2024

MANSOURA COLLEGE

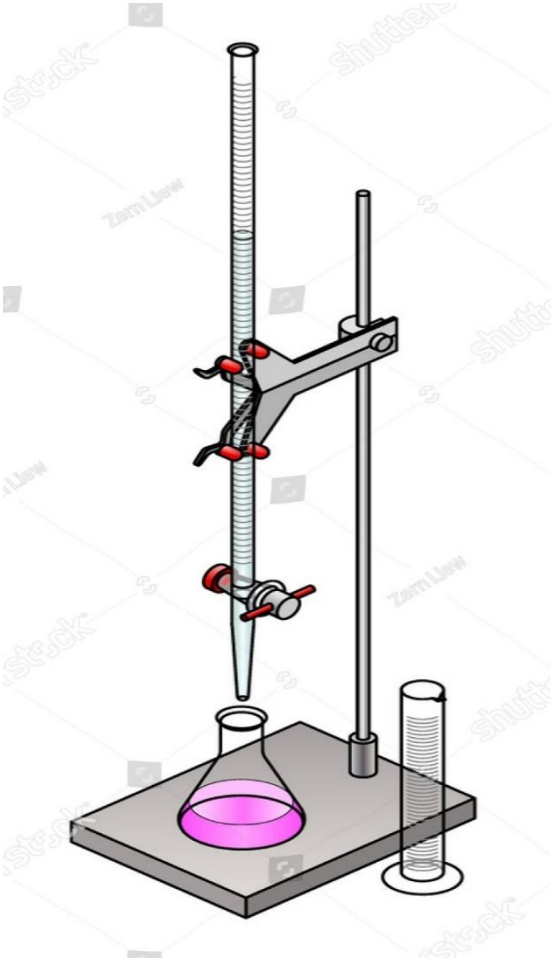
Mansoura High Institute Of Engineering And Technology

Chemistry Laboratory Manual

Practical

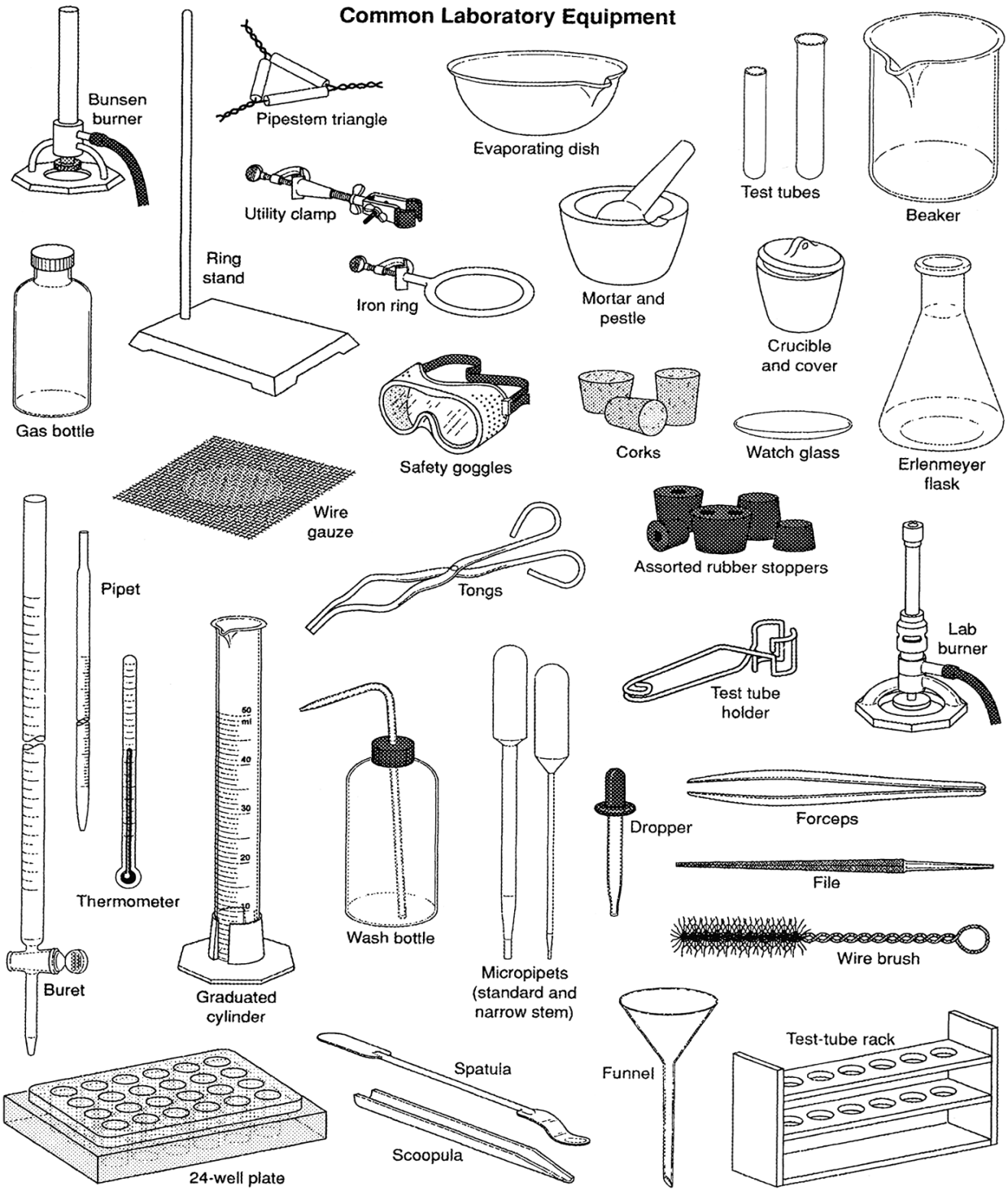
Engineering

Chemistry



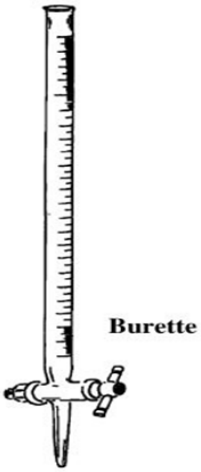
For

Engineering Chemistry

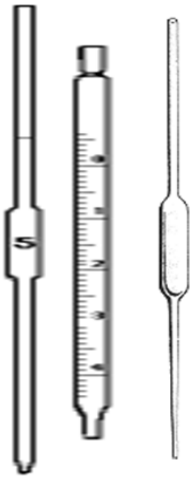

Common Laboratory Equipment
Bunsen burner
Pipestem triangle
Evaporating dish
Test tubes
Beaker
Utility clamp
Ring stand
Iron ring
Mortar and pestle
Crucible and cover
Gas bottle
Safety goggles
Corks
Watch glass
Erlenmeyer flask
Wire gauze
Tongs
Assorted rubber stoppers
Pipet
Lab burner
Test tube holder
Thermometer
Wash bottle
Dropper
Forceps
File
Wire brush
Buret
Graduated cylinder
Micropipets (standard and narrow stem)
Spatula
Funnel
Test-tube rack
Scoopula
24-well plate

Contents

COMMON LABORATORY GLASSWARES

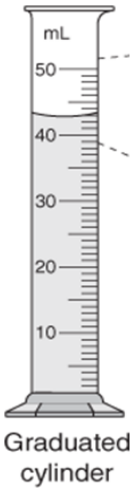


Burette

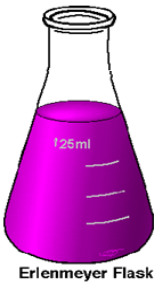


Pipette

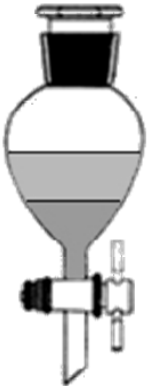
Test-tube



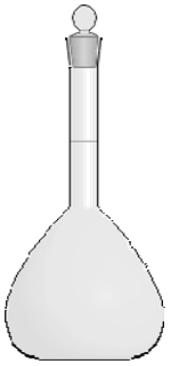
Measuring cylinder



Conical flask



Separating funnel

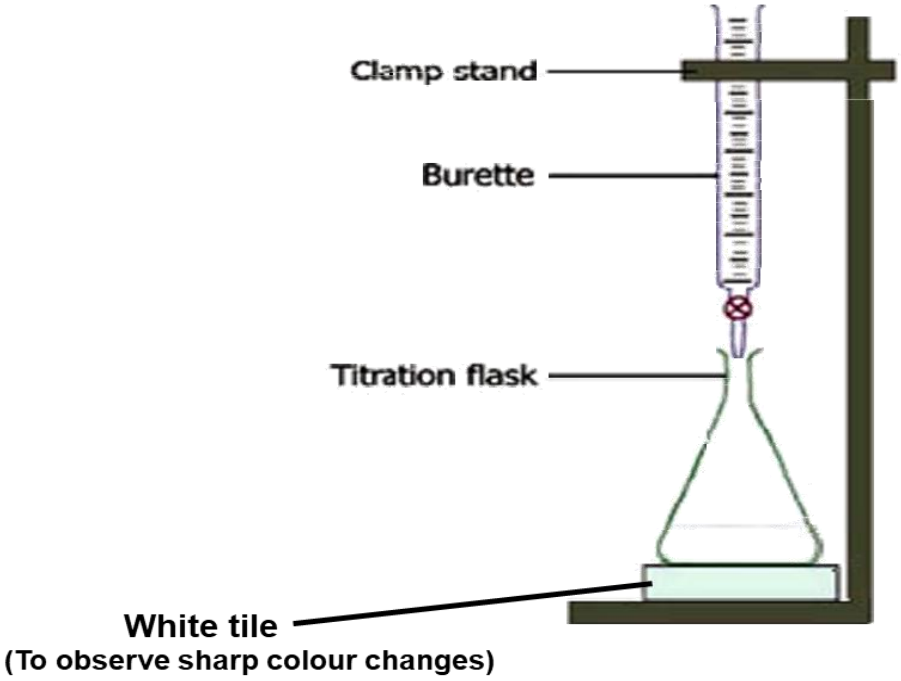


Volumetric flask

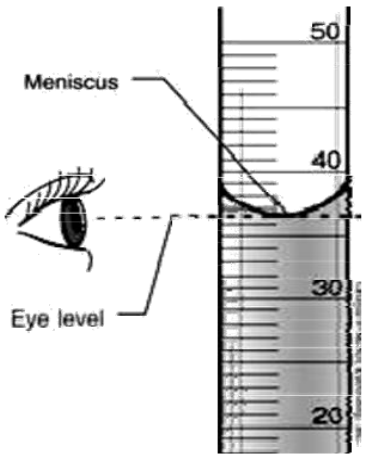
Beaker

Filter funnel

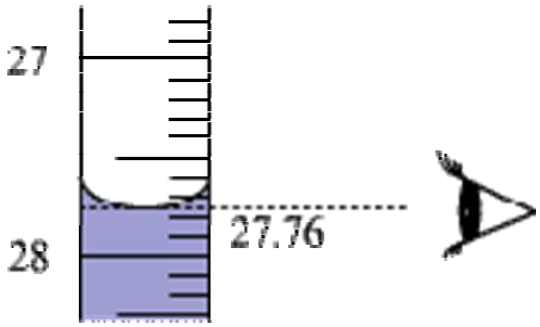
TITRATION ASSEMBLY



Correct method to note down the readings



Graduated Cylinder
The reading is 36.5 ml.



Burette
The reading is 27.8 ml

Constructions

A. GENERAL:

1. Keep your bags in the cupboards below the working table
2. First-aid kit is available for emergency use only in the laboratory. Band-aids for minor cuts are also available in the First-aid kit. Notify your instructor or the technicians if you use safety items.
3. Notify your instructor if any accidents and/or injuries, regardless of their severity. If you need medical treatment, you will be promptly taken to the nearby Health Center.
4. Learn the location and use of safety equipments, including the eyewash bottle, fire extinguisher, and sand bucket.
5. Work cautiously with chemicals only after you have learned about their potential hazards as well as the chemical properties. Laboratory has a catalogue of MSD (Material Safety Data Sheet) sheets that contain all the information about chemicals.
6. Wash your hands well before leaving the laboratory.
7. Keep your hands away from your face, while working.
8. Handle the apparatus and chemicals carefully.
9. Leave plenty of tap water after discarding the waste in the sink.
10. In the event of a chemical spill, large or small, consult your laboratory instructor or the technician as to the appropriate method of clean-up.

B. HANDLING OF CHEMICALS & WASTE DISPOSAL IN THE LABORATORY:

1. To avoid spattering of acids which can cause burns, always add acid to water. Never add water to acid.

2. Before taking any reagent, you must carefully read the label on the bottle. Many chemicals have similar names however they may exhibit different properties viz. concentration level, etc.

3. To avoid unnecessary waste, obtain only the required amount of chemicals in an experiment. Your instructor will tell you the proper procedure for dispensing liquids and solids.

4. Never return unused chemicals to the reagent bottle without prior permission of the instructor.

5. Follow scrupulously the instructor's instructions in case of disposing the chemicals. Dispose of non-hazardous, water soluble substances in the sink, and put insoluble materials such as filter paper in waste basket.

6. Broken glass must be put into the containers specified for that purpose.

7. Before leaving the Laboratory please ensure, clean off the surface. Remove matches & papers and wipe down the surface with wet cloth.

C. EYE PROTECTION:

1. If you get an irritating substance in your eye, move quickly to the eye washer, and wash your eyes thoroughly for at least 15 minutes. Do not take this incidence as a common one. Have someone notify the instructor of the accident so that you can be taken to the nearby Health Center immediately.

3. Remove contact lenses while performing experiment in the laboratory.

D. FIRE HAZARD:

1. In case of fire bring the fact immediately in the notice of concerned laboratory instructor.

2. Do not dry chemicals in a drying oven or heat any materials with an open flame unless specifically directed to do so by the laboratory instructor.

E. CONTACT & INGESTION HAZARD:

1. If you spill a corrosive substance on your skin or clothing, wash it off with plenty of water for 15 minutes. Notify the instructor of any spillage as soon as possible; he/she will provide any necessary secondary treatment and will arrange for your transportation to the Health Center, if necessary.

2. Never eat, drink, or taste anything in the laboratory.

3. Smoking & use of cell phones are strictly prohibited in the laboratory.

Experiment (1)

Titration of Acids and Bases

- **Objectives:**
  - Standardize a sodium hydroxide solution
  - Determine the molarity of an unknown hydrochloric acid solution
  - Understand the use of indicators in titrations
  -
  - Learn proper pipetting technique
  - Learn to titrate a strong acid with a strong base
- **Safety Notes:**
  - Eye protection must be worn at all times.
  - Hydrochloric acid and sodium hydroxide are caustic and should not come in contact with your skin or clothing.
  - Wear gloves when handling these chemicals. A lab coat or lab apron is recommended.
- **Discussion:**

Titration describes a process where the concentration of an unknown substance is determined by comparing it with a solution of known concentration. The concept that makes titrations possible is finding the equivalence point, i.e., identifying when the quantity of the unknown substance is equal to the quantity of the known substance.

المعايرة عملية يتم فيها تحديد تركيز مادة غير معروفة من خلال مقارنتها بمحلول تركيز معروف. المفهوم الذي يجعل المعايرة ممكنة هو إيجاد نقطة التكافؤ ، أي تحديد متى تكون كمية المادة غير المعروفة مساوية لكمية المادة المعروفة.

The equivalence point is found in a titration by adding trace amounts of a substance, called an indicator, which turns color when the equivalence point is reached. When a strong acid is titrated with a strong base, or vice versa, the pH of the solution will be about 7.0 at the equivalence point. Phenolphthalein **(Ph.Ph)** is the indicator used in this experiment. Phenolphthalein **(Ph.Ph)** is colorless in acidic solutions and turns pink in alkaline solutions.

تم العثور على نقطة التكافؤ في المعايرة عن طريق إضافة كميات ضئيلة من مادة ، تسمى الدليل ، والتي تحول اللون عند الوصول إلى نقطة التكافؤ. عندما يتم معايرة حمض قوي بقاعدة قوية ، أو العكس ، فإن الرقم الهيدروجيني للمحلول سيكون حوالي 7.0 عند نقطة التكافؤ. الفينول فثالين هو الدليل المستخدم في هذه التجربة. الفينول فثالين عديم اللون في المحاليل الحمضية ويتحول إلى اللون الوردي في المحاليل القلوية.

This experiment will be done in two parts:

(1) preparation and standardization of a 0.1 M sodium hydroxide solution and

(2) determination of the acid concentration in an unknown sample.

Standardization is the process of determining concentration in an unknown solution by titrating it with a solution of known concentration.

Ionic substances dissociate completely when placed in water. Strong acids are ionic substances that form H^+ (or more correctly, H_3O^+) when placed in water and strong bases are ionic substances that form OH when dissolved in water.

تنفصل المواد الأيونية تمامًا عند وضعها في الماء. الأحماض القوية عبارة عن مواد أيونية تشكل H^+ (أو بشكل صحيح ، H_3O^+) عند وضعها في الماء والقواعد القوية هي مواد أيونية تشكل OH عند إذابتها في الماء.

Hydrochloric acid, HCl, is the strong acid used in this experiment and sodium hydroxide, NaOH, is the strong base. The approximate concentrations of our solutions will be 0.1 M, and the goal of the experiment is to determine the exact concentration of the unknown acid solution.

Acids contain hydrogen and dissociate in water to give H^+:

$$HCl_{(aq)} \rightarrow H^+_{(aq)} + Cl^-_{(aq)}$$

Bases contain the hydroxyl group and dissociate in water to give OH^-:

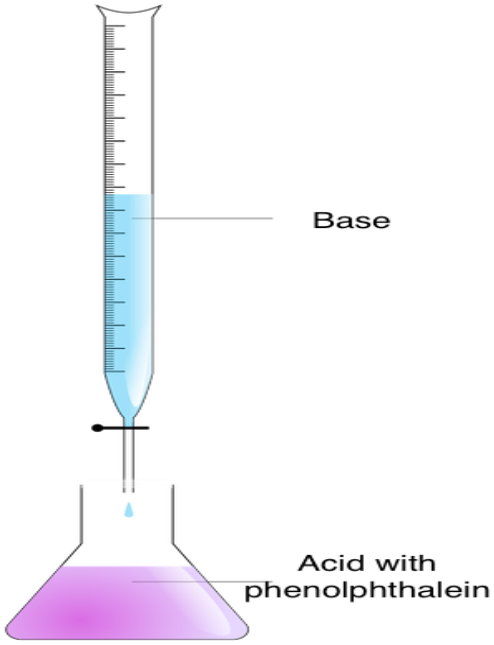
$$NaOH(aq) \rightarrow Na^+_{(aq)} + OH^-_{(aq)}$$

An acid (such as HCl) and a base (such as NaOH) react to form water and a salt. In this reaction, we say the acid and the base are **neutralized**:

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow Na^+_{(aq)} + Cl^-_{(aq)} + H_2O_{(l)} \text{ (balanced equation)}$$

Throughout this experiment the NaOH solution will be placed in the burette, and the hydrochloric acid solution will always be in the beaker. The phenolphthalein **(Ph.Ph)** indicator is colorless in acidic solution, and it will turn pink at the equivalence point. A small excess of OH^- is what causes the indicator to change from colorless to pink, which is the titration end point. *(Note: You should stop adding NaOH when the solution in the beaker holds a light pink color for about 10 seconds!).*

خلال هذه التجربة ، سيتم وضع محلول NaOH في السحاحة ، وسيكون محلول حمض الهيدروكلوريك دائمًا في الدورق. يكون مؤشر الفينول فثالين عديم اللون في المحلول الحمضي ، وسوف يتحول إلى اللون الوردي عند نقطة التكافؤ. الزيادة الصغيرة في OH^- هي ما يتسبب في تحول المؤشر من عديم اللون إلى اللون الوردي ، وهي نقطة نهاية المعايرة. (ملاحظة: يجب التوقف عن إضافة هيدروكسيد الصوديوم عندما يحتفظ المحلول الموجود في الدورق بلون وردي فاتح لمدة 10 ثوانٍ تقريبًا!).



- **Procedure:**

1. Rinse your burette twice with about 5 mL of the NaOH solution.

(يتم تنضيف السحاحة مرتين بحوالي 5 مل من محلول هيدروكسيد الصوديوم)

2. Fill the burette with the NaOH solution and position it above an Erlenmeyer flask (Note: be sure there are no air bubbles in the tip of the burette).

(املأ السحاحة بمحلول هيدروكسيد الصوديوم وضعه فوق دورق مخروطي (ملاحظة: تأكد من عدم وجود فقاعات هواء في طرف السحاحة).

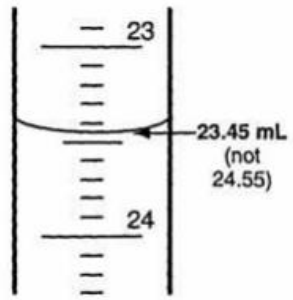


Figure 1. How to read a buret volume.

3. Obtain a 10.00 mL pipet and rinse it twice with about 5 mL of the unknown HCl acid solution.

(الحصول على ماصة 10.00 مل وتنظيفها مرتين بحوالي 5 مل من محلول الحمض)

4. Transfer 10.00 mL of the unknown HCl acid solution into the Erlenmeyer flask.

(يتم وضع 10.00 مل من محلول حمض الهيدروكلوريك المجهول التركيز إلى الدورق المخروطى)

5. Add 1-2 drops of phenolphthalein indicator solution into the flask.

(أضف 1-2 قطرات من دليل الفينول فثالين في الدورق المخروطى)

6. Before you begin the titration, be sure to record the initial volume of NaOH in the burette.

(قبل أن تبدأ المعايرة ، تأكد من تسجيل الحجم الأولي لـ NaOH في السحاحة)

7. Begin the titration, swirling the solution in the flask as you add NaOH in a drop-wise approach *(Note: Your solution will initially turn pink, and then fade back to colorless when swirled. The pink color will remain longer as you approach the end point of the titration).*

(ابدأ المعايرة ، وقم بتدوير المحلول في الدورق المخروطى أثناء إضافة هيدروكسيد الصوديوم (ملاحظة: سيتحول المحلول إلى اللون الوردي في البداية ، ثم يتلاشى مرة أخرى إلى عديم اللون عند الدوران. سيظل اللون الوردي أطول كلما اقتربت من نقطة نهاية المعايرة).

8. Record the final volume of NaOH in the burette when the solution in the Erlenmeyer flask remains light pink color for about 10 seconds.

(سجل الحجم النهائي لـ NaOH في السحاحة عندما يظل المحلول الموجود في الدورقالمخروطى بلون وردي فاتح لمدة 10 ثوانٍ تقريبًا)

9. Repeat this procedure two more times, refilling the NaOH in the burette as needed.

- **Results:**

Initial volume of NaOH (mL)	Final volume of NaOH (mL)	Total volume of NaOH used (mL)	Mean volume of NaOH used (mL)

Total volume NaOH used = Final volume NaOH – Initial volume NaOH

- **Calculations:**

Use the following relationship to calculate the HCl concentration:

$$\mathbf{V_a x\ N_a = V_b\ x N_b}$$

where, $\mathbf{V_a}$: The volume of NaOH solution, mL

$\mathbf{N_a}$**:** The Normality of the NaOH solution

$\mathbf{V_b}$**:** The volume of HCl solution, mL

$\mathbf{N_b}$**:** The Normality of the HCl solution

This simplifies to give:

$$\frac{\mathbf{V_a N_a}}{\mathbf{V_b}} = \mathbf{N_b}$$

Experiment (2)

Oxidation Reduction Titration

- **Introduction:**

Oxidation-reduction reactions (also known as redox reactions) are reactions that usually involve transfer of electrons. To determine the number of electrons transferred, oxidation states are assigned. Oxidation states of atoms are numbers that help chemists keep track of electrons during a reaction. Each atom in an equation can be assigned an oxidation state according to certain rules. If the oxidation state of an atom increases as you go from the reactants to the products in an equation, oxidation has occurred (electrons have been lost); if the oxidation state decreases, reduction has occurred (electrons have been gained).

تفاعلات الأكسدة والاختزال (المعروفة أيضًا باسم تفاعلات الأكسدة والاختزال) هي تفاعلات تتضمن عادةً إنتقال الإلكترونات. لتحديد عدد الإلكترونات ، يتم تعيين حالات الأكسدة. حالات أكسدة الذرات هي أرقام تساعد الكيميائيين على تتبع الإلكترونات أثناء التفاعل. يمكن تعيين حالة أكسدة لكل ذرة في المعادلة وفقًا لقواعد معينة. إذا زادت حالة أكسدة الذرة مع انتقالها من المواد المتفاعلة إلى النواتج في المعادلة ، فقد حدثت الأكسدة (فقدت الإلكترونات) ؛ إذا انخفضت حالة الأكسدة ، حدث اختزال (تم اكتساب الإلكترونات).

In this experiment you will use a standard solution of potassium permanganate ($KMnO_4$) to determine the iron (as Fe^{2+}) in an unknown solution. Permanganate ion reduces to a manganese (II) ion in the acidic solution. This reaction requires 5 electrons and 8 hydrogen ions:

$$MnO_4^- + 8H^+ + 5\ e^- \rightarrow Mn^{2+} + 4H_2O$$

Only one electron is necessary to reduce Fe (III) to Fe (II)

$$Fe^{3+} + e^- \rightarrow Fe^{2+}$$

Therefore, 1 mole of MnO_4^-(the oxidizing agent) reacts with 5 moles of Fe^{2+} (the reducing agent) to form 5 moles of Fe^{3+} and 1 mole of Mn^{2+}. Thus, in net ionic form:

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

The 1:5 mole ratio with respect to the amounts of $MnO_4^-$and Fe^{2+} consumed will provide the stoichiometric basis for all the calculations in this experiment.

The permanganate ion acts as its own indicator, as MnO_4^- is highly colored while Mn^{2+} is essentially colorless. The product of oxidation, the Fe^{3+} ion, is itself, slightly colored. To avoid any possible interference with the equivalence point determination a little phosphoric acid, H_3PO_4, is added to complex Fe^{3+} to a completely, colorless ion.

يعمل أيون البرمنجنات كدليل ذاتى ، حيث أن MnO_4^- ملون للغاية بينما Mn^{2+} عديم اللون.

- **Procedure:**

1. Rinse the burette with about 3 mL of your standard $KMnO_4$ solution.

(يتم تنضيف السحاحة بحوالي 3 مل من محلول $KMnO_4$ القياسي)

2. Fill the burette using a plastic funnel. Clear all bubbles from the tip. Remove any drops from the tip.

(املأ السحاحة باستخدام قمع بلاستيك. امسح كل الفقاعات من الحافة. قم بإزالة أي قطرات من الحافة).

3. Record the initial volume, before starting each titration. The top of the meniscus will be read at the beginning and end of the titration.

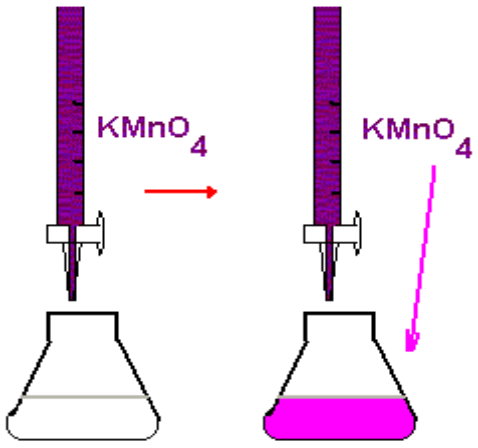
(سجل الحجم الأولي ، قبل بدء كل معايرة. سيتم قراءة الجزء العلوي من الغضروف المفصلي في بداية ونهاية المعايرة)

4. Transfer 10.00 mL of the unknown Ferrous ammonium sulfate into the Erlenmeyer flask.

(يتم وضع 10.00 مل من كبريتات الحديدوز النشادرية مجهوله التركيز إلى الدورق المخروطى)

5. Titrate with $KMnO_4$ until the appearance of a very faint pink color that persists for 30 seconds. *Note that as you approach this endpoint, the pink color will begin to persist for longer periods of time, before disappearing. At this point, it is advisable to add titrant slowly. You can even add partial drops from the burette tip - simply touch the flask to the droplet on the tip of the burette and then rinse the wall of the flask with a squirt of water from your wash bottle.*

(عاير باستخدام $KMnO_4$ حتى ظهور لون وردي باهت جدًا يستمر لمدة 30 ثانية. لاحظ أنه كلما اقتربت من نقطة النهاية هذه ، سيبدأ اللون الوردي في البقاء لفترات أطول من الوقت قبل أن يختفي. في هذه المرحلة ، من المستحسن إضافة المعايير ببطء).



6. Obtain the final volume reading from the calibration scale on the burette.

(احصل على قراءة الحجم النهائية من مقياس المعايرة على السحاحة)

9. Repeat this procedure two more times, refilling the $KMnO_4$ in the burette as needed.

- **Results:**

Initial volume of $KMnO_4$ (mL)	Final volume of $KMnO_4$ (mL)	Total volume of $KMnO_4$ used (mL)	Mean volume of $KMnO_4$ used (mL)

Total volume $KMnO_4$ used = Final volume $KMnO_4$ – Initial volume $KMnO_4$

- **Calculations:**

Use the following relationship to calculate the Ferrous ammonium sulfate concentration:

$$\mathbf{V_a x\ N_a = V_b\ x N_b}$$

where, $\mathbf{V_a}$: The volume of $KMnO_4$ solution, mL

$\mathbf{N_a}$**:** The Normality of the $KMnO_4$ solution

$\mathbf{V_b}$**:** The volume of FAS solution, mL

$\mathbf{N_b}$**:** The Normality of the FAS solution

This simplifies to give:

$$\frac{\mathbf{V_a N_a}}{\mathbf{V_b}} = \mathbf{N_b}$$

Experiment (3)

Chloride Titration

- **Introduction:**

This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

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

The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium chromate, to form a red-brown precipitate of silver chromate.

$$2\ Ag^{+}_{(aq)} + CrO_4^{2-}{}_{(aq)} \rightarrow Ag_2CrO_{4(s)}$$

تحدد هذه الطريقة تركيز أيون الكلوريد للمحلول بالمعايرة مع نترات الفضة. عند إضافة محلول نترات الفضة ببطء ، يتشكل راسب من كلوريد الفضة.

نهاية المعايرة تحدث عندما تترسب كل أيونات الكلوريد. ثم تتفاعل أيونات الفضة الإضافية مع الدليل المكون من أيونات الكرومات ، كرومات البوتاسيوم ، لتشكيل راسب بني أحمر من كرومات الفضة.

This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water. Seawater is used as the example here.

- **Safety:**

- Lab coats, safety glasses and enclosed footwear must always be worn in the laboratory. The chromate solution needs to be prepared and used with care as

chromate is a known carcinogen. Silver nitrate solution causes staining of skin and fabric (chemical burns). Any spills should be rinsed with water immediately.

- **Equipment Needed:**

- burette and stand.
- 250 mL conical flasks.
- 10 mL measuring cylinders.

- **Procedure:**

1. Rinse the burette with about 3 mL of your standard $AgNO_3$ solution.

(يتم تنضيف السحاحة بحوالي 3 مل من محلول نترات الفضة)

2. Record the initial volume, before starting each titration. The top of the meniscus will be read at the beginning and end of the titration.

(سجل الحجم الأولي ، قبل بدء كل معايرة. سيتم قراءة الجزء العلوي من الغضروف المفصلي في بداية ونهاية المعايرة)

3. Pipette a 10 mL aliquot of NaCl into a conical flask and add about 1 mL of chromate indicator.

(يتم وضع 10.00 مل من كلوريد الصوديوم إلى الدورق المخروطى وإضافة حوالى 1 مل من دليل الكرومات)

4. Titrate the sample with 0.1 mol L^{-1} silver nitrate solution. Although the silver chloride that forms is a white precipitate, the chromate indicator initially gives the cloudy solution a faint lemon-yellow color **(Figure 1)**.

(عاير العينة بمحلول من نترات الفضة. على الرغم من أن كلوريد الفضة الذي يتشكل هو راسب أبيض ، فإن دليل الكرومات يعطي في البداية لونًا أصفر ليموني باهتًا).

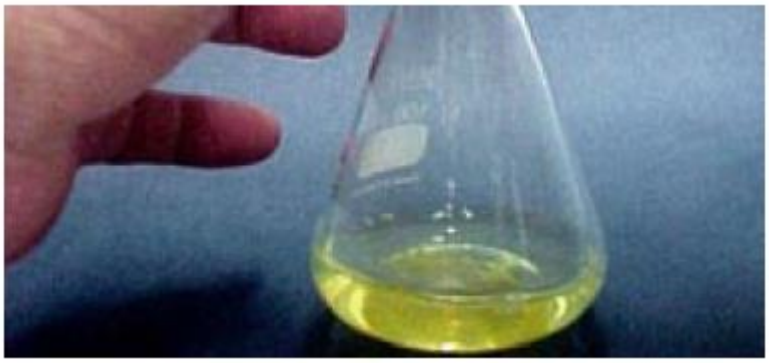


Figure 1 Before the addition of any silver nitrate the chromate indicator gives the clear solution a lemon-yellow color.

The endpoint of the titration is identified as the first appearance of a red-brown color of silver chromate **(Figure 2).**

(تم تحديد نقطة نهاية المعايرة على أنها أول ظهور للون الأحمر والبني لكرومات الفضة)

Figure 2: **Left flask:** before the titration endpoint, addition of Ag^+ ions leads to formation of silver chloride precipitate, making the solution cloudy. The chromate indicator gives a faint lemon-yellow color. **Centre flask:** at the endpoint, all the Cl^- ions have precipitated. The slightest excess of Ag+ precipitates with the chromate indicator giving a slight red brown coloration. **Right flask**: If addition of Ag^+ is continued past the endpoint, further silver chromate precipitate is formed and a stronger red brown color result. NB: The titration should be stopped

when the first trace of red brown color is observed. Using an incompletely titrated reference flask for comparison is a helpful way to identify the first appearance of red brown coloration.

5. Obtain the final volume reading from the calibration scale on the burette.

(احصل على قراءة الحجم النهائية من مقياس المعايرة على السحاحة)

9. Repeat this procedure two more times, refilling $AgNO_3$ in the burette as needed.

- **Results:**

Initial volume of $AgNO_3$ (mL)	Final volume of $AgNO_3$ (mL)	Total volume of $AgNO_3$ used (mL)	Mean volume of $AgNO_3$ used (mL)

Total volume $AgNO_3$ used = Final volume $AgNO_3$ – Initial volume $AgNO_3$

- **Calculations:**

Use the following relationship to calculate the Ferrous ammonium sulfate concentration:

$$\mathbf{V_a x\ N_a = V_b\ x\ N_b}$$

where, $\mathbf{V_a}$: The volume of $AgNO_3$ solution, mL

$\mathbf{N_a}$**:** The Normality of the $AgNO_3$ solution

$\mathbf{V_b}$**:** The volume of NaCl solution, mL

$\mathbf{N_b}$**:** The Normality of the NaCl solution

This simplifies to give:

$$\frac{\mathbf{V_a N_a}}{\mathbf{V_b}} = \mathbf{N_b}$$

Experiment (4)

Determine the Equivalent weight of Iron by Chemical Displacement method.

- **Object:**

To determine the Equivalent weight of Iron by Chemical Displacement method. The Equivalent weight of Copper is 63.5.

- **Theory:**

Iron displaces copper from a solution containing copper ions.

$$Fe + CuSO_4 \rightarrow FeSO_4 + Cu$$

(يزيح الحديد النحاس من محلول يحتوي على أيونات النحاس)

The quantity of copper sulphate remaining in solution, after the chemical displacement is estimated by Iodometric titration method.

$$2CuSO_4 + 4KI \rightarrow 2CuI_2 + 2K_2SO_4$$

$$2CuI_2 \rightarrow 2\ CuI + I_2$$

$$KI + I_2 \rightarrow KI_3$$

(يتم تقدير كمية كبريتات النحاس المتبقية في المحلول ، بعد الإزاحة الكيميائية بطريقة المعايرة)

The iodine so liberated remains dissolved in excess of KI and is proportional to the amount of copper sulphate which is then titrated against sodium thiosulphate using starch as indicator. At the end point the blue color disappears and a white ppt. of cuprous iodide is obtained.

$$2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI$$

Sodium Tetra Thionate

$$Starch + I_2 \rightarrow Starch.\ Iodide\ adsorption\ complex$$

Deep blue color

(يبقى اليود المحرر مذابًا فى الزيادة KI ويتناسب مع كمية كبريتات النحاس التي يتم معايرتها بعد ذلك مقابل ثيوسلفات الصوديوم باستخدام النشا كدليل. عند نقطة النهاية ، يختفي اللون الأزرق ويظهر راسب أبيض من اليوديد النحاسي).

Under chemical displacement conditions:

$$\frac{\text{Equivalent weight of copper}}{\text{Equivalent weight of Iron(x)}} = \frac{\text{Wt.of Cu.Deposited on Iron strip (C)}}{\text{Wt.of Iron goes into solution (D)}}$$

- **Chemicals:** N/10 $CuSO_4.5H_2O$, N/10 sodium thiosulphate ($Na_2S_2O_3.5H_2O$), solid KI, dilute H_2SO_4.
- **Glassware:** Burette, Pipette, Beaker, conical flask, funnel, measuring cylinder.
- **Indicator:** Freshly prepared starch solution.
- **End point:** Disappearance of blue color.
- **Procedure:**

1. Take an iron strip (4 cm x 1 cm) and clean it with sandpaper. Weigh the cleaned iron strip accurately and place it in a clean 250 ml beaker. Pour 100 ml of $CuSO_4$ solution (N/10) into it and allow the strip to stand in beaker for about 30 minutes.

(خذ شريطًا حديديًا (4 سم × 1 سم) ونظفه بورق الصنفرة. قم بوزن شريط الحديد النظيف بدقة وضعه في دورق نظيف 250 مل. نضع 100 مل من محلول $CuSO_4$ فيه واترك الشريط يقف في الدورق لمدة 30 دقيقة تقريبًا).

2. With the help of forcep, carefully withdraw iron strip from the beaker and place it on a porcelain plate contained in a desiccator (using $CaCl_2$ as desiccators).

3. The quantity of copper sulphate remaining in solution after the chemical displacement is estimated by Iodometric titration method.

(يتم تقدير كمية كبريتات النحاس المتبقية في المحلول بعد الإزاحة الكيميائية بطريقة المعايرة)

4. The dried iron strip (containing the deposited copper) is then carefully weighed.

(ثم يتم وزن شريط الحديد المجفف (الذي يحتوي على النحاس المترسب) بعناية)

5. Pipette out 25 ml of $CuSO_4$ solution (solution after chemical displacement). Now add 1 gm of KI in a conical flask, mix well and cover the mouth of conical flask with watch glass and allow the mixture to stand for 2 to 5 minutes. in the dark.

(يتم وضع 25 مل من محلول $CuSO_4$ ثم أضافة 1 جم من KI في دورق مخروطي ، واخلط جيدًا وقم بتغطية الدورق المخروطي واترك الخليط لمدة 2 إلى 5 دقائق. في الظلام)

6. Now titrate the liberated iodine with N/10 sodium thiosulphate solution. The brown color of iodine becomes fainter at the addition of sodium thiosulphate solution. When very light-yellow color remains add 5 drops of starch solution. It forms deep blue Iodine-starch complex Now add further Hypo solution drop by drop till blue color disappears. This is the end point.

(يتم معايرة اليود المحرر بمحلول ثيوسلفات الصوديوم . يصبح اللون البني لليود أكثر خفوتًا عند إضافة محلول ثيوسلفات الصوديوم. عندما يبقى اللون أصفر فاتح للغاية ، أضف 5 قطرات من محلول النشا. سوف يتكون مركب الأزرق العميق الآن أضف المزيد من محلول Hypo قطرة قطرة حتى يختفي اللون الأزرق. هذه هي نقطة النهاية.

- **Observation Table:**

S.No.		Burette reading	Concordant Volume of N/10

	Volume of $CuSO_4$ solution taken (ml)	Initial	Final	sodium thiosulphate solution used (ml)

- **Observations:**

1. Initial weight of Iron strip, A = _____gm
2. Weight of iron strip + copper after drying, B =_____gm.
3. Weight of copper deposited on iron strip, C = gm.
4. Weight of iron which goes into solution ($FeSO_4$), D = A + C – B _____gm.
5. Equivalent weight of copper = 63.5.

- **Calculations:**

1) Initial conc. of $CuSO_4$ solution = Normality x Equ.wt.

= 1 / 10 x 63.5 = 6.35 gm/lit.

2) $N_1V_1 = N_2 V_2 \Rightarrow \quad N_1 = \frac{N_2V_2}{V_1}$

3) Final conc. of $CuSO_4$ = Normality x Equ.wt.

4) $\frac{\text{Equivalent weight of copper}}{\text{Equivalent weight of Iron(x)}} = \frac{\text{Wt.of Cu.Deposited on Iron strip (C)}}{\text{Wt.of Iron goes into solution (D)}}$

5) Percentage error =

- **Result:**

1. The Equivalent of Iron =
2. Percentage error =

Experiment (5)

Estimation of CaO in Cement Solution by rapid EDTA method

- **Aim:**

To determine the percentage of calcium oxide in the given sample of cement solution using Standard EDTA solution.

- **Principle:**

Cement contains compounds of calcium, aluminum, magnesium, iron, and insoluble silica. When dissolved in acid, silica remains undissolved. On treating with ammonia, aluminum and iron can be precipitated as their hydroxides and separated. The provided cement solution contains calcium and magnesium ions. The constituents of Portland cement are: - CaO (60-67%), SiO_2 (17-25%), Al_2O_3 (3-8%), Fe_2O_3 (0.5-6%), MgO(0.1 -4%), SO_3(1-3%), K_2O & Na_2O (0.5-1.5%) and $CaSO_4$(3-5%).

يحتوي الأسمنت على مركبات الكالسيوم والألومنيوم والمغنيسيوم والحديد والسيليكا غير القابلة للذوبان. عندما تذوب في الحمض ، تبقى السيليكا غير ذائبة . عند المعالجة بالأمونيا ، يمكن ترسيب الألمنيوم والحديد على شكل هيدروكسيدات وفصلهما. يحتوي محلول الأسمنت على أيونات الكالسيوم والمغنيسيوم.

To estimate the calcium content in the given solution, a known volume of cement solution in presence of Magnesium, Calcium ions is titrated with standard EDTA solution using Patton & Reader's indicator in the pH range 12-14. The indicator combines with Calcium ions to form a wine-red colored Calcium-Indicator

complex (Wine red). Near the end point, when free calcium ions are exhausted in the solution, further addition of EDTA, dissociates Calcium Indicator complex, consumes the calcium ions, and release free indicator which is blue in color. Therefore, color change is wine red to blue.

لتقدير محتوى الكالسيوم في المحلول ، وهو حجم معروف من محلول الأسمنت في وجود المغنيسيوم ، تتم معايرة أيونات الكالسيوم بمحلول EDTA القياسي باستخدام دليل Patton & Reader في نطاق الأس الهيدروجيني 12-14. يتحد الدليل مع أيونات الكالسيوم لتكوين دليل بلون النبيذ الأحمر. بالقرب من نقطة النهاية ، عندما يتم استنفاذ أيونات الكالسيوم الحرة في المحلول ، فإن الإضافة الزائدة لـ EDTA ، تفكك دليل الكالسيوم ، وتستهلك أيونات الكالسيوم ، وتحرر دليل باللون الأزرق. لذلك ، تغيير اللون من النبيذ الأحمر إلى الأزرق.

- **Procedure:**

Part A: Preparation of standard solution of Disodium salt of EDTA

Weigh out the given EDTA crystals accurately into a 250 ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

قم بوزن بلورات EDTA المحددة بدقة في دورق سعة 250 مل. أضف ربع أنبوب اختبار من الأمونيا. يذوب في الماء المقطر ويخفف حتى العلامة ويخلط جيدا.

Part B: Estimation of CaO in the given cement solution

Pipette out 25 cm^3 of the given Cement solution into a clean conical flask. Add 5 ml of glycerol, 5 ml of diethyl amine and 10 ml of 4N NaOH solution. Add 3-4 drops of Patton and Reeder's indicator. Titrate against standard EDTA solution till the

color change from wine red to clear blue. Repeat the titration to get concordant values.

RESULT: CaO in the given sample of cement solution = ----------

يتم إضافة 25 سم^3 من محلول الأسمنت في دورق مخروطي نظيف. أضف 5 مل من الجلسرين ، 5 مل من ثنائي إيثيل أمين و 10 مل من محلول NaOH. أضف 3-4 قطرات من دليل Patton and Reeder's. يتم المعايرة مقابل محلول EDTA القياسي حتى يتغير اللون من الأحمر إلى الأزرق الصافي. كرر المعايرة للحصول على قيم متوافقة.

- **Observation:**

Part A: Preparation of standard solution of Disodium salt of EDTA

Weight of weighing bottle + EDTA salt (W_1) = ---------- g

Weight of empty weighing bottle (W_2) = ---------- g

Weight of EDTA transferred ($W_1 - W_2$) = ---------- g

$$\text{Molarity of EDTA} = \frac{\text{Weight of EDTA taken } (W_1 - W_2) \text{X } 4}{\text{Molecular weight of EDTA } (372)} = \text{----------}$$

Part B: Estimation of CaO in the given cement solution

Burette reading	**I**	**II**	**III**
Final burette reading			
Initial burette reading			
Volume of EDTA run down (ml)			

- **Observation:**

♦ Volume of EDTA consumed by 25 cm^3 of cement solution = ----------

- Weight of Cement in 25ml =0.1g
- 1000 cm^3 of 1 M EDTA = 56.08 g of CaO (Molecular mass of CaO =56.08)
- ------- cm^3 of -------- M EDTA = $\frac{X \quad X\ 56.08}{1000 \times 1}$ g of CaO = ----------
- 25 cm^3 of the Cement solution contains ---------- g of CaO
- % of CaO in the given sample of cement solution = $\frac{X\ 100}{0.1}$ =----------
- % of CaO in the given sample of cement solution = ----------

Experiment (6)

Estimation of hardness of water by EDTA method

- **Aim:**

To estimate the total, permanent and temporary hardness of water by complexometric (EDTA) method.

- **APPARATUS:**

1. Conical flask
2. Burette
3. Pipette
4. Burette stand
5. Spatula

- **CHEMICALS:**

1. Buffers solution
2. Eriochrome black-T indicator (EBT)
3. EDTA Solution

- **PRINCIPLE:**

EDTA forms colorless, stable complexes with Ca^{2+} & Mg^{2+} ions present in water at pH 9-10. To maintain pH at 9-10, NH_4Cl & NH_4OH buffer is used. Eriochrome Black-T (EBT) is used as Indicator. Hard water sample with EBT indicator forms unstable, wine red colored complexes with Ca2+ & Mg2+ present in water.

$$\begin{matrix} Ca^{2+} \\ (or) \\ Mg^{2+} \end{matrix} + EBT \xrightarrow{pH = 9-10} \begin{matrix} Ca-EBT \\ (or) \\ Mg-EBT \end{matrix}$$

Wine red colored complex

تشكل EDTA مجمعات ثابتة عديمة اللون مع أيونات Ca^{2+} & Mg^{2+} الموجودة في الماء عند درجة الاس الهيدروجينى 9-10. للحفاظ على الرقم الهيدروجيني عند 9-10 ، يتم استخدام NH_4Cl & NH_4OH buffer. يستخدم Eriochrome Black-T (EBT) كدليل . تشكل عينة الماء مع دليل EBT متراكبات غير مستقرة بلون أحمر مع +Mg2 & +Ca2 الموجودة في الماء.

The metal indicator complex is less stable than metal EDTA complex. So, this metal indicator complex is titrated with std. EDTA solution. Now colorless metal-EDTA complex is formed by releasing EBT indicator which is blue color. So, the color change from wine red to blue indicates the end point.

$$\begin{matrix} Ca-EBT \\ (or) \\ Mg-EBT \end{matrix} + EDTA \xrightarrow{pH = 9-10} \begin{matrix} Ca-EDTA \\ (or) \\ Mg-EDTA \end{matrix} + EBT$$

(Colorless stable Indicator)(blue Complex in color)

One mole of EDTA reacts with one mole of complex, hence equimolar reaction. (n=1).

- **Procedure:**

Preparation of standard hard water:

Dissolve 1 gm of $CaCO_3$ (dry) in minimum (2-3 drops) quantity of dilute HCl and evaporate the solution on water bath. Dissolve this Solution in small amount of distilled water and then transfer into 1000 ml standard flask. Make up the solution up to the mark and shake well for uniform concentration.

قم بإذابة 1 جم من كربونات الكالسيوم (جاف) بكمية لا تقل عن (2-3 قطره) من حمض الهيدروكلوريك المخفف ويتبخر المحلول في حمام مائي. قم بإذابة هذا المحلول في كمية صغيرة من الماء المقطر ثم انقله إلى دورق مخروطى 1000 مل . يستكمل المحلول حتى العلامة ويرج جيدًا للحصول على تركيز موحد.

Standardization of EDTA Solution:

Pipette out 20 ml of std. hard water solution into a conical flask, add 2 ml of buffer solution and 2-3 drops of EBT indicator and titrate the wine red color complex solution with EDTA solution taken in the burette, till the color changes to blue color. Note the reading. Repeat the titration to get concurrent values.

قم بإخراج 20 مل من محلول الماء القياسي في دورق مخروطي ، أضف 2 مل من buffer solution و2-3 قطرات من دليل EBT وعاير محلول اللون الأحمر مع محلول EDTA المأخوذ في السحاحة ، حتى يتغير اللون إلى اللون الأزرق اللون. لاحظ القراءة. كرر المعايرة للحصول على قيم متزامنة.

S.No.	Vol. of standard hard water	Burette reading		Vol. of EDTA required
		Initial	Final	

$$\frac{M_1V_1}{n_1} = \frac{M_2V_2}{n_2}$$ (Note that $n_1 = n_2 = 1$)

M_1V_1 (Hard water) = M_2V_2 (EDTA solution)

- **Estimation of Total Hardness:**

Pipette out 50 ml of hard water sample (tap water) into 250 ml conical flask, add 2 ml of buffer solution and 3 drops of EBT-Indicator. Titrate the wine-red colored solution with EDTA, till the blue color is obtained. Note the reading. Repeat the titration to get concurrent values.

يتم سحب 50 مل من عينة الماء (ماء الصنبور) بالماصة في دورق مخروطي 250 مل ، أضف 2 مل من buffer solution و 3 قطرات من دليل EBT. عاير المحلول الملون باللون الأحمر باستخدام EDTA ، حتى يتم الحصول على اللون الأزرق. لاحظ القراءة. كرر المعايرة للحصول على قيم متزامنة.

S.No.	Vol. of standard hard water	Burette reading		Vol. of EDTA required
		Initial	Final	

$$\frac{M_3V_3}{n_3} = \frac{M_2V_2}{n_2}$$ (Note that $n_3 = n_2 = 1$)

M_3V_3 (Tap water) = M_2V_2 (EDTA solution)

Total Hardness = M_3 x 100 x 1000 ppm

- **Estimation of Permanent Hardness:**

Pipette out 100 ml of tap water and boil the solution till the volume is reduced to half i.e. 50 ml (All bicarbonates of Ca^{2+} & Mg^{2+} decomposes to $CaCO_3$ and $Mg(OH)_2$ respectively). Cool the solution and filter the water into conical flask. Now

add 2 ml of buffer solution and 3 drops of EBT indicator, till the blue color is obtained. Note the readings. Repeat the titration to set concurrent values.

اسحب 100 مل من ماء الصنبور بواسطة الماصة وقم بغلي المحلول حتى ينخفض الحجم إلى النصف أي 50 مل (تتحلل جميع بيكربونات Ca^{2+} & Mg^{2+} إلى $CaCO_3$ and $Mg(OH)_2$ على التوالي). قم بتبريد المحلول وتصفيته في دورق مخروطي . أضف الآن 2 مل من buffer solution و 3 قطرات من دليل EBT ، حتى يتم الحصول على اللون الأزرق. لاحظ القراءات. كرر المعايرة لضبط القيم المتزامنة.

S.No.	Vol. of standard hard water	Burette reading		Vol. of EDTA required
		Initial	Final	

$$\frac{M_4V_4}{n_4} = \frac{M_2V_2}{n_2}$$ (Note that $n_4 = n_2 = 1$)

M_4V_4 (Heated tap water) = M_2V_2 (EDTA solution)

Permanent hardness = M_4 x 100 x 1000 ppm

Temporary hardness = Total hardness – Permanent hardness

- **RESULT:**

Temporary hardness of water = _____ ppm

Permanent hardness of water = _____ ppm

Total hardness of water = _____ ppm