**University of Baghdad**

**College of science**

**General Chemistry laboratory**

**Experiments in Analytical chemistry**

**For**

**Biotechnology Department**

**First semester**

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**إرشادات مختبرية عامة**

1. لأخذ مادة صلبة من قنينة استعمل ملعقة زجاجية او بلاستيكية لا تسكب المادة الصلبة فوق ورقة الترشيح او انبوبة اختبار او دورق او كاس زجاجي بينما السوائل فقط هي التي تسكب في الاوعية الزجاجية المناسبة لها.
2. لا تغمر قطارة او ماصة في قنينة سائل او محلول لتستخدمه في سائل او محلول اخر.
3. لا تضع ابدا سداد قنينة او محلول فوق الرف او المنضدة تحاشيا من التلوث.
4. ضع جميع قناني المواد الكيميائية في الاماكن المخصصة لها بعد اخذك ما تحتاج منها دون حملها الى مكان عملك فالطلبة الأخرين هم بحاجة لها أيضا.
5. لا تعيد الكمية الزائدة من المواد الكيميائية) الصلبة كانت ام سائلة الى قنينتها الاصلية (اعطها لزميل بحاجتها وأخبر أحد مسؤولي المختبر لمعرفة ما يجب عمله للاستفادة منها
6. لا تترك ابدا قنينة مواد كيميائية مفتوحة فهناك كثير من المواد الكيمياوية التي يتغير تركيبها عند تعرضها للجو او ضوء الشمس او الضوء الاصطناعي اضافة الى احتمال تلوثها وتأكد من ان القنينة قد سدت بإحكام عند اخذ جزء من المادة التي تحتويها.
7. ضع المواد الصلبة الزائدة التي لا يمكن الاستفادة منها واوراق الترشيح وبقايا عيدان الثقاب وغيرها قي وعاء المهملات ولا تضعها في الحوض او في مجرى المياه وتأكد قبل رمي المادة الصلبة انها لا تسبب اشعال النار فيما لو تعرضت للهواء او انها تعطي روائح غير مرغوب فيها عند رميها في الحوض او مجاري المياه.
8. لا تسكب الماء ابدا في حامض الكبريتيك المركز لغرض تخفيف الحامض الطريقة السليمة لتخفيف حامض الكبريتيك المركز هي بإضافة الحامض المركز ببطيء و عناية الى الماء في وعاء زجاجي) انبوب اختبار مناسب او اسطوانة مدرجة او كاس زجاجي او قنينة زجاجية الخ( مع التحريك المستمر فاذا اقتربت درجة حرارة المحلول من مرحلة الغليان عند ذلك يجب تبريده بسكب ماء الحنفية على الجدران الخارجية للوعاء و الاستمرار بالتحريك لحين انخفاض درجة الحرارة ثم يباشر بإضافة الحامض كالسابق.
9. احذر عند تسخين مادة صلبة او سائلة في انبوبة اختبار ان تكون فوهة الانبوبة موجهة نحوك او نحو زميل مجاور لك او باتجاه ادوات المختبر وكذلك نفس الحال عند غلي محلول او سائل
10. لا تحاول ابدا تذوق اي مادة كيميائية سائلة او صلبة تتعامل معها في المختبر ولا تشرب الماء في اي وعاء زجاجي تستخدمه في عملك داخل المختبر اذكر دائما ان من حولك العديد من المواد السامه.
11. كن حذرا عندما تريد شم سائل او غاز يتصاعد اثناء تفاعل ما ولا تشم بخار السائل او الغاز المتصاعد من قنينة او انبوبة اختبار او دورق التفاعل بتقريب الفوهة من الانف
12. اجراء عملية التسخين داخل الهود) خزانات الغاز (لان عملية التسخين يتضمن تحرير ابخرة او غازات ذات رائحة كريهة او سامة

ينبغي على الطالب عدم لبس الحلي الذهبية والفضية وغيرهما اثناء العمل خشية لتعرضها للتلف

**Experiment (1)**

**Preparation of approximately (0.1N) HCI** **and (0.IN) sodium carbonate**

**Calculations of volumetric analysis:**

Standard solution is a solution, which contains a known weight of the substances in a definite volume of the solvent.

Molar solution is a solution, which contains 1 gm molecular weight of the substances per liter of solvent.

Normal solution is a solution that contains l gm equivalent weight per liter of solvent.

Part per million(ppm): Milligrams of solute per liter of solution.

**Equivalent weights**

1. **Equivalent weight in neutralization reactions.**

The equivalent weight of acid is that weight of it which contains one-gram atom of replaceable hydrogen.

**Ex:**

equivalent weight of H2SO4 =M.Wt H2SO4/2

equivalent weight of H3PO4 =M.Wt H3PO4/3

The equivalent weight of Base is that weight of it which contains one replaceable hydroxyl group.

Ex: equivalent weight of NaOH = M.Wt NaOH/1

1. Equivalent weight in Oxidation -reduction reactions.

The equivalent weight of an oxidant or a reductant is the number of electrons which Imol of the substance gains or loses in the reaction.

Ex:

1. Equivalent weight of complex formation and precipitation reactions.

Here the equivalent weight is the weight of the substance which contains or reacts with lg. atm of a univalent cation M+.

Ex: When silver nitrate reacts with sodium chloride, to form silver chloride, the equivalent weight of AgNO3 is:

A: **Preparation of approximately (0.1N) HCI**

1. Calculate the normality of the concentrated HCl :
2. To prepare (500mL) 0f 0.1N HCL:

Transfer distilled water to a glass-Stoppard bottle and add about 4.14 mL of conc.HCI. Mix the contents of the bottle thoroughly. Dilute to the mark with

distilled water and make a label.

B: **Preparation of (0.IN) sodium carbonate:**

1. Dry a quantity of primary-standard sodium carbonate powder for 2 hours at 110 °C, cool in a desiccator Weigh 1.325gm of the salt into a small beaker, and dissolve in 25 to 50 mL of distilled water.
2. Transfer this solution to a 250mL volumetric flask. Wash the beaker and transfer this washing water to the volumetric flask. Dilute to the mark with distilled water and mix thoroughly. Make a label.

**Experiment (2)**

**Standardization of HCl solution with standard solution of Na2CO3**

**Volumetric (Titrimetric) analysis**

Volumetric analysis (or Quantitative chemical analysis) is determination the volume of a solution of accurately known concentration, which is required to react quantitatively with the solution of the substance being determined.

\* The solution of accurately known strength is called the **"standard solution".** \*The standard solution is usually added from a graduated vessel called a **"burette"**

\* The process of adding the standard solution until the reaction is just complete is termed a **"titration".**

\*The reagent of known concentration is called the **"titrant"** and the substance being titrated is termed the **"titrate".**

\***Equivalent point** is the point in a titration where the amount of standard titrant added is chemically equivalent to the amount of analyte in sample.

\***End point** is the point in a titration when an observable change that signals that the amount titrant added is chemically equivalent to the amount of analyte in sample.

\***Indicator** is a reagent used to indicate when the end point has been reached.

\* A primary standard is a highly purified compound that serve as a reference material in all volumetric titrimetric methods.

Important requirements for a primary standard are :

1. High purify.
2. Stability toward air.
3. Absence of hydrate water.
4. Ready availability at modest cost.
5. Reasonable solubility in the titration medium.
6. Reasonable large molar mass so that the relative error associated with weighing the standard is minimized.

**Volumetric(titrimetric)analysis reaction must fulfill the following conditions:**

1. There must be a simple reaction which can be expressed by a chemical equation and the substance to be determined should react completely with the reagent.
2. The reaction should be proceeding with very great speed.
3. An indicator should be available.
4. Equillibrium constant must be very large.

**Calculations for titrimetric reaction**:

titrant titrant

at equivalent point:

no.mmol of titrant(A) = no.mmol of titrant(B)

**The following are required for volumetric analysis:**

1. Calibrated measuring vessels, including burettes, pipettes, and measuring flasks.
2. Substances of known purity for the preparation of standard solution.
3. A visual indicator or an instrumental method detecting the completion of the reaction.

**Classification of reactions in volumetric (titrimetric) analysis:**

1. Neutralisation reaction or acidimetric and alkalimeteric .(reaction between base and acid).
2. Complex formation reaction (Ions or compound combination to form stable-soluble slightly dissociated ion or compound).
3. Precipitation reactions.(form a simple precipitate).
4. Oxidation-reduction reactions (include all reactions involving change in oxidation number or transfer of electrons among the reaction substances).

**Procedure**

1. Clean the burette and rinse with HCl solution.
2. Fill the burette with HCI.
3. Pipet 10 mL of standard solution (Na2CO3) into a 250 mL conical flask. Add 3 drops of Methyl orange indicator.
4. Titrate by adding HCl drop wise until the solution just beings to change from yellow to red.
5. Reapeat the titration a few times until you gets approximate results. Take the average of the results and subtract 0.05mL. (This result represents the volume of extra drop which change the color of indicator.
6. Calculate the normality of HCI :

Nacid \* Vacid = N base \* V base

1. Make label.

**Experiment (3)**

**Analysis of a mixture (sodium hydroxide + sodium carbonate)**

1. Transfer a (10mL) of a mixture solution to 250 mL conical flask and add 1 drop of phenolphthalein indicator.
2. Clean the burette and rinse with hydrochloric acid.
3. Fill the burette with HCI.
4. Titrate with standard hydrochloric acid solution until the pink color disappears (the solution will be colorless).

\* At this stage: all the hydroxide and half the carbonate have been neutralized. Let us assume that the volume of acid be “X” mL.

1. Now add 3drops of Methyl Orange indicator into the solution above and continue the titration until the solution just begins to change from yellow to red.

\* At this stage :another half of carbonate has been neutralized. Let the volume of acid be "Y" mL.

1. Repeat the titration a few times until you get approximate results.

**Calculations:**

(X-Y)=ZVolume of HCI which equivalent to OH-……..(1)

2\*YVolume of HCl which equivalent to ………….(2)

Nacid \* Vacid = N base \* V base

Nacid \* Z = N OH \* 10 …….(1)

Nacid \* 2Y = N CO3 \* 10…..(2)

Concentration of [OH-](ppm) = N OH-\* eq.wt\*1000

Concentration of [CO3](ppm) = N CO3 \*eq.wt \*1000

**Experiment (4)**

**Analysis of a mixture (sodium bicarbonate + sodium carbonate)**

1. Repeat the same steps of the experiment no.3.
2. Volume of acid at the end point of Ph.Ph indicator=X.
3. Volume of acid at the end point of M.O indicator=Y.

(Y-X)=Z → Volume of HCl which equivalent to

2\*X Volume of HCl which equivalent to

Nacid \* Vacid = N base \* V base

Concentration of [](ppm) = \* eq.wt\*1000

Concentration of [](ppm) = \*eq.wt \*1000

**Experiment (5)**

**Determination of chloride ion by Mohr method**

Precipitation titration: is titration depend upon the combination of ions to form a simple precipitate. Mohr method is a method depend upon formation a colored precipitate for the determination of chloride ion.

Chloride ion is reacted with silver nitrate solution to form AgCl precipitate.

AgNO3+NaCl AgCl + NaNO3 Ksp AgCl =1.2x10-10

A small quantity of potassium chromate (K2CrO4) solution is added to serve as indicator. The first excess of titrant results in the formation of a red silver chromate precipitate which signal the end point.

2AgNO3+K2Cro4Ag2CrO4+2KNO3 . Ksp Ag2Cro4=1.7x10-12

**Procedure:**

1. Clean the burette and fill it with silver nitrate(0.1N).
2. Pipet 10mL of chloride ion solution into 250mL conical flask, add 5drops of potassium chromate.
3. Titrate chloride solution against silver nitrate until arrive to equivalent point(the point in which the number of moles of AgNO3 equal to the number of moles of chloride ion)[Notice a white precipitate in the yellow solution] After this point the excess of AgNO3 will react with potassium chromate leading to formation of red precipitate Ag2CrO4 (the end point). The difference between equivalent and end point is the volume of AgNO3 reacted with the indicator.
4. Repeat the titration and calculate the average and subtract the indicator blank(equal to 0.1mL).Calculate the normality.

Concentration of [CI-](ppm) = x eq.wt x1000

**Questions:**

1. Why is Mohr method carried out in neutral solution?
2. On which does Mohr method depend?
3. Enumarate methods used for determination of chloride ion?

**Experiment (6)**

**Oxidation-reduction reactions**

**Oxidation-reduction reactions**: are all the reactions involving change in oxidation number or transfer of electrons among the reacting substances.

\* **Reduction agent:** is one that loses electrons and is oxidized to a higher valiancy conditions.

Ex: Ferrous, stannous compounds, sodium thiosulphate and oxalate ion.

\* **Oxidation agent:** is one that gains electrons and is reduced to a lower valiancy conditions.

Ex: Potassium dichromate, potassium permanganate.

The permanganate ion in acid solution is a strong oxidizing agent, because the standard potential in acid solution E˚ has been calculated to be 1.51V.

**Preparation of solutions:**

**A: Preparation of 0.1N potassium permanganate.**

\* Weigh out about 3.2gm of KMnO4 on a watch glass and transfer it to a 1500mL beaker and add 1L distilled water, cover the beaker with a clock glass then heat the solution to boiling, boil gently for 1hr and allow the solution to cool to the laboratory temperature, filter the solution through a funnel containing a plug of purified glass wool, collect the filtrate in a vessel. The solution should be stored in a clean, glass stoppered bottle of a dark-brown-colored glass and kept in the dark.

**B: Preparation of 0.1 N sodium oxalate (Na2C2O4).**

\* Weigh out about 1.675gm of Na2C2O4 and dissolved it in distilled water in a 250mL volumetric flask.

**Standardization of permanganate solution with oxalate ion:**

1. Pipet 10mL of 0.1 N of sodium oxalate (Na2C2O4) in a 250mL conical flask
2. Add by a graduated cylinder 5mL of H2SO4 [1:8].
3. Add some drops of potassium permanganate and heat to 55-60°C and complete the titration by adding permanganate solution until a faint pink color persists for 30sec.
4. Calculate the normality of permanganate ion.

**Determination the concentration of ferrous ion:**

1. Pipet 10mL of ferrous ion solution in a 250mL conical flask.
2. Add 5mL of sulphuric acid H2SO4 [1:8].
3. Titrate the solution with standard (0.1N) KMnO4 until the pink color persists.
4. Repeat the titration and then calculate the normality of ferrous ion.

**Questions:**

1. Why do we consider KMnO4 as a strong oxidizing agent?
2. Why do we heat the prepared solution of KMnO4 to a boiling boil for 1 hr?
3. Why do we not use an indicator in this experiment?

**Experiment (7)**

**Complexometric titration**

This kind of titration is called complexometric because it involved formation of complex especially by using the compound EDTA (Ethylene Diamine Tetra Acetic acid) which forms a stable complex with metal ions especially with calcium and magnesium ions. In addition the users indicator with EDTA have also capable to form a complex with the same ions (Mg2+ and Ca2+) but less stable than the complex formed between the metal ions and EDTA.

The total hardness of water is generally due to dissolved calcium and magnesium salts Water hardness was defined in terms of the capacity of cations in the water to replace the sodium and potassium ions in soaps and form sparingly soluble products.

There are two types of water hardness, temporary and permanent, **Temporary hardness** is due to the bicarbonate ion,,(sodium and magnesium bicarbonate)being present in the water. This type of hardness can be removed by boiling the water to expel the CO2.

**Permanent hardness** is due to the presence of the ions Ca2+, Mg2+ and SO4 This type of hardness cannot be eliminated by boiling.

Hardness is now expressed in terms of the concentration of calcium carbonate that is equivalent to the total concentration of all the multivalent cations in the sample. The determination of hardness is a useful analytical test that provides a measure of water quality for household and industries uses.

**Determination of total hardness (permanent and temporary) of water**

**A: Determination of total hardness of water.**

1. Prepare 250mL of 0.02N of EDTA solution.
2. Clean the burette and rinse with EDTA solution.
3. Pipet 10mL of hardness water and add 2mL of ammonia buffer solution (PH=10)[prepared from ammonium chloride +conc. ammonia].
4. Add 3-4 drops of Eriochrome black-T indicator and titrate with standard EDTA solution until the color changes from wine red to pure blue color.
5. Repeat the titration a few times and calculate the total hardness (VA) of the sample of water (Ca2+ + Mg2+).

**Calculations**

VEDTA= Volume of EDTA which equivalent to (Ca2+ + Mg2+) VA

**Or**

**B: Determination of the permanent hardness**

1. Pipet 10mL of the hardness water and add 2mL of sodium hydroxide solution 4N (PH=12) which is sufficiently high to precipitate the magnesium quantitatively as the hydroxide.
2. Add some crystals of Murexide indicator.
3. Stirr the solution and titrate it with standard EDTA solution until the color changes from pink to a purple.
4. Repeat the titration a few times and calculate the (VB), which is the volume of EDTA equivalent to only.

**Calculations**

VEDTA =Volume of EDTA which equivalent to only VB

**Or**

**C: Determination of temporary hardness**

Calculate the temporary hardness of water by subtracting the permanent hardness from the total hardness.

**Or**

**Questions**:

1. What is the benefit of determination of water hardness?
2. Write the structure of EDTA compound and mention the ions that react with?
3. Why do we use Murexide and EBT as indicators in complexometric titrations?
4. Define the water hardness and give the main solutions to this problem?
5. Why should we prepare EDTA solution in ionized water?
6. Why do we use sodium hydroxide solution(PH=12)when we determine the permanent hardness?

|  |  |  |
| --- | --- | --- |
| No |  |  |
|  | clamp | ماسك معدني |
|  | Iron stand | حامل حديدي |
|  | Water bath | حمام مائي |
|  | Beaker | قدح |
|  | Washing bottle | قنينة غسيل |
|  | Balance | میزان |
|  | Conical flask | دورق مخروطي |
|  | Glass rod or stirrer | محرك زجاجي |
|  | Desiccator | مجفف |
|  | Graduated cylinder | اسطوانة مدرجة |
|  | spatula | ملعقة وزن |
|  | pipet . | ماصة |
|  | Burette clamp | ماسك سحاحة |
|  | Burette | سحاحة |
|  | Glass stoppered Bottle | قنينة بسداد زجاجي |
|  | Separating funnel | قمع فصل |
|  | Crucible | جفنة خزفية |
|  | Dropper | قطارة |
|  | Volumetric flask | قنينة حجمية |
|  | Test tube | انبوبة اختبار |
|  | Test tube Rack | حامل انابيب اختبار |
|  | Test tube Brush | فرشة انابيب اختبار |
|  | Reagent Bottle | قنينة كواشف |
|  | Tong | ماسك يدوي |
|  | Centrifuge | جهاز الطرد المركزي |
|  | Evaporating dish (porcelain) | جفنة تبخير |
|  | condenser | مكثف زجاجي |
|  | Adapter | مكيف (وصلة زجاجية) |
|  | Rubber tube | انبوب مطاطي |
|  | Round bottom flask | دورق دائري القعر |
|  | flat bottom flask | دورق مستو القعر |
|  | Fractional column | عمود التقطير التجزيئي |
|  | pipet | ماصة |
|  | Burette clamp | ماسك سحاحة |
|  | Burette | سحاحة |
|  | Glass stoppered Bottle | قنينة بسداد زجاجي |
|  | Separating funnel | قمع فصل |
|  | Evaporating dish (porcelain) | جفنة تبخیر |
|  | Crucible | جفنة خزفية |
|  | Dropper | قطارة |
|  | Volumetric flask | قنينة حجمية |
|  | Test tube | انبوبة اختبار |
|  | Test tube Rack | حامل انابيب اختبار |
|  | Test tube Brush | فرشة انابيب اختبار |
|  | Reagent Bottle | قنينة كواشف |
|  | Tong | ماسك يدوي |
|  | Centrifuge | جهاز الطرد المركزي |







































