless colourless solution against NaOH with constant swirling against a white tile until a pink colour obtained. Note burette final reading. Repeat to get at least 2 concurrent readings.

c) Titration of HCl v/s standardized NaOH.

Same procedure as above titration is followed burette contains NaOH and pipetted 10mL of HCl is filled in 100mL flask. Phenolphthalein indicator is added and swirled till pink colour occurs. Final reading is recorded and repeated for concurrent readings.

When a thermos flask or a beaker is used as a calorimeter for above determinations first step will be determination of heat capacity of calorimeter and second step will be determination of heat of neutralisation using same calorimeter.

2) Determination of heat capacity of the calorimeter.

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Take thermos flask and fix cork having 2 holes, one for fitting thermometer and second loose for stirring with glass stirrer. Measure 100ml of distilled water and add into the flask. At same time, 100ml of hot water is taken in a beaker outside the flask. At same time, 100ml of hot water is taken in a beaker outside the flask. Temperature of hot water should be about 20°-25°C higher than that of cold water.

Note the temperature of hot and cold water simultaneously.

Start the stop watch and continue noting the temperature of hot and cold water simultaneously after every half minute for 3 minutes, and both mixtures has to be stirred continuously.

At 3-5 minutes, transfer the hot water to the cold water inside the flask. Stir the mixture vigorously and note the temp of the mixture every half minute for next 4 minutes.

3. Determination of heat of neutralisation of strong acid (HCl) and strong base ($NaOH$)

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Take a thermos flask (500mL) and fit a cork having two holes, one for fitting a thermometer and another loose for operating a glass stirrer. Measure 100 mL of HCl and place it in a same flask used in step 1. At the same time, 100mL of NaOH is taken in a beaker outside the flask. Start the stop watch and continue. The temperature of HCl and NaOH simultaneously after every half minute for 3 minutes.

At 3-5 min, transfer the NaOH to HCl inside the flask. Stir mixture vigorously and note the temp of the mixture every half minute for the next 4 minutes.

Result

The heat of neutralisation of strong acid (HCl) with strong base is found to be _____ KJ/mole

Actual value = 57.3 KJ/mol.

Inference

1 Enthalpy of solution or solution heat is expressed in KJ/mol; and when a solution is formed it is the amount of heat energy

that is released.

2. Enthalpy of neutralisation is always constant for a strong acid and a strong base : this is because all strong acids and strong bases are completely ionised in dilute solution. Enthalpy changes in neutralisation are always negative when an acid and alkali react heat is given out.
3. Enthalpy of neutralization is exothermic in nature since heat is released when bonds are made