LAB-ANALATICAL CHEMISTRY

MIRACLE 2023

BY MALAK AL-ALWAN

AND SHAHEERA ALSHBEILAT

((CONTENTS))

{ وَأَنْ لَيسَ لِلإنسَانِ إلَّا مَا سَعَى , وَأَنَّ سَعْيَهُ سَوْفَ يُرَى , ثُمَّ يُجْزَاهُ الْجَزَاءَ الأَوْفَى }

المصادر الي اخذت منها المعلومات ورتبتها
[بناسيا وترياق والمانيوال + ملاحظاتي من الاب]
malak.moh.alalwan@gmail.com

أكيد المانيوال المرجع الأساسي 👍

The Hashemite University

Analatical Chemistry (1)

EXPERIMENT (1) : Volumetric glass ware and Balances

* Analatical glass wares only :

1.volumetric flask
2.volumetric piptte
3.Burets
4.piptte

* Graduated cylinder is not Analatical glass ware .

لانه مش أداة دقيقة لحساب حجم السائل بس بنستخدمها لنقل العينات فقط

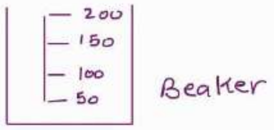
* Analatical Balance is the balance that measure 3 points of right come 0.000 (..........g ± 0.0001)

* نسبة الخطأ في الأدوات المدرجة = $\frac{\text{أقل تدريج}}{2}$

مثلاً

150 - 100 = 50

$\frac{50}{2} = 25$



معنى ذلك عند استخدام ال Beaker للقياس قد تكون القراءة زائد عليها أو منقوص منها 25ml مثلاً 150ml + 25 هكذا تكتب القراءة .

* ال Burets , نسبة الخطأ فيها $\frac{0.1}{2} = 0.05$

القراءة من فوق لل Burets , تنظيف بال Tap water ثم بال D.W ثم بالحمض أو القاعدة الذي نود ان نعبيه فيها .
لأن قطرات ال D.W تعمل ع تخفيف المحلول لذلك نغسل بالحمض أو القاعدة الذي نريد استخدامها للتخلص من هذه القطرات , ولأنه قطرات ال D.W يصعب تجفيفها .

تجربة اليوم :

- خذ 0.05ml من الماء (volume) وال mass 0.05
- احسب الكثافة (Density)

$$\text{الحجم} = \frac{\text{الوزن}}{\text{الكثافة}}$$

- كثافة ال D.W نستخرجها حسب درجة الحرارة من الجدول ص٢ في المانيوال .
- كل قيم الكثافة قريبة من 1 ... لاب التحليلية لأنه يحتاج دقة , يجب الالتزام ببيانات الجدول .

This type of calibration involves the use of on analytical balanc .

mL/g الرجاء التأكد من الجدول وقيمة والوحدة القياسية لانه يتغير مع السنوات حسب آخر تحديث للمانيوال المتبع وقتها

Table 2. Volume of 1g of water weighed in air at various temperatures

°C	mL	°C	mL	°C	mL
10	1.0016	19	1.0026	28	1.0046
11	1.0017	20	1.0028	29	1.0048
12	1.0018	21	1.0030	30	1.0051
13	1.0019	22	1.0032		1.0051
14	1.0020	23	1.0034		
15	1.0021	24	1.0036		
16	1.0022	25	1.0038		
17	1.0023	26	1.0041		
18	1.0025	27	1.0043		

* إذا كانت وحدة الكثافة (g \ ml) إذا كانت بالامتحان بهذا الشكل احسب الحجم عن طريق ال V = mass \density

*إذا كانت معكوسة (ml \ g) نحسب الحجم عن طريق ال V = mass * density

مهم. م. م.

$$\boxed{V_{actual} = mass \times \frac{1}{d}}$$

← ترتيب الأدوات مهم بالامتحان

volumetric piptte > piptte > Burette > graduated cylinder > Beaker & Erlenmeyer flask

هذا الترتيب حسب دقة الأدوات .

← قوانين التجربة :

Apparent volume (ml) = final burette reading - initial burette reading

weight of water (g) = final of flask - initial of flask

الكثافة Density

Actual volume (ml) = weight of water × volume of 1g of water

من الجدول ص2

بالمانيوال mg / L

correction (ml) = | Actual volume - Apparent volume |

((errors))

(1) initial & final reading ناتجة من الشغل العملي

القراءة فردية ، وبتالي ال error للجهاز ..

- Burette $\frac{0.1}{2} = 0.05$
- Balance (± 0.0001) تصفير القراءة

(2) Actual volume , Apparent volume & weight of water ... يعني اي شيء ناتج من قانون

- قانون جمع أو طرح

$$error = \sqrt{(error)^2 + (error)^2}$$

- قانون ضرب أو قسمة

$$error = 2 \times \sqrt{\left(\frac{error}{\text{القيمة}}\right)^2 + \left(\frac{error}{\text{القيمة}}\right)^2}$$

مثل حساب ال error لل Actual volume

تطبيق :

Q1. Order in increasing accuracy of volumetric measurement tools :

أدوات القياس الحجمية
رتبها حسب دقتها

a) burret , beaker , graduated cylinder , pipette
b) graduated cylinder , beaker , pipette , burret
c) beaker , graduated cylinder , pipette , burret
d) beaker , graduated cylinder , burret , pipette

أقل دقة ... اعلى دقة

Answer : D

Q2. A 10ml pipette was used to deliver 10 ml of four substances : A, B ,C and D. If the mass of 10 ml of each substances was found to be :(8.9 g , 9.2 g , 9.1 g , 9.1 g) respectively . order the substances in order of density decreasing .

رتبا هذه المواد حسب نقصان الكثافة

اعلى كثافة ⟶ اقل كثافة

$d = \frac{m}{\underset{ثابت}{v}}$

Answer : B , C , D , A

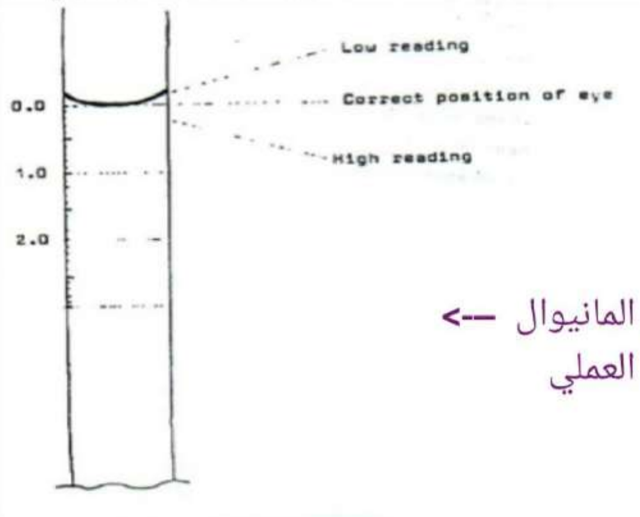
$d \propto m$
الاعلى كثافة
هو الاعلى وزن

A. Cleaning.

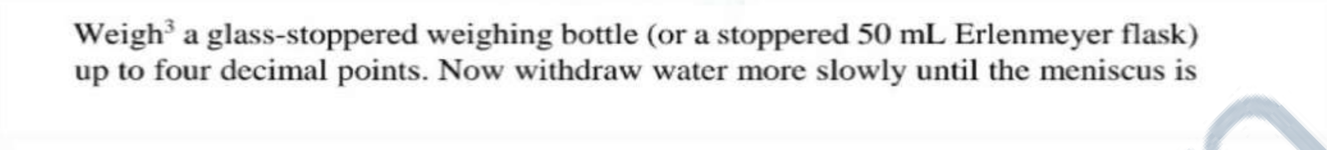
Clean the buret using a mild detergent and brush. Thoroughly rinse and test the buret for dirt by allowing water to drain from the buret. If the buret is clean, an even film of water will develop on the surface, if the buret is still dirty, water droplets will form as the liquid is allowed to drain from the buret. Thus, it is necessary to clean to buret with dichromate cleaning solution. However, care must be taken when using this cleaning solution since it can cause burns.

B. Calibration of a buret.

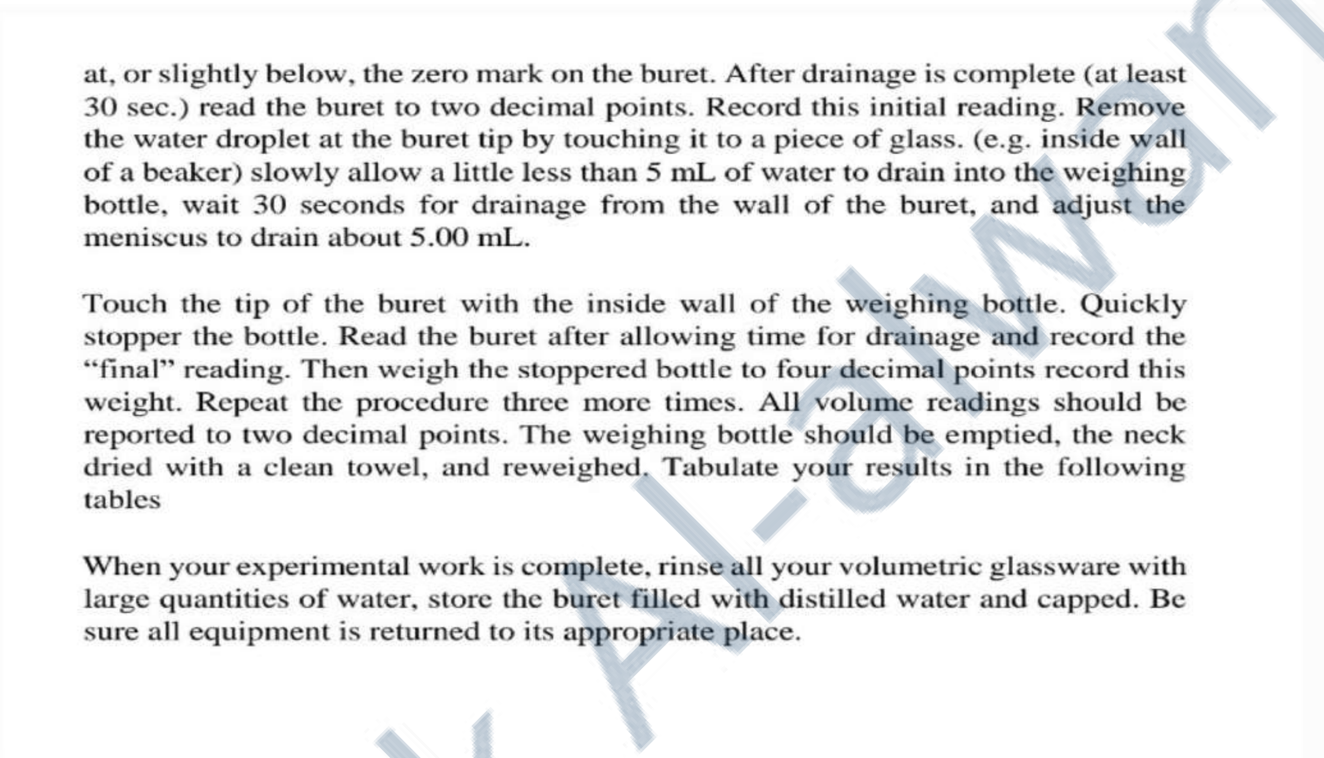
After the buret has been cleaned and rinsed, fill it with distilled water so that the meniscus is above the zero mark. Then slowly open the stopcocks and allow the solution to fill the tip. If air bubbles appear in the tip, these must be removed by quickly turning the stopcock open and closed until the bubbles leave through the tip. By opening the stopcock, adjust the level of solution in the buret so that the meniscus is above the zero mark, Fig (1). (Be sure there are no air bubbles in the buret tip). Record the temperature of the water.



الشغل العملي من المانيوال <--
أو فيديو التجربة العملي



Weigh[3] a glass-stoppered weighing bottle (or a stoppered 50 mL Erlenmeyer flask) up to four decimal points. Now withdraw water more slowly until the meniscus is



at, or slightly below, the zero mark on the buret. After drainage is complete (at least 30 sec.) read the buret to two decimal points. Record this initial reading. Remove the water droplet at the buret tip by touching it to a piece of glass. (e.g. inside wall of a beaker) slowly allow a little less than 5 mL of water to drain into the weighing bottle, wait 30 seconds for drainage from the wall of the buret, and adjust the meniscus to drain about 5.00 mL.

Touch the tip of the buret with the inside wall of the weighing bottle. Quickly stopper the bottle. Read the buret after allowing time for drainage and record the "final" reading. Then weigh the stoppered bottle to four decimal points record this weight. Repeat the procedure three more times. All volume readings should be reported to two decimal points. The weighing bottle should be emptied, the neck dried with a clean towel, and reweighed. Tabulate your results in the following tables

When your experimental work is complete, rinse all your volumetric glassware with large quantities of water, store the buret filled with distilled water and capped. Be sure all equipment is returned to its appropriate place.

EXPERIMENT (1)

VOLUMETRIC GLASSWARE AND BALANCES

Name:	**Group NO.:**

III DATA

		Trial 1	Trial2	Trial 3	Trial4
قبل إضافة الماء	Initial burette reading, mL	0.0 +- 0.1 mL	10.0 +- 0.1 mL	20.0 +- 0.1 mL	
بعد إضافة الماء	Final burette reading, mL	10.0 +- 0.1 mL	20.0 +- 0.1mL	30.0 +- 0.1 mL	
	Apparent volume, mL	10 mL	10 mL	10 mL	
قبل إضافة الماء W1	Initial weight of flask, g	0.0000 +- 0.0001 g	0.0000 +- 0.0001 g	0.0000 +- 0.0001 g	
بعد إضافة الماء W2	Final weight of flask, g	9.7436 +- 0.0001 g	10.1384 +- 0.0001 g	9.9698 +- 0.0001 g	
W2 - w1	Weight of water, g	9.7436 +- 0.0001 g	10.1384 +- 0.0001 g	9.9698 +- 0.0001 g	
	Temperature, ^{o}C	26.5 +- 0.5 °c	26.9 +- 0.5 °c	26.5 +- °c	
	Actual volume, mL	9.7855 +- 0.0001 mL.	10.1820 +- 0.0001 mL.	10.0127 +- 0.0001 mL	
	Correction, mL	0.2145 +- 0.0001 mL.	0.1820 +- 0.0001 mL.	0.127 +- 0.0001 mL	

Instructor's Signature

Actual volume = weight of water × volume of 1 g of water (Density) من جدول ص٢

Trial 1 = 9.7436 g × (1.0043 mL ÷ 1 g) = 9.7855 +- 0.0001 mL

Trial 2 = 10.1384 g × (1.0043 mL ÷ 1 g) = 10.18199 +- 0.0001 mL

Trial 3 = 9.9698 g × (1.0043 mL ÷ 1 g) = 10.01267 = 10.0127 +- 0.0001 mL

Correction = | Actual volume - Apperent volume |

Trial 1 = I 9.7855 - 10 I = I - 0.2145 I = 0.2145 +- 0.0001 mL

Trial 2 = I 10.1820 - 10 I = I 0.1820 I = 0.1820 +- 0.0001 mL

Trial 3 = I 10.0127 - 10 I = I 0.0127 I = 0.0127 +- 0.0001 mL

Note: use significant figures and decimal places in the calculations

IV. CALCULATION[4]

$\Delta x = \left(\frac{\text{error } x}{x}\right)^2$

(Trial No ....)

1- Apparent Volume (mL) = Final burret reading – Initial burret reading

2- Weight of water(g) =

3- Actual Volume (mL)

4- Correction (mL) =

ERRORS

- **According to the following rules:**

جمع. z = x + y or طرح z = x - y ⇒ $\Delta z = \sqrt{\Delta X^2 + \Delta y^2}$

ضرب z = x * y or قسمة $z = \frac{x}{y}$ ⇒ $\Delta z = z * \sqrt{\left(\frac{\Delta X}{x}\right)^2 + \left(\frac{\Delta y}{y}\right)^2}$

Calculate the following errors:

خطأ ال Burret ① Initial (ΔR_i) and final (ΔR_f) burret reading errors= $\frac{0.1}{2}$ = ± 0.05

ناتجة من قانون طرح

2- Apparent volume error (ΔA) = $\sqrt{(0.05)^2 + (0.05)^2}$ = ± 0.07071

3- Initial (ΔW_i) and final (ΔW_f) weight of the flask errors= Error in the balance= ± 0.0001

ناتجة من قانون طرح

4- Weight of water error (ΔW)= $\sqrt{(0.0001)^2 + (0.0001)^2}$ = ± $1.414 * 10^{-4}$

مش مطالبين فيها إجباراً ⑤ Temperature error (ΔT) ± 0.5

6- Actual volume error(ΔAct) =

.

. QUESTIONS

Apparent volume

Q1) A 25 mL pipette was found to deliver 24.876 g of water when calibrated against stainless steel weight at 25 ^{o}C. Use the data in Table 2 to calculate the volume delivered by the pipette at this temperature, repeat the calculation at 19 ^{o}C.

عند درجة حرارة 25

☆ Actual volume = weight of water × volume of 1 g of water
= 24.876 g × (1.0038 mL ÷ 1 g) = 24.970528 = 24.971 mL

☆ correction (mL) = | Actual volume - Apparent volume | = | 24.971 - 25 | = 0.029 mL

عند درجة حرارة 19

☆ Actual volume = weight of water × volume of 1 g of water
= 24.876 g × (1.0026 mL ÷ 1 g) = 24.941 mL

☆ correction (mL) = | Actual volume - Apparent volume | = | 24.941 - 25 | = 0.059 mL

Q2) Suggest some sources of error in this experiment.

مش مطلوب إجمالًا

- from devices such as Analytical Balance & thermometer

- measurement Errors resulting from parallax effect from graduated cylinder (must be taken from meniscus)

- equipment is not clean or containing precipitating

إجابة آخرى

1) systemetic error

2) personal error

EXPERIMENT (2) : Determination of sampling error

هدف هذه التجربة : حساب ال standard diviation (الانحراف المعياري) لعينة نقية وعينة غير نقيه.
من خلال الحسابات , ستلاحظ الفرق الذي يحدثه وجود ال error في العينة وتشويش .

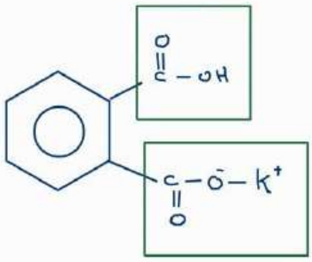
SAMPLE

HOMOGENEOUS نقية

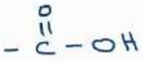
HETROGENEOUS غير نقية

acid + base → salt + water
KHP + NaoH → salt + water

KHP : Potassium Hydrogen Phthalate



" الاختصارات مهمة في لاب التحليلية "
لا يهمنا ال structure في الاب , لكن يهمنا معرفة انه حمض
والدال ع ذاك وجود الحمض الكربوكسيلي



$-\overset{O}{\overset{\|}{C}}-OH$

فكرة التجربة :

- نريد مفاعلة حمض مع قاعدة معاً , يحدث تعادل , نقطة التعادل سوف نسجلها بالاب .
- نبدأ بإضافة كمية من ال Base شوي شوي فوق كمية معينة من ال Acid , عند الوصول لنقطة التعادل , يجب إيقاف إضافة ال Base . نقطة التعادل سوف تلاحظها عن طريق تغير اللون .

Burette contains (Base NaoH)

Flask contains Acid + Indicator
(KHP + ph.ph)

- نعبي ال Burette بال NaoH لأي رقم .
- ال KHP عبارة عن powder لذلك نذوبه بالماء داخل ال Flask ونضع فوقه الكاشف (Indicator) .

- ما فائدة ال Indicator ؟ له لون في المحلول الحمضي يختلف عن لونه في القاعدي , فعند حدوث تغير في اللون , يدل ذلك ع تحول وسط المحلول .

(عند حدوث تغير في اللون معناته انا وصلت لنقطة التعادل - end point)

- نوزن 0.25 غرام من ال KHP .
- **إرشادات لل Titration :**
ال flask الذي نوزن به ال KHP , خطأ ان يكون مبلول من الخارج , يحدث error في القراءة .

الميزان حساس لذالك نختار flask صغير الحجم للتوزين .
يفضل نفس الشخص ان يقوم بعملية ال مُعايرة بالكامل .

تذوب ال KHP في كمية من ال (water (30 - 40 ml ولا يهم كمية الماء لانه غير داخله في الحسابات , المهم ان تذوب كل كمية ال KHP .

بضل افتح واسكر في الصمام ال burette حتى يصبح لون المحلول في ال flask زهري فاتح (pink) , لون المحلول ال pink دليل ع ان المحلول تحول الى القاعدي , لكن انا اريد اخذ القراءة عند نقطة التعادل قبل التحول للقاعدي , لكن هذه النقطة صعب ايجادها عملياً , لذلك عندما يصبح اللون زهري فاتح , يجب التوقف عن ال Titration .

المفروض عندما يتغير اللون لل pink أن يثبت اللون لمدة 30 ثانية ثم يختفي .

إذا صار اللون pink غامق فهذا يدل ع ← تم إضافة كمية كبيرة

← ضفت Base زيادة عن اللزوم .

Ph.Ph

pink in base media | بدون لون , شفاف **colurless in acid media**

الحسابات .... عند نقطة التعادل

$$\text{Moles of KHP} = \text{Moles of NaoH}$$

هدف هذا الجزء من التجربة هو حساب ال (M (Molarity لل NaoH .

عن طريق هذه العلاقة , نحسب ال M لل NaoH .

$$\text{Moles of KHP} = \text{Moles of NaoH}$$

$$\frac{\text{mass}}{\text{M.m}} = \boxed{M} \times V$$

الذي وزناه بالاب 0.25 g

204.2 g/mol

تُعطى بالإمتحان

مطلوبه ?!

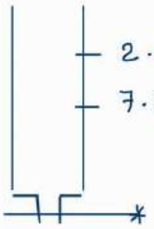
mol / L

بالسحب من البيوريت وهو الحجم الذي حدث عنده نقطة التحول

كيفية قياس الحجم من ال Burette , كانت ممتلئه بال NaoH لحد 2.00ml

عند عمل ال Titration قل الحجم , فاصبحت 7.3ml فرق الحجم هوه (النهائي - البدائي)

نعوضه بالقانون وهو الحجم الدال ع حصول نقطة التعادل .



$$7.3 - 2 = 5.3 \text{ mL}$$

الذي يدخل في الحسابات

→ Part two of EXP (Hetro geneous sample)

نفس الجزء الاول , الفرق ان ال KHP مخلوط معه Sugar **(بنسبة 1 : 1)**
نوزن 0.5g منه , ونذوبه في ال flask وبنعمل Titration طبيعي

ملاحظات ع حل الريبورت

فرع B لا تنسى القسمة ع 2 ؛ لانه متوقع عند التصفيه ان يكون ال KHP لوحده لذلك عند الحسابات نقسم ع 2 .
السكر لا نحتاجه في الحسابات , فقط نحتاجه ليشوش العينة وبتالي سيأثر ع ال (V) المحسوبه من ال Burette .

Part 1 - Homogenous sample

نحسب لها Sa حيث a : analysis
الانحراف المعياري الناتج من التحليل (analysis) ولسى من العينة لأنه(pure KHP نقي)
أي خطأ سيكون من التحليل .

Part 2 - Hetrogenous sample

نحسب لها So حيث o : overall
هنا قد يحدث خطئين .. في التحليل أو في التحضير للعينة

يهمنا حساب ال Ss وهي الإنحراف المعياري الناتج عن :

part 2 الكلي   part 1 التحليل

$$S_o^{2} = S_a^{2} + S_s^{2}$$

الانحراف المعياري

$$S_s = \sqrt{S_o^{2} - S_a^{2}}$$

لا تنسى التربيع والجذر ، دائمًا ال So أكبر من ال Sa
لانه ناتجة عن جمع $S_a^2 + S_s^2$ ولانها إحتوت على تشويش 🤪

خطوات إخراج ال SD من الآلة الحاسبه نوعها casio . كل آله حاسبه لها طريقة خاصة بالحساب لذلك تدرب ع آلتك قبل الامتحان

1) MODE
2) 2 : STAT
3) 1 : 1- VAR
4) ونضغط على(=) , ندخل القيم
5) AC
6) shift + 1
7) 4 : VAR
8) 4 : SX

لا تنسى تعديل ال mode الى ال 1 : comp بعد الإنتهاء من الحل .

تطبيق :

سنوات مكرر وبنفس الأرقام

Q1 : A student found the standard deviation for molarities of a homogenous sample to be 0.04 & the standard deviation for molarities of heterogeneous sample to be 0.09

Find the standard deviation of sampling .

Ss ?!

$S_a = 0.04 \qquad S_o = 0.09$

$$S_s = ?? = \sqrt{S_o^2 - S_a^2} = \sqrt{(0.09)^2 - (0.04)^2} = 0.0806 = 0.081$$

Q2 : A student has calculated the standard deviation for molarities of a homogenous sample to be 0.04 & the standard deviation for molarities of heterogenous sample to be 0.06

Find the standard deviation of sampling .

?!

$S_a = 0.04 \qquad S_o = 0.06$

$$S_s = \sqrt{S_o^2 - S_a^2} = \sqrt{(0.06)^2 - (0.04)^2} = 0.0447 = 0.045$$

II. EXPERIMENTML PROCEDURE

a. Homogenous sample

1. Titration of potassium hydrogen phthalate (KHP)

Wight accurately about 0.5g of pure KHP into a 250 mL Erlenmeyer flask. add 25 mL distilled water and three drops of phenolphtalein. Titrate with 0.10 M NaOH till the indicator color becomes pink. Repeat 5 times.

b. Hetrohomogenous sample

1. Mixture of KHP and sugar.

Wight accurately about 0.5gof the KHP and sugar mixture into 250 mL Erlenmeyer flask, add 25 mL distilled water and three drops of phenolphthalein. Titrate against 0.10 M NaOH. Repeat 5 times.

$n_{KHP} = n_{NaOH}$

$$\boxed{\frac{mass}{M.mass} = M * V} \rightarrow M = \frac{mass}{M.mass * V}$$

EXPERIMENT (2)

SAMPLING AND STATISTICAL HANDLING OF DATA

Name:	**Group NO.:**

a) Homogenous sample

	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Mass of KHP(g)	0.250 g ± 0.001	0.240 g ± 0.001	0.295 g ± 0.001		
Volume of NaOH (mL)	10.70 mL ± 0.07	11.5 mL ± 0.07	12.80 mL ± 0.07		
Mokarity of NaOH (M)	0.114	0.102	0.113		
Avarage molarity of NaOH	0.110 M				AV
Standered diviation (S_a)	0.00665				Sa

mol/L ??

عملياً

عملياً

نظرياً

b) Hetrohomogenous sample

(50% KHP + 50% diluent)

$\frac{0.50}{2}$

بدون سكر

	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5
Mass of mixture(g)	0.251 g ± 0.001	0.250 g ± 0.001	0.247 g ± 0.001		
Volume of NaOH (mL)	5.50 mL ± 0.07	6.00 mL ± 0.07	6.30 mL ± 0.07		
Mokarity of NaOH (M)	0.112	0.102	0.096		
Avarage molarity of NaOH	0.103 M				AV
Standered diviation (S_t)	0.00794				So

mol/L ??

عملياً

عملياً

نظرياً

$$\boxed{M_{NaOH} = \frac{mass\ KHP}{V_{NaOH} * M.mass_{KHP}}}$$

$M.m_{KHP} = 204.2$ g/mol

$x_i = M_{NaOH}$

في ال Homogeneous sample

كل قراءة على حدا

$$\boxed{mass\ of\ KHP = \frac{ratio\ of\ KHP\ in\ HS}{100} * mass\ of\ HS}$$

$S_s = \sqrt{So^2 + Sa^2} = \sqrt{(0.0074)^2 - (0.00665)^2}$

$= 4.34 * 10^{-3} = 0.00434$

النهاية

c) Calculation the sampling error (Ss)

a) Homogeneous sample:

1- Molarity of NaOH for trial 1 = $M_{NaOH} = \frac{m\ KHP}{M.m_{KHP} * V_{NaOH}} = \frac{0.25\ g}{204.2\ g/mol * 10.7\ mL}$

$= \frac{1.144\ mol}{\cancel{mL}} * \frac{1000\ \cancel{mL}}{1L} = 0.144\ M$

$= 0.144\ mol/L$

2- Average molarity of NaOH (Ave.x) =

= (trial 1 + trial 2 + trial 3) ÷ 3 = (1.144 + 0.102 + 0.113) ÷ 3 = 0.110 M

نطلع
الآلة الحاسبة

3- The standard deviation of Homogeneous sample (S_a): = 6.65 × 10 -³ = 0.00665

Standared diviation (S) = $\sqrt{\frac{\sum(x_i - Ave.x)^2}{n-1}}$ $\bar{x}$

Sa =

$M_{NaOH} = \frac{m\ KHP}{M.m_{NaOH} * V_{NaOH}} = \frac{0.126\ g}{204.2\ g/mol * 5.5\ mL}$

b) Heterogeneous sample (HS):

1- Molarity of NaOH for trial 1 $= \frac{1.121 * 10^{-4}\ mol}{\cancel{mL}} * \frac{1000\ \cancel{mL}}{1L} = 0.112\ mol/L$

$\bar{x}$

نطلع
الآلة الحاسبة
أسرع

2- Average molarity of NaOH = 0.1033

3- The standard deviation of Heterogeneous sample (S_t): $n = 3 \longrightarrow n-1 = 3-1 = 2$

$\Sigma(x_i - \bar{x})^2 = (0.112 - 0.103)^2 + (0.102 - 0.103)^2 + (0.096 - 0.103)^2 = 0.00013$

Sampling error (S_s):

Remember that: $\boxed{S_o^2 = S_a^2 + S_s^2}$

$S_o = \sqrt{\frac{\Sigma(x_i - \bar{x})^2}{n-1}} = \sqrt{\frac{0.00013}{2}}$

$= 8.062 * 10^{-3}$

$= 0.00806$

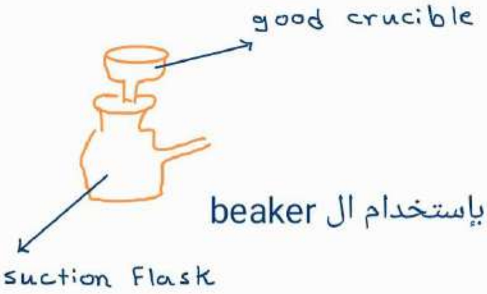
$$\boxed{S_s = \sqrt{So^2 - Sa^2}}$$

EXPERIMENT (3) : Gravimetric Analysis

بشكل عام هذه التفاعلات تعتمد ع الجاذبية الارضية gravity
فكرة هذه التفاعلات : ينتج لدي راسب , اوزنه , كل حساباتي تعتمد ع وزن هذا الراسب

خطوات التجربة :

- احضر beaker حجم 600ml
- عبيه (100 - 150 ml) water , دائماً للسلامة العامة يفضل ان تملأ ال beaker بكمية لا تتعدى منتصفه إذا اردنا تسخينه , لكي لا يؤذيك .



- داخل ال beaker نضع ال anon ← الهدف حساب وزنه
- نضع 0.5ml HNO3 (nitric acid) لأنه يزيد الذائبية
- نضيف 20ml AgNO3 in the cylinder ونقيس هذه الكمية , نضيفها بإستخدام ال beaker
- نحرك بشكل متواصل وقوي جداً لقبل الغليان بشوي , اجعله يبرد
- عملية الفلتره : عن طريق ال suction flask & good crucible

- عملية غسل المتبقي من المحلول .... (HNO3 (nitric acid مخفف بالماء المقطر D.W , نغسل المحلول
- ضعهُ في الفرن oven , يحتاج ربع ساعة
- قبل ان نوزنه يجب ان يبرد , استخدم المجفف Desicater , يحتوي بقاعدته سيليكا جل , هذه الحبيبات تعشق الرطوبة مثل المركب (salt .5H2O) مثل الموجودة بعلب الأحذية

الحسابات :

$$\text{Mass of CL}^- = \text{Mass of Agcl} \times \frac{\text{M.m of CL}^-}{\text{M.m of Agcl}}$$

$$\text{Mass of Anon} = \text{Mass of ppt الراسب} \times \frac{\text{M.m of Anon}}{\text{M.m of ppt}}$$

Mass (g)

$$\frac{g}{L} = \frac{\text{Mass}}{10 \times 10^{-3}}$$

حجم الأنون بال mL

لتحويل للتر

ppm (mg / L)

$$\frac{\text{Mass} \times 10^3}{10 \times 10^{-3}}$$

لتحويل الـ g إلى mg

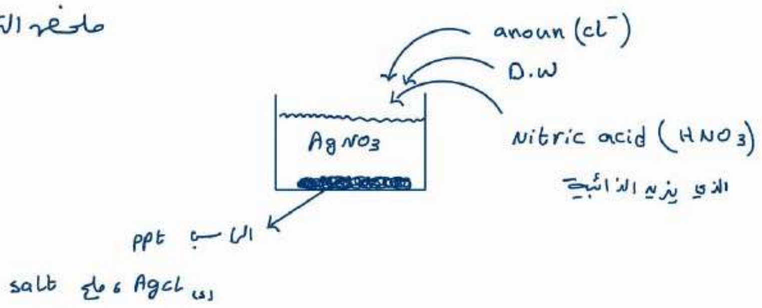
حجم الأنون بال mL

لتحويل إلى اللتر

Mass of empty crucible = 29.307
Mass of crucible + ppt = 29.469
Mass of AgcL ppt = 29.469 - 29.307 = 0.162

معروف إنه ال CL يترسب على شكل AgCL

ملخص التفاعل



the processes of gravimetric analysis :

1. preparation of analyte solution
2. precipitation الترسيب
3. digestion ⟶ For one hour
4. filitration ⟶ collection of ppt using gooch crucibles.
5. washing ⟶ the ppt AgCl washed with HNO_3 in order to (prevent piptization)
6. drying
7. weighing

Desired properbies of product :

1.law solubility
2. easy to filter& wash
3. large particles
4. unreactive
5. known composition

properties of collored ppt :

1. hard to settled & filtered
2. impure غير نقي because adsorption of contamination (coperecipitation).

coagulation of collorde ppt :

1.heating
2. stirring
3. adding electrolyte solution

HNO3 (nitric acid) employed for washing ppt :

1. it is prevent piptization

تفكك عكس coguلation

2. readrly volatile

AgCl is Light sensitive 8- $AgCl \xrightarrow{h.r} 2\,\boxed{Ag} + Cl_2$

remain collordly dispersed in the AgCl and imparts a purple color.

$$Cl_2 + 5Ag^+ + 3H_2O \rightleftharpoons 5AgCl + ClO_3^- + 6H^+$$

II. EXPERIMENTAL PROCEDURE

Pipet 10.0 mL of the unknown chloride solution into a 600 mL beaker. Dilute with 100-150 mL of distilled water. Add about 0.5 mL of conc. nitric acid. Cover the beaker with a clean watch class.

Slowly and with string add about 20 mL of the 0.1 M $AgNO_3$ solution. Heat the suspension nearly to boiling with frequent stirring[4] to coagulate the silver chloride. Let the precipitate settle, and test for complete precipitation by carefully adding few drops of silver nitrate to the clear supernatant liquid. If more precipitate or cloudiness appears, add a few more milliliters of silver nitrate solution, stir well, heat, let the precipitate settle, and test again. Continue in this way until precipitation is complete. Let the covered beaker stand in the desk, protected from light, for 30 min. before filtration.

Preparation of the gooch crucible:

Clean the crucible from surface contamination using soap and water, rinse, then place it in a crucible holder in a suction flask, if chemical cleaning is required, draw a little concentrated HNO_3 slowly through the porous filtering disc. With gentle suction, draw several small portions of distilled water through the filter. Mark the crucible with your name, and put it the oven at the drying temperature of the precipitate (120-130 ^{0}C) for half an hour. With clean crucible tongs, transfer the hot crucible to a desiccator, cool for 10 minutes, and weigh. Repeat drying until constant weights within ± 0.3 mg are obtained.

Filtration and washing of the precipitate

Decant the solution through the weighed crucible, pouring the solution down by stirring rod, and using gentle suction.
Prepare the wash solution by mixing 5 mL of concentrated HNO_3 with 200 mL water. To the precipitate in the beaker add about 25 mL of the wash solution, stirr well, let the precipitate settle, and decant the solution through the filter crucible.

Repeat the washing by decantation two times, and finally bring the precipitate into the filter; use small portions of the wash solution for transfer. Remove with a rubber policeman any solid particles adhering to the beaker Continue washing the precipitate in the crucible with the wash solution until the last portions of washing give a negative test for silver ions. Silver ion is tested for by adding a drop of hydrochloric acid.

Drying and Weighing of the Precipitate:

Place the crucible containing the precipitate in the oven for (25-30) minutes at 120 -130 ^{0}C. Cool the crucible in the desiccator, and weigh accurately. Repeat for 15 minutes periods to obtain constant weights of ± 0.3 mg.

---

[4] The stirring helps prevent bumping of the solution during heating and the danger of loss of precipitate

تطبيق :

Q1 : An excess of $AgNO_3$ was added to a 0.24 g sample of 70.6 % $CaCL_2$ (111 g /moL)
What is the mass of AgCL precipitated ??
M.m AgCL = 143.5 g / moL
M.m CL = 35.5 g / moL

answer : 0.44 g

$AgNO_3$ فائض

$m_{sample} = 0.24\ g \longrightarrow 70.6\ \%\ of\ cacl_2$

$M.m_{cacl_2} = 111\ g/mol$

$m_{AgcL} = ??\ g$ راسب ppt

$M.m_{AgcL} = 143.5\ g/mol$

$M.m_{cL} = 35.5\ g/mol$

$$\boxed{1\ cacl_2 + 2\ AgNO_3 \longrightarrow 2\ AgcL}$$

① anoun

$$\frac{mass\ of\ cacl_2^{\ ?!}}{mass\ sample} * 100 \cancel{\%} = 70.6 \cancel{\%}$$

$$mass\ of\ cacl_2 = \frac{70.6 * m_{sample}}{100} = \frac{70.6 * 0.24}{100} = 0.16944\ g$$

② $n_{cacl_2} = ?? = \frac{0.16944}{111} = 1.5264 * 10^{-3}\ mol$

③ من المعادلة

$$1\ mol\ cacl_2 \longrightarrow 2\ mol\ AgcL$$
$$1.5264 * 10^{-3}\ mol\ cacl_2 \xrightarrow{\times} ?!\ mol\ AgcL$$

$$n_{AgcL} = 3.0528 * 10^{-3}\ mol$$

④ $m_{AgcL} = M.m * n = 143.5 * 3.0528 * 10^{-3}$
$= 0.43807\ g$
$= 0.44\ g$

Q2 : What is the mass of AgCL (143.5 g / moL) precipated when excess AgNO3 was added to a 0.24 g sample that contains 50.6 % CaCL2 (111 g / moL) ??

M.m CL = 35.5 g / moL

answer : 0.31 g

$m_{AgcL} = ??\ g$

$M.m_{AgcL} = 143.5\ g/moL$

(فائض)

$m_{sample} = 0.24\ g$

50.6 % $cacl_2$ تحتوي على

$M.m_{cL^-} = 35.5\ g/moL$

$M.m_{cacl_2} = 111\ g/moL$

① $\frac{m_{cacl_2}\ ?!}{m_{sample}} * 100 \cancel{\%} = 50.6 \cancel{\%}$

$m_{cacl_2} = \frac{m_{sample} * 50.6}{100}$

$= \frac{0.24 * 50.6}{100} = 0.12144\, g$

المعادلة

② $1\, cacl_2 + 2\, AgNO_3 \longrightarrow 2\, AgcL$

$n_{cacl_2} = \frac{m}{M.m} = \frac{0.12144}{111}$

$= 1.09405 * 10^{-3}\ mol$

$1\ moL\ cacl_2 \longrightarrow 2\ moL\ AgcL$

$1.09405 * 10^{-3}\ mol\ cacl_2 \longrightarrow ?!\ mol\ AgcL$

$n_{AgcL} = 2.188 * 10^{-3}\ mol$

③ $m_{AgcL} = n * M.m = 2.188 * 10^{-3} * 143.5$

$= 0.313978\ g = 0.31\ g$

EXPERIMENT (3)
GRAVIMETRIC DETERMINATION OF CHLORIDE

Name:	**Group NO.:**

III DATA

رقم الأنون 29

① Volume of unknown solution used, mL	10.00 mL ± 0.05
② Mass of empty crucible, g	29.9370 g ± 0.0001
③ Mass of crucible + precipitate, g	30.2795 g ± 0.0001
④ Mass of AgCl precipitate, g	0.3425 g ± 0.0001

$= ③ - ② = 30.29795 - 29.9370 = 0.3425$

IV CALCULATION

ppt AgCl ⑤

1- Mass of Cl in the volume used, g = $\text{Mass of AgCl} * \frac{\text{M.m of Cl}^-}{\text{M.m of AgCl}}$

$= 0.3425\ g * \frac{35.5\ g/mol}{143.3\ g/mol} = 0.0848\ g$

[illegible] $\frac{⑤}{①}$

2- Mass of Cl in 1L (g/L) = $\frac{0.0848\ g}{10 * 10^{-3}\ L} = \frac{0.0848}{10} * 10^3 = 8.48\ g/L$

$M.m_{Cl^-} = 35.5\ g/mol$

$M.m_{AgCl} = 143.3\ g/mol$

V QUESTIONS

مهم جدًا جدًا 📌

Q1) What is the effect of the following phenomena on the chloride determination:

فيزكغا بة

a) Insufficient amount of $AgNO_3$ added. ⬇️ dec.

b) Incomplete transfer of the precipitate. ⬇️ dec. لم يتم نقلها بشكل صحيح

c) Coprecipitation ⬆️ inc. المحلول تكتل كويس

d) Washing too little ⬆️ inc. الغسل قليل ، يعني بشكل غير كافي

e) Washing too much. ⬇️ dec. الغسل بشكل زائد عن اللزوم

f) Incomplete drying of precipitate. ⬆️ inc . تجفيف الراسب بشكل غير كافي

حفظ g) Photodecomposition of silver chloride. ⬆️ inc. تتأثر بالضؤ وبتالي يزيد الوزن

☆ طيب completedrying التجفيف بشكل كافٍ ، الجواب رح يكون ... no effect لا تأثير 👍

Q2) What is digestion of a precipitate? Why is it necessary?

it's necessary to coagulation & heat for long time to increase particle size .

Q3) Dilute HNO_3 is used as a wash solvent. Explain?

1) prevent peptization التفكك
وهي عكس التخثر اي التكتل coagulation

2) Rebidly volitile يتطاير بسرعه
يتطاير بسرعه لكي لا يؤثر على وزن ال PPt

☆ التخثر بسرعه يمنع ال
Digestion = Coagulation زيادة التكتل

EXPERIMENT (4) :
نفس تجربة 3 لكن الفرق في المولات

$$Ni^{+2} + \boxed{HDMg} \rightarrow Ni(DMg)_2$$

anon (unknown nickel salt solution)   ppt. agent excess

- لكي نضمن ذوبان ال Ni+2 كامل نضيف معه HCL & D.W
- ال DMG ما بيرسب ال Ni+2 , إلا في حاله واحده إذا كان المحلول slight basic (بسيط بعض الشيء) لذلك بجيب أمونيا NH3 وبضيفها .

- بعد ان يترسب ال Ni(DMG) اغسل المحلول بالماء والإثانول , لماذا ؟

لكي اتخلص من ال DMG لانه Excess .
لكي لا يحدث coprecipitation (تكتل او تخثر) , وال DMG (Bulk) كبير يقوم بعمل ال coprecipitation ومعناها إلتصاق بالأيون .

DMG : Dimethylglyoxime

مهم جدًا جدًا

* DMG is specific for Ni^{+2} in basic media .
* DMG is specific for pd^{+2} in acid media .

- الراسب Ni (DMG)2 :

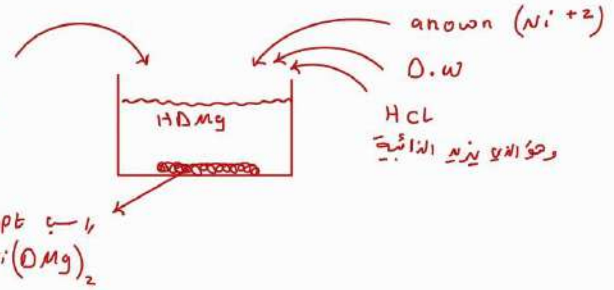
1. bulky ppt
2. tends to creep up wavs of yhe beaker contaning it
3. has a bright strawberry color

☆ ال Ni^{+2} عكس ال pd^{+2}

Exp 3 --> HNO3 to prevent peptization
Exp 4 --> HCL add on Ni^{+2} + D.W عشان أضمن تذويبه بالكامل

ملخص التفاعل

أمونيا NH3 سبب الإضافة لأنه DMG مابيرسب ال Ni لحاله يحتاج إلى وسط قاعدي خفيف .



وهو الذي يزيد الذائبية

تطبيق : (من ترياق وبناسيا)

Q3 : In a gravimetric determination of nickel , 10 mL was taken from an unknown sample contains Ni^{+2} (58.7 g / moL) . the mass of the empty crucible was 30.107 g and the mass of the crucible with the Ni(DMG)2 (288.7 g / moL) was 30.3 g
Find the concentration of Ni^{+2} in mg / L ??

Answer : 3.9×10^{-3} mg / L

السؤال قوي جدًا

$V_{anoun}(Ni^{+2}) = 10 \text{ mL}$

$M.m\,(Ni^{+2}) = 58.7 \text{ g/moL}$

فاضي $m_{crucible} = 30.107 \text{ g}$

$m\begin{pmatrix}\text{crucible} + \underset{\text{ppt راسب}}{Ni(DMg)_2}\end{pmatrix} = 30.3 \text{ g}$

$M.m_{ppt} = 288.7 \text{ g/mol}$

$M_{Ni+2} = ?! \text{ mg/L}$

① $m_{ppt} = 30.3 - 30.107$
$= 0.193 \text{ g}$

② $\overset{!?}{m}_{Ni+2} = \dfrac{m_{ppt} * M.m_{Ni+2}}{M.m_{ppt}}$

$= \dfrac{0.193 \text{ g} * 58.7 \text{ g/moL}}{288.7 \text{ g/moL}}$

$= 0.03924 \text{ g}$

③ $\dfrac{0.03924 \cancel{g}}{10 \cancel{mL}} * \dfrac{1000 \text{ mg}}{1 \cancel{g}} * \dfrac{1000 \cancel{mL}}{1 L} = 3.924 * 10^{3} \text{ mg/L}$

ترياق مقربين الـ 3.924 لـ 3.9 g/L

SD $3.9 * 10^{-3} = 3900 \text{ mg/L}$
مهم التقريب ، يمكن يكون هيك الخيار

Q4 : A 10 g sample of soil contain MgCL2 (95.3 g / moL) was dissolved in 500 mL D.W , A 25 mL aliquote was taken & titrated with AgNO3 , wich produces 0.0411 g silver chloride (143.5 g / moL) .
Find the concentration of MgCL2 in the soil sample .

Answer : 2.7 %

$m_{sample} = 10\ g$ تحتوي على $MgCl_2$ → أذيب في 500 mL D.W

$M.m_{MgCl_2} = 95.3\ g/mol$

أخذنا منه

ونتج التالي

(وتايرناه مع ال $AgNO_3$) $V = 25\ mL$

$m_{AgCl} = 0.0411\ g$

$M.m_{AgCl} = 143.5\ g/mol$

$M_{MgCl_2} = ?!$

$$\boxed{1\ MgCl_2 + 2\ AgNO_3 \longrightarrow \underset{\text{راسب ppt}}{2\ AgCl}}$$

① $n_{AgCl} = \frac{m}{M.m} = \frac{0.0411\ g}{143.5\ g/mol} = 2.8641 * 10^{-4}\ mol$

② 1 mol $MgCl_2$ ⟶ 2 mol AgCl

?! mol $MgCl_2$ ⟶ $2.841 * 10^{-4}$ mol AgCl

$n_{AgNO_3} = \frac{2.841 * 10^{-4}}{2} = 1.432 * 10^{-3}\ mol$

③ $m_{MgCl_2} = ?! = n * M.m = 1.432 * 10^{-4} * 95.3 = 0.01364\ g$

④ $\overset{?!}{[\text{التركيز}]}_{\substack{MgCl_2 \\ \text{in sample}}} = \frac{[MgCl_2]\ g/L}{[sample]\ g/L} * 100\ \% = \frac{0.5456}{20} * 100\% = 2.728\%$

$\frac{10\ g}{500\ mL * 10^{-3}\ L} = 20\ g/L$

$\frac{?\ g}{25\ mL * 10^{-3}\ L} = \frac{0.01364\ g}{25 * 10^{-3}\ L} = 0.5456\ g/L$

Q5 : A 1.8636 g sample contain Ni^{+2} (58.7 g / moL) was dissolved in 250 mL distilled water . A 50 mL aliquot was precipitated into Ni(DMG)2 (2.88.7 g / moL) .

The mass of the precipitated was 0.4016 g
Find the concentration of Ni^{+2} in the sample (%)

a) 22 %   b) 44 %   c) 74 %

Answer : A

$m_{sample} = 1.8636\ g$ Ni^{+2} تحتوي على
$M.m_{Ni^{+2}} = 58.7\ g/mol$
$\rightarrow$ إذابة ب D.W
$V = 25\ mL$

$V_{ppt} = 50\ mL = V_{Ni^{+2}}$
راسب $Ni(DMg)_2$

$M.m_{ppt} = 288.7\ g/mol$
$m_{ppt} = 0.4016\ g$

$\%\ ?! = [\ ]\ Ni^{+2}$ in the sample احسب تركيز

دايما g/L أو mol/L

$$\frac{\overset{1.64\ g/L}{[Ni^{+2}\ \text{in the sample}]}}{[\text{sample}]} * 100\ \% = ?!$$

① $$m_{Ni^{+2}} = \frac{m_{ppt} * M.m_{Ni^{+2}}}{M.m_{ppt}} = \frac{0.4016\ g * 58.7\ g/mol}{2.887\ g/mol} = 0.08165\ g = 0.082\ g$$

② $$\frac{0.082\ g}{50\ \cancel{mL}} * \frac{1000\ \cancel{mL}}{1\ L} = 1.64\ g/L$$

③ $$\frac{1.8636\ g}{250\ \cancel{mL}} * \frac{1000\ \cancel{mL}}{1\ L} = 7.4544\ g/L$$

④ $$\frac{1.64}{7.4544} * 100\ \% = 22\ \%$$

II. EXPERIMENTML PROCEDURE:

Pipet 10 mL from the unknown nickel salt solution into a 400 mL beaker. Add about 5 mL of dilute HCl then dilute to about 200 mL with distilled water. Heat to 70-80 ^{0}C (use a thermometer).

Reduce the flame and add 30 mL of DMG. With good stirring, add dilute ammonia dropwise, until precipitation takes place and then add a slight excess. Heat very gently on a small flame and test the solution for complete precipitation when the red precipitate has settled out.

Allow the precipitate to stand for 20-30 minutes at room temperature. Clean up a gooch crucible and heat it to constant weight at 110-120 ^{0}C. Filter the cold solution through the crucible, wash the precipitate with warm water and then with 30% alcohol, to dissolve any excess DMG from the precipitate.

Finally, put the crucible in an oven at 110-120 ^{o}C for 30 minutes. Allow to cool in a desiccator and weigh. Repeat drying until constant weight is achieved.

EXPERIMENT 4

GRAVIMETRIC DETERMINATION OF NICKEL AS BIS(DIMETHYLGLOXIMATO)-NICKEL(II) NI(DMG)$_2$

Name:	**Group NO.:**

III DATA

① Volume of unknown solution used, mL	10.00 mL ± 0.05
② Mass of empty crucible, g	29.001 g ± 0.001
③ Mass of crucible + Ni(DMG)$_2$, g	29.661 g ± 0.001
④ Mass of Ni(DMG)$_2$ precipitate, g	0.660 g ± 0.001

$= ③ - ② = 29.661 - 29.001 = 0.660$

IV CALCULATION

1- Mass of Ni in the volume used, g = $\overset{④}{\text{mass of Ni(DMG)}_2} * \frac{\text{M.mass of Ni}}{\text{M.mass of Ni(DMG)}_2}$ → ⑤

$= 0.66\ g * \frac{58.69\ g/mol}{288.91\ g/mol} = 0.1341\ g$

2- Mass of Ni in 1L (g/L) = $\frac{0.1341\ g}{10\ \cancel{mL}} * \frac{1000\ \cancel{mL}}{1L} = 13.41\ g/L$

$⑥ = \frac{⑤}{①}$

$M.m_{Ni} = 58.69\ g/mol$

$M.m_{Ni(DMG)_2} = 288.91\ g/mol$

V QUESTIONS

لماذا يتم الترسيب في محلول الأمونيا NH_3

Q1) Why precipitation is carried out in an ammonia solution?

1) DMG is specific for Ni $^{+2}$ in weakly Basic solution

2) DMG is specific for Pd $^{+2}$ in acid Medium

Q2) Why a large excess of DMG should not be added?

Because Ni(DMg)2 is bulky PPt when first precipitate .

30%

Q3) Why ethanol is used for washing?

To get red excess HDMG using rubber policeman to remove any solid particles hering to the beaker .

اسألة إضافية بناسيا 📌

تجربة 2

1) A student found the standard deviation for molarities of a homogenous sample to be 0.04, and the standard deviation for molarities of heterogeneous sample to be 0.09. Find the standard deviation of sampling.

Answer: 0.081

تجربة 1

2) Order in increasing accuracy of volumetric measurement tools:

a) burret, beaker, graduated cylinder, pipette

b) graduated cylinder, beaker, pipette, burret

c) beaker, graduated cylinder, pipette, burret

d) beaker, graduated cylinder, burret, pipette

Answer: D

تجربة 4
قوية 🔥

3) In a gravimetric determination of nickel, a 10 ml was taken from an unknown sample contains Ni^{+2} (58.7 g/mol). the mass of the empty crucible was 30.107 g, and the mass of the crucible with the $Ni(DMG)_2$ (288.7 g/mol) was 30.3 g. Find the concentration of Ni^{+2} in mg/L?

Answer: $3.9*10^{+3}$ mg/L

تجربة 4
قوية 🔥

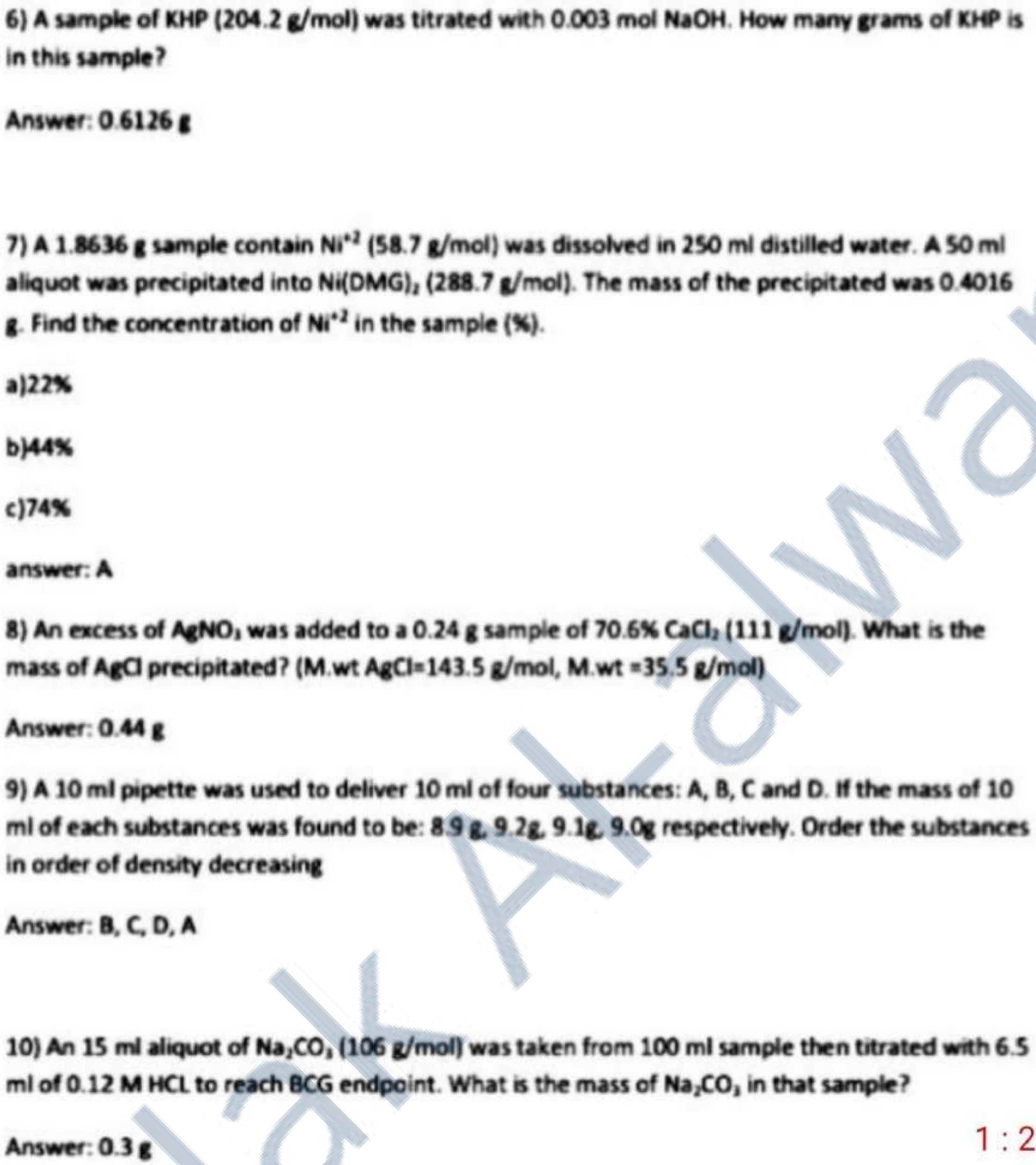
4) A 10 g sample of soil contain $MgCl_2$ (95.3 g/mol) was dissolved in 500 ml distilled water. A 25 ml aliquote was taken and titrated with $AgNO_3$,which produces 0.0411 g silver chloride (143.5 g/mol). Find the concentration of $MgCl_2$ in the soil sample.

Answer: 2.7%

تجربة 5

5) 10 ml of phosphoric acid (98 g/mol) needed 32 ml of 0.1 M NaOH to reach bromo cresol green end point .Find the concentration of H_3PO_4 (g/L)

Answer : 31.36 g/L



تجربة 5

6) A sample of KHP (204.2 g/mol) was titrated with 0.003 mol NaOH. How many grams of KHP is in this sample?

Answer: 0.6126 g

تجربة 4
قوية 🔥

7) A 1.8636 g sample contain Ni^{+2} (58.7 g/mol) was dissolved in 250 ml distilled water. A 50 ml aliquot was precipitated into Ni(DMG)$_2$ (288.7 g/mol). The mass of the precipitated was 0.4016 g. Find the concentration of Ni^{+2} in the sample (%).

a)22%

b)44%

c)74%

answer: A

تجربة 3

8) An excess of AgNO$_3$ was added to a 0.24 g sample of 70.6% CaCl$_2$ (111 g/mol). What is the mass of AgCl precipitated? (M.wt AgCl=143.5 g/mol, M.wt =35.5 g/mol)

Answer: 0.44 g

تجربة 1

9) A 10 ml pipette was used to deliver 10 ml of four substances: A, B, C and D. If the mass of 10 ml of each substances was found to be: 8.9 g, 9.2g, 9.1g, 9.0g respectively. Order the substances in order of density decreasing

Answer: B, C, D, A

تجربة 5

10) An 15 ml aliquot of Na$_2$CO$_3$ (106 g/mol) was taken from 100 ml sample then titrated with 6.5 ml of 0.12 M HCL to reach BCG endpoint. What is the mass of Na$_2$CO$_3$ in that sample?

Answer: 0.3 g

النسبة 2 : 1

EXPERIMENT (5) : Neutralisation titration in aqueous medium

(المانيوال)

→ two indicator (كواشف) :

1. bromocresol green (BCG) وسط حمضي
2. phenol phthalein (Ph.Ph) وسط قاعدي

→ in this experiment a solution of about 0.1M HCL is going to be standardized against a primary standard solution of sodium carbonate (NaCo3) .

→ the standardized HCL solution will then be used for standardized of about 0.1M NaOH solution .

→ the standardized NaOH will be used for determination of phospheric acid (H_3PO_4) in commercial acid and for determination of the equivalent weight of an unkown or ganic acid .

A) Standarization of sodium hydroxide (NaOH) .

خطوات التجربة :

Na_2CO_3 مو

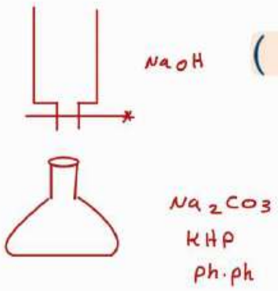
- وزن من ال potassium hydrogen phthalate (KHP)(primary standard) بحدود ال 1.5g
- ذوبه بال D.W وصفيه في ال volumetric flask بحدود ال 100ml

- احسب ال MKHP
- بإستخدام ال Pipete 10ml اسحب من ال volumetric flask 10ml وضعه في ال Erlenmeyer flask

- ضع فوقه 3 قطرات من ال phenolphalein indicator (Ph.Ph) وضع 20ml من ال D.W
- اعمل لهُ مُعايره من الNaOH solution

(unit the solution changes from colorless to pink)

- احسب التركيز لل NaOH



B) Determination of phosphoric acid in commercial acid .

- phospheric acid H3PO4 : H_3PO_4
  is a tribasic acid having three replicable hydrogen atoms.

- Neutralization of this acid leads to the production of the :

  1. dihydrogen phosphate
  2. the monohydrogen phosphate
  3. the tribasic phosphate

- the PH at the equivalence point at each stage is 4.6 , 9.7 and 12.6 respectively

- thuse in the titration of this acid an appropriate indicator with atransition PH range

- which matches PH of the stage at the equivalenice point should be selected

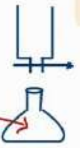
- no satisfactory indicator is known for the third stage

لون أزرق BCG + لونه شفاف H_3PO_4 ⟶ يطلع لون أصفر + NaOH(titrant)

⟵ ٢, يطلع شندي لون أزرق مش أخضر

خطوات التجربة :- ① NaOH 0.1M 10 mL

٦ ثم أحسب M H_3PO_4 .



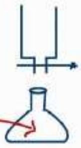
3, 5 قطرات من methyl orage
or bromocresol green indicator (BCG)

10 mL من الدورق الذي يستخدم في التجربة الثانية من H_3PO_4
unkown phosphoric acid solution

لونه بالأصل أزرق blue وفي الوسط الحمضي يتغير لونه لحد pH = 4

② NaOH

٦ ثم احسب M H_3PO_4 .



يحتوي على الـ H_3PO_4
uknown phosphoric acid solution

3, 5 قطرات من phenolphthalein indicator (ph.ph)

لونه شفاف تقريبًا (colorless)
في الوسط القاعدي يتغير لونه لحد pH = 9

اللون ٢, يطلع (pink)

C) Determination of the equivalent weight of an unknown organic acid . (theoretical)

- equivalent weight of a weak acid is useful for identification puppose .
- it can be determined by titration with astrong base using an indicator that changes color frome (**PH = 8** to **PH = 10**) .

- it is necessary to carry out preliminary solubility tests .

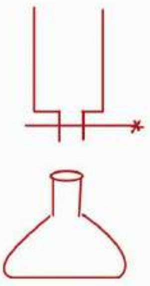
خطوات التجربة : (1) نوزن من العينة الحمض المجهول 0.3g
ضعهم في Beaker 200mL
وذوبهم في D.W 50mL

- in some cases heating maybe required to dissolve the sample .

- also sometimes it is necessary to dissolve the sample in an alcohol water mixture .

(2) ضعهم في ال volumetric flask ، ثم خففه بال D.W
لحد 100mL ثم أخلطه جيدًا ... نوخذ منه 10mL بال pipete

(3)



3-4 drops من ال
phenolphalein indicator

ثم احسب تركيز ال
equivalent weight of
the unknown acid.

(EXP.5 بناسيا)
ملخص مهم جداً

عايرت ال anon (الماده المتفاعله) ← H3PO4
titrant ← NaOH (2˙standard)
حسب الكاشف (indicator) .

H3PO4
(titrant NaOH is 2˙standard)

Ph.Ph indicator
2:1 ratio

BCG indicator
1:1 ratio

شرح $1\,H_3PO_4 + 2\,NaOH \xrightarrow{ph.ph} HPO_4^{2-} + ----$

$1\,H_3PO_4 + 1\,NaOH \xrightarrow{BCG} H_2PO_4^{-} + ----$

المحاليل نوعين : (١ 1° standard
ومعنا ما إنه إذا حضرت محلول تركيزه يبقى ثابت مع الزمن

(٢ 2° standard
تحتاج معايرة مرة آخرى

عايرت ال HCL الذي يعتبر (2˙ standard) بإستخدام مادة أولية (2˙standard)
وهي ال Na2CO3 .

Na2CO3 (1˙standard)
(HCL is 2˙standard)

Ph.Ph indicator
1:1 ratio

BCG indicator
2:1 ratio

شرح $1\,HCl + 1\,Na_2CO_3 \xrightarrow{ph.ph} NaCl + NaHCO_3$

$2\,HCl + 1\,Na_2CO_3 \xrightarrow{BCG} 2\,NaCl + \underbrace{H_2CO_3}_{CO_2 + H_2O}$

BCG : blue (Basc)
Yellow (acid)
PH = 3.8 - 5.4

تحضيره : 1) نوزن $NaCO_3$
2) تذوب المادة بال D.W
3) نسحب 10mL من ال solu الذي حضرناه ، ثم نقوم بال titration مع ال HCL

الهدف : معرفة تركيز ال HCL بواسطة ال Na_2CO_3

((ملخص لل 2° & 1° الميد والفاينل))

1° standard

KHP , $NaCO_3$ & $K_2Cr_2O_7$ (EX.7).

2° standard

HCL , KSCN (EX.6) , NaOH , EDTE (EX.9) , $NaSO_3$, Ag^+ (EX.6) ,
$S_2O_3^{-2}$ & I_2 (EX.8) .

عايرت ال HCL الذي يعتبر (2° standard)
إستخدام مادة ثانوية وهي (2° standard) NaOH .

الهدف أعاير NaOH وافاعله مع مادة أخرى لكي احسب تركيزه .

$$1\,HCL + 1\,NaOH \longrightarrow NaCL + H_2O$$

ratio 1 : 1
PH = 8.3 - 10

هنا لا فرق لو إستخدمنا ال indicator سواء ال ph.ph or BCG
لانه العلاقة للتنتين نفس الإشي 1 : 1

مهم جدًا جدًا

acid حمض	Base قاعدة	
Color less بدون لون	pink وردي	ph.ph لونه بالأصل شفاف بلا لون
Yellow أصفر	Blue أزرق	BCG لونه بالأصل أزرق

ملخصات سريعة تساعد على فهم النسب

(1) $M\,HCL \times V\,HCL = M\,Na_2CO_3 \times V\,Na_2CO_3$ in precense of ph.ph

(2) $\frac{M\,HCL \times V\,HCL}{2} = M\,Na_2CO_3 \times V\,Na_2CO_3$ in precense of BCG

(3) $M\,HCL \times V\,HCL = M\,Na_2CO_3 \times V\,Na_2CO_3$ ratio 1:1 , BCG or ph.ph

(4) $M\,NaOH \times V\,NaOH = M\,H_3PO_4$ BCG

(5) $\frac{M\,NaOH \times V\,NaOH}{2} = M\,H_3PO_4 \times V\,H_3PO_4$ ph.ph

BCG in acidic media
ph.ph in bacic media

لتسهيل الحفظ، لأن الBCG حمض فإنه مع HCL بنسبة 1 : 2

$$\frac{M_{HCL} \times V_{HCL}}{2}$$

ولأن ال ph.ph قاعدي فإنه مع NaOH بنسبة 1 : 2

$$\frac{M_{NaOH} \times V_{NaOH}}{2}$$

عايرت ال NaOH (2°standard) بإستخدام مادة أولية (standard 1°)
وهي KHP = KHC8H4O4

KHP (KHC8H4O4)
(NaOH)

Ph.Ph indicator
1: 1 ratio

BCG indicator

شرح

$$1KHp + 1NaOH \xrightarrow[1:1]{ph.ph} NaKp + H_2O$$

$$n_{KHp} = n_{NaOH}$$

$$M_{KHp} * V_{KHp} = M_{NaOH} * V_{NaOH}$$

شرح بالتوضيح والتفصيل لأول شغلة أخذناها بالتجربة

Determination of H3PO4 concentration

1) using BCG :

$$1H_3PO_4 + 1NaOH \xrightarrow{BCG} H_2PO_4^- + \text{------}$$

ratio 1:1

pH = (2-4) BCG

end point $n_{NaOH} = n_{H_3PO_4}$

$$M_{NaOH} * V_{NaOH} = M_{H_3PO_4} * V_{H_3PO_4}$$

2) using Ph.Ph : $1H_3PO_4 + 2NaOH \xrightarrow{ph.ph} HPO_4^{2-} + \text{-----}$

ratio 1 : 2

pH = (9-11) ph.ph

end point $\frac{n_{NaOH}}{2} = n_{H_3PO_4}$

$$\frac{M_{NaOH} * V_{NaOH}}{2} = M_{H_3PO_4} * V_{H_3PO_4}$$

Neutralization titration in aqueos medium :

HCL & NaOH are not standard solution

$\boxed{M = n / V}$

$H_3PO_4 + 2\,NaOH \xrightarrow{ph.ph} Na_2HPO_4 + 2H_2O$

$H_3PO_4 + 1\,NaOH \xrightarrow{BCG} H_2O + NaH_2PO_4$

مهم جدًا جدًا

$H_3PO_4 \longrightarrow H^+$ نزع $+ H_2PO_4^-$ PH = 4.6

$H_2PO_4^- \longrightarrow H^+ + HPO_4^{-2}$ PH = 9.7

$HPO_4^{-2} \longrightarrow H^+ + PO_4^{-3}$ PH = 12 .6

(تطبيق ع دوسية بناسيا وترياق)

Q1 : 10 ml of phosphoric acid (98 g\mol) needed 32ml of 0.1M NaOH to reach bromo cresol green end point . find the concentration of H3PO4 (g\L) .

answer : 31.36 g\L

$V_{H_3PO_4} = 10\ mL$ $V_{NaOH} = 32\ mL$

$M.m\ H_3PO_4 = 98\ g/mol$ $M_{NaOH} = 0.1\ M$

$M_{H_3PO_4} = ?!\ g/L$ $1 : 1$ النسبة

$$① \quad M_{H_3PO_4}^{?} = \frac{M_{NaOH} * V_{NaOH}}{V_{H_3PO_4}} = \frac{0.1\ mol/L * 32\ mL}{10\ mL}$$

$$= 0.32\ mol/L \text{ of } H_3PO_4$$

$$② \quad \frac{0.32\ \cancel{mol}}{1L} * \frac{98\ g}{1\ \cancel{mol}} = 31.36\ g/L$$

Q2 : A sample of KHP (204.2 g\mol) was titrated with 0.003 mol NaOH how many grams of KHP is in this sample ?

answer : 0.6126 g

$M.m_{KHp} = 204.2$ g / moL

$m_{KHp} = ?!$ g

$n_{NaoH} = 0.003$ moL

① $n_{NaoH} = n_{KHp} \longrightarrow 0.003\ mol = \frac{m_{KHp}}{M.m_{KHp}}$

$$0.003\ moL = \frac{\overset{?!}{m_{KHp}}}{204.2\ g/moL}$$

$$\overset{?!}{m_{KHp}} = 0.003\ moL * 204.2\ g/moL = 0.6126g$$

Q3 : An 15ml aliquot of NaOH (106 g\mol) was taken from 100 ml sample then titrated with 6.5 ml of 0.12 M HCL to reach BCG endpoint . what is the mass of Na2CO3 in that sample ?

answer : 0.3 g

$V_{NaCO_3} = 15$ mL

$M.m_{NaCO_3} = 106$ g / moL

$m_{NaCO_3} = ?!$ g

in the sample $\longrightarrow$ g / 100 mL

$V_{HcL} = 6.5$ mL

$M_{HcL} = 0.12$ Ⓜ $\swarrow$ moL / L

النسبة 2 : 1

① $\frac{n_{HcL}}{2} = n_{NaCO_3} \longrightarrow \frac{M_{HcL} * V_{HcL}}{2} = \overset{?!}{M_{NaCO_3}} * V_{NaCO_3}$

$$\overset{?!}{M_{NaCO_3}} = \frac{M_{HcL} * V_{HcL}}{2 * V_{NaCO_3}} = \frac{0.12\ moL/L * 6.5\ mL}{2 * 15\ mL}$$

$$= 0.026\ moL/L \text{ of } NaCO_3$$

② $\frac{0.026\ \cancel{\text{mol of NaCO3}}}{\cancel{1L}} * \frac{106\text{ g of NaCO3}}{1\text{ mol }\cancel{\text{of NaCO3}}} * \frac{\cancel{1L}}{1000\text{ mL}}$

$= \frac{0.026 * 106\ \boxed{\text{g}}}{10 * \boxed{100\text{ mL}}} = 0.275\ \text{g}/100\text{ mL}$

في السؤال طالب ←

$\approx 0.3\ \text{g}/100\text{ mL}$

Q4 : A 15ml aliqout was taken from 100ml solution of Na2CO3 (106 g\mol) and titrated with 0.12 M HCL . it required 9.5ml to reach the bromo cresol green end point . what is the mass of Na2CO3 in this 100ml solution .

جديد تراكة answer : 0.4028

$100\text{ mL sample} \xrightarrow{\text{أخذنا منه}} 15\text{ mL sample } Na_2CO_3 = V_{Na_2CO_3}$

$M.m_{Na_2CO_3} = 106\ \text{g/mol}$

$m_{Na_2CO_3} = ?! \longrightarrow \text{g}/100\text{ mL}$

(in the 100 mL solution)

$V_{HCl} = 9.5\text{ mL}$

$M_{HCl} = 0.12\text{ M}$

2 : 1 النسبة

① $\frac{n_{HCl}}{2} = n_{Na_2CO_3} \longrightarrow \frac{M_{HCl} * V_{HCl}}{n} = M^{?!}_{Na_2CO_3} * V_{Na_2CO_3}$

$= \frac{0.12\text{ mol/L} * 9.5\text{ mL}}{2 * 15\text{ mL}} = 0.038\ \text{mol/L}$

② $\frac{0.03\ \cancel{\text{mol}}}{\cancel{1L}} * \frac{106\text{ g}}{1\ \cancel{\text{mol}}} * \frac{\cancel{1L}}{1000\text{ mL}} = \frac{0.038 * 106\text{ g}}{10 * 100\text{ mL}}$

$= 0.4028\ \text{g}/100\text{ mL}$

II. EXPERIMENTAL PROCEDURE

a) Standardization of Sodium hydroxide

Weigh accurately about 1.50g of primary standard KHP in a weighing bottle. Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Adjust the volume to the mark and mix well, calculate the molarity of the KHP solution.

Pipet 10 mL of the KHP solution into an Erlenmeyer flask, add 3 drops of phenonaphthalene indicator and 20 mL distilled water then titrate with the NaOH solution until the solution changes from colorless to pink. Repeat three times and calculate the exact molarity of the NaOH solution.

b) Determination of Phosphoric acid in commercial acid

Phosphoric acid H_3PO_4 is a tribasic acid having three replicable hydrogen atoms. Neutralization of this acid leads to the production of the dihydrogen phosphate, the monohydrogen phosphate and the tribasic phosphate. The pH at the equivalence point at each stage is 4.6, 9.7 and 12.6 respectively. Thus in the titration of this acid an appropriate indicator with a transition pH range which matches the pH of the stage at the equivalence point should be selected. No satisfactory indicator is known for the third stage.

1- Titrate 10.0 mL from the provided phosphoric acid with the 0.1M NaOH solution using 3-5 drops of methyl orange or bromocresol green as indicator. Repeat 2 times. Calculate the concentration of H_3PO_4 in the unknown phosphoric acid solution in grams per liter.

2- Titrate another 10.0 mL from the provided phosphoric acid with the 0.1 M NaOH using 3-5 drops of phenolphthalein indicator. Repeat two times. Calculate the concentration of H_3PO_4 in the unknown phosphoric acid solution in grams per liter.

c) Determination of the equivalent weight of an unknown organic acid. **(theoretical)**

Equivalent weight of a weak acid is useful for identification purposes. It can be determined by titration with a strong base using an indicator that changes color from pH 8 to pH 10. It is necessary to carry out preliminary solubility tests.

1- Weigh accurately about 0.3 g sample of the unknown acid. Transfer the sample into 200 mL beaker and dissolve it in about 50 mL of distilled water. In some cases heating may be required to dissolve the sample. Also sometimes it is necessary to dissolve the sample in an alcohol water mixture. Transfer the solution quantitatively into a 100 mL volumetric flask and dilute to the mark. Mix well.

2- Titrate 10 mL of the above solution with the provided 0.1M NaOH solution using 3-4 drops of phenolphthalein indicator. Repeat three times. Calculate the equivalent weight of the unknown acid.

EXPERIMENT 5

NEUTRALISATION TITRATION IN AQUEOUS MEDIUM

Name:	**Group NO.:**

III DATA

d) a) Standardization of Sodium hydroxide

Weight of KHP (g)…………………………

	Trial 1	Trial2	Trial 3
Volume of KHP mL			
Volume of NAOH, mL			

Average vol. of NaOH……………… Molar Conc. NaOH…………………

b) Determination of Phosphoric acid in commercial acid

	Volume of H_3PO_4	Volume of NaOH	Indicator
Trial 1	10.00 mL ± 0.05	6.00 mL ± 0.05	BCG
Trial 2	10.00 mL ± 0.05	6.00 mL ± 0.05	BCG
Trial 3	10.00 mL ± 0.05	12.00 mL ± 0.05	Phph
Trial 4	10.00 mL ± 0.05	11.50 mL ± 0.05	Phph

Molar conc. H_3PO_4 ..0.06 M Average vol. NaOH ..6.00 mL	BCG
Molar conc. H_3PO_4 0.0588 M Average vol. NaOH 11.75 mL	Phph

no. of unknown = 2

III. CALCULATIONS

$$n = \frac{m}{M.m}$$

$$M = \frac{n}{V}$$

a) .a Standardization of Sodium hydroxide

- The balanced equation for the reaction is:

$M_{NaOH} = 0.1$ mol/L

$M.m_{H_3PO_4} = 97.99$ g/mol

$V_{H_3PO_4} = 10$ mL

- Molarity of KHP(mol/L) =

- Molarity of NaOH (mol/L) =

b) (Determination of Phosphoric acid in commercial acid)

.b1 (Using BCG indicator)

- The balanced equation for the reaction is:

$$1\,NaOH + 1\,H_3PO_4 \xrightarrow{BCG} NaH_2PO_4 + H_2O$$

- Molar Conc. of H_3PO_4 (mol/L) =

$$\frac{M_{NaOH} * V_{NaOH}}{V_{H_3PO_4}} = \frac{0.1\ mol/L * 6\ \cancel{mL}}{10\ \cancel{mL}} = 0.06\ mol/L$$

- Conc. of H_3PO_4 (g/L) = $\frac{0.06\ \cancel{mol}}{1L} * \frac{97.99\ g}{1\ \cancel{mol}} = 5.879\ g/L$

b2 (Using Phph. indicator)

The balanced equation for the reaction is:

$$2\,NaOH + 1\,H_3PO_4 \xrightarrow{ph.ph} Na_2HPO_4 + 2\,H_2O$$

- Molar Conc. of H_3PO_4 (mol/L) = $\frac{1}{2} * \left(\frac{M_{NaOH} * V_{NaOH}}{V_{H_3PO_4}}\right)$

$$= \frac{1}{2} * \left(\frac{0.1\ mol/L * 11.75\ mL}{10\ mL}\right) = 0.0588\ mol/L$$

- Conc. of H_3PO_4 (g/L) =

$$\frac{0.0588\ \cancel{mol}}{1L} * \frac{97.99\ g}{1\ \cancel{mol}} = 5.762\ g/L$$

تمَّ تفريغ هذه التجربة حسب المانيوال الجديد و تمَّ جمع المعلومات من أكثر من مصدر لا تنسوا الإطلاع على المعلومات و التأكد من مروركم على جميع المعلومات و إن كان هنالك أي خطأ أرجو منكم أن تخبروا زملائكم و ادعوا لي في ظهر الغيب و جعلها الله صدقة جارية عني و عن جميع المسلمين🤍

مع تمنياتي للجميع بالتوفيق🤍

مسافرٌ أنت و الآثار باقية
فاترك وراءك ما تحي به أثرك ✨

Volumetric experiment

EXPERIMENT (6)

The Neutralizing Capacity of Antacid Tablets

مضاد للحموضة

Objectives

- Determine the amount of base in commercial antacids
- Determine the Neutralizing capacity of antacid
- Understand the Back titration

مكونها الأساسي مادة قاعدية

- **Antacids** are a products which neutralize stomach acidity and are used to relieve heartburn.

Antacids ingredients

Active ingredients

Strong base
رايح يحرق
أعضاءنا

Weak bases as
- ✓ $Mg(OH)_2$,
- ✓ $NaHCO_3$,
- ✓ $CaCO_3$
- ✓ $Al(OH)_3$

Other ingredients

- Flavorings
- Binders

لازم يكون في منكهات عشان
الواحد يقدر يستسيغ الحبة

حتى تكون قريبة من بعضها
البعض بحيث تكون على شكل
دائري أو كبسولة

- ✓ **Base** هي مادة حارقة
- ✓ في المعدة PH=2.5~3.5
- ✓ في المعدة لدينا حمض ال HCL
حتى يذوب الطعام و يسهل
للخلايا أن تمتصها

Neutralizing

حمض + قاعدة ⟵ ملح + ماء

يعمل على تهدئة المعدة

- **Neutralization reaction** is the reaction of acid and base
- The reaction between metal hydroxide and the stomach acid is an acid base reaction

ماء + ملح

$Mg(OH)_2 + 2\,HCl \longrightarrow MgCl_2 + 2\,H_2O$

$Al(OH)_3 + 3\,HCl \longrightarrow AlCl_3 + 3\,H_2O$

$CaCO_3 + 2\,HCl \longrightarrow CaCl_2 + H_2O + CO_2$

القاعدة: Weak base الموجود في antiacid

الحمض : الموجود في المعدة

تصاعد غاز ثاني أكسيد الكربون

كل المركبات القاعدية تفاعلها ملح +ماء إلّا

قاعدة :

إذا كان عندي كربونات CO_3^{-2} و ليس هيدروكسيد OH هون رايح يصير عندي تصاعد لغاز ثاني أكسيد الكربون و هذا رايحة ألاحظ إنه بصير عندي تجشؤ

● كربونات
● هيدروكسيد

Compound	Chemical Formula	Chemical Reaction
Aluminum hydroxide	$Al(OH)_3$	$Al(OH)_{3(s)} + 3HCl_{(aq)} \rightarrow AlCl_{3(aq)} + 3H_2O_{(l)}$
Calcium carbonate	$CaCO_3$	$CaCO_{3(s)} + 2HCl_{(aq)} \rightarrow CaCl_{2(aq)} + H_2O_{(l)} + CO_{2(g)}$
Magnesium carbonate	$MgCO_3$	$MgCO_{3(s)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + H_2O_{(l)} + CO_{2(g)}$
Magnesium hydroxide	$Mg(OH)_2$	$Mg(OH)_{2(s)} + 2HCl_{(aq)} \rightarrow MgCl_{2(aq)} + 2H_2O_{(l)}$
Sodium bicarbonate	$NaHCO_3$	$NaHCO_{3(aq)} + HCl_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)} + CO_{2(g)}$

● ماء
● الملح
● ثاني أكسيد الكربون

Neutralizing Capacity of Antiacid:
Is the amount of HCl that one gram tablet can neutralize

In this experiment, you will determine the neutralizing capacity of antacid

Determined by back titration

$$neutralizing\ capacity\ of\ ant\ acid = \frac{moles\ of\ HCl\ neutralized}{mass\ of\ antacid}$$

Determined by balance

كل ما كان هاد الرقم كبير كل ما كان قدرة الحبة /gram على معادلة كمية كبيرة من HCl و هاد مُفضل

NOTES

معظم هذه الحبة مواد غير ذائبة في الماء عشان هيك أنا
بذوبها بـ HCl محلول معروف التركيز و الحجم و عدد المولات

- **Most active ingredients in the antacid tablet are <u>water insoluble</u> Therefor direct dissolution in water is not effective**

- **Active ingredients would react and dissolve in <u>HCl solution</u>**

Therefore, Method of $\boxed{\text{back}}$ titrations is used to do the experiment
Which involves:

الكميّة الباقية من HCl التي أضفتها لإذابة ال Antiacid

☆ نويت 0.1g من antiacid
في كمية كبيرة من HCl ليس فقط كمية تكافئها
بل أكثر من حاجتها

- **Dissolving <u>known amount of antacid in known $\boxed{\text{excess}}$ amount of HCl</u>**
- **Determine the excess of HCl by titration with standard NaOH**

✕ و الجزء الباقي من ال HCl هو الي رايحة أعمله Titration مع NaOH   عشان هيك اسمه Back Titration

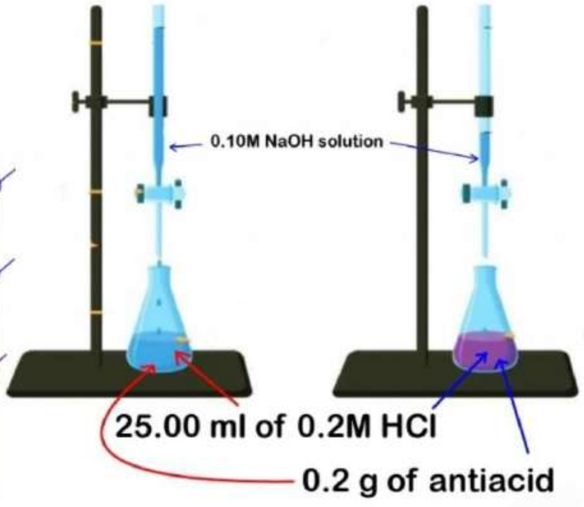
---

Experiment:

- In a titration flask place accurately weighted antacid sample (~0.20 g)
- Add 25.00 mL of 0.200M HCl solution → To react with the bases in the antacid
- Boil for few seconds → To get rid of CO2
- Let cool, add indicator and titrate to the end point with 0.100M NaOH solution

عدد المولات = تركيز المحلول *حجمه(باللتر)
n=M*V
0.2*0.025 = $\boxed{\text{0.005 moles}}$
بعض هذه المولات سوف يتفاعل مع
0.2 g of antiacid
و الباقي من هاي المولات رايحة أعمل إلهم
back titration مع NaOH

From colourless to pink



25.00 ml of 0.2M HCl

0.2 g of antiacid

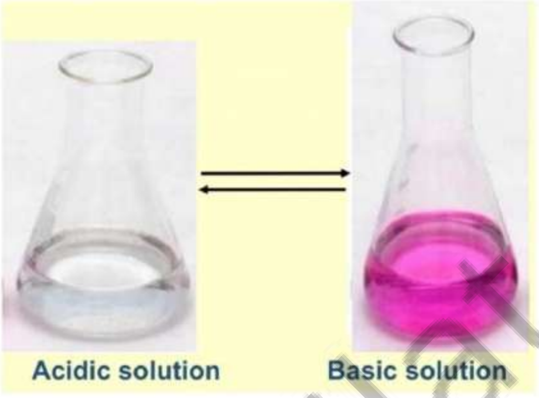
40ml amount of HCl needed to dissolve antiacid

أمّا الي تفاعل مع antiacid فهو مجهول

✓ في الحمض Ph.ph :-
Colourless
✓ في القاعدة Ph.ph :-
زهري
✓ الحبة لازم نطحنها عشان لو ما طحناها HCl يتفاعل فقط مع السطح

Phenolphthalein indicator

- The color changes from colorless to pink
- An acid base indicator



سؤال :

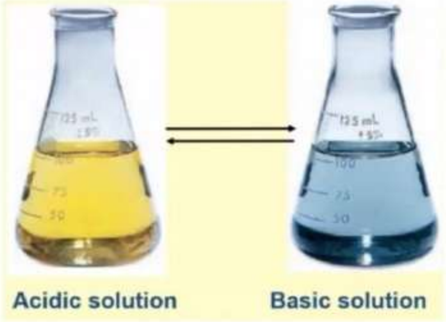
لمّا عملت **titration** بين **NaOH** و ال **HCl** هل أنا فاعلت **NaOH** مع **HCl**
الي ذوب **Antiacid** و لا الباقي من **HCl** الي ضفته ل **Antiacid** ؟

طبعًا الباقي من **HCl** الي ضفته ل **Antiacid**

moles of NaOH = moles of HCl

Bromothymol blue

- **An acid base indicator**
- **the colour of Bromothymol blue changes from Yellow to Blue**



Calculations:

The total amount of HCl is neutralized by two ways

- **Reacting with the antacid**
- **Reacting with NaOH**

moles of acid = moles of base in the antacid + moles of NaOH

moles of base in the antacid = moles of acid – moles of NaOH

moles of base in the antacid = $MHCl \ast V\,HCl - MNaOH \ast V\,NaOH$

الحجم الي أضفته *تركيز الأسيد

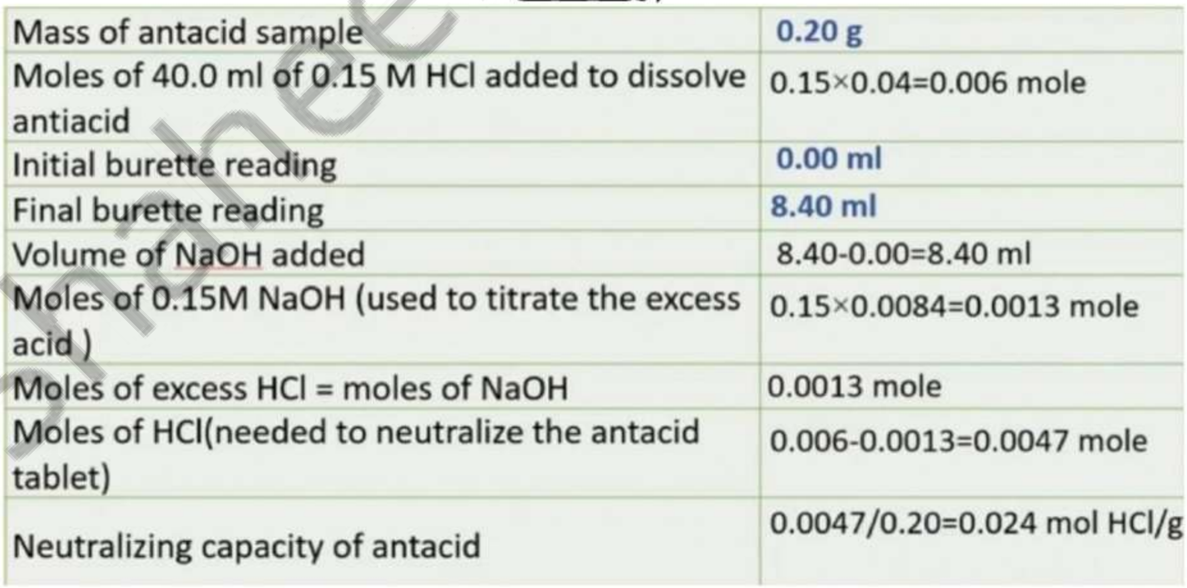
Moles of acid added = moles of acid neutralised with antiacid + moles of acid reacted with NaOH

نفسها عدد مولات NaOH الي نزلت من burette

Neutralizing capacity of antacid = $\dfrac{moles\ of\ base\ in\ antacid}{mass\ of\ antacid}$

أمثلة

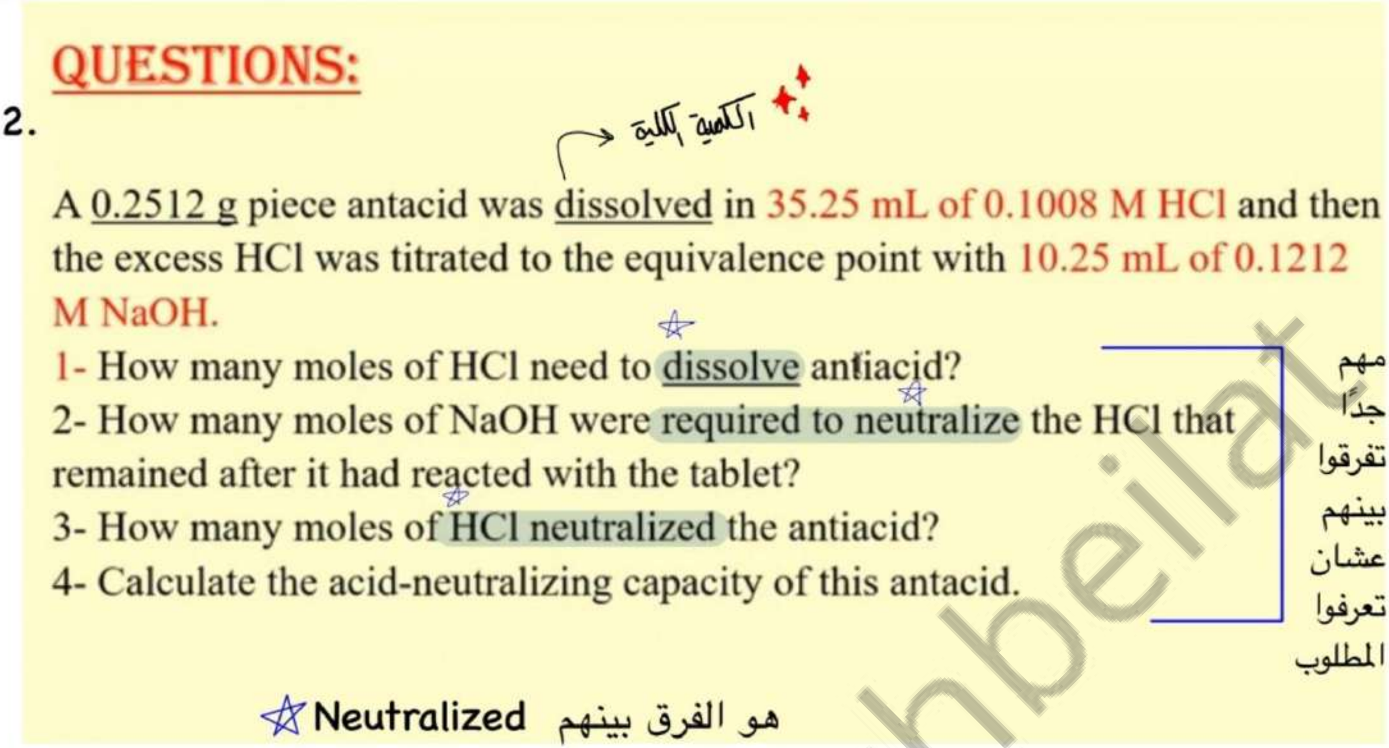
1.



Mass of antacid sample	**0.20 g**
Moles of 40.0 ml of 0.15 M HCl added to dissolve antiacid	0.15×0.04=0.006 mole
Initial burette reading	**0.00 ml**
Final burette reading	**8.40 ml**
Volume of NaOH added	8.40-0.00=8.40 ml
Moles of 0.15M NaOH (used to titrate the excess acid)	0.15×0.0084=0.0013 mole
Moles of excess HCl = moles of NaOH	0.0013 mole
Moles of HCl(needed to neutralize the antacid tablet)	0.006-0.0013=0.0047 mole
Neutralizing capacity of antacid	0.0047/0.20=0.024 mol HCl/g

40ml amount of HCl needed to dissolve antiacid

أمّا الي تفاعل مع antiacid فهو مجهول



QUESTIONS:

2.

الكمية الكلية

A 0.2512 g piece antacid was dissolved in 35.25 mL of 0.1008 M HCl and then the excess HCl was titrated to the equivalence point with 10.25 mL of 0.1212 M NaOH.

1- How many moles of HCl need to dissolve antiacid?

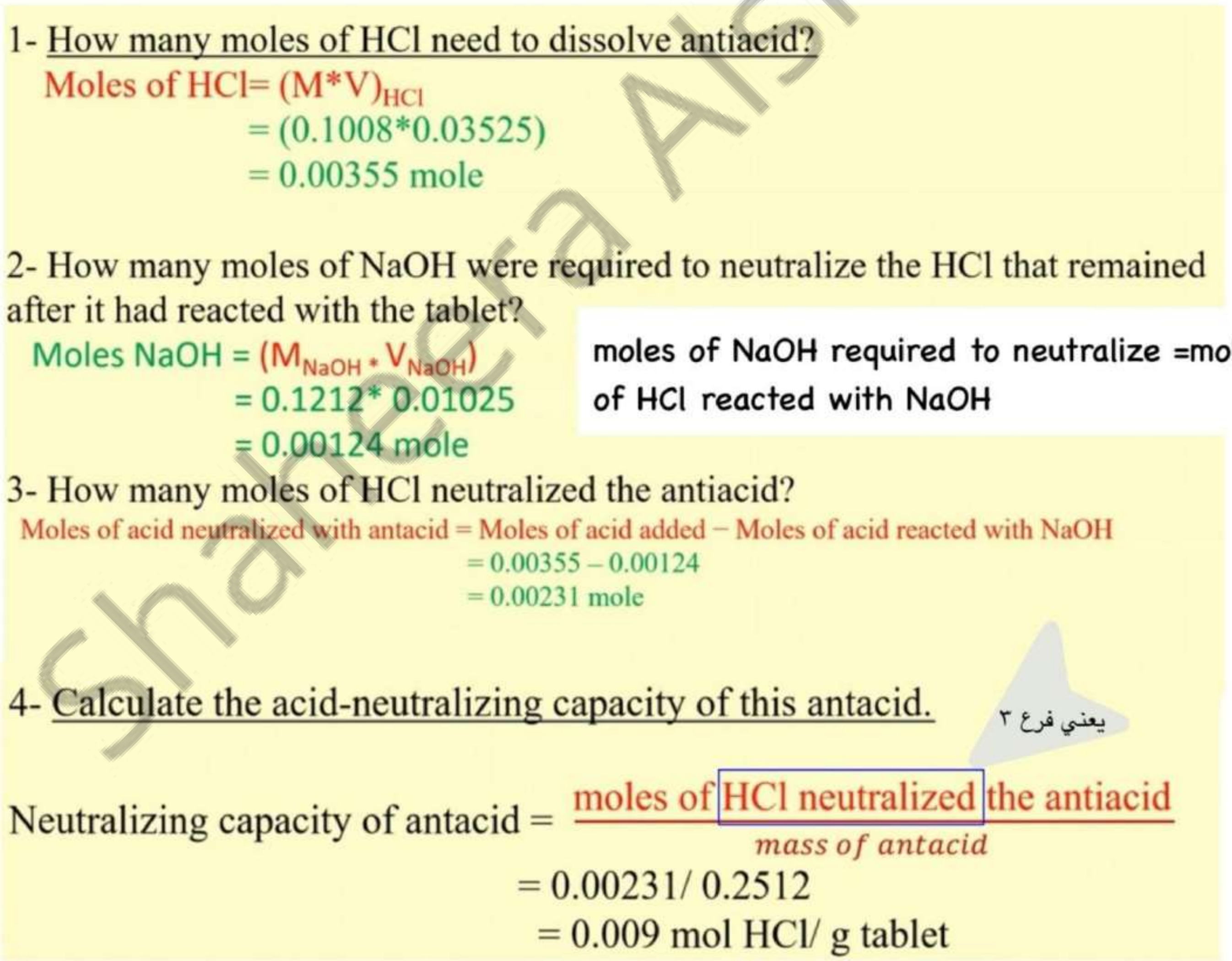
2- How many moles of NaOH were required to neutralize the HCl that remained after it had reacted with the tablet?

3- How many moles of HCl neutralized the antiacid?

4- Calculate the acid-neutralizing capacity of this antacid.

مهم جدًا تفرقوا بينهم عشان تعرفوا المطلوب

☆ **Neutralized** هو الفرق بينهم



1- How many moles of HCl need to dissolve antiacid?

$$\text{Moles of HCl} = (M*V)_{HCl}$$
$$= (0.1008*0.03525)$$
$$= 0.00355 \text{ mole}$$

2- How many moles of NaOH were required to neutralize the HCl that remained after it had reacted with the tablet?

$$\text{Moles NaOH} = (M_{NaOH} * V_{NaOH})$$
$$= 0.1212 * 0.01025$$
$$= 0.00124 \text{ mole}$$

moles of NaOH required to neutralize =moles of HCl reacted with NaOH

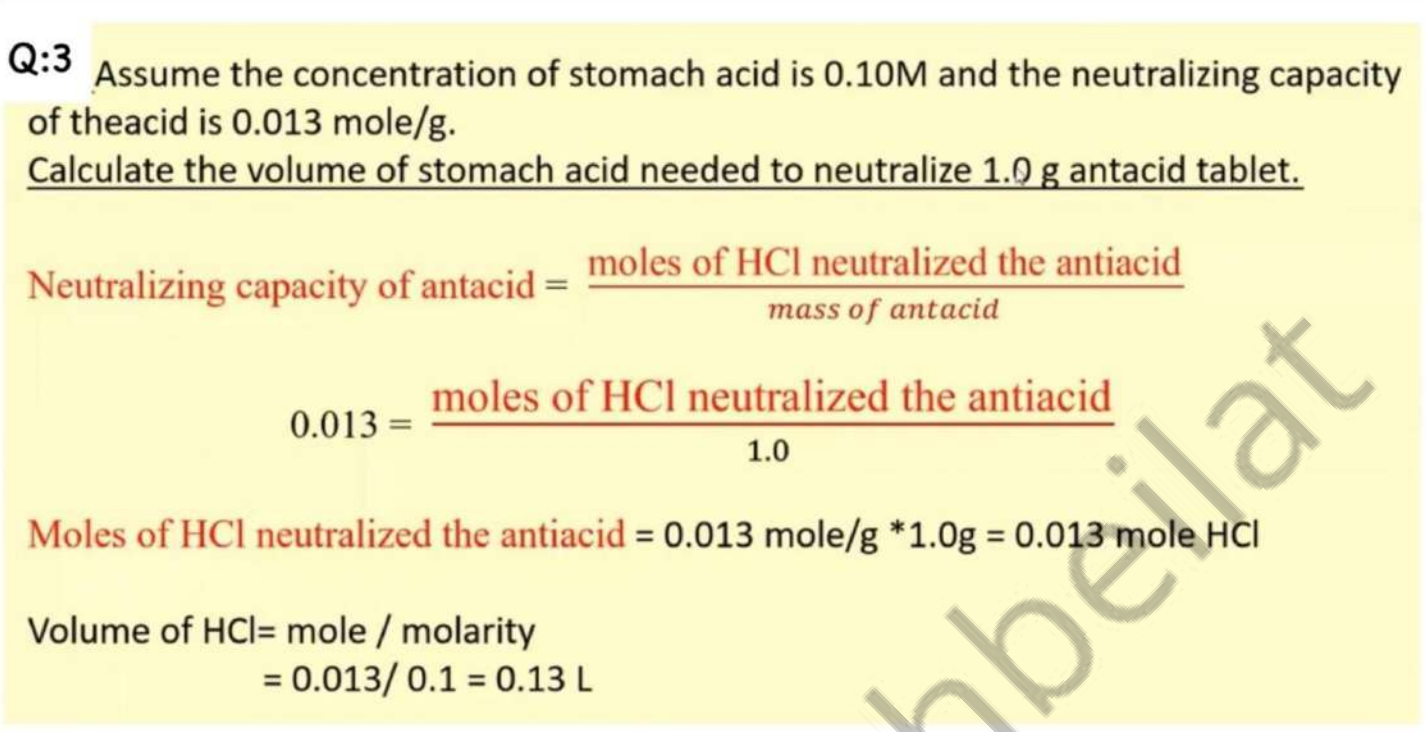
3- How many moles of HCl neutralized the antiacid?

$$\text{Moles of acid neutralized with antacid} = \text{Moles of acid added} - \text{Moles of acid reacted with NaOH}$$
$$= 0.00355 - 0.00124$$
$$= 0.00231 \text{ mole}$$

4- Calculate the acid-neutralizing capacity of this antacid.

يعني فرع ٣

$$\text{Neutralizing capacity of antacid} = \frac{\text{moles of } \boxed{\text{HCl neutralized}} \text{ the antiacid}}{\textit{mass of antacid}}$$
$$= 0.00231 / 0.2512$$
$$= 0.009 \text{ mol HCl/ g tablet}$$



Q:3 Assume the concentration of stomach acid is 0.10M and the neutralizing capacity of theacid is 0.013 mole/g.

Calculate the volume of stomach acid needed to neutralize 1.0 g antacid tablet.

$$\text{Neutralizing capacity of antacid} = \frac{\text{moles of HCl neutralized the antiacid}}{mass\ of\ antacid}$$

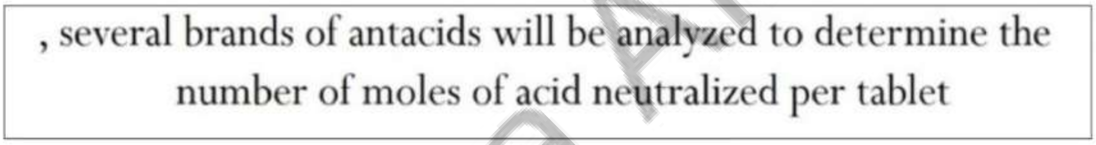
$$0.013 = \frac{\text{moles of HCl neutralized the antiacid}}{1.0}$$

Moles of HCl neutralized the antiacid = 0.013 mole/g *1.0g = 0.013 mole HCl

Volume of HCl= mole / molarity

= 0.013/ 0.1 = 0.13 L

سلايدات مس إيناس

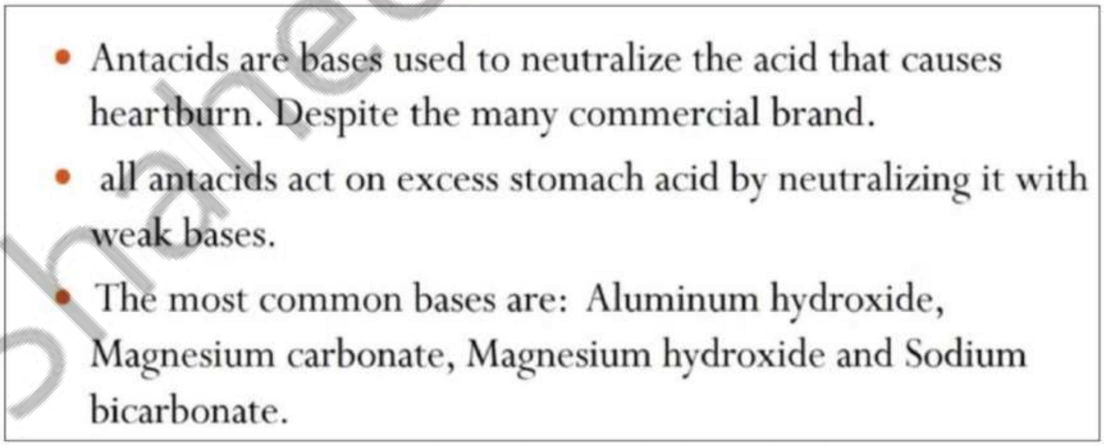


, several brands of antacids will be analyzed to determine the number of moles of acid neutralized per tablet

معادلة للحموضة الموجودة في المعدة Antiacid

انه يكون اشي قابل انه يدخل جسم الإنسان base وال base بالضرورة تكون Antiacid

فيها مادة فعالة بنسبة معينة plate كل



- Antacids are bases used to neutralize the acid that causes heartburn. Despite the many commercial brand.
- all antacids act on excess stomach acid by neutralizing it with weak bases.
- The most common bases are: Aluminum hydroxide, Magnesium carbonate, Magnesium hydroxide and Sodium bicarbonate.

بدي أحول الـ tablet إلى شكل solution بدي

أذوب القاعدة تبعتي و أحلل القاعدة الموجودة فيها

Experiment procedure

- Clean, rinse, and fill a buret with 0.100 M NaOH solution.
- Weigh a sample of about (0.150-0.200) grams of crushed antacid table in a weighing bottle.
- Transfer the sample to a clean 250 mL Erlenmeyer flask.
- Add exactly 25.0 ml of 0.200 M HCl
- Swirl to help dissolve the antacid
- Boil the solution to get rid of CO2
- Leave to cool
- Add 3 - 4 drops of the ph.ph indicator to the flask, and titrate the acidic solution to the endpoint.
- Record NaOH add

طحنها مع مي و عملت
titration
Base تحت / acid فوق
... في احتمال انه تكون
الذائبية تاعتها قليلة

الذائبية في وسط
حمضي أفضل من
الذائبية في وسط مائي

الغليان له فائدتين :
١-بتأكد انه ذابت تمامًا
٢- CO2 إذا في زيادة في المحلول بطير لأنه CO2 مع الماء بعطي
حمض H2CO3 لما نسخنها بنضمن إنه النتيجة أدق

عشان أحصل على minimum error انه أذوبها بِـ acid و بعدين
أعادل ال acid المتبقي و هذا هو مبدأ التجربة

زيادة عن الي بحتاجه
تفاعل جزء و ضل جزء

- The 25ml of 0.20M HCl (total mole) is excess :

React with base (anti acid)

Remain in the solution

الكمية الي ضلت من HCl بقدر أعرفها من خلال titration لما عملت
titration بالـ NaOH أنا فعليًا طلعت كمية HCl الي ضلت بالمحلول

نسبتهم ١:١

Titrate with NaOH

بكشف عنه بال titration مع NaOH

- Mole of the base = total mole of HCl – mole remain of HCl
- so this titration called **back titration**

بطلع moles ناتج طرح
※ Direct titration بطلع moles مباشرة
Moles=moles

Calculation

- Total moles of HCl added

$$= M_{HCl} * V_{HCl}$$
$$= 0.2 * 0.025$$

- Moles of NaOH needed to neutralize excess HCl (remain HCl)

$$= M_{NaOH} * V_{NaOH}$$

from titration

  - Moles of excess HCl (remain) = mole of NaOH
- Moles of acid needed to neutralize antacid= (total moles of acid – moles of excess acid)
- Capacity of antacid = (Moles of acid needed to neutralize antacid / mass of antacid)

EXPERIMENT (6) :

precipitation titrations (Argentimetry)

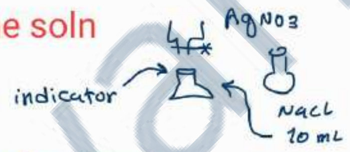
(مانيوال + ترياق)

← القوانين المُستخدمة بالتجربة حسب كل عالم وطريقتة :

1) Standarization of silver nitrate (AgNO3) by Mohr`s method .

$$\boxed{n_{AgNO_3} = n_{NaCl}} \longrightarrow M_{AgNO_3} * \overset{Burret}{V_{AgNO_3}} = \overset{?!}{M_{NaCl}} * \overset{10\ ml}{V_{NaCl}}$$

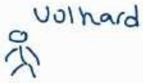
chromate indicator (HCO3-) : nutralsoln , the color of the soln (the end point is reddish brown بني مُحمرّ) .



2) Standarization of silver nitrate (AgNO3) by fajan`s method .

dichlorofluorescein indicator (F-) : the color become pink .

3) Standarization of potassium thiocyanate (KSCN) by Volhard`s method .



$$\boxed{n_{AgNO_3} = n_{KSCN}} \longrightarrow \overset{B\ or\ A\ in}{M_{AgNO_3}} * \overset{10\ ml}{V_{AgNO_3}} = \overset{?!}{M_{KSCN}} * \overset{Burret}{V_{KSCN}}$$

ferric indicator solution (Fe+3 ferric ion) : the color becomes (reddish brown بني مُحمرّ) .

FeSCN2+ كاشف يعمل كوسط حمضي

4) Determination of a mixture of halides (NaCL+ KCL) .

- our unknown is (NaCL + KCL) mixture .
- in the 10ml unknown sample you have 0.065g from (NaCL + KCL).

- suppose إفترض : mass of NaCL = X mass of KCL = Y

then in your sample 10ml → X + Y = 0.065g

A) According or using Mohr`s method :

X + Y = 0.065 g in 10ml sample

end point : $\underset{(NaCl + KCl)}{n_{mixture}} = n_{AgNO_3} \longrightarrow \frac{m_{mixture}}{M.m_{mixture}} = \frac{m_{AgNO_3}}{M.m_{AgNO_3}}$

$$\frac{m_{NaCL}}{M.m_{NaCL}} + \frac{m_{KCL}}{M.m_{KCL}} = M_{AgNO_3} * V_{AgNO_3} \longrightarrow \frac{X}{58.5} + \frac{Y}{74.5} = \underset{\text{B or A من}}{M_{AgNO_3}} * \underset{\text{Burret (L)}}{V_{AgNO_3}}$$

B) According or using Volhard`s method : (back titration)

$X + Y = 0.065g$ 1

end point : total moles of AgNO3 = moles (NaCL+KCL) + moles KSCN

$$n_{(NaCL + KCL)} = n_{AgNO_3} - n_{KSCN}$$

$$\left(\frac{X}{58.5} + \frac{Y}{74.5}\right) = \left(\underset{\text{B or A من}}{M_{AgNO_3}} * \underset{\substack{\text{15 mL} \\ \text{لازم باللتر}}}{V_{AgNO_3}}\right) - \left(\underset{\text{من فرع c}}{M_{KSCN}} * \underset{\text{من البيوريت (L)}}{V_{KSCN}}\right)$$

Argentimetry : silver nitrate (AgNO3) is used as titran in this method .
and used for determine different anons
such as halides (NaCL + KCL) .

*** the general methodes of argentimetry are :**

1. Mohr`s method : the determination of halides anown . in the titration of neutral or very alkaline solution (6.5 - 9) of halued with silver nitrate solution . potassium chromate (KCO3) solution is added as indicator .

$$Ag^+ + cl^- \rightarrow AgCL_{(s)}$$ **(white ppt راسب أبيض)**

but at the end point :

$$2\,Ag^+ + CrO_4^{2-} \rightleftharpoons Ag_2Cr_7O_4\,(s)$$ **(raddish brown ppt)**

why neutral or very alkaline solution with in (6.5 - 9) ?!

in acidic : $2CrO_4^{3-} + 2\,H^+ \rightleftharpoons Cr_2O_7^{2-} + H_2O$

and then : $2Ag^+ + Cr_2O_7^{2-} \rightarrow Ag_2Cr_2O_7\,(s)$ ppt

الذائبية قليلة يصير
يترسب بالأول

and because solubility of $Ag_2Cr_7O_4 > AgCL > Ag_2Cr_2O_7$

then we can`t use $Cr_2O_7^{2-}$ as indicator because it will precipitate before AgCL .

in basic : $Ag^+ + OH^- \rightarrow 1\backslash 2\ Ag_2O_{(s)} + 1\backslash 2\ H_2O$

2. Fajan`s method : in this method , at equivalence point the indicator is absorped by ppt & due to this process , the color of ppt is changed .

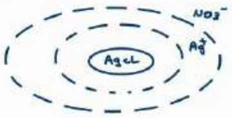
adsorption indicator كواشف ماصة

actual dyes أصباغ فعلية
such as : fluorescein sries

it is either
basicdyes أصباغ قاعدية
such as : Rhodamine series.

$$HIn \rightleftharpoons H^+ + In^-$$ (fluorescein ion).

During titration , just one drop excess of $AgNO_3$, enough to make a double layer around AgCL



, but (In^-) is adrobed more than (NO_3^-)

then due to that adsorption the color of ppt change to pink .

in acidic : $HIn \rightleftharpoons H^+ + In^-$ (the form of indicator always HIn)
in basic : $Ag^+ + OH^- \rightleftharpoons 1\backslash2\ Ag_2O_{(s)} + 1\backslash2\ H_2O$

Note : fajan`s method is more accurate than mohr`s method .

3. Volhard`s method : it is indirect method , where aknown excess of silver nitrate (AgNO3) is added to the halide solution and the residual silver nitrate (AgNO3) is back titrated with a standard thiocyanate solution (SCN-) using ferric ion (Fe+3) as indicator .

rxn : $Fe^{+3} + 3OH^- \rightleftharpoons Fe(OH)_3$
$2Fe(OH)_3 \rightleftharpoons Fe_2O_{3\,(s)} + 3H_2O$

indicator كاشف : Fe^{+3}
chemical rxns : $Ag^+{}_{excess} + CL^- \rightarrow AgCL_{(s)}$
$Ag^+{}_{remained} + SCN^- \rightarrow AgSCN_{(s)}$ witht ppt

at the end point : $Fe^{+3} + SCN^- \rightarrow FeSCN^{2+}$ (red complex ion)

nitrobenzene is added to coat AgCL to prevent the following rxn :
$AgCL + SCN^- \rightarrow AgSCN + CL^-$

and if this happen , it will cause a **ve+ error** in volume of SCN^- solution during titration .

تطبيق ترياق :

أمثلة على القوانين : (من الريبورت الخاص بالاب)

1. $(M * V)\ AgNO_3 = (M * V)\ NaCL$

2. $(M * V)\ AgNO_3 = (M * V)\ KSCN$

EXAMPLE : M for $AgNO_3$ = 0.0714 — V for AdNO3 = 10 ml — V for KSCN = 11.5ml — calculate the M for KSCN

$(M * V)\ AgNO_3 = (M * V)\ KSCN$

$0.0714 * 10\ ml = M * 11.5\ ml$

M of KSCN = 0.062 mol\L

3. Moles (NaCL + KCL) = Moles $AgNO_3$

التطبيق ع هذا القانون كان الجزاء D1 من التجربة وهذا الجزاء محذوف من الريبولات , لان الحل يشبه فرع D2.

4. $(M * V)\ AgNO_3$ = Moles (NaCL + KCL) + $(M * V)\ KSCN$

EXAMPLE : M for $AgNO_3$ = 0.0714 — M for KSCN = 0.061 — V for $AgNO_3$ = 15 * 10 -3 — V for KSCN = 1.8 * 10 -3

calculate the mass of anon (NaCL) (KCL)

المطلوب هو مجهولين , لذلك نحتاج معادلتين .

① المعادلة $(M * V)_{AgNO_3} = \text{Moles } (NaCl + KCl) + (M*V)_{KSCN}$

$$(0.0714 * 15 * 10^{-3}) = \left(\frac{\overset{ⓧ}{\text{mass of NaCl}}}{58.5} + \frac{\overset{ⓨ}{\text{mass of KCl}}}{74.5}\right) + (0.061 * 1.8 * 10^{-3})$$

$$\frac{\text{Mass of NaCl}}{58.5} + \frac{\text{Mass of KCl}}{74.5} = 9.612 * 10^{-4}$$

② المعادلة $X + y = 0.0650$ حفظ من المانيوال

x = Mass of NaCl = 0.024

Y = mass of KCl = 0.0408

بتحل المعادلتين بالحذف أو التعويض .
أو بإستخدام الآلة الحاسبة أسرع

حل المعادلتين بإستخدام الآله الحاسبه :
حسب نوع آلتك بتلاقي الطريقة على اليوتيوب
casio fx - 99IES plus

mode --> 5 : EQN --> 1 : an x + bn y = c

<-- أدخل القيم <-- = <-- بيطلع الناتج

طبعًا بدون ال Moles ,

$$\text{Moles} = \frac{\text{mass}}{\text{M.mass}}$$

calculate the conc. of NaCL / KCL in g / L <-- مرات بيطلب

فبتقسم على الحجم باللتر وبكون مُعطى بالسؤال الحجم .

Q.1 : 50.5 ml of 0.092 M $AgNO_3$ was added to 10ml of KCL .
the excess $AgNO_3$ was titrated with 10ml of 0.088M KSCN .
what is the concentration of KCL in (g\ L).

M.m = 74.5 g\ mole
answer : 28.0567 g\L

سؤال سنوات

الحل نلاحظ ان المعطيات فيها KSCN لذلك القانون الرابع هو القانون المناسب ، لكن السؤال مخفف علينا وطالب بس معلومات عن ال KCL ومش ذاكر ال NaCL ليش ؟!

عشان ما تضطر تحل مجهولين وتستخدم معادلتين ، لذلك المطلوب هو مجهول واحد بكفيه معادله وحده

$$(M * V)_{AgNO_3} = \text{Moles KCL} + (M * V)_{KSCN}$$

$$0.092 * \frac{50.5}{100} = \text{Moles KCL} + 0.088 * 10\,\cancel{ml} * \frac{1L}{1000\,\cancel{ml}}$$

$$\text{Moles KCL} = 3.766 * 10^{-3}$$

يجب ان يكون الحجم باللتر

$$\text{concentration of KCL in } \frac{g}{L} = \frac{\text{mass (g)}}{10 * 10^{-3}\ (L)}$$

$$= \frac{\text{moles} * \text{M.mass}}{10 * 10^{-3}} = \frac{3.766 * \cancel{10^{-3}} * 74.5}{10 * \cancel{10^{-3}}}$$

$$= 28.057\ g/L$$

Q.2 : In titratins involving $AgNO_3$, it shouldn`t be done in alkaline medium ,why ?

A) to prevent production of Ag_2O .
B) to inhance ionization of NO_3^- .
C) to avoid the formation of $Fe(OH)_3$
D) to gain a distinct indicator color.

answer : A

Q.3 : 0.24 g sample
contains 50.6 % $CaCL_2$ (111 g\ mole)
CL (35.5 g\ mole)
excess AgNO3
AgCL (143.5 g\mole)
mass ??

$\boxed{n_{AgCl} = 2 * n_{CaCl_2}}$

1 g ⟶ 1000 mg

① $\text{mass } CaCl_2 = \frac{50.6}{100} * 0.24 \text{ g} = 0.12144 \text{ g}$

② $\text{mol } CaCl_2 = 0.12144 \cancel{g} * \frac{1 \text{ mol}}{111 \cancel{g}} = 0.001094 \text{ mol}$

$= 1.094 * 10^{-3} \text{ mol}$

③ $\underline{1 CaCl_2} + 2 AgNO_3 \longrightarrow \underline{2 AgCl} + Ca(NO_3)_2$

$1 \text{ mol } CaCl_2 \longrightarrow 2 \text{ mol } AgCl$
$1.094 * 10^{-3} \text{ mol } CaCl_2 \longrightarrow ?? \text{ mol } AgCl$

$\text{mol } AgCl = 2 * 1.094 * 10^{-3} = 2.188 * 10^{-3} \text{ mol of } AgCl$

④ $\text{mass of } AgCl = 2.188 * 10^{-3} \cancel{mol} * \frac{143.5 \text{ g}}{1 \cancel{mol}}$

$= 313.978 * 10^{-3} \text{ g} = 0.313978 \text{ g}$

$= 313.9 \text{ mg}$

Q.4 : Which of the followings is correct in using hydrochloric acid (HCL) as the titrant for determination of bases (AgNO3) ?? answer : A

A) the titrant is hydrochloric acid .
B) Ph.Ph indicator has pink color in the acidic medium .
C) Ph.Ph was the only indicator we used in this experiment .
D) sodium phosphoratea is one of the bases we wanted to determine .

II. EXPERIMENTAL PROCEDURE

(A) Standardization of silver nitrate by Mohr's method.

1. Dry sodium chloride at 105 ºC 1-2 hours and weigh accurately about 0.60g. Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Complete to the mark. Shake well.
2. Pipet 10 mL of the standard chloride solution into a 250 mL conical flask and titrate with the silver nitrate solution using 1 mL of the chromate indicator until a faint but distinct change in color occur (faint reddish-brown). Repeat the titration using 1 mL of the mixed indicator (chromate and dichromate) and titrate to the first permanent color change.
3. Calculate the molarity and weight per liter of silver nitrate.
   Use the calculated concentration only in part D.1

(B) Standardization of silver nitrate by Fajan's method

Pipet 10 mL of the standard chloride solution into a conical flask, add 5 drops of the dichlorofluorescein indicator and titrate using the above standardized silver nitrate solution until the precipitate suddenly assumes a pronounced pink color. Repeat the titration and calculate the molarity and weight per liter of $AgNO_3$. Use the calculated concentration in parts C and D.2.

(C) Standardization of potassium thiocyanate by Volhard's method

Pipet 10 mL of the standard silver nitrate solution into a conical flask. Add about 2-3 mL of the 6 M HNO_3 and 1 mL of the ferric indicator solution. Titrate with thiocyanate solution. Shake vigorously during the titration until the red brown color of $FeSCN^{2+}$ is permanent for one minute.
Calculate the molarity and weight per liter of the thiocyanate solution.

(D) Determination of a mixture of halides (NaCl + KCl)

1. According to Mohr's method

Pipet 10 mL of the unknown mixture solution into a conical flask and add 1 mL chromate indicator (or the mixed indicator) and titrate with the standard silver nitrate. Find the volume of silver nitrate which reacts with the mixture. Duplicate the titration.

If $W_1 = 0.0650$ g is the weight of the mixture in 10.0 mL which contains x g of NaCl and y g of KCl then

$$W_1 = x + y = 0.0650 \text{ g}$$

$$\frac{x}{58.5} + \frac{y}{74.5} = M.V$$

Where M is the molarity of $AgNO_3$ and V is the volume of $AgNO_3$ required for the titration.
By solving the above two equations determine the weight of each of NaCl and KC1 per liter.

2. According to Volhard's method

Pipet 10.0 mL of the unknown mixture into a conical flask. Add an excess volume of 0.1 M $AgNO_3$ (15.0 mL exactly), about 2-3 ml of 6 M HNO_3 and 1 mL of the ferric indicator solution, and 1 mL of nitrobenzene. Titrate with the standardized potassium thiocyanate solution according to Volhard's method. Duplicate the titration. Determine the weight of NaCl and KCl per liter. Compare the two results obtained from steps (1) and (2)

M.m NaCL = 58.45 g / moL
M.m KCL = 74.5 g / moL
M.m AgNO3 = 169.87 g / moL
M.m KSCN = 97.18 g / moL

ملاحظة مهمة جدًا : التجربة دقيقة جدًا بالشغل العملي بالاب
أي خطأ لحظة أخذ القراءة يغير جزاء من الحل بالريبورت
وتصبح أحد الكتل من x or y تطلع بالقيمة السالبة

تجنب الخطأ قدر الإمكان 🫡

في علاقات بدهم حفظ لتسهيل الحل ✍️

$$n_{AgCL} = 2 \times n_{CaCL2}$$

$$n_{AgCL} = 2 \times n_{MgCL2}$$

شرح لمعرفة الحل على الآلة الحاسبة

$$\underset{(NacL + KcL)}{\underset{(x+y)}{n_{\substack{mixture \\ unknown}}}} = \underset{(NacL)}{n_x} + \underset{(KcL)}{n_y} = \frac{m_{NacL\ (x)}}{M.m_{NacL\ (x)}} + \frac{m_{KcL\ (Y)}}{M.m_{KcL\ (y)}}$$

$$n_{unknown} = \frac{m_x}{58.44} + \frac{m_y}{74.55} \longrightarrow 0.00099 = \frac{x}{58.44} + \frac{y}{74.55}$$

$$\overset{\text{معامل } x}{\left(\frac{1}{58.44}\right)} x + \overset{\text{معامل } y}{\left(\frac{1}{74.55}\right)} y = 0.00099$$

$$\boxed{0.017\,x + 0.0134\,y = 0.00099} - - - - - ②$$

تم الحل على الآلة الحاسبة ، بإدخال المعادلات والحساب.

EXPERIMENT (7)
PRECIPITATION TITRATIONS (ARGENTIMETRY)

Name:	**Group No.:**

رقم الأنون 10

III DATA

Standardization of silver nitrate

Weight of NaCl..0.600..±..0.001..g

a) ***Mohr's Method***	Trial 1	Trial 2	Trial 3
Volume of NaCl	10.00 ± 0.05 mL	10.00 ± 0.05 mL	
Volume of $AgNO_3$	7.15 ± 0.05 mL	7.10 ± 0.05 mL	

Average vol. of $AgNO_3$…..7.13….mL Molar conc. $AgNO_3$..0.144..M…

b) ***Fajan's Method***	Trial 1	Trial 2	Trial 3
Volume of NaCl	10.00 ± 0.05 mL	10.00 ± 0.05 mL	
Volume of $AgNO_3$	6.90 ± 0.05 mL	6.80 ± 0.05 mL	

Average vol. of $AgNO_3$…6.85…mL Molar conc. $AgNO_3$….0.15.M…

Standardization of potassium thiocyanate

c) ***Volhard's Method***	Trial 1	Trial 2	Trial 3
Volume of $AgNO_3$	10.00 ± 0.05 mL	10.00 ± 0.05 mL	
Volume of KSCN	15.10 ± 0.05 mL	14.90 ± 0.05 mL	

Average vol. of KSCN………15….mL Molar conc. KSCN……0.100 M

Determination of a mixture of halides (NaCl + KCl)

a) ***Mohr's Method***	Trial 1	Trial 2	Trial 3
Volume of unknown	10.00 ± 0.05 mL	10.00 ± 0.05 mL	
Volume of $AgNO_3$	6.85 ± 0.05 mL	6.75 ± 0.05 mL	

Average vol. of $AgNO_3$.......6.8... mL

b) ***Volhard's Method*** Volume of $AgNO_3$……………..

	Trial 1	Trial 2	Trial 3
Volume of unknown	10.00 ± 0.05 mL	10.00 ± 0.05 mL	
Volume of KSCN	12.65 ± 0.05 mL	12.55 ± 0.05 mL	

Average vol. of KSCN .....12.6...... mL

Instructor's Signature

IV CALCULATIONS

• Part A. (Mohr's method)

- Molarity of NaCl (mol/L) =

$$\frac{m_{NaCl}}{M.m_{NaCl} * V_{NaCl}(L)} = \frac{0.6\ \cancel{g}}{58.44\ \frac{\cancel{g}}{mol} * 1000\ \cancel{mL}} * \frac{1000\ \cancel{mL}}{1L} = 0.103\ mol/L$$

- Molar Conc. of $AgNO_3$ (mol/L) =

$$!?\ M_{AgNO_3} = \frac{M_{NaCl} * V_{NaCl}}{V_{AgNO_3}} = \frac{0.103\ mol/L * 10\ \cancel{mL}}{7.13\ \cancel{mL}} = 0.144\ M$$

- Weight of $AgNO_3$ (g/L) = $\frac{0.144\ \cancel{mol}}{L} * \frac{169.87\ g}{1\ \cancel{mol}} = 24.46\ g/L$

$$\boxed{\begin{gathered} n_{AgNO_3} = n_{NaCl} \\ (M * V)_{AgNO_3} = (M * V)_{NaCl} \end{gathered}}$$

• Part B. (Fajan's method)

- Molar Conc. of $AgNO_3$ (mol/L) =

$$\frac{M_{NaCl} * V_{NaCl}}{V_{AgNO_3}} = \frac{0.103\ mol/L * 10\ \cancel{mL}}{6.85\ \cancel{mL}} = 0.150\ mol/L$$

- Weight of $AgNO_3$ (g/L) = $\frac{0.150\ \cancel{mol}}{1L} * \frac{169.87\ g}{1\ \cancel{mol}} = 25.48\ g/L$

$$\boxed{\begin{gathered} n_{KSCN} = n_{AgNO_3} \\ (M * V)_{KSCN} = (M * V)_{AgNO_3} \end{gathered}}$$

• Part C. (Volhard's method) (Use the concentration of $AgNO_3$ from part B)

- Molar Conc. of KSCN (mol/L) = $\frac{M_{AgNO_3} * V_{AgNO_3}}{V_{KSCN}} = \frac{0.150\ mol/L * 10\ \cancel{mL}}{15\ \cancel{mL}}$

$= 0.1\ mol/L$

- Weight of KSCN (g/L) =

$$\frac{0.1\ \cancel{mol}}{1\ L} * \frac{97.18\ g}{1\ \cancel{mol}} = 9718\ g/L$$

x y

- **Part D. (Mixture of halides $\boxed{\text{NaCl}}$ and $\boxed{\text{KCl}}$)**

$$\boxed{1x + 1y = 0.0650}$$

معادلة رقم واحد

- Weight of mixture in one liter = 6.50 g
- Weight of mixture in 10 mL (W_1) = 0.0650 g

α o **Part D.1 (Mohr's Method)** (Use the concentration of $AgNO_3$ from part A)

- Moles of the unknown =

- Weight of NaCl (x) and KCl (y) =

- Concentration of NaCl and KCl (g/L)=

$$\boxed{V_{\underset{\text{الفائض}}{AgNO_3}} = 15\ mL}$$

o **Part D.2 (Volhard's Methods)** (Use the concentration of $AgNO_3$ from part B)

- Moles of $AgNO_3$ added = $M * V = \frac{0.150\ mol}{1\cancel{L}} * \frac{1\cancel{L}}{1000\cancel{mL}} * 15\cancel{mL}$

$= 0.00225\ mol$

$$\boxed{\begin{array}{l}\text{part .c من} \\ M_{KSCN} = 0.1\ M\end{array}}$$

- Moles of KSCN = $M * V = \frac{0.1\ mol}{1\cancel{L}} * 12.6\cancel{mL} * \frac{1\cancel{L}}{1000\cancel{mL}}$

$= 0.00126\ mol$

- Moles of Unknown = mole of $AgNO_3$ − mole $KSCN$

$= 0.00225 - 0.00126 = 0.00099\ mol$

- Weight of NaCl (x) and KCl (y) =

من معادلة
① & ②

$x = 0.032$ g of NaCl

$y = 0.033$ g of KCl

$$\boxed{V_{\substack{x\\y}} = 10\ mL}$$

- Concentration of NaCl and KCl (g/L)=

$$x \longrightarrow \frac{0.032\ g}{10\ \cancel{mL}} * \frac{1000\ \cancel{mL}}{1L} = 3.2\ g/L$$

$$y \longrightarrow \frac{0.033\ g}{10\ \cancel{mL}} * \frac{1000\ \cancel{mL}}{1L} = 3.3\ g/L$$

V **QUESTIONS**

Q1) Why precipitation titrations by the Mohr's method require a neutral solution?

in acidic CrO4 $^{-3}$ convert to Cr2O7 $^{-2}$ (can't use)

in basic Ag + + OH - ----------------> 1/2 Ag2O + 1/2 H2O

Q2) What is the effect of using acidic solution in the Fajan's method?

Conc. of indicator ⬇️
لانه حدث لهُ إمتصاص من القاعدة
H + + FL - ----------------> HFL

Q3) What is the effect of using $K_2Cr_2O_7$ instead of K_2CrO_4 as an indicator on the titration result?

بدلاً من

Ag2CrO > AgCL > Ag2Cr2O7 ((Solubility))

Q4) What is the effect of using basic solution on the results in precipitation titration?

Ag + + OH - ----------------> 1/2 Ag2O + 1/2 H2O

Q5) Why nitrobenzene is added in case of titration of Cl^- by Volhard's method?

To prevent the following rxn.

AgCL - + SCN - ------------> AgSCN + CL -

and to protect AgCL preciptation

EXPERIMENT (7) :
Redox titration (dichromate titration) .

(ترياق)

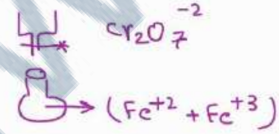
- الكاشف في هذه التجربة هو (diphenylamine) ثنائي فينيل أمين .
(هو عباره عن محلولين مع بعض Fe^{+2} & Fe^{+3})
ال Fe^{+2} فقط الذي يتفاعل منه 10ml .

- سوف نفاعل 10ml من الأنون مع ال $K_2Cr_2O_7$ بوجود وسط حمضي . ونضيف H_3PO_4 \ H_2SO_4
حمضين ونعمل titration .

B1] at the end point , the color change to violet - blue using diphenylamine indecator .

this indecator is an oxidizing agent & will oxidise Fe^{+2} to Fe^{+3} & Fe^{+3} that presrnt in the unknown sample will not react .

end point : $\frac{\text{moles } Fe^{+2}}{6} = \text{moles } Cr_2O_7^{-2}$



moles Fe^{+2} = 6 * (M $Cr_2O_7^{-2}$ * V $Cr_2O_7^{-2}$) = ... g\ L Fe^{+2}

B2] we will first reduce all Fe^{+3} in the 10.00 ml unknown sample to Fe^{+2} , using johnes reductor (zinc amalgam) (ZnHg).

end point : moles Fe^{+2} + moles Fe^{+3} = 6 * (M $Cr_2O_7^{-2}$ * V $Cr_2O_7^{-2}$)

by subbtract → we will obtain moles Fe^{+3}
then find (g \L of Fe^{+3}) .

* potassium dichromate ($K_2Cr_2O_7$) is oxidizing agent .
→ is primary standard

* Fe^{+2} is red agent .

* the chemical rxn :

$$Cr_2O_7^{-2} + 6Fe^{+2} + 14H^{+} \xrightarrow[\text{diphenylamine}]{} 2Cr^{+3} + 6Fe^{+3} + 7H_2O$$

(يتغير لون ال diphenylamine من اللون color less لل violet to blue).

لا تيأس وأنت تعلم أن الله دوماً يخلق نوراً جديداً بعد كل ظلام .

Determination of Fe+2 & Fe+3 in amixture :

1] Determination of Fe^{+2} in the mix. In this part, $Cr_2O_7^{-2}$ reacts only with Fe^{+2} So :

$$\text{moles of } Fe^{+2} = 6 \times (\mathbf{M}\ Cr_2O_7^{-2} \times \mathbf{V\,1}\ Cr_2O_7^{-2})$$

$$\mathbf{M}\ Fe^{+2}\ (g/L) = \frac{\text{moles}}{0.01\ L} \times 55.85\ \ g/mole$$

2] Determination of Fe^{+3} in the mix .

A. Preparation of the **Johnes reductor (ZnHg)** :

$$2\ Zn_{(s)} + Hg^{+2} \rightarrow Zn^{+2} + ZnHg_{(s)}$$

B. Reduction of Fe+2 to Fe+3 :

$$2\ Fe^{+2} + ZnHg \rightarrow 2\ Fe^{+3} + Zn + Hg^{+2}$$

To test presence of Fe^{+3} , we use SCN^- (thiocyanate ion).

$$Fe^{+3} + SCN^- \rightarrow FeSCN^{+2} \quad \text{(red complex)}$$

Then we contine the titration as in part.1 :

$$\text{total moles } Fe^{+2} + Fe^{+3} = 6\ M \times V_2$$

$$M_{Fe^{+3}}\ (g/L) = \frac{\text{moles of } Fe^{+3}}{0.01} \times 55.85\ \ g/mol$$

$$\boxed{M = \frac{n}{V}} \qquad \boxed{n = \frac{m}{M.m}}$$

NOTES : 1- why you add H2SO4 , H3PO4 ??
H2SO4 : to get acidic medium
H3PO4 : to minimize the potential .
between Cr_2O_7 -2 & Fe +2 and H3PO4 from complex with Fe+3 , so we get shap end point for this rxn.

2- diphenyl amine : is a readox indicator , change from color less to violet to blue in colo in color.

تطبيق :

Q.1 : A 20 ml alqoul of 16.4 g/L mixture of Fe^{+2} / Fe^{+3} was titrated to with 13.1 ml of 0.03M $K_2Cr_2O_7$.
What is the mass of Fe^{+2} In one liter (g/L) ?!
(Molar weight of Fe^{+2} = 55.8 g/mol)

$m_{Fe^{+2}} = ?!\ g/L$

① $\text{mole } Fe^{+2} = 6 * (\text{mole } K_2Cr_2O_7) = 6 * (13.1 * 0.03 * 10^{-3})$
$= 2.358 * 10^{-3} \text{ mol}$

② $\text{mass } Fe^{+2} = M_{Fe^{+2}} * n_{Fe^{+2}} = \frac{55.8\ g}{1\ \cancel{mol}} * 2.358 * 10^{-3}\ \cancel{mol}$

$= 0.1315\ g$ of Fe^{+2}

③ السؤال طلب الكتلة لل Fe^{+2} بوحدة مقاس ال g/L

$\frac{0.1315\ g}{20\ \cancel{ml}} * \frac{1000\ \cancel{ml}}{1L} = 6.575\ g/L$

{ وَأَنْ لَيسَ لِلإنسَانِ إلَّا مَا سَعَى , وَأَنَّ سَعْيَهُ سَوفَ يُرَى , ثُمَّ يُجْزَاهُ الْجَزَاءَ الأَوْفَى }

حكي ترياق ، خذ المفيد منه 🤷‍♀️

* كيف بدي أطلع مولات أيون ال Fe+3 ؟ $Fe^{+2} \rightarrow Fe^{+3}$ بتأكسد

* احسن طريقة لتحليل الFe+3 هي : اختزاله الى Fe+2 ، وارجع من جديد آخذ عينة جديدة من الانون ، ونعمل titration .
بشكل عام ينتج (moles new for Fe).

وبتالي :

New moles - moles of part (β_1) = moles of Fe+3

* نحسب عينة جديدة من ال anon ، ونختزل Fe+3 لل Fe+2 (β_2).
اصبحت كل العينة Fe+2 ، وبرجع بعمل ال titration وبحصل ع مولات جديدة ، هذه حجمها اكبر لانها جايه من مولات ال Fe+2 الاصلية & مولات ال Fe+3 التي اختزلت الى Fe .

$\beta_2 - \beta_1 = Fe^{+3}$ بالطرح مولات

* كيف اقوم بالاختزال ؟ سوف أحضر العامل المختزل ، يجب ان يكون fresh
(Johues reductor - zinic amalgam)

zinic pure ⟶ نلصق عليها أيونات الزئبق

* بنحط 10ml zinic جوا ال flask ، ونضيف عليه 10ml HgCL3 ، وبنعمل تحريك قوي جداً ، لمدة خمس ثواني .

* حبة الزنك (لونها اسود ويحتفظ بال zinic amalgam)
بيصير إلتصاق او ترسب الايونات الHg+2 عليها .
هاذ عامل مختزل يختزل ال Fe+3 لل Fe+2 ، عن طريق التخزين .

* أضف عليه 10ml من الأنون وال 40ml H2SO4 & acid . وأبدأ التسخين حتى يغلي - للسلامه العامه - لازم يغلي بدون ما ينشف ؛ لانه لو نشف بيطلع غاز سام .
لذلك يجب ان نغطية بال Filter funnel .

* كيف نتأكد انو تم الاختزال ؟ بنطلع قطرة وحدة من المحلول ونحطها ع wash class ، ثم نضع عليها KSCN ع المحلول . لازم بيطلع لون أحمر ، دليل ع وجود ال fe ، لذلك برجع بعمل تسخين اذا كان اللون color less معناته خلص الامور تمام .

* الان بدنا نتخلص من ال zinic amalgam ونفلتره ع قطنة ، تسمى فلتر فينيل الذي غطيت فيه .
احضر 2flask ، واغسل ال فلتر فينيل منبح فوق ال flask لكي ينزل كل ال fe العالق فيها .
وفلتر ال flask القديم ع الجديد ، وحبيبات الزنك بخليها تنزل ع القطنة .
السوائل حامله معها ال anon & acid .

* النوع الثاني من ال (H3PO4 - acid) وبنرجع بنعمل titration .
الطلاب بينسو ال indicator . ما حدا يفلتر وهوه ساخن ؛ لأن عملية ال titration مش رح تزبط .
وبعدين بنطرح الحجوم او نطرح المولات .

وفي شوي ورق بيناسيا كلام فاضي عن حل الريبورت .

* أهم شي تتذكر التالي :

1] $M_{fe^{+2}} \times V_{fe^{+2}} = 6 \times (M_{Cr2O7^{-2}} \times V_{Cr2O7^{-2}})$

2] moles of fe^{+3} = total moles - moles of fe^{+2}

$$= 6 (M * V)_{Cr2O7^{-2}} - (M * V)_{fe^{+2}}$$

هذه التجربة هي التي تحتوي على (zinic amalgam)
Zinic amalgam - Johuns reductor

Indicator for EXP is diphenyl amine

II. EXPERIMENTAL PROCEDURE

(a) Determination of chromium in a soluble chromic salt:

Chromic salts are oxidized to dichromate by boiling with an excess of a persulphate solution in the presence of a little $AgNO_3$. The dichromate resulted is determined by adding an excess of a standard ferrus solution and titration of the excess of Fe^{2+} with standard $K_2Cr_2O_7$.

1. Weigh out accurately about 0.5 g of the provided chromic salt, and dissolve it in about 30 mL of distilled water. Add about 5 mL of 0.1M $AgNO_3$ solution, followed by 10 mL of 10% solution of ammonium persulfate. Boil the liquid gently for 20 minutes. Cool and dilute to 100 mL in a volumetric flask

2. Pipet 10 mL of the above solution into a 250 mL conical flask, add 10 mL of the standard ferrous ammonium sulfate solution, 40 mL of 1M H_2SO_4, 2-4 drops of diphenylamine indicator and 3-5 mL of syrupy phosphoric acid. Titrate the excess ferrous ion with the standard $K_2Cr_2O_7$. Duplicate the titration. Calculate the percentage of chromium in the sample

(b) Determination of Fe^{2+} and Fe^{3+} in a mixture

1- Determination of Fe^{2+} in the mixture

Pipet 10 mL of the provided mixture and add about 10 mL of 1M H_2SO_4 into a conical flask. Add 2-4 drops of diphenylamine indicator and about 5 mL of phosphoric acid (H_3PO_4). Titrate with the standard $K_2Cr_2O_7$ solution until the color changes to violet-blue. Repeat the titration and calculate the amount of iron (II) in a liter of mixture.

$$Cr_2O_7^{2-} + 6Fe^{2+} + 14H^+ \longrightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

2- Determination of Fe^{3+} in the mixture

2.1.Preparation of the Johnes reductor (Zinc amalgam)

Into 250 mL conical flask, cover about 10 g of pure zinc (20-30 mesh) with 2 percent-mercuric chloride solution (about 10 mL). The mixture is stirred vigorously for about 5 minutes, and the solution is decanted off. Wash the zinc amalgam with distilled water 3-4 times by decantation.

2.2. Reduction procedure:

Add 10 mL of the unknown mixture and about 40 mL of 1M H_2SO_4 on the amalgamated zinc granules. Insert a funnel in the neck of the flask and boil the solution until the yellow color has disappeared entirely, this confirms that all Fe^{3+} has been reduced to Fe^{2+}.

To check the complete reduction of Fe^{3+}, one drop of the solution is added to a drop of NH_4CNS on a porcelain tile.
No pink color should appear.

When the solution has become colorless cool it thoroughly under the tap, wash the drops of liquid from the funnel into the flask. Insert a piece of glass wool (cotton wool as an alternative) into the funnel and filter the solution into a large conical flask (500 mL).

Wash the original flask, funnel and the glass wool several times with distilled water. Add about 5 mL of syrupy H_3PO_4 and 2-4 drops of diphenylamine indicator; titrate with the standard $H_2Cr_2O_7$ as in procedure (b1) above.

EXPERIMENT (8)
REDOX TITRATIONS (DICHROMATE TITRATIONS)

Name:	**Group NO.:**

III DATA

unknown number = 5

بيورية

من البيورية

(b) Determination of Fe^{2+} and Fe^{3+} in a mixture		
Molarity of $K_2Cr_2O_7$……0.02 M		
b.1) Determination of Fe^{2+} in the mixture		
	Trial 1	Trial2
Volume of unknown	10.00 ± 0.08 mL	10.00 ± 0.08 mL
Volume of $K_2Cr_2O_7$	12.00 ± 0.05 mL	11.90 ± 0.05 mL
Average vol. of $K_2Cr_2O_7$……11.95 mL……		
b.2) Determination of Fe^{3+} in the mixture		
Volume of unknown	10.00 ± 0.08 mL	
Volume of $K_2Cr_2O_7$	13.20 ± 0.05 mL	
Weight of Fe^{2+} ..8.0089….. g / L	Weight of Fe^{3+} ……0.8378…… g / L	

mass of zinc = 10.350 ± 0.001 g

Zn Hg

Instructor's Signature

$M.m_{K_2Cr_2O_7} = 0.02\ M$

IV. CALCULATIONS

b. Determination of Fe^{2+} and Fe^{3+} in a mixture

b.1 Determination of Fe^{2+} in the mixture

- Molar conc. of Fe^{2+} (mol/L) = $\overset{!?}{(M * V)}_{Fe^{+2}} = 6\ (M * V)_{Cr_2O_7^{-2}}$

$$\frac{M_{Fe^{+2}} * 10}{10} = \frac{6 * 0.02 * 11.95}{10} = 0.1434\ \text{Molar}$$

- Weight of Fe^{2+} (g/L) = $\text{Molarity} * M.m = \frac{\text{mass}}{V}$

$$55.85 * 0.1434 = \frac{\text{mass}}{V}$$

$$\frac{\text{mass}}{V} = 8.0089\ g/L \text{ of } Fe^{+2}$$

$$\text{mol of } Fe^{+3} = \frac{\text{mass}}{M.\text{mass}}$$

$$\text{Molarity} * V * M.m = \text{mass}$$

b.2 Determination of Fe^{3+} in the mixture

- Moles of Fe^{2+} in the mixture = $\text{mol of } Fe^{+2} = \frac{\text{mol}}{V\ (L)}$

$$0.1434 * \frac{10}{1000} = \text{mol of } Fe^{+2}$$

$$\text{mole of } Fe^{+2} = 1.434 * 10^{-3}\ \text{mol}$$

- Moles of Fe^{2+} and Fe^{3+} in the mixture =

$$6\ \text{mol } Cr_2O_7^{-2} = \text{mole of mixture}$$

$$6 * (0.02 * 13.2 * 10^{-3}) = \text{mol of } Fe^{+3} \longrightarrow \text{mole of mixture} = 1.584 * 10^{-3}$$

- Moles of Fe^{3+} in the mixture =

$$\text{Molar conc. of } Fe^{+3} = \frac{\text{mol}}{V(L)} = \frac{15 * 10^{-4}}{10 * 10^{-3}} = 0.015\ \text{mol}/L$$

- Molar conc. of Fe^{3+} (mol/L) =

$$\text{mole of mixture} = \text{mole of } Fe^{+3} + \text{mole of } Fe^{+2}$$

$$\text{mole of } Fe^{+3} = 1.584 * 10^{-3} - 1.434 * 10^{-3} = 1.5 * 10^{-4}\ \text{mol}$$

Weight of Fe^{3+} (g/L) =

$$\text{mole of } Fe^{+3} = \frac{m}{M.m}$$

$$\text{Molar} * M.m = \frac{m}{V}$$

$$0.015 * 55.85 = 0.8378\ g/L \text{ of } Fe^{+3}$$

V. QUESTIONS

Q1) What is the role of H_3PO_4 in determination of Fe^{2+} by titration with $K_2Cr_2O_7$?

to decrese the difuclt poriutial & that malse the change in color more clearly.

Q2) Can you determine iron ores using $K_2Cr_2O_7$?

Yes , Fe $+^2$ can be determine diricly by titrat with K2Cr2O7 .

Fe $+^2$ can be determinded by changenow

Fe $+^2$ & all the solut by K2Cr2O7 .

EXPERIMENT (9) :
Complexometric titration (titration with EDTA)

- ال EDTA لما يكون حوله أيون بحاصره الايون وبعمل معه Complex ←
بيكون بين ال metal وبين ال ion وبين ال titrant وبين ال analyte

- والعلاقة (النسبة) 1 : 1 مع الأيون

- EDTA has tetra protic acid (يعني عليه 4H)
- **EDTA : Ethylen Diamine Tetra Acetic acid**

Dissociation constant تفكك ثابت :

Ka for 1H → Ka = 10-3

10^{-5} وأقل (حمض ضعيف) ← Ka → (حمض قوي) وأكثر 10^{-5}

- ال EDTA يتفاعل مع أي معدن (metal) شحنته (+2) , مثل : Zn^{+2} , Mg^{+2} , Ca^{+2}
- بالسؤال معطي ال M &V لل EDTA وطالب حساب ال V لل Zn^{+2} .
فالحل تطبيق مباشر $(M_{Zn+2} * V_{Zn+2}) = (M_{EDTA} * V_{EDTA})$
✓ ✓ ✓ ?

A. standardization of EDTA .

end point : $\text{moles}_{Zn+2} = \text{moles}_{EDTA}$

معطى بالسؤال $M_{Zn+2} * V_{Zn+2} = M_{EDTA} * V_{EDTA}$
10 ml ?? Burret

بدك تطلع $CaCO_3$

H_2O

Ⓑ determination of water hardness .

1] end point : $\text{moles}_{Ca+2} + \text{moles}_{Mg+2} = \text{moles}_{EDTA}$

?? $\text{moles}_{Ca+2} + \boxed{\text{moles}_{Mg+2}} = M_{EDTA} * V1_{EDTA}$
من فرع Ⓐ من البيورتة

لتتخلص منها

2] adding 1ml of 5.0 M NaOH will react with Mg^{+2} to precipitate it is as $Mg(OH)_{2(s)}$, then we have only Ca^{+2} (end point) .

$\text{moles } Ca^{+2} = \text{moles}_{EDTA}$
$\text{moles } Ca^{+2} = M_{EDTA} * V2_{EDTA}$

(V2 − V1)
بعد ال NaOH بوجود ال NaOH

بعد التفاعل كاملاً مع ال Mg^{+2} وترسيبه ppt .
ببداية وضعه قبل ما يتفاعل مع ال Mg^{+2} ويترسب .

توضيح

3] $\text{moles } Mg^{+2} = \text{moles}_{EDTA} - \text{moles}_{Ca+2}$
$(M_{Mg+2} * V_{Mg+2}) = (M_{EDTA} * V_{EDTA}) - (M_{Ca+2} * V_{Ca+2})$

(V2 − V1) or (V1 − V2)

عادي المهم تطلع الإشارة موجبة ، لأنه خطأ تطلع سالب إنتبه مهم!!!.

$ZnSO_4 . 7H_2O$
zinc sulfate

ppm (mg\L) of $CaCO_3$

400

اقل من 400 لايوجد تعكر في الماء .
NO water hardless.

اكثر من 400 يوجد تعكر في الماء .
yes water hardless.

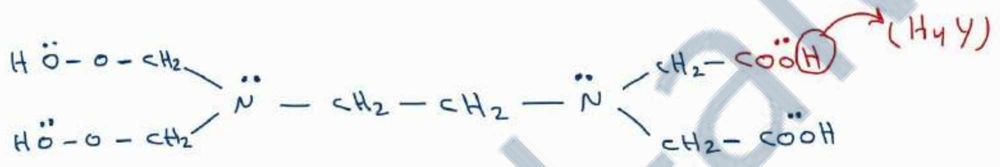
ppm from Ca_{+2} * $\frac{M.m\ CaCO_3}{M.m\ Ca_{+2}}$ = القيمة المحددة لعُسر الماء water hardness

M.m (ZnSO4.7H2O) = 287.97
161.97 + (7* 18) = 287.97

* إذا جاب M.m لل (ZnSO4) لحالها ← أضف عند الحل (7 * 18)

→ Ethylendiamine tetra acetic acid (EDTA)

(حكي المانيوال أخذ المفيد منه 😉)



- the usefulness of EDTA as a titrant is due to the presence of four or six atoms which are available for coordination to a metal cation .
in such a way that stable five membered rings are produced .

⬆ acidity ⬇ PH ⬆ stable

$MY^{(n-4)}$ complex

⬆ ionization ⬇ PH ⬇ stable

⬆ PH ⬆ reaction slightly

- EDTA has four acid dissociation constants , $PKa_1 = 2.0$, $PKa_2 = 2.7$, $PKa_3 = 6.2$, $PKa_4 = 10.3$.

PKa_1 & PKa_2 : for two strongly acidic protons .
PKa_3 & PKa_4 : for two weakly acidic protons .

disodium salt of EDTA . ملح ثنائي الصوديوم

- EDTA forms stable , water soluble 1:1 chelates with polyvalent metal ions .

$$M^{n+} + \boxed{H_2Y^{2-}} \rightleftharpoons MY^{(n-4)+} + 2H^{+}$$

- ionization of MY^{n-4} complex depend upon the PH of solution so lowering the PH decrease the stability of complex.
  also increasing PH of reaction tend to form slightly.

- soluble metallic hydroxide is enhanced :

$$MY^{n-4}\,n + nOH^{-} \rightleftharpoons M(OH)n + Y^{4-}$$

and this depend on KSP for $M(OH)_n$

- for that reasones, it is advisable to do the reaction in a buffer medium.

- **indicators :** organic dyes that form colored chellates with total ions which must be weake, that chelate formed by the metal with EDTA.
  most of these indicators possess acid - base indicator porperties.
  (depend on PH value)

examples of indicators :
- **Murexide**
- **Eriochrome black T.**

- the acidic proton of the sulfonic acid group dissociates in the PH- range of interest 7-12.

- the formula of the **Eriochrome black T indicator** can be written as (H_2 Ind).

two protons يعني عندي

- the color of the indicator changes as follows :

at PH < 6.3 : H_2Ind^{-} has red color.

at 7 < PH < 11 : $HInd^{2-}$ has blue color.

at PH > 11.5 : Ind^{3-} has orange color.

wine red ⟶ deep blue ⟶ orange

$$[H_2Ind]^{-1} \underset{+H^+}{\overset{-H^+}{\rightleftharpoons}} [HInd]^{-2} \underset{+H^+}{\overset{-H^+}{\rightleftharpoons}} [Ind]^{-3}$$

$pH < 6.3$ $7 < pH < 11$ $pH > 11.5$

- at PH range (7-11), the color of ECBT (Eriochrome black T) is blue, but addition of metal ions. produces change in coior to red.

$$M^{2+} + \underset{\text{blue}}{HIn^{2-}} \rightarrow \underset{\text{red}}{MIn^{-}} + H^{+}$$

Murexide indicator change color from pink to violet.

Calculations :

1] $$M_{Ca^{+2}} = \frac{(M_{EDTA} * \overset{mL}{V}_{2\ EDTA})}{10\ \cancel{ml}} * 40.1 * \frac{1000\ \cancel{ml}}{1\ L}$$

2] $$M_{Mg^{+2}} = \frac{M\,(V_1 - \overset{mL}{V_2})_{EDTA}}{10\ \cancel{ml}} * 24.3 * \frac{1000\ \cancel{ml}}{1\ L}$$

3] $$M_{Zn^{+2}} = \frac{(\text{mass of } ZnSO_4.7H_2O \ \backslash\ M.m)}{0.1}$$

4] $$M_{EDTA} = \frac{M_{Zn^{+2}} * V_{Zn^{+2}}}{V_{EDTA}}$$

5] $$M_{Ca^{+2}} = \frac{(M * \overset{mL}{V_2})_{EDTA}}{50\ \cancel{ml}} * \frac{40.1\ \cancel{g}}{1\ mol} * \frac{1000\ mg}{1\ \cancel{g}} = (\quad)\ ppm$$

6] $$M_{Mg^{+2}} = \frac{M_{EDTA} * (V_2 - V_1)_{EDTA}}{50\ ml} * \frac{24.3\ g}{1\ ml} * 1000 = (\quad)\ ppm$$

7] $$\text{Total water } \overset{Ca^{+2}}{\text{hardness}} = \frac{(M * \overset{mL}{V_2})_{EDTA}}{50\ ml} * \overset{\longrightarrow M.m\ CaCO_3}{100.1} * 1000 = (\quad)\ ppm$$

Q.1 : A 20 ml of EDTA was titrated with 45 ml of 0.05 M Zn +2 . what is the milarity of this EDTA solution ?? :تطبيق

$20\,mL$ EDTA $\longrightarrow$ $M_{EDTA} = ?!$

$V_{zn^{2+}} = 45\ mL$

$M_{zn^{2+}} = 0.05\ M$

$$\overset{?!}{M} * V_{EDTA} = M * V_{zn^{2+}}$$

$$\frac{M * \cancel{20}\ mL}{\cancel{20}} = \frac{0.05 * 45\ mL}{20}$$

$$M_{EDTA} = 0.1125\ M \approx 0.113\ M$$

Q. 2 : A 0.5 g sample contains both Ca +2 (40.08 g\mole) and Mg +2 (24.3 g \ mole) was titrated with 60 ml of 0.01 M EDTA with the presence of NaOH .
find the percentages of Ca+2 and Mg+2 . (g \ g)

① Total mol EDTA $= M * V = 0.01 * 60 * 10^{-3} = 6 * 10^{-4}$ mol of EDTA

② mole of Ca^{+2} = mole of EDTA $= 6 * 10^{-4}$ mol

③ mass of $Ca^{+2} = n * M.m = 6 * 10^{-4}\ \cancel{mol} * \frac{40.08\,g}{\cancel{mol}}$

$= 0.024\ g$ of Ca^{2+}

④ $\%\ Ca^{2+} = \frac{\text{mass of } Ca^{2+}}{\text{mass of sample}} * 100\ \% = \frac{0.024\ g}{0.5\ g} * 100\ \%$

$= 4.8\ \%$ of Ca^{2+}

⑤ $\%\ Mg^{+2} = 100\ \% - \%\ Ca^{2+} = 100\ \% - 4.8\ \%$

$= 95.2\ \%$

1. Weigh accurately about 0.60-0.70 g of A.R. Zinc sulfate ($ZnSO_4.7H_2O$). Dissolve in distilled water and transfer quantitatively into a 100 mL volumetric flask. Complete the volume to the mark using distilled water.
2. Pipet 10 mL of $ZnSO_4$ solution into a titration flask and add 5 mL of ammonia-ammonium chloride buffer solution followed by 5-6 drops Eriochrome black T indicator.
3. Titrate with about 0.01 M EDTA solution until the color changes from wine red through purple to pure blue. Repeat the titration three times and calculate the exact molarity of the EDTA solution.

B. Determination of water hardness

1. Pipet 50 mL of tap water into a conical flask. Add 2 mL of ammoniaammonium chloride buffer solution and 5-6 drops of Eriochrome black-T indicator.
2. Titrate with the standard EDTA solution until the color changes from wine red to pure blue. This titration determines the sum of calcium and magnesium (V_1), repeat one more time.
3. Pipet another 50 mL of the tap water into a titration flask. Add 1 mL of 5.0 M NaOH to precipitate the Mg^{2+} ions as $Mg(OH)_2$. Then add 5-6 drops murexide indicator.
4. Titrate until the color changes from pink to purple. This titration determines the calcium only (V_2). The volume corresponding to magnesium is ($V_1 - V_2$). Repeat the titration one more time.

Calculate the calcium and magnesium content in ppm. Calculate the total water hardness as mg $CaCO_3$/L.

C. Determination of the concentration of calcium and magnesium in an unknown solution:

II. EXPERIMENTAL PROCEDURE

A. Standardization of about 0.01M EDTA solution with a standard zinc sulfate solution:

1. Pipet 10 mL of your unknown solution into a conical flask. Add about 25 mL distilled water, 2 mL of ammonia-ammonium chloride buffer and 5-6 drops of the Eriochrome black-T indicator. Titrate with the standard EDTA solution until the color changes from wine red to pure blue. Repeat one more time.

2. Pipet another 10 mL of your unknown solution into a conical flask. Add about 25 mL distilled water, 1 mL of 5 M NaOH and 5-6 drops murexide indicator. Titrate with the standard EDTA solution until the color changes from pink to purple. Repeat the titration one more time.
   Calculate the concentration of calcium and magnesium in your unknown solution in ppm.

EXPERIMENT (9)
COMPLEXOMETRIC TITRATIONS (TITRATIONS WITH EDTA)

Name:	**Group NO.:**

III DATA

Standardization of about 0.01M EDTA

Weight of $ZnSO_4.7H_2O$ ....g

	Trial 1	Trial 2	Trial 3
Volume of $ZnSO_4.7H_2O$			
Volume of EDTA			

Average vol. of EDTA……………..mL Molar conc. EDTA .... mol/L

Determination of water hardness

	Volume of H_2O	Volume of EDTA	Note
Trial 1 (V1)			------------
Trial 2 (V1)			
Trial 1 (V2)			In present of NaOH
Trial 2 (V2)			

Averg. V1 =... Averg. V2 =..

Conc. of $CaCO_3$..mg/L

Determination of the concentration of Ca and Mg in an unknown solution

	Volume of unknown	Volume of EDTA	Note
Trial 1 (V1)	10.00 mL ± 0,01	11.00 mL ± 0.05	------------
Trial 2 (V1)	10.00 mL ± 0,01	11.80 mL ± 0.05	
Trial 1 (V2)	10.00 mL ± 0,01	7.00 mL ± 0,05	In present of NaOH
Trial 2 (V2)	10.00 mL ± 0,01	6.60 mL ± 0.05	

EDTA Averg. V1 =.........11.4 mL......................... Averg. V2 =......6.8 mL.............................

ppm Ca………817.63 ppm……. ppm Mg….335.4….ppm……

EDTA = 0.03 M

un.k = 13

Instructor's Signature

IV. CALCULATIONS

- Part II.A. (Standardization of about 0.01M EDTA solution)

  - Molar conc. of Zn^{2+} (mol / L)

  - Molar Conc. of EDTA (mol / L)

- Part II.B. (Determination of water hardness)

  - Conc. of Ca (ppm)

  - Conc. of Mg (ppm)

  - Total water hardness, as mg $CaCO_3$ /L

- Part II.C. (Determination of the conc. of Ca and Mg in an unknown solution)

  - Conc. of Ca (ppm) $(M * V)_{Ca^{2+}} = (M * V)_{EDTA}$

$$M_{Ca^{2+}} = \frac{M * V_2}{V_{Ca^{2+}}} = \frac{0.03 * 6.8 * \cancel{10^{-3}}}{10 * \cancel{10^{-3}}} = 0.0204\ M$$

حجم الأيون

  - Conc. of Mg (ppm)

$$\frac{0.0204\ mol}{1L} * \frac{40.089}{1\ mol} * \frac{10^3\ mg}{1g} = 817.63\ ppm$$

اكمل تحت

$$\text{Mole } Mg^{+2} + \text{mole } Ca^{+2} = (M * V_1)_{EDTA}$$

$$\text{Mole}_{Mg^{+2}} = (M * V)_{EDTA} - \text{mole } Ca^{+2}$$

$$= M * V_1 - M * V_2 = M(V_1 - V_2)$$

$$= 0.03 * (11.4 - 6.8) * 10^{-3}$$

$$= 1.38 * 10^{-4} \text{ mol of } Mg^{+2}$$

$$\text{conc. of } Mg \text{ (ppm)} = \frac{1.38 * 10^{-4}}{10 * 10^{-3}} * 24.3 * 10^{-3}$$

$$= 3.3547 * 10^{2} = 335.4 \text{ ppm}$$

EXPERIMENT (10) :
Determination of unknown Bases by standardized HCL .

(ترياق)

Constituents in sample — Relation between **V ph.ph & V BCG**

1] NaOH — **V ph.ph = V BCG**

moles NaOH = M HCL × V BCG
moles NaOH = M HCL × V ph.ph

2 ضعف 4|

2] Na2CO3 — **V ph.ph = 1/2 V BCG**

moles Na2CO3 = M HCL × V ph.ph
moles Na2CO3 = M HCL × 1/2 V BCG

أكبر

3] NaHCO3 — **V ph.ph = 0 ; V BCG > 0**

moles NaHCO3 = M HCL × V BCG

أصغر

4] NaOH , Na2CO3 — **V ph.ph > 1/2 V BCG**

moles NaOH = moles Na2CO3 = M HCL × (V BCG - V ph.ph)
moles NaOH + moles Na2CO3 = M HCL × V ph.ph

5] Na2CO3 , NaHCO3 — **V ph.ph < 1/2 V BCG**

moles Na2CO3 = M HCL × V ph.ph
moles Na2CO3 + moles NaHCO3 = M HCL × (V BCG - V ph.ph)

القواعد التى ستكتشفها من هذه التجربة

NaOH
Na2CO3
NaHCO3

تطبيق ترياق :

Q.1 : A 0.2 g base was titrated with 0.3 M HCL . It required 25ml to reach ph.ph end point and required 50ml to reach the BCG end point .
Find the components and moles of this base ??

base : Na_2CO_3

$n_{Na_2CO_3}$ = ?! moles

m_{base} = 0.2 g vs 0.3 M HCL

$V_{ph.ph}$ = 25 mL V_{BCG} = 50 mL

$\boxed{V_{BCG} = 2 * V_{ph.ph}} \longrightarrow Na_2CO_3$ إذاً القاعدة هي الـ

$\boxed{M = \frac{n}{V(L)}}$ moles Na_2CO_3 = $M_{HCL} * V_{(L)\,ph.ph}$

$= 0.3 * 25\,\cancel{mL} * \frac{1\,L}{1000\,\cancel{mL}}$

$= 7.5 * 10^{-3}$

= 0.0075 mol of Na_2CO_3

⑤ , ③

Q.2 : Which of the following gives off the results of the titratiin of sodium hydrogen ($NaHCO3$) carbonates with hydrochloric acid (HCL) . The first number is the Volume required to reach ph.ph end point and the second number indicates the volume required to reach BCG end point .

A) 25 , 25 [✓ B) 0 , 25] C) 17 , 25 D) 28 , 50 E) 25 , 50

ph.ph , BCG

ph.ph = 0 ; $V_{BCG} > 0$ ---- الحالة الأولى ③

$V_{ph.ph} < 1/2\ V_{BCG}$ ---- الحالة الثانية ⑤

I. EXPERIMENTAL PROCEDURE

a) Standardation of hydrochloric acid against sodium carbonate

1. Weight out four 0.2 g sodium carbonate sample by difference from the weighing bottle (to avoid absorption of moisture) into 250 mL flasks and dissolve each of them in 50-100 mL of distilled water.

2. Add 2-3 drops of phenolphthalein indicator to each of the titration flask, and then titrate with the HCl solution until the solution changes from red to colorless. Calculate the molarity of the HCl solution, repeat one more time.

$$Na_2CO_3 + HC1 \longrightarrow NaHCO_3 + NaC1$$

b) Determination of the composition of the unknown

1. Weight out four 0.20-0.21 g samples by difference from the weighing bottle into 250 mL flasks and dissolve each of them in 50-100 mL of distilled water. Titrate the first two samples with HCl using bromocresol green indicator and the second two samples using phenolphthalein indicator. Use the data in Table (1) to determine the composition of the unknown, and then calculate the percentage of each component in the unknown.

EXPERIMENT (10)
DETERMINATION OF UNKNOWN BASES BY STANDARDIZED HCI

Name:	**Group NO.:**

II DATA

a) Standardation of hydrochloric acid against sodium carbonate			
	Trial 1	Trial2	Trial 3
Weight of Na_2CO_3, g			
Volume of HCl, mL			
Molar Conc. HCl, mol/L			
Average molar conc. of HCl ………………..			

b) Determination of the composition of the unknown						
	Phph			BCG		
	Trial 1	Trial2	Average	Trial 1	Trial2	Average
Volume, mL						
Wt. of unknown						
The first component of the unknown is .... and its percentage ....%						
The second component of the unknown is .... and its percentage .... %						

Instructor's Signature

III. CALCULATIONS

- Molar conc. of HCl (mol / L)

- Percentage of the first component

- Percentage of the second component

- IV. QUESTIONS

Q1) If a basic unknown solution was titrated with Phenolphthalein and need 20ml and when it titrated with bromocresol green needs 40ml, the unknown will consist of which type of basic material?

Q2) where you can find the mix of these basic materials in the environments?

1A 2A

3A 4A 5A 6A 7A

8A

1 **H** 1.008																	2 **He** 4.003
3 **Li** 6.941	4 **Be** 9.012											5 **B** 10.81	6 **C** 12.01	7 **N** 14.01	8 **O** 16.00	9 **F** 19.00	10 **Ne** 20.18
11 **Na** 22.99	12 **Mg** 24.31											13 **Al** 26.98	14 **Si** 28.09	15 **P** 30.97	16 **S** 32.07	17 **Cl** 35.45	18 **Ar** 39.95
19 **K** 39.10	20 **Ca** 40.08	21 **Sc** 44.96	22 **Ti** 47.88	23 **V** 50.94	24 **Cr** 52.00	25 **Mn** 54.94	26 **Fe** 55.85	27 **Co** 58.93	28 **Ni** 58.69	29 **Cu** 63.55	30 **Zn** 65.38	31 **Ga** 69.72	32 **Ge** 72.59	33 **As** 74.92	34 **Se** 78.96	35 **Br** 79.90	36 **Kr** 83.80
37 **Rb** 85.47	38 **Sr** 87.62	39 **Y** 88.91	40 **Zr** 91.22	41 **Nb** 92.91	42 **Mo** 95.94	43 **Tc** (98)	44 **Ru** 101.1	45 **Rh** 102.9	46 **Pd** 106.4	47 **Ag** 107.9	48 **Cd** 112.4	49 **In** 114.8	50 **Sn** 118.7	51 **Sb** 121.8	52 **Te** 127.6	53 **I** 126.9	54 **Xe** 131.3
55 **Cs** 132.9	56 **Ba** 137.3	57 **La*** 138.9	72 **Hf** 178.5	73 **Ta** 180.9	74 **W** 183.9	75 **Re** 186.2	76 **Os** 190.2	77 **Ir** 192.2	78 **Pt** 195.1	79 **Au** 197.0	80 **Hg** 200.6	81 **Tl** 204.4	82 **Pb** 207.2	83 **Bi** 209.0	84 **Po** (209)	85 **At** (210)	86 **Rn** (222)
87 **Fr** (223)	88 **Ra** 226	89 **Ac**† (227)															

*Lanthanides

58 **Ce** 140.1	59 **Pr** 140.9	60 **Nd** 144.2	61 **Pm** (145)	62 **Sm** 150.4	63 **Eu** 152.0	64 **Gd** 157.3	65 **Tb** 158.9	66 **Dy** 162.5	67 **Ho** 164.9	68 **Er** 167.3	69 **Tm** 168.9	70 **Yb** 173.0	71 **Lu** 175.0
90 **Th** 232.0	91 **Pa** (231)	92 **U** 238.0	93 **Np** (237)	94 **Pu** (244)	95 **Am** (243)	96 **Cm** (247)	97 **Bk** (247)	98 **Cf** (251)	99 **Es** (252)	100 **Fm** (257)	101 **Md** (258)	102 **No** (259)	103 **Lr** (260)

†Actinides