Republic of Iraq

Ministry 0f Higher Education and scientific Research

Al-Ayin University

Petroleum Eng. College
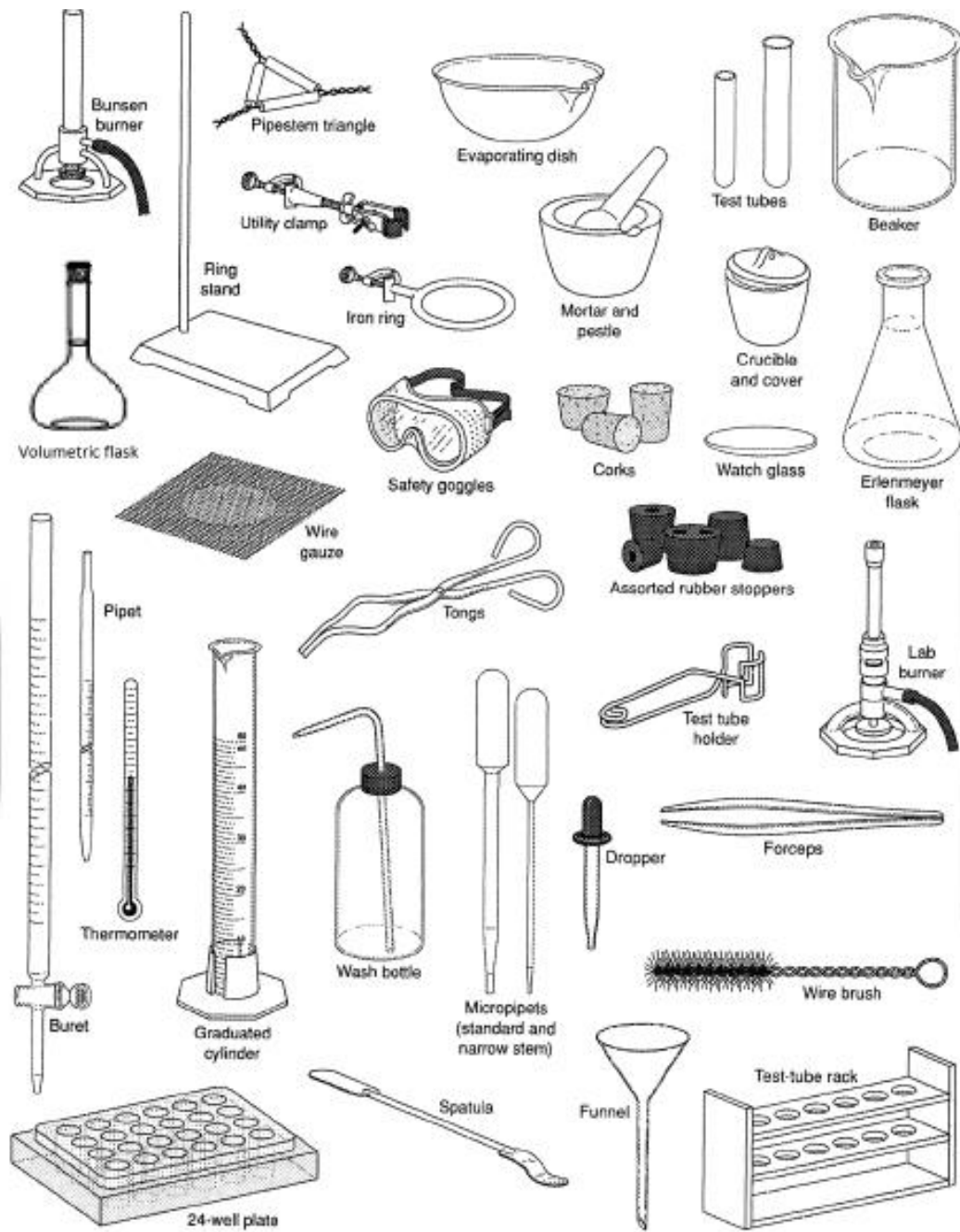
Practical

Analytical Chemistry

First year

2017-2018

Edited by

Montather Farid

Bunsen burner

Pipestem triangle

Evaporating dish

Test tubes

Beaker

Utility clamp

Ring stand

Iron ring

Mortar and pestle

Crucible and cover

Volumetric flask

Safety goggles

Corks

Watch glass

Erlenmeyer flask

Wire gauze

Tongs

Assorted rubber stoppers

Pipet

Test tube holder

Lab burner

Thermometer

Wash bottle

Dropper

Forceps

Buret

Graduated cylinder

Micropipets (standard and narrow stem)

Wire brush

Spatula

Funnel

Test-tube rack

24-well plate

Volumetric Analysis

Volumetric analysis, any method of quantitative chemical analysis in which the amount of a substance is determined by measuring the volume that it occupies.

Chemical procedure used for determining the concentration by measured of it is volume A known volume of a solution of unknown concentration is reacted with a known volume of a solution of known concentration (standard). This technique is known as titration.

Titration:

A titration is process for determining the amount of a substance by measurement of the quantity of a reagent required to react completely with that substance.

There are many types of titrations:

1. Acid-base titration

2. Reduction-oxidation reaction titration

3. Precipitation titration

4. Complexometric titration

equivalence point :

the equivalence point is the point where the amount of standard solution added is chemically equivalent to substance with which it reacts.

At the equivalence point of a titration, an exactly equivalent amount of titrant has been added to the sample. The experimental point at which the completion of the reaction is marked by some signal is called the end point. This signal can be the color change of an indicator during the titration.

Primary standard solutions :

Primary standard solutions are used in analytical chemistry and is a highly purified chemical compound . Features of a primary standard include:

1- High purity

2- High Stability and non-sensitive to atmospheric oxygen.

3- Not be hygroscopic.

4- Should be available and not too expensive.

5- Should have high molecular weight for weighing errors are minimized.

Indicator:

Is a chemical compound that exhibits a change in color as a result of concentration changes occurring near the equivalence point.

Indicator	*pH*	*Indicator color*	
		Acidity medium	*Basics medium*
Methyl orange (M.O)	3.1 – 4.4	Red	Yellow
Bromocresol Green	3.3 – 4.5	Yellow	Blue
Methyl Red (M.R)	4.2 – 6.3	Red	Yellow
Bromo Thymol Blue	6 – 7.6	Yellow	Blue
Phenol Red (P.O)	6 - 8	Yellow	Red
Cresol Indigo	7.4 - 9	Yellow	purple
Phenol Phthalin (Ph. Ph)	8 – 9.8	Colorless	Red
Thymol Blue	8 – 9.8	Yellow	Blue

Acid-base titration

Experiment No. (1)

Preparation and Standardization of an

Acid and base

Hydrochloric acid is produced in solutions up to 38% HCl (concentrated grade). Higher concentrations up to just over 40% are chemically possible, but the evaporation rate is then so high that storage and handling need extra precautions, such as pressure and low temperature.

Laboratory grade hydrochloric acid is not sufficiently pure to be used as a primary standard, because it evaporates easily . In this experiment, a standard solution of sodium carbonate is used to determine the exact concentration of a hydrochloric acid solution.

Procedure :

Part I: Preparation of 0.1 N HCl

1- From the bottle of HCl you have in your lab ,record the following data: Concentration (%), specific gravity (sp.gr), and calculate the molecular and equivalent weights of HCl.

2- Calculate the normality of this bottle from the following relation:

$$N = \frac{\text{sp.gr} * \% * 1000}{\text{eq.wt}}$$

3- To prepare another concentration from this bottle, calculate the required volume from this relation:

(N*V) before = **(N*V)** after

where:

N_{before} = the calculated normality of HCl bottle

V_{before} = the volume to be taken (required)

N_{after} = the required normality of the solution

V_{after} = the final volume of the prepared solution

4- Take V_{before} from the bottle and complete it with distilled water to the final volume. This way you will have the required concentration of HCl.

Part II: Standardization of the prepared HCl using sodium carbonate

1- Transfer 10 ml exactly 0.1 N sodium carbonate Na_2CO_3 (primary standard) into a conical flask .

$$N = \frac{wt}{eq.wt} * \frac{1000}{V}$$

2- Add 2 drops of methyl orange as an indicator (the solution will have a yellow color) .

3- Wash the burette with HCl before filling it with the prepared solution of HCl. Record the volume on the burette (V_1).
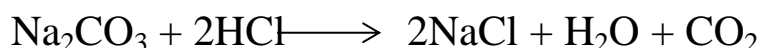
4- Titration with HCl solution drop by drop from the burette into the conical flask to react with sodium carbonate until a faint orange color is obtained . Record the burette volume(V_2).
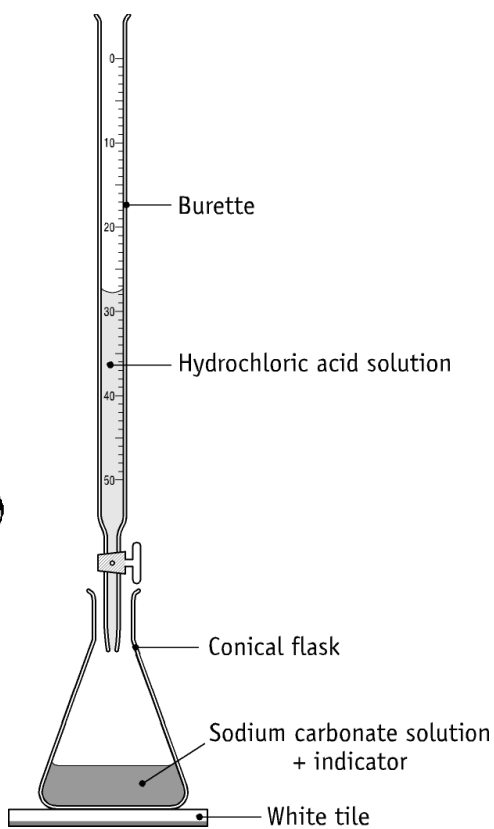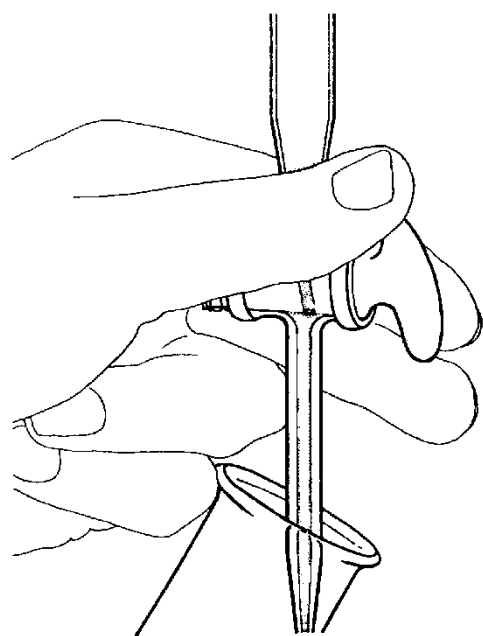
5- Calculate the consumed HCl volume:

$V_{total} = V_2 - V_1$

6- Calculate the concentration of HCl using the following equation:

$(N_1 * V_1)$ $_{HCl}$ = $(N_2 * V_2)$ $_{Na2CO3}$

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + H_2O + CO_2$$

Burette

Hydrochloric acid solution

Conical flask

Sodium carbonate solution + indicator

White tile

Discussion:

1- How would you prepare

   a- 500 ml of 0.1 M HCl ?

   b- 1 L of 0.9 N HCl ?

      Note : atomic weight for Cl=35.5, H=1

      Sp.gr (HCl)=1.18   %w/w HCl= 37%

2- Why HCl is not primary standard material ?

Experiment No. (2)

Determination of the normality of 0.1N NaOH solution

Titration of a strong acid with a strong base is the simplest of the four types of titrations as it involves a strong acid and strong base that completely dissociate in water. this titration requires the use of a burette to dispense a strong base into a container of strong acid, or vice-versa, in order to determine the equivalence point

Procedure :

1- *Preparation of an ≈ 0.1 N NaOH*

2- *Transfer 10ml of standard HCl solution to a conical flask.*

3- *Add 1-2 drops of phenolphthalein (ph.ph) as an indicator.*

4- *Fill the burette with the prepared NaOH solution.*

5- *Add NaOH drop by drop into the conical flask until the color of the solution is faint pink.*

6- Calculate the concentration of NaOH using the following equation :

$$HCl + NaOH \longrightarrow NaCl + H_2O$$
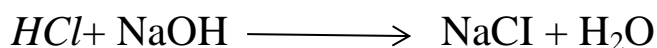
$$(N_1 * V_1)_{HCl} = (N_2 * V_2)_{NaOH}$$

Discussion:

1- How would you prepare 500 ml of 0.1 N NaOH ?

   Note : atomic weight for Na=23 , O=16 , H=1

2- It is usually impossible to obtain NaOH of sufficient purity to use it as a primary standard ?

3- A student sets up the apparatus shown below for the titration of sodium hydroxide solution with a standard hydrochloric acid solution :



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funnel

sodium hydroxide solution
of unknown concentration

standard hydrochloric acid
solution (0.1 M) 25 cm³
+ 2 drops of phenolphthalein
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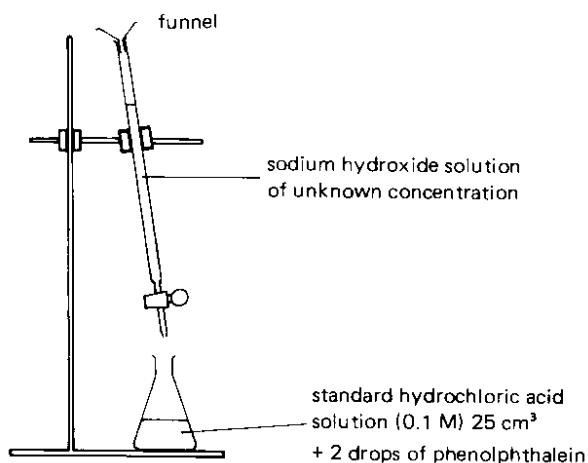
What errors has be made in the set up ?

Experiment No. (3)

Determination of Acetic Acid Content of Vinegar

Acetic acid is the major acid that is found in vinegar. Acetic acid is also known as ethanoic acid, and its molecular formula is $C_2H_4O_2$. It is found in many other substances other than vinegar, such as explosives, sugars, and starch. Acetic acid can be formed in the atmosphere and it can also be produced when biological waste decomposes. Humans are regularly exposed to acetic acid through foods and also from the air and are not in any danger from this exposure . Acetic acid by itself is clear and colorless. It gives vinegar its distinct odor

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$
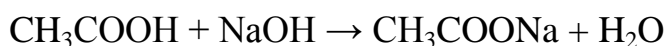
Colorless   **Red** .

Procedure :

Part I: Preparation of unknown acetic acid solution :

1-Transfer 10 ml unknown into 100 ml volumetric flask.

2- Complete the volume with distilled water .

3- Stopper the flask and shake well .

Part II: Determination of the % w/v of acetic acid in vinegar :

1- Transfer 10 ml of the prepared solution to conical flask .

2- add one or two drops of ph. ph indicator to this solution.

3- Fill the burette with 0.1 N NaOH solution.

4- Titrate the prepared acetic acid solution with standard NaOH until the color of the solution passes from Colorless to faint red /pink.
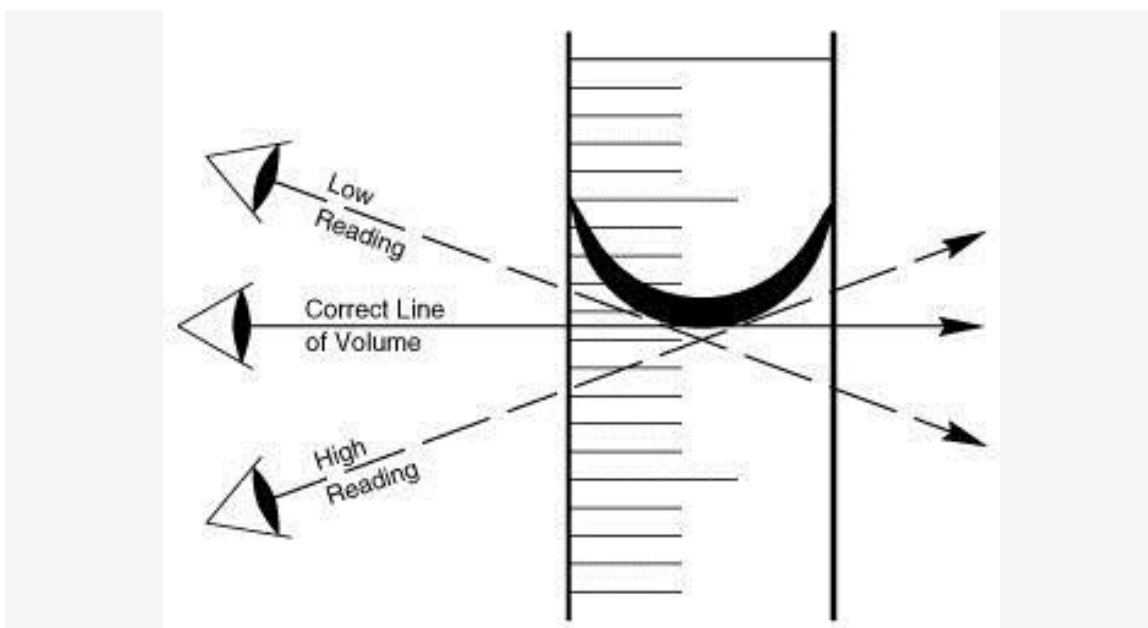
Calculation:

$$(N * V)_{HAc} = (N * V)_{NaOH}$$

Weight of acetic acid (HAc) in 10 ml of the diluted unknown

$$(N * V)_{NaOH} = \frac{wt\ of\ HAc}{eq.wt\ of\ HAc} * 1000$$

$$\%\ ^W/_V\ of\ HAc = \frac{wt\ of\ HAc}{V\ of\ dil\ HAc} * 100$$

Discussion:

1- Calculate the W/V% HAc for a 10 ml sample of HAc in vinegar that required 30 ml of 0.4 N NaOH solution ?

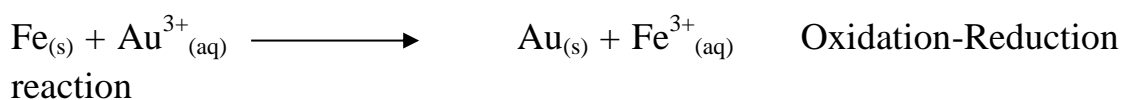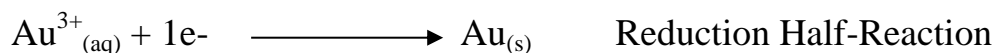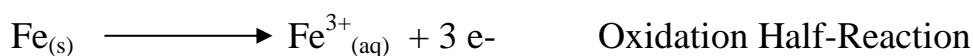Note : at.wt for Na=23 , O=16 , H=1 , C= 12

2- Which indicator is suitable for titration of NaOH against HAc solution ? and why?

Oxidation-Reduction Titrations

Reactions in which electrons are transferred from one species to another are called oxidation-reduction (redox) reactions. When one species loses electrons by an oxidation process another species simultaneously gains electrons by a reduction process in a chemical reaction. The balanced chemical reaction can be written as the combination of two half-reactions representing the oxidation reaction and the reduction reaction , respectively.

For example, if solid iron is placed in a solution of gold (III) ions, the gold (III) ions are reduced to solid gold and the iron is oxidized to iron(III) ions , according to the following half-reactions:

$Fe_{(s)} \longrightarrow Fe^{3+}_{(aq)} + 3\ e-$ Oxidation Half-Reaction

$Au^{3+}_{(aq)} + 1e- \longrightarrow Au_{(s)}$ Reduction Half-Reaction

$Fe_{(s)} + Au^{3+}_{(aq)} \longrightarrow Au_{(s)} + Fe^{3+}_{(aq)}$ Oxidation-Reduction reaction

Experiment No. (4)

Standardization of potassium permanganate KMnO$_4$

Potassium permanganate is not primary standard substance because it contains reduced products like manganese oxide MnO$_2$ where the concentration of KMnO4 changed after preparation because it dissociated via reducing agents such as ammonia and organic substances in water ;therefore, potassium permanganate must be standardized before use it and keep it at dark place and dark bottle.

## *Procedure* :

Part I: Preparation of ≈ 0.1 N KMnO$_4$ solution :

1- Dissolve 0.8 gm of solid KMnO$_4$ in 100 ml of distilled water .

2- Heat the solution on hotplate for 10 minutes at 70-80 C^0 .

3- Cool the solution to room temperature , then add 2.5 ml of concentration sulfuric acid.

4- Complete the volume up to 250 ml by distilled water.

5- Store the solution in the dark.

Part II: Standardization of KMnO$_4$ solution against sodium Oxalate

1- Fill the burette with the KMnO$_4$ solution .

2- Transfer 10 ml of 0.1 N sodium oxalate solution to a conical flask

3- Add 5 ml concentrated H$_2$SO$_4$.

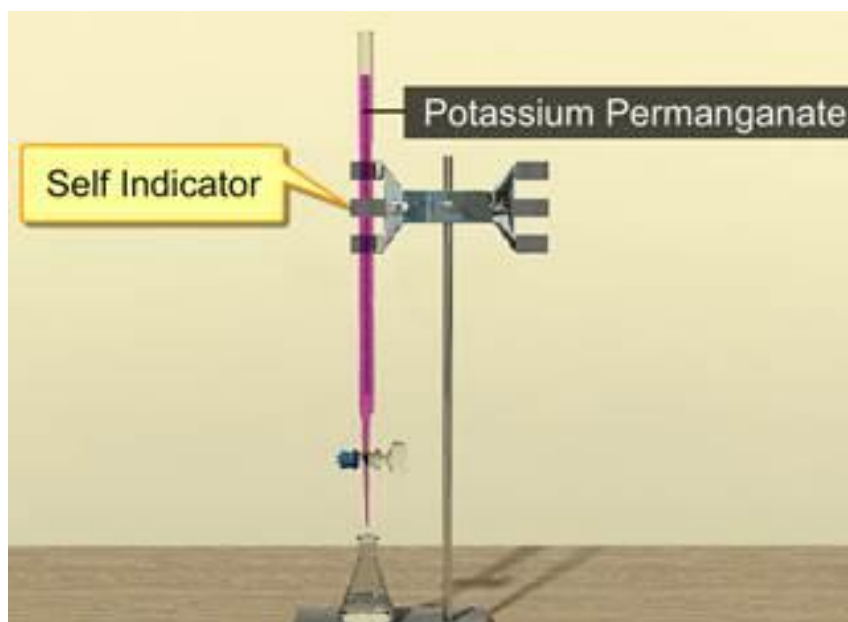4- Heat to 65 C^0 (as needed) titrate against $KMnO_4$. the end point is the first permanent pink color .

$$2MnO_4^- + 5C_2O_4^{2-} + 16H^+ \rightleftharpoons 2Mn^{2+} + 10CO_2 + 8H_2O$$
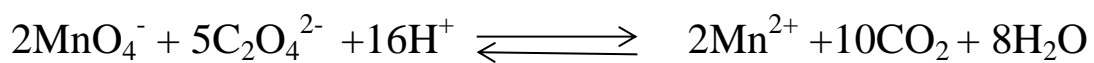
Calculation:

$(N_1 * V_1)_{KMnO4} = (N_2 * V_2)_{Na2C2O4}$

Discussion:

1- Cooling the $KMnO_4$ solution before added concentrated sulfuric acid ?

2- Heat the solution (sodium oxalate + sulfuric acid) to 65 C^0 ?

3- What means "self indicator" ?

Petroleum Eng. College   *Practical Analytical chemistry*

Precipitation titrations

Precipitation titrations are a form of titration useful in the determination of halides such as chlorides, bromides and iodides. These titrations involve the use of a precipitating agent such as silver nitrate, and are therefore also known as argentimetric titrations. Depending on the method of detecting the end point of the titration
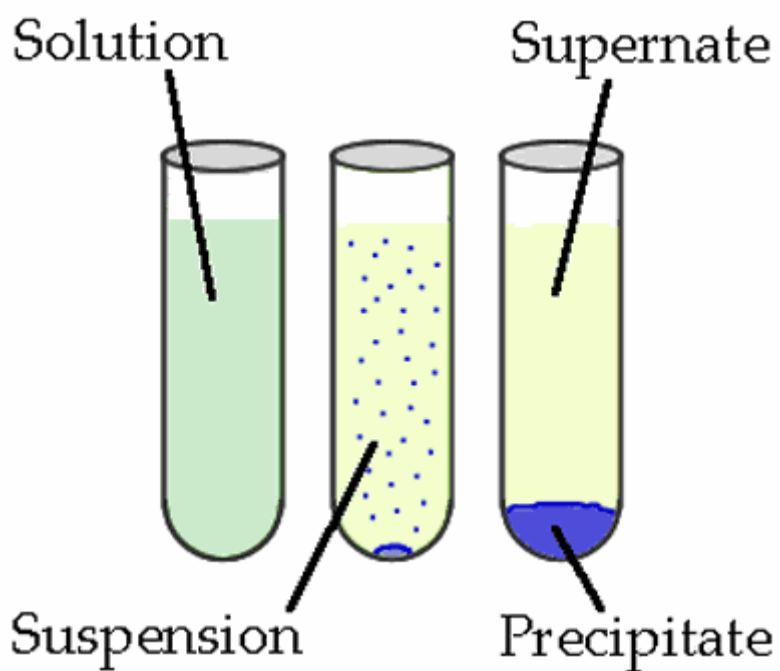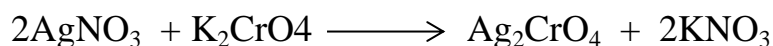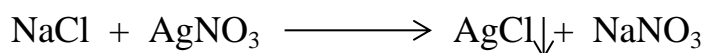
Experiment No. (5)

Determination of Chloride by the Mohr Method

The chloride ion is titrated with standard silver nitrate , AgCl is formed as a precipitate . the end point is the appearance of brick-red color of silver chromate $AgCrO_4$.

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

$$2AgNO_3 + K_2CrO4 \longrightarrow Ag_2CrO_4 + 2KNO_3$$

Procedure :

Part I: Standardization of AgNO₃ solution:

1- Transfer 10 ml of standard 0.1 N NaCl solution into a conical flask .

2- Add 4 drops of K_2CrO4 indicator and titrate slowly with silver nitrate solution until a faint red color is formed .

Calculation :

$$(N_1 * V_1)_{AgNO3} = (N_2 * V_2)_{NaCl}$$

Part II: Determination of Chloride

1- Transfer 10 ml of unknown solution containing KCl or NaCl into a conical flask .

2- Add 1 ml of K_2CrO4 as an indicator .

3-Titrate with standard 0.1 N silver nitrate solution , slowly , until a faint reddish color appears .

Burette containing
silver nitrate solution

Conical flask

Seawater and chromate
indicator
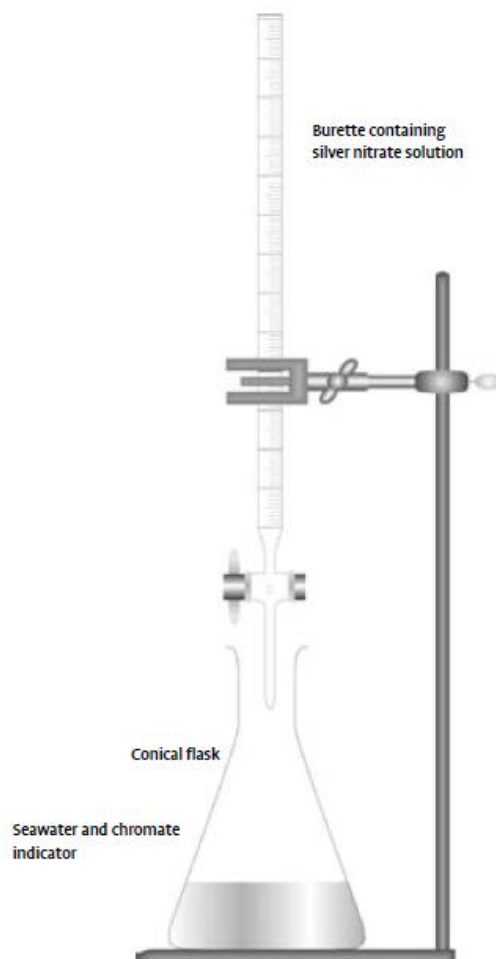
Calculation:

At. wt of Cl^- = 35.45 g/mole

mmole of $Cl^- = M_{AgNO3} * V_{AgNO3}$

wt of Cl^- = mmoles of Cl^- * At.wt of Cl^-

Discussion:

1- Why $AgNO_3$ is not primary standard material ?

2- What is a Mohr Method ?

Complexometric Titration

Complexometric Titration or chelatometry is a type of volumetric analysis wherein colored complex is used to determine the endpoint of the titration. Complexometric Titration is in the detection of mixtures of different metal ions present in the solution.

When each drop of titrant is added, the reaction reaches an equilibrium state swiftly. There would be no chance for any interfering situations. The equivalent point can be identified very accurately using a complexometric titration. ethylenediaminetetraacetic (EDTA) is used as a titrant and it is pretty much well established.

$$HOOC-CH_2 \diagdown \qquad\qquad\qquad\qquad CH_2-COOH$$
$$:N-CH_2-CH_2-N:$$
$$HOOC-CH_2 \diagup \qquad\qquad\qquad\qquad CH_2-COOH$$

structure of EDTA

Experiment No. (6)

Determination of total hardness in tap water

Water hardness is a measure of the amount of calcium and magnesium salts dissolved in water. There are no health hazards associated with water hardness, however, hard water causes scale, as well as the reduced lathering of soaps. Hard water should be not used for washing (it reduces effectiveness of detergents) nor in water heaters and kitchen appliances like coffee makers (that can be destroyed by scale). It is also not good for fish tanks. In general, there are many applications where ability to easily determine water hardness is very important.

Procedure :

1- Transfer 50 ml of the tap water into a conical flask .

2- Add 10 ml of buffer 10 .

3- Add 0.2 gm of Eriochrome black –T as an indicator .

4- Titrate against 0.01 F EDTA solution until the color of the solution changes from red to blue .

Note: $CaCO_3$ represents the total hardness of H_2O

$$V * M_{EDTA} = \frac{wt.of\ CaCO3}{M\ .wt} * 1000$$

$$M\ .wt\ of\ CaCO_3 = 100$$

$$ppm_{CaCO3} = \frac{wt.of\ CaCO3}{50} * 10^6$$

Discussion:

1- Why added buffer solution to tap water?

 2- What is the difference between tap water and distilled ?

Reference :

1- Gary D. Christion , Purnendu K. Dasgupta ,"Analytical Chemistry ", Wiley ,seventh edition, (2014).

2- F. P. Treadwell , " Analytical Chemistry" , Polytechnic Institute of Zurich , (1903).

3- Najib Al-Sabea , " Practical analytical chemistry " , university of Baghdad,(2011).

4-Ruba F. Abbas , "Practice Volumetric Chemical Analysis", Al-Mustansiriya university, Baghdad, (2012).