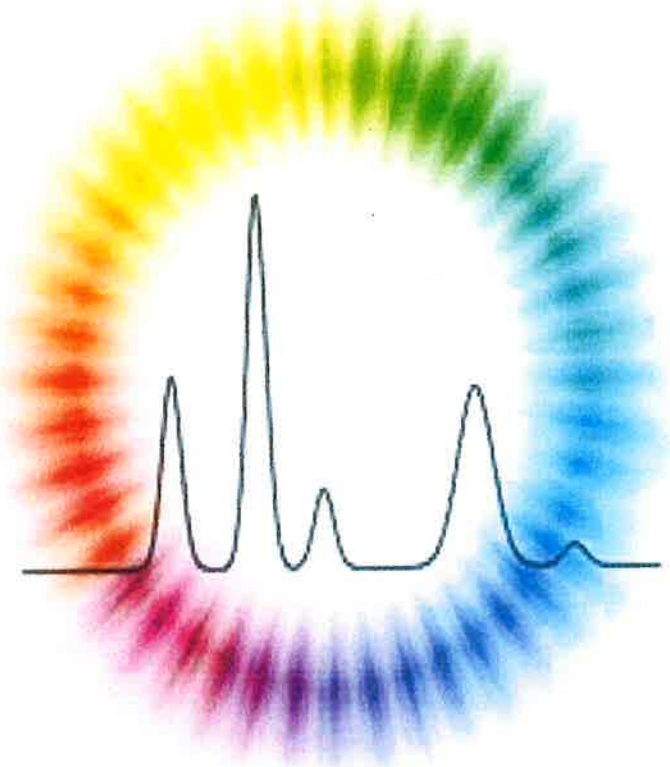
Instrumental Analytical Laboratory
4th year
Department of Chemistry
College of Science
Mustansiriyah University

ANALYTICAL CHEMISTRY



Written by:
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Experiment No.: 1

Experiment name: Determination the Concentration of $K_2Cr_2O_7$

The purpose of the experiment:

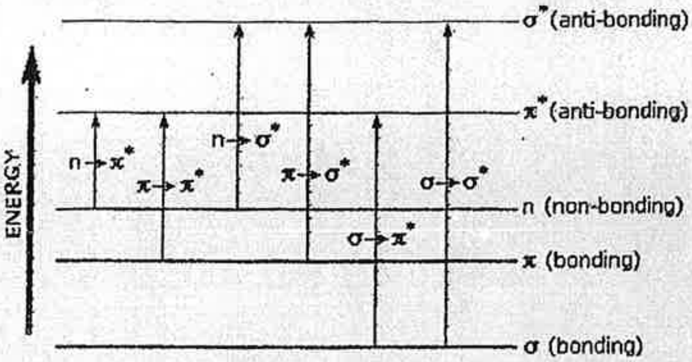
A-Determination wavelength of maximum absorbance λ_{max} of $K_2Cr_2O_7$.
B-Determination the unknown concentration of $K_2Cr_2O_7$.

Apparatus &Tools: Spectrophotometer (single beam), balance, pipette, volumetric flask(V.F.) and beakers.

Reagents and Supplies: Potassium dichromate($K_2Cr_2O_7$) and Distilled water (D.W).

Theory:

UV-Vis spectroscopy is routinely used in analytical chemistry for the quantitive determination of different analytes, such as transition metal ions, conjugated organic compounds and biological molecules. When sample molecules are exposed to light having an energy that matches a possible electronic transition within the molecule, some of the light energy will be absorbed as the electron is promoted to a higher energy orbital (Molecules containing π-electron or non-bonding electrons (n-electrons) can absorb the energy in the form of ultraviolet or visible light to excite these electrons to higher anti-bonding molecular orbital as shown in the diagram below). An optical spectrometer records the wavelengths at which absorption occurs, together with the degree of absorption at each wavelength. The resulting spectrum is presented as a graph of absorbance (A) versus wavelength.



Procedure:

0.01g

1- Prepare a stock solution of $K_2Cr_2O_7$ by weighing accurately about (0.025 gm).

$$M = \frac{Wt\,g \times 1000}{M.Wt\ (292) \times Vol,\,ml}$$

2- Dissolve in D.W. and transfer to 100 ml volumetric flask, complete the volume to the mark (with D.W).

3- In set of five (50 ml) volumetric flask , using a 5 ml pipette, transfer 1, 2, 3, 4 and 5 ml of the above stock solution (in step1) and complete the volume to the mark with D.W and shake well. Calculate the concentration (in ppm) for each solution.

$$M_1 V_1 = M_2 V_2$$

$$C\ ppm = M \times M.Wt \times 1000$$

4- Fill the first cell with the reference solution and the second cell with solution No.1, and clean the outside surface of each cell.

5- Following the instruction, obtain the absorbance for both solution in the region of (380- 330nm), make change of 5nm till 330 nm for each absorbance reading, obtain the wavelength of maximum absorbance λ_{max}.

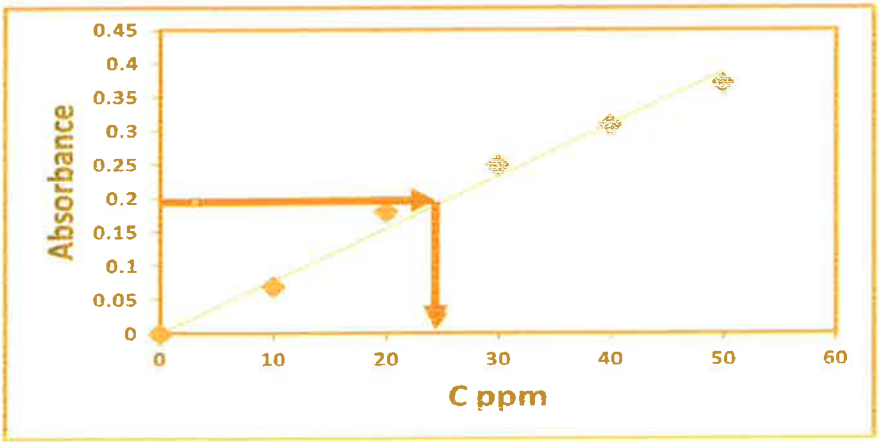
6- Set the spectrophotometer at λ_{max} .

7- Fill the 2^{nd} cell with solution No.2 and the 3^{ed} cell with solution No.3 and then read the absorbance at the λ_{max}.

8- Repeat the same steps using solutions No.4 and 5 and the unknown sample and record the absorbance reading.

9- Plot calibration curve between A versus concentration

10- Find the concentration of the given unknown sample.



Questions :

1-Why Potassium dichromate gives an absorbance within UV region instead of visible region?

2-What are the kinds of cells used in UV region and why?

Experiment No.: 2

Experiment name: Determination the Molecular formula of Fe(II)-2,2'-Bipyridine complex by using the mole-ratio method.

The purpose of the experiment: Determination of molecular formula for the complex of Fe(II) with Bipyridine.

Apparatus &Tools: Spectrophotometer (single beam), balance, pipette, V.F and beakers.

Reagents and Supplies: $Fe(NH_4)_2(SO_4)_2.6H_2O$, 0.1% Bipyridine solution,

Theory:

To understand the coordination chemistry for the complex (FeII-2,2-Bipyridine) formed by the reaction of Iron(II) with Bipyridine as a ligand ,the Mole-Ratio method has been employed. This method depends on fixing the concentration of one substance (Fe (II)) and changing the other substance (Bipyridine).

Procedure:

1-Prepare (100ml) of (0.002M) Fe^{+2} by dissolving the calculate amounts of $Fe(NH_4)_2(SO_4)_2.6H_2O$, then dilution to the mark with D.W.

$$M = \frac{\text{Wt g} \times 1000}{\text{M.Wt } (392.14) \times \text{Vol, ml}}$$

2-Prepare (50 ml) of (0.002 M) Bipridine from the prepared (0.1 % Bipyridine wt/v) concentration solution.

$$M = \frac{\text{Wt g} \times 1000}{\text{M.Wt } (156.19) \times \text{Vol, ml}}$$

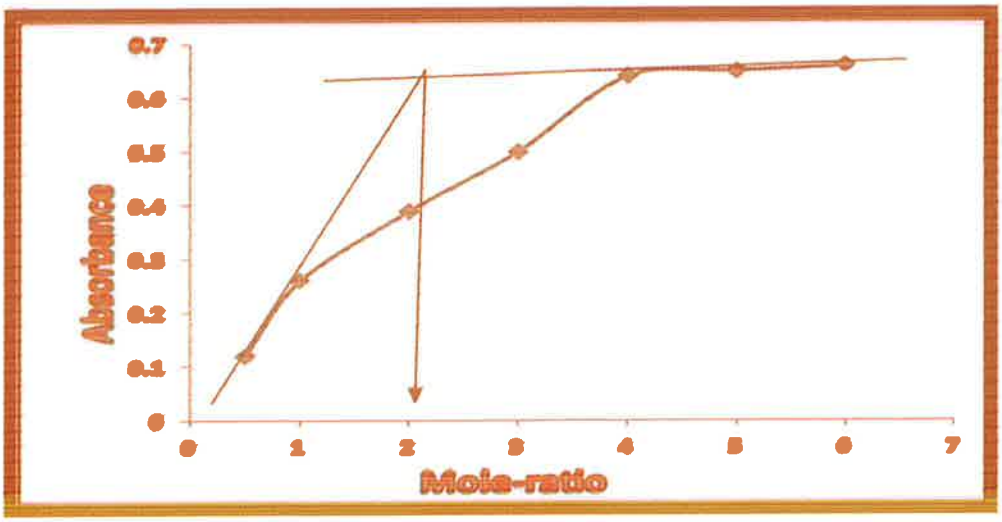
$$\frac{\text{Wt\%}}{\text{Vol}} = \frac{0.1\text{g}}{100\text{ ml}}$$

3-To each of seven 25ml volumetric flasks, transfer 1 ml of the Fe^{+2} solution then added consequently 0.5, 1, 2, 3, 4, 5 and 6 ml of the Bipyridine solution prepared in (2) above, dilute to the mark with D.W.

4-Measure the absorbance's of the solutions at (522 nm) after calibrating the apparatus on blank solution.

5-Find the Molecular formula after plotting the absorbance record against the mole-ratio(C_x/C_m).

Where C_x=Nomber of Bipyridine moles.
C_m=Number of Fe(II) moles.



Questions:

1-What is the benefit of using Mole-Ratio method?
2-Explain the Mole-Ratio method?

Experiment No.: 3

Experiment name: Employing the proportional-method in Fe(II) determination .

The purpose of the experiment: Colorimetric determination of Fe(II) concentration .

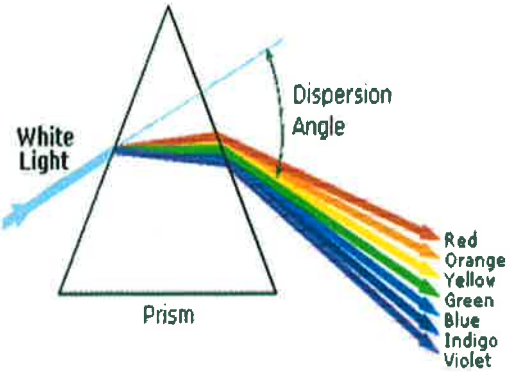
Apparatus &Tools: Spectrophotometer (single beam), balance, pipette, V.F. and beakers.

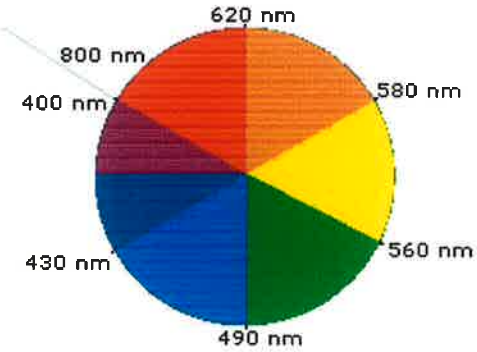
Reagents and Supplies: Fe $(NH_4)_2$ $(SO_4)_2.6H_2O$, 0.1% Bipyridine solution, 3M H_2SO_4, 10% Na (CH_3COO), NH_2OH and D.W.

Theory:

When white light passes through or is reflected by colored substance, a characteristic portion of the mixed wavelengths is absorbed. The remaining light will then assume the complementary color to the wavelengths absorbed. This relationship is demonstrated by the color wheel shown in the figure below. There, complementary colors are diametrically opposite each other. Thus absorption of (420-430) nm light renders a substance yellow and absorption of (500-520) nm light makes it red.

The principle of this method is that Iron(II) must be reacted with another substance to form a colored substance which can be spectrophotometrical determine in the visible region using the Ratio method which leg to precision although it has the advantage of being fast.





Procedure:

1-Prepare (100 ml) of (0.002 M) Fe^{+2} by dissolving the calculate amounts of $Fe(NH_4)_2\ (SO_4)_2.6H_2O$, then added 3 ml of 3 M H_2SO_4 before dilution to the mark with D.W.

$$M = \frac{Wt\,g \times 1000}{M.Wt\ (392.14) \times Vol,\ ml}$$

2-Pipet (5 ml) of the first above solution into (50 ml) V.F. and added 2 ml of 3 M H_2SO_4 and dilute to the mark with D.W.

$M_1V_1=M_2V_2$

3-To each three (25 ml) V.F. transfer:

1-(5 ml) of the previous prepared solution Fe(II)[Standard].

$M_1V_1=M_2V_2$

2-(5 ml) from the Unknown solution.

3-(5 drops) of 3M H_2SO_4 [Balnk].

To each of the three flasks add:

A- 0.5 ml of (0.1 %) Hydroxyamin. Hydrochloride solution.

B- 5 ml of (0.1 %) Bipyridine solution.

C- 2 ml of (10 %) Sodium acetate solution.

4-Shake well these solutions after each reagent solution added, then dilute to the mark with D.W.

6- Set the absorbance reading to zero with blank solution, then measure the absorbance of the each solutions at (522 nm).

6-Determine the concentration of unknown solution using the following equation:

$$\frac{A_{st}}{A_{unk.}} = \frac{C_{st}}{C_{unk.}}$$

Questions:

proportional

1-Which is the best ↗ method or other calibration method?

2-What is the reason for adding each material: 3M H_2SO_4, 0.1% Bipyridine solution, 10% Na (CH_3COO), NH_2OH, D.W.

3-What does the number (522 nm) represent?

4-What is the lamp kind and cell measurement in this Apparatus?

Experiment No.: 4

Experiment name: Determination of Fe^{+3},by Colorimetric method .

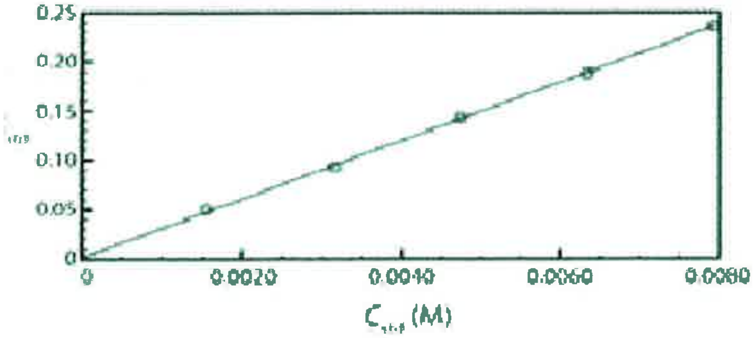
The purpose of the experiment: Determination the Concentration of Fe^{+3} by calibration curve method.

Apparatus &Tools: Spectrophotometer (single beam), balance, pipette, V.F. and beakers.

Reagents and Supplies: perchloric acid, $HClO_4$, $Fe(NO_3)_2.9H_2O$ and Sulpho-salicylic acid $C_6H_3(OH)(COOH)$- $SO_3.2H_2O$.

Theory:

In analytical chemistry, a calibration curve is a general method for determining the concentration of a substance in an unknown sample by comparing the unknown to a set of standard samples of known concentration. The calibration curve is a plot of how the instrumental response, changes with the concentration of the analyte (the substance to be measured). The operator prepares a series of standards across a range of concentrations near the expected concentration of analyte in the unknown. The concentrations of the standards must lie within the working range of the technique (instrumentation) they are using. Analyzing each of these standards using the chosen technique will produce a series of measurements. For most analyses a plot of instrument response vs. analyte concentration will show a linear relationship. The operator can measure the response of the unknown and using the calibration curve; can interpolate to find the concentration of analyte.



Procedure:

1-Prepare (250 ml) of (0.1 M) perchloric acid ($HClO_4$).

$$M = \frac{\text{Sp.gr } (1.65) \times \% \ (70/100) \times 1000}{\text{M.Wt } (100.5)}$$

$$M = \frac{\text{Sp.gr } (1.65) \times 70 \times 10}{\text{M.Wt } (100.5)}$$

$M_1V_1=M_2V_2$

2-Prepare (50 ml) a stock solution of (0.005 M) Fe^{+3} by dissolving the calculated amounts of $Fe(NO_3)_2.9H_2O$ in small beaker containing about 10ml of 0.1 M $HClO_4$, dilute to mark with 0.1 M $HClO_4$.

$$M = \frac{\text{Wt g} \times 1000}{\text{M.Wt } (404) \times \text{Vol, ml}}$$

3-Prepare (100 ml) of (0.01 M) Sulphosalicylic acid by dissolving the calculated amounts of Sulphosalicylic acid in small beaker containing appropriate volume of 0.1 M $HClO_4$, dilute to mark with 0.1 M $HClO_4$.

$$M = \frac{\text{Wt g} \times 1000}{\text{M.Wt } (254.22) \times \text{Vol, ml}}$$

4-Calibration graph prepared as follows: To each of four 25 ml volumetric flasks, transfer 0.5, 1.5, 2.0 and 2.5 ml of 0.005 M Fe^{+3} solution and added 10 ml of 0.01 M Sulphosalicylic acid solution to each 25 ml volumetric flasks respectively, dilute to mark with 0.1 M $HClO_4$.

$M_1V_1=M_2V_2$

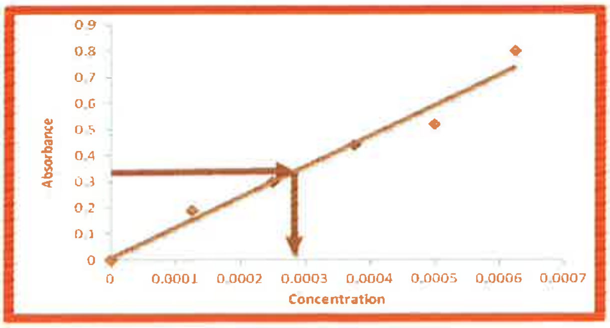
5-To the 25 ml volumetric flask contain unknown sample, add (10 ml) Sulphosalicylic acid and diluted with $HClO_4$.

6-Allow standing all standards and unknown Solutions about half an hour.

7-Measure the absorbance in a (1 cm) cell at (500 nm) for all standards and unknown Solutions.Tabulate the results obtained.

9-Plot the calibration curve between absorbance A vs. concentration, then determine the concentration of Fe^{+3} in unknown sample.

10-Calculate the original concentration of unknown sample.



Questions :

1-Why do you add perchloric acid?

2-Why do you add Sulphosalicylic acid?

Experiment No. 5

Experiment name: Simultaneous determination of mixture Co^{+2} and Ni^{+2}.

The purpose of the experiment:

A-Determination molar absorptive (ε) for Co^{+2} & Ni^{+2}.
B-Determination the concentration of Co^{+2} & Ni^{+2}.

Apparatus &Tools: Spectrophotometer (single beam), balance, pipette, V.F. and beakers..

Reagents and Supplies: $Co(NO_3)_2.6H_2O$, $Ni(NO_3)_2.6H_2O$ and D.W.

Theory:

Depending on the molar absorptive (ε), Cobalt and Nickel ions concentrations are determined sensitively and separately (Due to their close chemical properties resulting from their lies in one transition period) without any chemical interference or processing neither a chemical masking nor separation operation.

Procedure:

1-Prepare (50 ml) of (0.15 M) Co^{+2} from solid $Co(NO_3)_2.6H_2O$.

$$M = \frac{Wt\,g \times 1000}{M.Wt\ (291) \times Vol,ml}$$

$$M_1V_1=M_2V_2$$

2-Dilute to prepare (25 ml) of two solutions contain (0.09 & 0.06 M) from (0.15 M) Ni^{+2} solution.

3-Prepare (0.15, 0.09 &0.06 M) Ni^{+2} from $Ni(NO_3)2.6H_2O$ salt by the same way in step 1 and 2.(M. wt=290 $g.mol^{-1}$)

4-Measure the absorbance of the above solutions of Co^{+2} and Ni^{+2} and unknown separately at both (510 & 660 nm).

5-Calculate the molar absorptive ε for each solution by:

A-Using the equation $A= \varepsilon bc$

B-Drawing A vs. Conc. Then the slop of straight line :

$$slope = \frac{\Delta A}{\Delta C}$$

6-Calculate the Conc. of Co^{+2} & Ni^{+2} by using equations below:

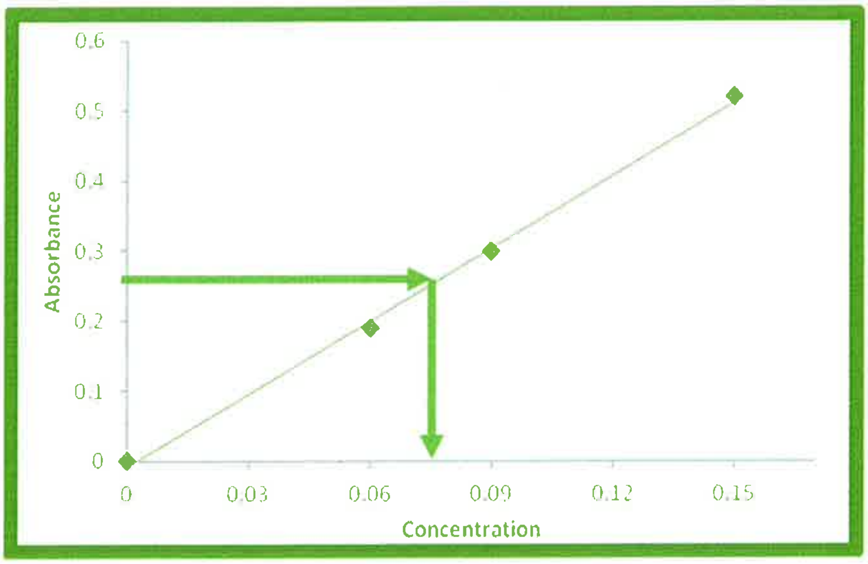
$$C_1 = \frac{\varepsilon_{22} A_1 - \varepsilon_{12} A_2}{\varepsilon_{11} \varepsilon_{22} - \varepsilon_{12} \varepsilon_{21}}$$

$$C_2 = \frac{\varepsilon_{11} A_2 - \varepsilon_{21} A_1}{\varepsilon_{11} \varepsilon_{22} - \varepsilon_{12} \varepsilon_{21}}$$

Where $\varepsilon11$ is the Molar absorptive of Co^{2+} at 510nm, $\varepsilon12$ is the Molar absorptive of Ni^{2+} at 510nm, $\varepsilon21$ is the Molar absorptive of Co^{2+} at 660nm, $\varepsilon22$ is the Molar absorptive of Ni^{2+} at 660nm, A_1 is the absorbance of Co^{2+} at 510nm and A_2 is the absorbance of Ni^{2+} at 660nm.

$$A_1 = \varepsilon_{11} C_1 + \varepsilon_{12} C_2$$

$$A_2 = \varepsilon_{12} C_1 + \varepsilon_{22} C_2$$



Questions :

1-Define Simultaneous Determination?

2-Is there are any interference between Cobalt and Nickel ions?

3-Define Molar absorptive ε?

4-What are the λ_{max} for both Cobalt and Nickel, Why?

Experiment No.: 6

Experiment name: Determination of Sodium by Flame Emission photometry.

Purpose of the experiment: Determination the concentration of sodium by Flame Emission photometry.

Apparatus & Tools: Flame photometry, balance, V.F., beakers and pipette.

Reagents and Supplies: NaCl, KCl and D.W.

Theory:

When a solution of a metal ion is placed in the flame of a Bunsen burner, the flame turns a color that is characteristic of the metal ion. A sodium solution gives a yellow color; the phenomenon just described is an "atomic emission" phenomenon. The first step of the process is an atomization step. That is, the flame converts the metal ions into atoms. When a solution of sodium chloride is placed in a flame, the solvent evaporates, leaving behind solid crystalline sodium chloride. This evaporation is then followed by the dissociation of the sodium chloride crystals into individual ground state atoms -a process that is termed atomization. The atomic emission is a result of electrons dropping from an excited state to lower states. Following atomization, a small percentage of the atoms absorb sufficient energy from the flame (as opposed to a light beam) so as to be promoted to an excited state, these atoms quickly return to a lower state and light corresponding to the energy that is lost in the process is generated. It is this light that our eye perceives. The complete sequence of events is depicted in Figure below.

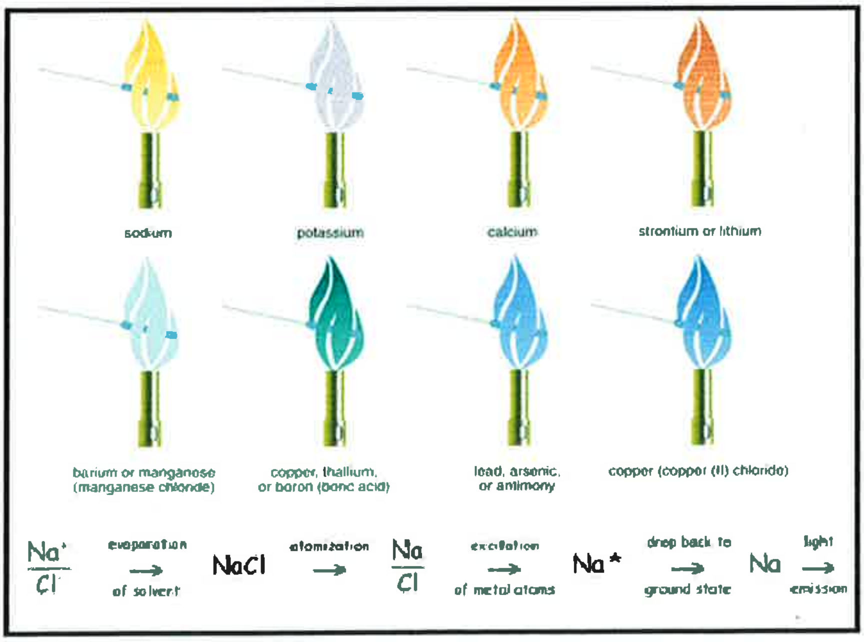


Figure: Observation of the flame color& the sequence of events occurring in the flame test.

Procedure:

1-Prepare (50 ml) of (500 ppm) Na standard solution from NaCl .

$$C\ ppm = \frac{Wt\ g \times 10^{6}}{Vol,\ ml}$$

2-Prepare (50 ml) 0f (2000 ppm) K standard solution from KCl.

$$C\ ppm = \frac{Wt\ g \times 10^{6}}{Vol,\ ml}$$

3-From a 500 ppm Na standard solution, 50 ml of each of the following solutions were prepared, 0, 2.5, 5, 10, 15 and 20 ppm Na in a 50 ml V.F., each of them contain (200) ppm K.

$M_1V_1=M_2V_2$

4-Set the Flame photometer to the Na filter under its proper conditions.

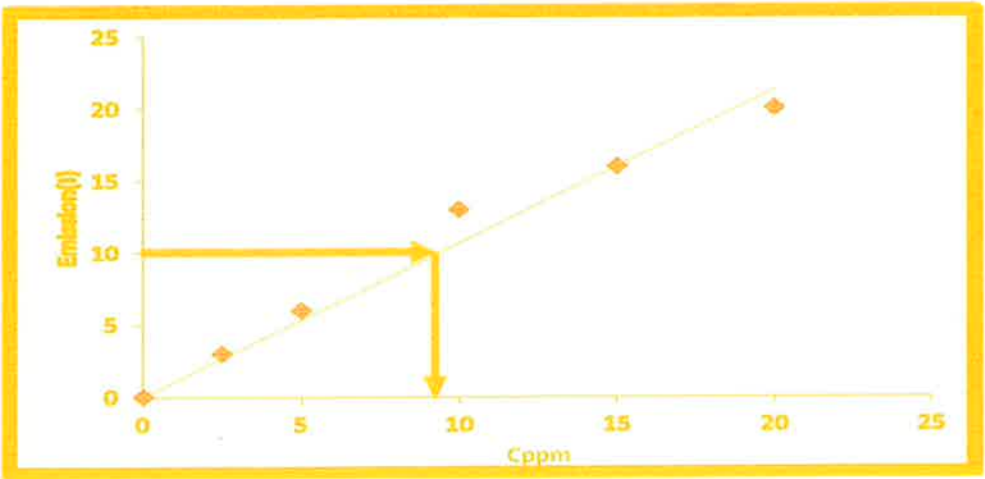
5- Calibrate with Blank (0 ppm Na +200 ppm K), the apparatus was set to zero by means of the zero control against a reagent blank solution.

6-The reagent blank was replaced by a standard solution of conc. Corresponding to a reading of 100 (20 ppm) or the calibration curve is use and the sensitivity control was adjusted to give exactly full-scale deflection,100.

7-The emission intensity of the other standard solutions were recorded also for the unknown solution.

8-Plot the curve between emission intensity(I) and the concentration of standard Na solutions (Conc.).

9-Determine the Conc. of Na in unknown sample.



Questions :

2-Explain the addition of KCl to the solutions?

2-What is the benefit of making the filter tool on Na filter?

Instrumental Analysis Lab.

Experiment No : 7

Experiment name : Determination of sulphates by barium chloride .

The purpose of experiment : Determination of sulphates using Turbidity meter .

Apparatus & Tools : Turbidity meter , balance , pipette , volumetric flask and beakers .

Reagents and supplies : potassium sulphate , conditioning solution , barium chloride and D.W .

Theory : This constitutes an extremely sensitive method of analysis the quantity of a solid material dispersed as a colloid can be determined by measurement either of the transmitted light (turbidimetry) or of the scattered light (nephelometry) .

Sulphat are found in appreciable quantity in all natural waters , particularly high in arid and semi arid regions where natural waters in general have high salt content . Sulphate salts are mostly soluble in water and import hardness . water with high concentration has a bitter test . Sulphate may cause intestinal disorders .

The sulphate data is used in determining applicability of different water types for their public industrial applications . It indicates extent of problems that can arise due to reduction of sulphates to hydrogen sulfides . In addition , sulphate content of organic matter fed to anaerobic digester is important information as it gives idea of generation of hydrogen sulfides , which needs to be removed .

Unites of measurements :

Nephelometric Tubidity Unit (NTU)

Formazin Nephelometric Unit (FNU)

Procedure :

Conditioning solution : dissolve 120g NaCL in about 400ml of D.W. , add 10ml conc . HCl and 500ml glycerol and dilute to 1000ml.

1. Prepare a standard solution of (0.01N) k_2SO_4 , (250 ml . is required) .
2. In five volumetric flask (50 ml) pipette 4,8,12,16 and 20ml .of the stock solution (in 1) then continue the volume up to the mark with dist . water .
3. Transfer 10ml . of the first dilution into a small beaker , add 10 ml. of the conditioning solution and mix well . Add 0.3gm . of $BaCL_2$ as crystals , then stir for 1min . , allow to stand for 4 mins . ,then stir for 15 secs . just before measurements .
4. Using the Turbidity meter , read the turbidance immediately after the above 15secs .
5. Repeat step 3 and 4 for the other dilutions and record the turbidance .
6. To the given unknown sample add dist . water and continue the volume up to the mark .
7. Repeat steps 3 and 4 and record the unknown readings .
8. Construct a calibration curve for the relation of turbidance on the vertical axis and the relative concentration on the horizontal axis

9. From the above calibration curve , evaluate the concn. of the given unk. sample .

Questions :

1. List conditioning solution contents used in the experiment .
2. What is the benefit of adding conditioning solution .
3. Why do we remove the bubbles from the cell .
4. what is the light source used in turbidity meter .

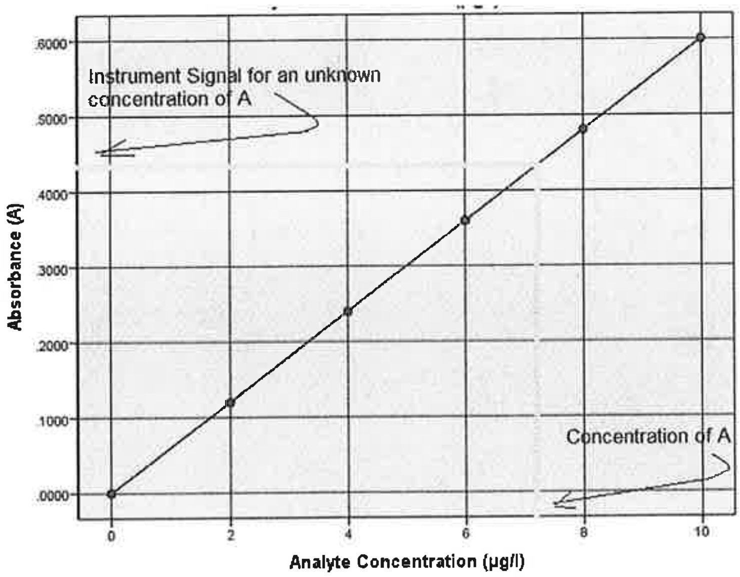
Calibration Methods:

1. Proportional calibration method is a method for estimation the concentration of an unknown sample by means of comparison to a known concentration standard, through applying the following equation:

$$\frac{A_{st}}{A_{unk}} = \frac{C_{st}}{C_{unk}}$$

2. Calibration curve method

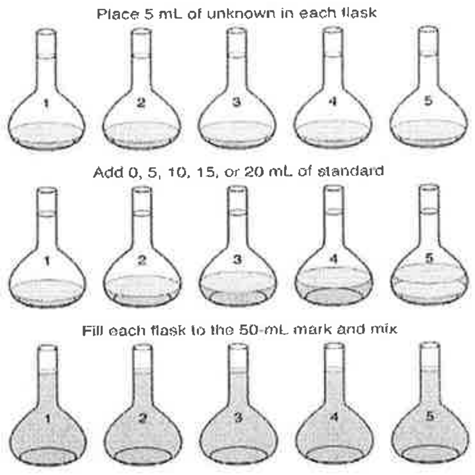
In analytical chemistry, a normal calibration curve is a general method for determining the concentration of a substance in an unknown sample by comparing the unknown to a set of standard samples of known concentration. A series of standards are prepared across a range of concentrations near the expected concentration of analyte in the unknown. The concentrations of the standards must lie within the working range of the technique (instrumentation) they are using.

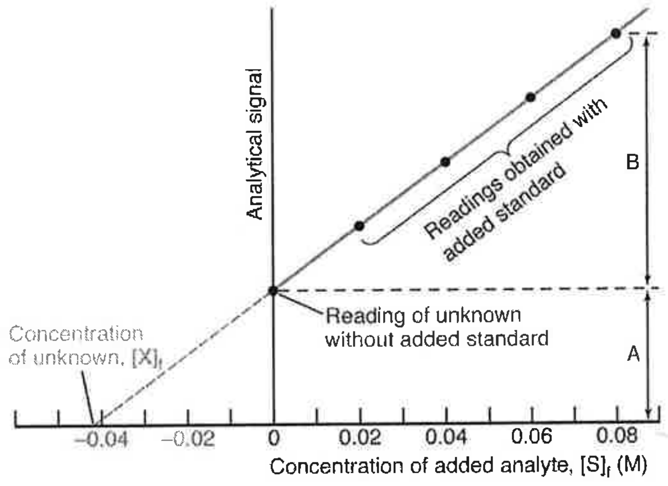


3. Standard Addition Method

The method of standard addition is a type of quantitative analysis approach often used in analytical chemistry whereby the standard is added directly to the aliquots of analyzed sample. This method is used in situations where sample matrix also contributes to the analytical signal, a situation known as the matrix effect, thus making it impossible to compare the analytical signal between sample and standard using the traditional calibration curve approach.

A typical procedure involves preparing several solutions containing the same amount of unknown, but different amounts of standard. For example, five 25 mL volumetric flasks are each filled with 10 mL of the unknown. Then the standard is added in differing amounts, such as 0, 1, 2, 3, and 4 mL. The flasks are then diluted to the mark and mixed well.





Standard addition is often used when the sample is unknown or complex and when species other than the analyte affect the signal. The matrix is everything in the sample other than the analyte and its affect on the response is called the matrix effect. The matrix effect problem occurs when the unknown sample contains many impurities.

If impurities present in the unknown interact with the analyte to change the instrumental response or themselves produce an instrumental response, then a calibration curve based on pure analyte samples will give an incorrect determination

Experiment No.1

Potentiometric titration of strong acid with strong base

The purpose of experiment:

Part A: Determination the concentration of NaOH (calibration the strong base)
Part B: Determination the concentration of poly protic acid, H_3PO_4 and (K_1 , K_2)

Part A:

Apparatus: pH meter, combine glass electrode, magnetic stirrer, stirrer bar, burette and beaker.

Reagents: NaOH solution, standard solution of HCl solution, buffer solutions (pH4, pH7) and D.W.

Theory: In an acid-base titration the change in $[H_3O^+]$ may be very large, i.e. from 10^{-1} to 10^{-10} mol/liter. It is inconvenient to plot such curve. However the change in pH is only from 1.0 to 10.0 and such numbers can be plotted readily. The result of plotting pH versus the volume of titrant added is called atitration curve. The pH can be measured by use of a pH meter, an instrument with an electrode sensitive to H_3O^+. Near the equivalence point the pH changed sharply and this change in pH readily seen on the titration curve as asharp change in the slope of the curve. You will now titrate astrong acid (HCl) with the base (NaOH) of known molarity.

Procedure:

1-Calibrate pH meter using buffer pH4 and pH7

2- Transfer a (   )ml (   )N of HCl to 250ml beaker and complete the volume to 150ml with D.W., put the stirrer bar in the bottom and immerse the combined glass electrode (glass electrode and reference electrode) in to the solution, we make sure that when the stirrer is turned on, the stirring bar will not hit the electrode.
3- Titrate with NaOH solution (in the burette), using the magnetic stirrer and stirrer bar to stir the solution. approximately 0.2ml of NaOH solution was added at each time when the pH value changing slowly, from aburette to the solution and about two drops near the expected equivalent point, stirred for few seconds then the pH value was read.

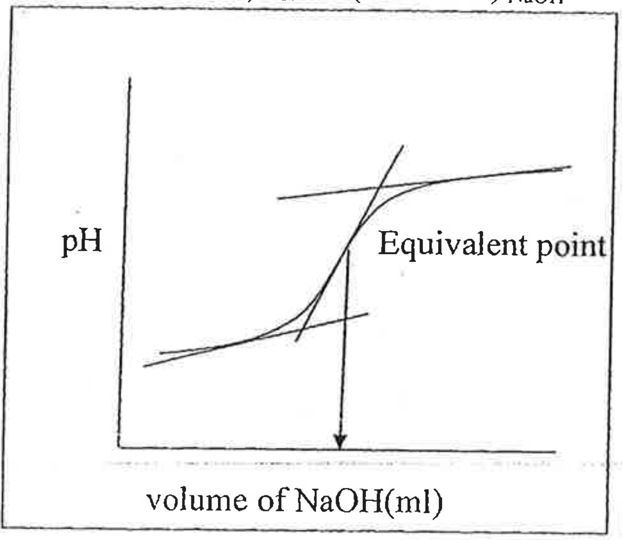
4- Tabulate all the results obtained as below:

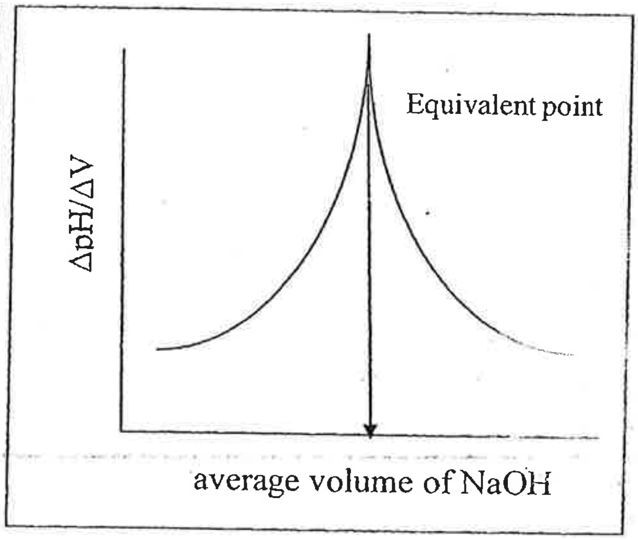
Added volume of NaOH (ml)	pH	ΔV	ΔpH	ΔpH/ΔV	Average volume of NaOH (ml)

5- Plot atitration curve between pH values at X-axis versus the volume of NaOH added at Y-axis or beween ΔpH/ΔV at X-axis versus average volume of NaOH at Y-axis, then show and determine the equivalent point.

6- Calculate the concentration of NaOH using the equation:

$$(N \times V)_{HCl} = (N \times V)_{NaOH}$$





Discussion:

Answer the following questions:

1-What is the pH value at the equivalent point?

2-What ions are present in the solution at the equivalent point (only those in significant amounts)?

3-What is the type of electrode used? Mention the reason?

4-Why you stop the titration at pH11?

5-What is the difference between the potentiometric titration and normal titration?

6-What is the difference between the end point and equivalent point?

7-Why you put about two drops each time near the equivalent point?

Part B:

Apparatus: pH meter, combine glass electrode, magnetic stirrer, stirrer bar, burette,beaker

Reagents: NaOH solution, H_3PO_4 solution (unknown) and D.W.

Theory: Many substances which act in aqueous solution can give up more than one hydrogen ion per molecule these are called poly protic acids and have two or more dissociation constants. It will be possible to neutralize essentially all of the acid from the first ionization before asignificant amount of hydrogen ion is produced by the second ionization. One might in this case hope to see the neutralization of the hydrogen ion from the first and second ionization show up aseparate and distinct features of the titration curve. In this experiment you will titrate phosphoric acid with standard NaOH. As the pH electrode doesn't give satisfactory reading at high (pH>12). You will not be able to distinguish the third end point.

Procedure:

1- Pipit and transfer a (   )ml of H_3PO_4 solution (unknown) to 250ml beaker and complete the volume to 150ml with D.W., put the magnetic bar in the bottom, immerse the combined electrode in to the solution, make sure that when the stirrer is turned on the stirrer bar will not hit the electrode.

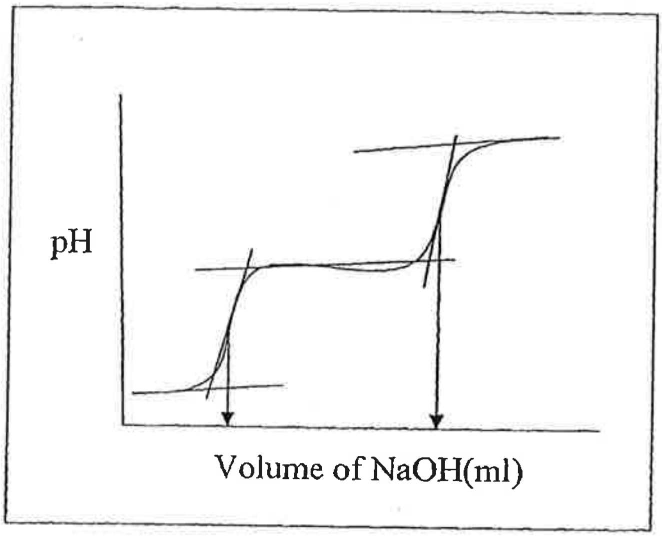
2-Titrate with NaOH solution (in the burette), using the magnetic stirrer and stirrer bar to stir the solution, approximately 0.2ml of NaOH was added at each time in regions where the pH is changing slowly, but about two drops at atime in the region near the expected end point, stop the titration at pH11

3- Tabulate all the results obtained as below:

Added volume of NaOH (ml)	pH	ΔV	ΔpH	ΔpH/ΔV	average volume of NaOH (ml)

4- Plot atitration curve between the pH values at X-axis versus the added volume of NaOH in ml at Y-axis or ΔpH/ΔV at X-axis versus average volume of NaOH at Y-axis, then show and determine the equivalent point.

5-Calculate the concentration of H_3PO_4 at the two equivalence points using the equation: $(N \times V)_{H3PO4} = (N \times V)_{NaOH}$



Discussion:

Answer the following questions:

1-What is the pH at each equivalence point?

2- What ions are present at each equivalence point in significant amounts?

3- How many equivalence points does H_3PO_4 has?

Experiment No.2

Potentiometric titration of weak acid with strong base:

The purpose of experiment: Determination the concentration of acetic acid in vinegar

Apparatus: pH meter, combine glass electrode, magnetic stirrer, stirrer bar, burette, beaker

Reagents: vinegar, NaOH solution, buffer solutions (pH4, pH7), D.W.

Theory: Vinegar normally contain 5% acetic acid, so we can determine the total acid content by potentiometric titration with standard base.

$$CH_3COOH + NaOH \rightleftharpoons CH_3COONa + H_2O$$

Procedure:

1-Calibrate pH meter using buffer pH4 and buffer pH7

2- Calibrate NaOH solution by titrate ( )ml, ( )N of standard HCl solution with the base as in part (A), experiment (1)

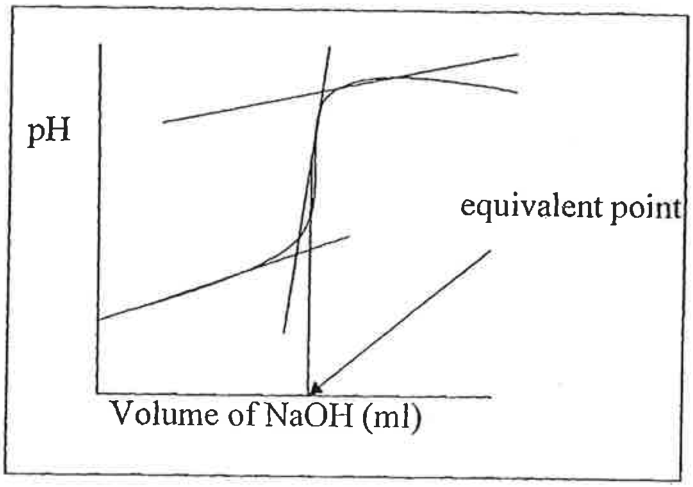
3- Tabulate all the results obtained in this titration, plot the titration curve between of pH values versus added volume of NaOH or between ΔpH/ΔV versus average volume of NaOH, determine the concentration of NaOH

4- Pipit and transfer (5)ml from vinegar to weighted small beaker, then weight the beaker with the sample, calculate the weight of the sample (vinegar)

5- Transfer the sample quantitatively to 250ml beaker then dilute to 150ml with D.W.

6- Titrate the sample with standard NaOH solution, continue the titration until pH (10.5).

7- Tabulate all the results obtained, plot the titration curve between pH values versus added volume of NaOH or ΔpH/ΔV versus average volume of NaOH



8- Select the equivalent point from the calibration curve

9-Calculate the concentration of CH_3COOH from the equation:

$$(N \times V)_{NaOH} = (N \times V)_{CH3COOH}$$

10-Calculate the weight of CH_3COOH from the equation

$$N = \frac{Wt_{acetic\ acid}}{Eq.wt} \times \frac{1000}{V(ml)}$$

11 -Calculate the percentage of CH_3COOH in vinegar

$$\%CH_3\ COOH\ \frac{Wt}{V} = \frac{Wt_{CH3COOH}}{V_{sample}} \times 100$$

$$\%CH_3\ COOH\ \frac{Wt}{Wt} = \frac{Wt_{CH3COOH}}{Wt_{sample}} \times 100$$

Experiment No.3

Precipitation- conductometric titration

The purpose of experiment:

Part A: Calibration of $AgNO_3$ solution
Part B: Determination of NaCl in table salt

PartA:

Apparatus: Conductivity meter, magnetic stirrer, stirrer bar, burette, beaker.
Reagents: $AgNO_3$ solution, standard solution of HCl and D.W.

Procedure:

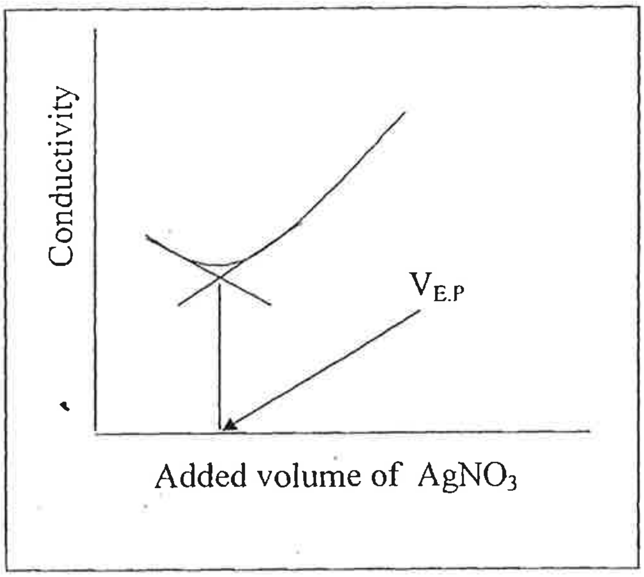
1- Calibrate the conductivity meter with standared solution

2- Put (   )ml, (   )N of HCl was pipette and transfer to 250ml beaker, dilute to 150ml with D. W., put the stirrer bar inside the solution, immerse the electrode

3- Titrate with $AgNO_3$ solution by adding approximately 0.2ml at each time, record conductance value after each addition

4- plot the titration curve between conductivity and volume of $AgNO_3$ solution
5- show and determine the equivalent point and volume of $AgNO_3$ at the equivalent point, calculate the concentration of $AgNO_3$ using this equation:

$$N \times V)_{HCl} = (N \times V)_{AgNO3}$$



Part B:

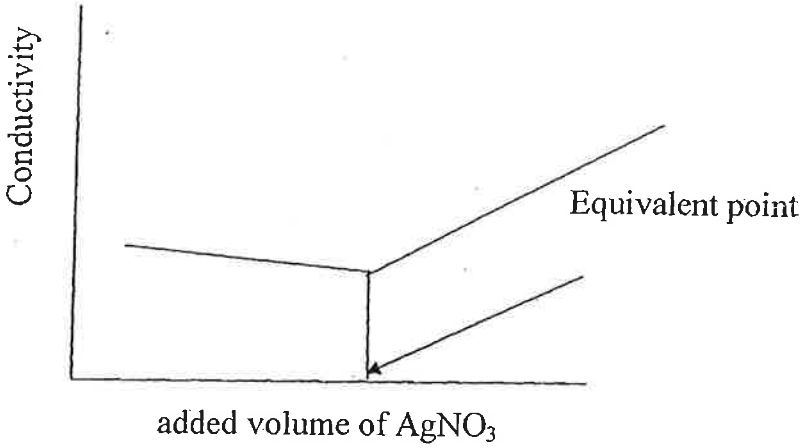
Apparatus: conductivity meter, magnetic stirrer, stirrer bar, burette, pipette, beaker

Reagents: NaCl salt (unknown), $AgNO_3$ solution, D.W.

Theory: Figure below illustrates the titration of sodium chloride with silver nitrat. The initial additions of reagent in effect cause substitution of chloride ions by the somewhat less mobile nitrate ions of reagent.

$$NaCl + AgNO_3 \longrightarrow AgCl + NaNO_3$$

Aslight decrease in conductance result. After the reaction is complete arapid increase occurs, owing to the addition of excess silver nitrate.



Procedure

1-Weight 0.5g of table salt, dissolve it in D.W., transfer the solution to 100ml volumetric flask, dilute to the mark

2- Pipette and transfer 1ml of this solution to 250ml beaker, dilute to 150ml with D.W.

3- Titrate against $AgNO_3$ solution as in part A

4- Show and determine the equivalent point and volume of $AgNO_3$ at the equivalent point

I- Calculate the concentration of NaCl using this equation:

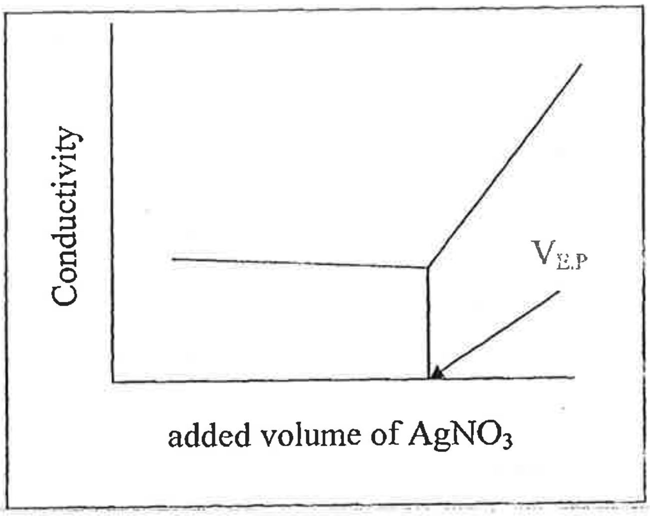
$$(N \times V)_{NaCl} = (N \times V)_{AgNO3}$$

II -Calculate the weight of NaCl in 100ml NaCl solution using this equation:

$$N_{NaCl} \frac{Wt_{NaCl}}{Eq.wt_{NaC}} \times \frac{1000}{V(ml)}$$

III-Calculate the percentage of NaCl in 0.5g of NaCl salt

$$\% \, NaCl = \frac{Wt_{NaCl}}{0.5} \times 100$$



Experiment No.4

conductometric titration (acid-base)

The purpose of experiment:

Part A: Calibration of NaOH using strong acid HCl

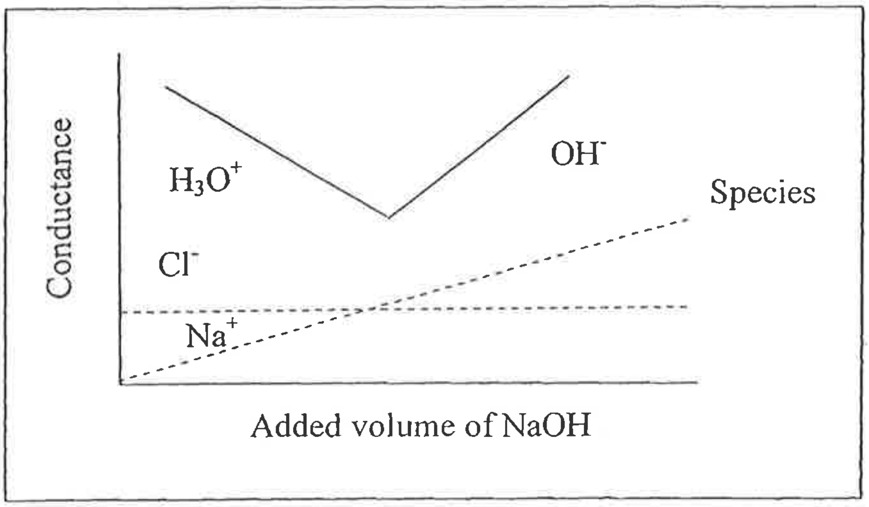
Part B: Determination the concentration of weak acid CH_3COOH

PartA:

Apparatus: Conductivity meter, magnetic stirrer, stirrer bar, burette, beaker.
Reagents: NaOH solution, standard solution of HCl and D.W.

Theory: A conductometric titration involves measurement of conductance of the sample after successive additions of reagent. The end point is determined from aplot of either the conductance or specific conductance as afunction of the volume added of titrant. These titration curve take avariety of shapes depending upon the chemical system under investigation.

Neutralization titration are particularly well adapted to conductometric end point because of the very high conductance of the hydronium and hydroxide ions compared with the conductance of the reaction products. Figure bellow represents acurve obtained when strong acid (HCl) is titrated with strong base (NaOH). During neutralization,-hydronium ions are being replaced by an equivalent number of loss mobile sodium ions and the conductance changes to lower values as aresult of this substitution. At the equivalence point the concentration of hydronium ions and hydroxide ions are at minimum and the solution exhibits its lowest conductance. After end point has been passed, areversal of slope occur as the sodium ion and the hydroxide ion concentration from the excess base increase



Conductometric titration of strong acid with strong base

Procedure

1- Calibrate the conductometer with standard solution

2- Put (   )ml, (   )N HCl was peptide and transferred to 250ml beaker, diluted with 150ml D.W., add the stirrer bar and dipped the conductivity cell, we make sure that when the stirrer is turned on, the stirrer bar will not hit the cell.

3- The solution was stirred for afew seconds, then the initial conductance was read.

4- Approximately 0.2ml of NaOH solution was added at each time from the burette to the solution, stirred for afew seconds, then the conductance was read.

5- The titration was continued until suitable volume (5-6)ml of NaOH was added and the conductance was read after each addition.

6- Tabulate all the results obtained in this titration.

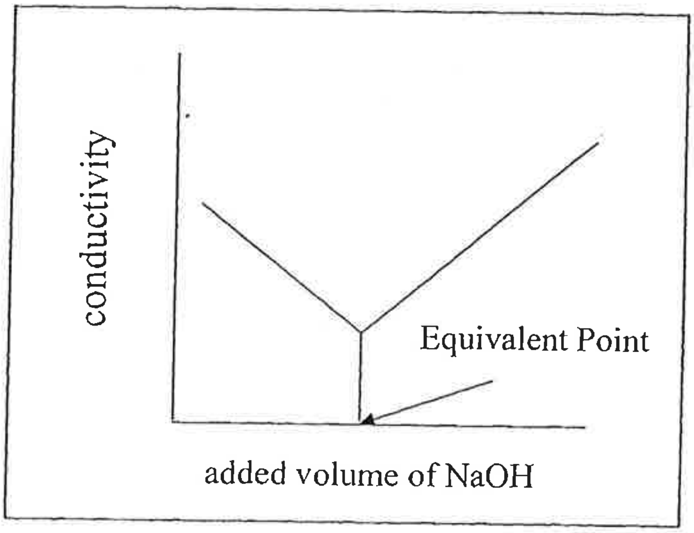
7-Plot the titration curve between conductivity versus volume of NaOH add as atitrant.

8-Show and determine the equivalent point and volume of NaOH at the equivalent point

9-Calculate the concentration of NaOH using this equation:

$$(N \times V)_{HCl} = (N \times V)_{NaOH}$$

10-Remove the conductivity cell and rinsed with distilled water.



PartB:

Apparatus: conductivity meter, conductivity cell, magnetic stirrer, stirrer bar, burette, beaker

Reagents: CH_3COOH solution(unknown), NaOH solution and D.W.

Theory: Figure bellow illustrates the titration of moderately weak acid, such as acetic acid ($K_a = 10^{-5}$) with sodium hydroxide. Nonlinearity in the early portions of titration curve causes difficulty in establishing the end point; with concentrated solution, however the titration is feasible. As before, we can interpret this curve in light of the changes in the composition that occur. Here the solution initially has amoderate concentration of hydronium ions. Addition of base results in the establishment of buffer system and aconsequent dimination in the hydronium ion concentration. Concurrent with this decrease in the increase in the concentration of sodium ion as well as the anion of the acid. These two factors act in apposition to one another, At first the decrease in hydronium ion concentration predominates and adecrease in conductance is observed. As the titration progresses, however, the pH becomes stabilized (in the buffer region); the increase in the salt content become the more important factor and alinear increase in conductance finally results. Beyond the end point, the curve become steepens because of the great ionic conductance of hydroxide.

Procedure:

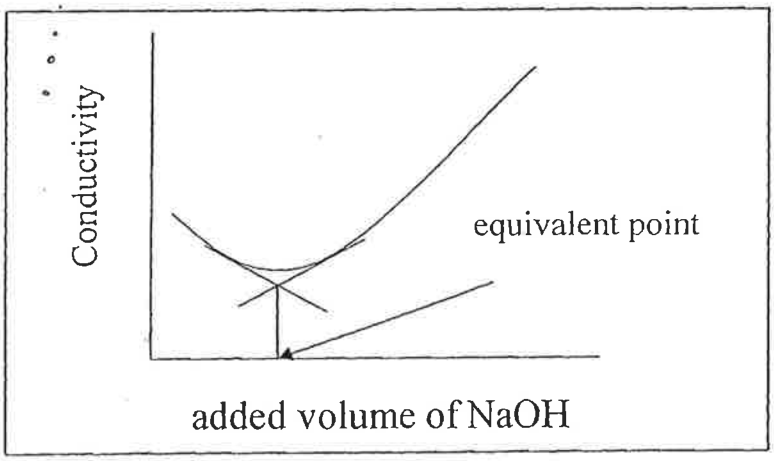
1- A (1)ml of unknown acetic acid was pipetted and transfer to 250ml beaker and diluted with 150ml D.W. and put the magnetic bar and dipped the electrode, make sure that when the stirrer is turned on, the stirrer bar will not hit the conductivity cell, the solution was stirred for afew seconds, then the initial conductance was read.
2- Approximately 0.2ml of NaOH 0.1N solution was added at each time from aburette to the solution, stirred for afew seconds then the conductance was read
3- The titration was continued until suitable volume of NaOH was added and the conductance was read after each addition.
4- Tabulate all the results obtained in this titration

5- plot the titration curve between conductivity versus volume of NaOH added as atitrant

6-Show and determine the equivalent point and volume of NaOH at the equivalent point

7-Calculate the concentration of CH_3COOH using this equation:

$$(N \times V)_{CH3COOH} = (N \times V)_{NaOH}$$



Experiment No.5

Conductometric determination of aspirin

The purpose of experiment: Determination of acetyl salicylic acid in aspirin tablet

Apparatus: Conductivity meter, magnetic stirrer, stirrer bar, burette, beaker, mortar, pipette, volumetric flask

Reagents: Aspirin tablet(unknown), NaOH solution, ethanol, D.W.

Procedure:

1-Weight one tablet of aspirin

2- Grind it very well using amortar, dissolved the powder in 12ml ethanol, transfer the solution to 100ml volumetric flask and dilute to the mark with D.W.

3- Transfer 40ml of the solution (step1) in to 250ml beaker, dilute to 150ml with D.W., put the magnetic bar inside the solution and immerse the electrode. Make sure that when the stirrer is turned on, the magnetic bar will not hit the conductivity cell.

4- Titrate with 0.1N NaOH, approximately 0.2ml 0f 0.1N NaOH solution was added at each time from aburette to the solution and stirred for afew seconds then read the conductance, continued the titration and the conductivity was read after each addition

5- Tabulate all the results obtained in this titration, plot the titration curve between conductivity and the volume of NaOH solution added as atitrant

6- Determination and show the equivalent point and volume of NaOH at the equivalent point.

I- Calculate the concentration of acetyl salicylic acid using this equation:

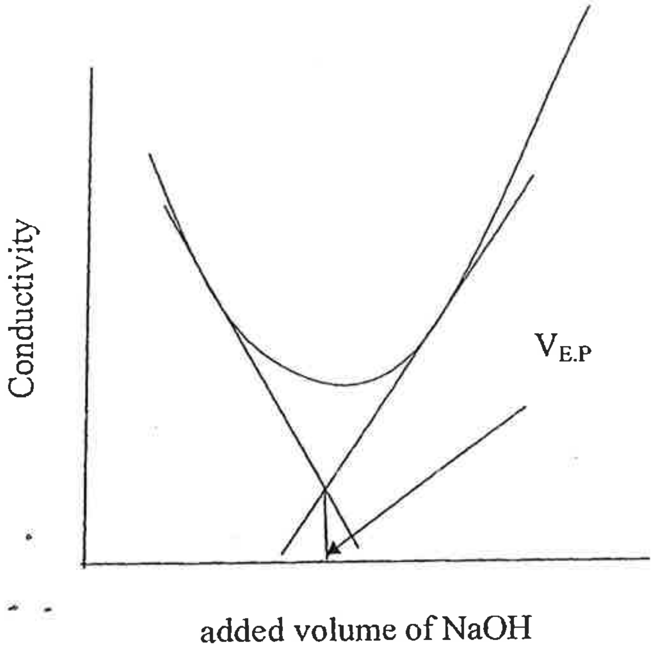
$$(N \times V)_{\text{acetyl salicylic acid}} = (N \times V)_{NaOH}$$

II-Calculate the weight acetyl salicylic acid ($C_6H_5OCOCH_2COOH$) (M.Wt = 180 g/mol) using the equation:

$$N = \frac{Wt}{Eq.wt} \times \frac{1000}{V(ml)}$$

III -Calculate the percentage of acetyl salicylic acid in aspirin tablet

$$\% = \frac{Wt_{\text{acetyl sal.}}}{Wt_{\text{aspirin}}} \times 100$$



Experiment No.6

Precipetation-Conductometric titration of OCl^- in bleach

The purpose of experiment:

Part A: Calibration of $AgNO_3$ solution
Part B: Determination of OCl^{-1} Levels in bleach

PartA:

Apparatus: conductivity meter, magnetic stirrer, stirrer bar, burette, beaker.
Reagents: $AgNO_3$ solution, standard solution of HCl and D.W.

Procedure:

1- A ( )ml of ( )N of HCl was transfer to 250ml beaker and diluted with 150ml D.W. , put the magnetic bar and dipped the electrode, make sure that when the stirrer is turned on, the stirrer bar will not hit the conductivity cell, the solution was stirred for afew seconds, then the initial conductance was read.

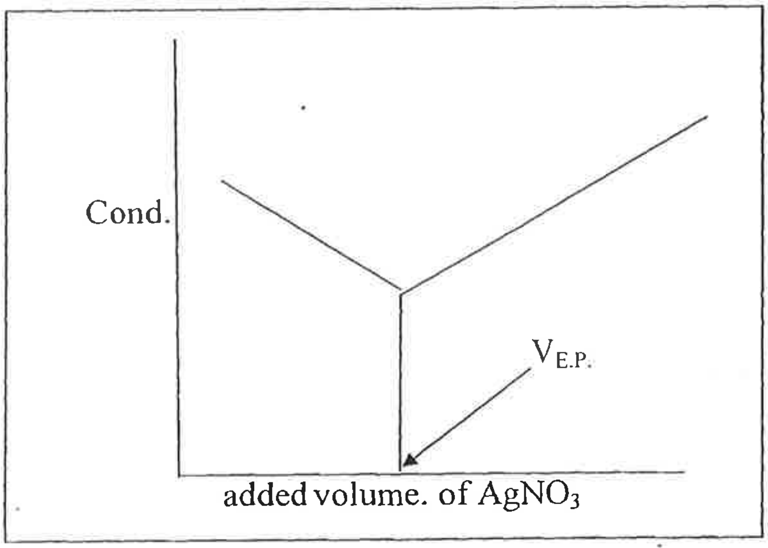
2- Approximately 0.2ml of $AgNO_3$ 0.1N solution was added at each time from aburette to the solution, stirred for afew seconds then the conductance was read
3- The titration was continued until suitable volume of $AgNO_3$ was added and the conductance was read after each addition.
4- Tabulate all the results obtained in this titration

5- plot the titration curve between conductivity versus volume of $AgNO_3$ added as atitrant

6-Show and determine the equivalent point and volume of $AgNO_3$ at the equivalent point

7-Calculate the concentration of $AgNO_3$ using this equation:

$$(N \times V)_{HCl} = (N \times V)_{AgNO3}$$



Part B: Determination of OCl^- conductivity in unknown bleach solution

Apparatus: conductivity meter, magnetic stirrer, stirrer bar, beaker, volumetric flask, burette, cylinder

Reagents: Bleach solution, $AgNO_3$ solution, D.W.

Theory: The active ingredient in house hold bleach is sodium hypochlorite, or NaOCl. This family is represented below:

$$ClO^- + 1/2O_2 \xrightarrow{Energy} ClO_2^- + 1/2O_2 \xrightarrow{Energy} ClO_3^- + 1/2O_2 \xrightarrow{Energy} ClO_4^-$$

Hypochlorite Chlorite Chlorate Per chlorate

Procedure:

1-Weight 2ml of bleach solution and transfer the solution quantitatively to 100ml volumetric flask and dilute to the mark with D.W.

2- pipette 2ml from the diluted solution above (step1) to 250ml beaker, dilute with 150ml D.W.

3- Titrate with $AgNO_3$ solution as in part A

4- Tabulate all the results obtained in this titration

5- plot the titration curve between conductivity versus volume of $AgNO_3$

6- Show and determine the equivalent point and volume of $AgNO_3$ at the equivalent point.

I-Calculate the concentration of $AgNO_3$

$$(N \times V)_{NaOCl} = (N \times V)_{AgNO3}$$

II-Calculate the weight of NaOCl:

$$N_{NaOCl} = \frac{Wt}{Eq.wt} \times \frac{1000}{V(ml)}$$

III-Calculate the weight of OCl^- :

$$\frac{Wt_{NaOCl}}{Eq.wt_{NaOCl}} = \frac{Wt_{OCl^-}}{Eq.wt_{OCl^-}}$$

VI-Calculate the weight ratio:

$$\frac{Wt}{Wt_{bleach}} = \frac{Wt_{OCl^-}}{Wt\ of\ 2ml} \times 100$$

Discussion:

Answer the following questions:

1-Why we are must calibrate $AgNO_3$ solution ?

2- $AgNO_3$ solution is not standard solution ?why?