Subject Date 195 No

"Practical Analytical Chemistry"

Name:

Reg.No :

locker No. : 163

Section No: 9

Instructor: Dr- Safwan Frehat . د.صفوان فريحات

Date : 17th - oct - 2023

Exp.No: Experiment "1"

Title of the experiment : Volumetric Glassware & Balances

Experiment 1
Data sheet and calculations

Name: Section:
Reg. No.: Date:

	Trial (1)	Trial (2)	Trial (3)
Initial burette reading	0,00 ± 0,05	5,10 ± 0,05	10,20 ± 0,05
Final burette reading	5,10 ± 0,05	10,20 ± 0,05	15,33 ± 0,05
Apparent volume, ml	5,10 ± 0,07	5,10 ± 0,07	5,13 ± 0,07
Weight of empty flask	6,6446 ± 0,0001	6,6446 ± 0,0001	6,6446 ± 0,0001
Weight of flask and water	11,6150 ± 0,0001	11,7052 ± 0,0001	11,7579 ± 0,0001
Weight of water, g	4,9704 ± 0,0001	5,2602 ± 0,0001	5,3119 ± 0,0001
Temperature, °C	17,0 ± 0,5	17,0 ± 0,5	17,0 ± 0,5
Actual (experimental) volume , ml	4,9818 ml	5,2723 ml	5,3241 ml
Correction (ml)	-0,1182	0,1723	0,1941

Calculate the volume of water contained in each 5 ml portion from the above data

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culations :-

1) Apparent volume = final burette reading - initial burette reading

2) Weight of water = weight of flask and water - weight of empty flask

3) Correction = actual volume - apparent volume

4) actual volume from Table

* Calculating of the results :- $T = 17^\circ C$

- Trial 1 :-
- Initial burette reading = $(0,00 \pm 0,05)$ ml
- final burette reading = $(5,10 \pm 0,05)$ mL
- Apparent volume = $5,10 - 0,00 = 5,10 \pm 0,07 \rightarrow \sqrt{(0,05)^2 + (0,05)^2} = 0,07$
- Weight of empty flask = $(6,6446 \pm 0,0001)$ g
- Weight of flask and water = $(11,6150 \pm 0,0001)$ g
- Weight of water = $(4,9704 \pm 0,0001)$ g
- Actual volume = $4,9704 * 1,0023 = 4,9818$ ml
- Correction = $-0,1182$ ml

Trial 2 :- $T = 17^\circ C$

- initial burette reading = $(5,10 \pm 0,05)$ ml
- final burette reading = $(10,20 \pm 0,05)$ ml
- Apparent volume = $10,20 - 5,10 = 5,10 \pm 0,07 \rightarrow$
- Weigh of empty flask = $(6,6446 \pm 0,0001)$ g
- weight of flask and water = $(11,7062 \pm ,0001)$ g
- weight of water = $(5,2602 \pm 0,0001)$ g
- Actual volume = $5,2602 * 1,0023 = (5,2723)$ ml
- Correction = $0,1723$ m

Trial 3 :- $T = 17^\circ C$

- initial burette reading = $(10,20 \pm 0,05)$
- final burette reading = $(15,33 \pm 0,05)$
- Apparent volume = $(5,13 \pm 0,07)$ ml
- Weight of empty flask = $6,6446 \pm 0,000$
- weight of flask and water = $11,7579 \pm 0,0$
- weight of water = $(5,3119 \pm 0,0001)$
- Actual volume = $5,3241$ ml
- correction = $0,1941$ ml

* Sources of error :-

1- There was air bubles in the burette

2- There was an error by recording the initial and final burette reading

3- The weighting bottle was holded by fingers

4- weighting the bottle wasn't proffesionly done because it wasn't cleaned very well.

Question

A 25.00 ml pipette was found to deliver 24.976 g of water when calibrated against stainless steel weight at 25°C. Use the data in Table 2 to calculate the volume delivered by the pipette at this temperatu and at 20°C.

at 25°C :-

1 g → 1,0036 ml

24,976 → ?? ml

Actual volume = 25,0659 ml

at 20°C :-

1 g → 1,0028 ml

24,976 g → ?? ml

Actual volume = 25,0459 ml

Name :-

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9.5

Section :- 9

[redacted]

Instructor :- Dr. Safwan AL-Freihat

Date :- 20 ~ Mar ~ 2023

Exp. No :- 2

Title of the exp :- Sampling and Statistical Treatment of Data

Experiment 2
Data Sheet and Calculations

Name: Section: 9

Reg. No.: Date:

A. Homogenous sample

Result no.	V_S ±0.01	V_B	V_S-V_B
1	10,80 ±0,05	0,05 ±0,07	10,75 ± 0,09
2	12, 30 ±0,07	0,05 ±0,07	12,25 ± 0,09
3	10,10 ±0,07	0,05 ±0,07	10,05 ±0,09
4	10.55 ±0,07	0,05 ± 0,07	10,50 ± 0,09
5	11.15 ±0,07	0,05 ± 0,07	11,10 ±0,09

B. Non-homogenous sample

Result no.	V_S	V_B	V_S-V_B
1	6,50 ±0,07	0,05 ±0,07	6,45 ± 0,09
2	7,10 ±0,07	0,05 ± 0,07	7,10 ± 0,09
3	6,10 ±0,07	0,05 ± 0,07	6,10 ± 0,09
4	7.75 ± 0,07	0,05 ± 0,07	7,80 ± 0,09
5	6.85 ±0,07	0,05 ± 0,07	6,80 ± 0,09

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Calculations :-

(A) Homogenouse sample

(1) The results : (10,05 , 10,50 , 10,75 , 11,10 , 12,25)

(2) Q-test (The value of Q critical in confidence limit 90% = 0,64)

→ 10,05 Q calculated = $\frac{|10,05 - 10,50|}{12,25 - 10,05} = 0,20$ Q calc < Q critical so retained

Keep the value 10.05

→ 12,25 Q calculated = $\frac{|12,25 - 11,10|}{12,25 - 10,05} = 0,52$ < Q critical so retained

// = = = 12.25

(3) Mean = $\frac{\Sigma x}{n} = 10,93$

(4) Median = 10,75

(5) Average deviation from the mean = $\frac{\Sigma |x_i - \bar{x}|}{N} = \frac{(0,88 + 0,43 + 0,18 + 0,17 + 1,32)}{5}$

(6) Relative average deviation from the ~~median~~ mean = $(AD/\bar{X}) * 100\% = 5,4\%$ = 0,596

(7) Average deviation from the median = $(0,7 + 0,25 + 0,00 + 0,35 + 1,5)/5 = 0,56$

(8) Relative avarage deviation from the median = $\frac{0,56}{10,75} * 100\% = 5,2\%$.

(9) standard deviation = $S = \sqrt{\frac{\Sigma (x_i - \bar{x}_e)^2}{N-1}} = 0,83$

(10) Relative standard d'viation = $\frac{S}{\bar{X}} * 100\% = 7,6\%$.

(B) Non Homogenous Sample

(1) The results :- (6,10 , 6,45 , 6,80 , 7,10 , 7,80)

(2) Q-test → 6,05 → $\frac{|6,10 - 6,45|}{7,80 - 6,05} = 0,22$ < Q critical so retained

Keep the value 6,10

→ 7,80 → $\frac{|7,80 - 7,10|}{7,80 - 6,05} = 0,41$ < Q critical so retained

// // // 7,80

(3) Mean = $\frac{\Sigma x_i}{N} = 6,85$

(4) Median = 6,80

Average deviation from the mean = $\frac{\sum |x_i - \bar{x}|}{N}$ = $\frac{(0,75 + 0,40 + 0,05 + 0,25 + 0,15)}{5}$

= 0,48

⑥ Relative Average deviation from the mean = $\frac{AD}{\bar{x}} * 100\%$ = 7,00%

⑦ Average deviation from the median = (0,7 + 0,35 + 0,00 + 0,30 + 1,00)/5

= 0,47

⑧ Relative average deviation from the median = $\frac{AD}{6,8} * 100\%$ = 6,9%

⑨ standard deviation = $s = \sqrt{\frac{\sum (x_i - \bar{x})^2}{N-1}}$ = 0,65

⑩ Relative standard deviation = $\frac{s}{\bar{x}} * 100\%$ = 9,5%

Questions 1- compare the results of two groups in terms of:-

ⓐ Mean homogenous 10,93 > 6,85 Non Homogenous $-\frac{1}{2}$

ⓑ standard deviation Homogenous 0,83 ≯ 0,65 Non Homogenous

ⓒ Relative standard deviation Homogenous 7,6% < 9,5% Non Homogenous

② Calculated the molar concentration of acitic acid for both groub results

↳ Homogenous $M_{(NaOH)} * V_{(NaOH)} = M_{(CH_3COOH)} V_{(CH_3COOH)}$

$0,10 * 10,93 = M\ (10,00)$

$M = 0,1093\ M$

↳ Non homogenous

$M_{(NaOH)} V_{(NaOH)} = M_{(CH_3COOH)} V_{(CH_3COOH)}$

$(0,10)\ (6,85) = M\ (10,00)$

$M = 0,0685\ M$

③ Assume the concentration of acitic acid is 0,1 M ; Make comments on the results in terms of accuracy and precision Accuracy → ralative error < for Homo → $\frac{0,1093 - 0,1}{0,1} * 100\%$ = 9,3%

for non Homo → $\frac{0,0685 - 0,1}{0,1} * 100\%$ = -31,5%

presision → RSD ↪ for Homo = 7,6%

↪ for Non Homo = 9,5%

NAME :

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Section No: 9

Instructor : Dr. Safwan Al-Freihat.

Date : 7-11-2023.

Exp No: 3

Title : Neutralization Titration in Aqueous Medium

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Experiment 3
Date Sheet and Calculations

Name: Section: 9
Reg. No.: Date:

Mass of Na_2CO_3		0.5005 ∓0.0001
(A) Vol. of Na_2CO_3		**Vol. of HCl**
1. 10.00 ∓0.05		9.9 ∓0.07
2. 10.00 ∓0.05		9.70 ∓0.07
3. 10.00		
(B) Vol. of NaOH ∓0.05		**Vol. of HCl** ∓ 0.07
1. 10.00 ∓0.05		9.3 ∓0.07
2. 10.00 ∓0.05		8.90 ∓0.07
3. 10.00		
(C) Vol. of H_3PO_4	**Vol. of NaOH**	**Indicator** bromo cresol
1. 10.00 ∓0.05	14.2 ∓0.07	green indicator
2. 10.00 ∓0.05	13.4 ∓0.07	Phenophtalin

∓0.07

Calculate the molarity of Na_2CO_3, HCl and NaOH solutions, and calculate the concentration of H_3PO_4 in g/L.

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Instructor's name: .....

31/10/2023

→ Calculation ←

Part (A)

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

- mass of Na_2CO_3 = 0.5005g ∓ 0.0001
- Volume of Na_2CO_3 = 10.00 ∓ 0.05 mL

$$M = \frac{0.5005 \quad (\times 10^3 \leftarrow \text{For V(L)})}{\text{molar mass of } (Na_2CO_3) \times 10.00 \times 10^{-3}} = 0.047\ M$$

$105.988 \times 10.00 \times 10^{-3}$

$\frac{4.722 \times 10^{-3}}{0.01} = 0.47$

→

* Volume of HCl = (9.90 + 9.70)/2 = 9.8 mL ∓ 0.1 → $\sqrt{(0.07)^2+(0.07)^2}$

* Stiochiometry → 1mol of Na_2CO_3 : 2mol HCl

$2 M \bar{V}_{(Na_2CO_3)} = M \bar{V}(HCl)$ ($\bar{V}$ NaOH = (10.00 + 10.00)/2 = 10.00mL ∓ 0.07)

$$\frac{2 \times 0.047 \times 10.00}{9.8} = M(HCl) \Rightarrow \boxed{M(HCl) = 0.096\ M}$$

— The indicat that I have been used in this part: bromocresol green indicator → For changes From blue (base) → yellow (acid).

Part (B)

* $NaOH + HCl \longrightarrow NaCl + H_2O$

$\bar{V}$ HCl = (9.30 + 8.90)/2 = 9.1mL ∓ 0.1 → (Same as $\bar{V}$ HCl in Part A)

$\bar{V}$ NaOH = (10.00 + 10.00)/2 = 10.00 mL ∓ 0.07 → $\sqrt{(0.05)^2+(0.05)^2} = 0.07$

* Stoichiometry 1mol NaOH = 1mol HCl

$M \bar{V}(NaOH) = M \bar{V}(HCl)$

$$M(NaOH) = \frac{0.096 \times 9.1}{10.00} = 0.097\ M$$

* The indicator : phenophthalein from pink (acidic) → colorless (basic)

Part C : unknown number : 43

(1) Neutralization of one proton:-

$H_3PO_4 + NaOH \rightarrow NaH_2PO_4 + H_2O \rightarrow pH = 4.6$

$\bar{V}$ NaOH = (14.2 + 13.4)/2 → 13.8 mL ∓ 0.1

$\bar{V}$ H_3PO_4 = (10.00 + 10.00)/2 = 10.00 mL ∓ 0.07

$M \bar{V}(H_3PO_4) = M \bar{V}(NaOH)$

Stoichiometry → 1mol H_3PO_4 = 1mol NaOH

$$\Rightarrow M(H_3PO_4) = \frac{0.097 \times 13.8}{10.000} = 0.131\ M$$

Molar mass of H_3PO_4 = 97.994 g/mol

$0.131 \frac{mol}{L} \times 97.994 \frac{g}{mol} = 12.83 \frac{g}{L}$

- The indicator has been used : Bromocresol green indicator
  (acidic) yellow → blue (basic)

For indicator : Phenophthalin.

Change from colorless (acidic) → pink (basic)

For Calculation same as above → the result → = 6.67 g/L

$H_3PO_4 + 2NaOH \rightarrow Na_2HPO_4 + 2H_2O \rightarrow pH = 9.7$

$\bar{V}_{NaOH} = (13.8 mL \mp 0.1)$

$\bar{V}_{H_3PO_4} = 10.00 mL \mp 0.07$

Stoichiometry $1 mol\ H_3PO_4 = 2 mol\ NaOH$

$M\bar{V}(H_3PO_4) = \frac{1}{2} M\bar{V}(NaOH)$

$M(H_3PO_4) = \frac{0.97 \times 13.8}{2 \times 10.00} = 0.069\ M$

→ $0.069 \frac{Mol}{L} \times 97.994 \frac{g}{mol} = 6.67\ g/L$

Avery = $\frac{6.67 + 6.67}{2}$ = 6.67 g/L

9.75

4.5 without of NaOH

---

* Sources of error :-
- Error while detecting the colorchange of the indicator
- Determining the initial & final volume of the burette
- using the balance as a result of holding the weighing bottle.

Conclusion :

[1] we choose the indicator depending on the numerical value of pH which the indicator changes it's color at a certain pH

[2] Titration is 3types : 1) strong acid & Strong base
2) weak " & " "
3) strong " & weak base

[3] in this experiemont Na_2CO_3 was a primary standard solution

[4] we can standarize an acid, then use it with other bases in titration & complete calculation

Questions:

[1] How do you determine the solubility of $Ca(OH)_2$ by a simple acid-base?

(a) prepare a solution of $Ca(OH)_2$

(b) pipette a certain volume of the $Ca(OH)_2$ standard solution

(c) Titrate against a weak or strong acid with a known concentration

(E.g) HCl & put a porpriate indicator

(d) Find the solubility of $Ca(OH)_2$ by the equation of titration.

$2HCl + Ca(OH)_2 \rightarrow CaCl_2 + 2H_2$

here ... 1 mole of $Ca(OH)_2$ → 2 mol of HCl

(E) According to the above equation.

$$M(Ca(OH)_2) = \frac{M_{HCl} V_{HCl}}{2 V(Ca(OH)_2)} \Rightarrow M_{Ca(OH)_2} = [Ca^{+2}] = \frac{1}{2}[OH^-]$$

---

[2] How do you determine the water of crystallization of washing soda? you need x in → $Na_2CO_3 \times H_2O$ (percentage of water percent)

[A] prepare a solution by putting a known mass of hydrate Na_2CO_3 in a distilled water.

[B] mark the volume of the solution that has been prepared

[C] Put Methyl orange indicator, or any appropriate indicator

[D] Titrate with a known molarity with HCl (acid) at the end point the color will change yellow → peach pink

[E] According to the equation:

$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$ $\quad \frac{1}{2} M_1V_1 = M_2V_2$

[F] Calculate the mass of H_2O = mass of $Na_2CO_3 \times H_2O$ − mass of Na_2CO_3

[g] Calculate the mole of H_2O ($n = m/Mm$)

[H] x water of crystallization = moles of H_2O / moles of Na_2CO_3

[3] ~~don~~ why two indicators can be used for the determination of phosphoric acid?

Because phosphoric acid has 3 protons (H_3PO_4) and each proton gets titrated ~~at~~ a specific pH, and choosing indicators depend on the ph medium (color changes at certain pH)

[4] No there is no titration between weak acid & weak base because there will not be a abrupt change in pH at the equivalence point. Also, we will need a large amount of titrate in order to get to the equivalence point) NH_3: weak base, CH_3COOH: weak acid

Practical Analytical Chemistry

Name :

Reg No :

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Taker No :

Course : experiemental analytical chemistry (0303216)

section No : 9

Instructor : Dr- Safwan Frehat

Date : 14-11-2023 وتم التسليم ←

Exp No : 4

Title : Precipitation Titration Argentometry

unknown No : 22

Experiment 4
Data Sheet and Calculations

Name:
Reg. No.:

Section: 9
Date: 7-11-2023

Mass of NaCl: ..... 0.6163 ± 0.0001

(A) Mohr's method	Trial (1)	Trial (2)
Vol. of NaCl solution Vol. of $AgNO_3$ solution	10.00mL ± 0.05 10.10 ± 0.07	
(B) Fajan's method		
Vol. of NaCl solution Vol. of $AgNO_3$ solution	10.00mL ± 0.05 10.30 ± 0.07	
(C) Volhard's method		
Vol. of $AgNO_3$ solution Vol. of KSCN solution	10.00mL ± 0.05 10.50 ml ± 0.07	10.00mL ± 0.05 10.30 ± 0.07

Unknown mixture ID: 22		
(I) According to Mohr's method	Trial 1	Trial 2
Vol. of unknown solution Vol. of $AgNO_3$ solution	10.00mL ± 0.05 10.90ml ± 0.07	→ 10.00 ml ± 0.05 10.80 mL ± 0.07
(II)According to Volhard's method		
Volume of unknown solution Vol. of KSCN solution	10.00ml	
Vol. of $AgNO_3$ added in excess		

Instructor's Signature: .....

Instructor's Name: .....

7/11/2023

Calculations :-

"PART A" Standardization of $AgNO_3$ by Mohr's method

Equations: $Ag^+_{(aq)} + Cl^-_{(aq)} \rightleftharpoons AgCl_{(s)}$

$2Ag^+ + CrO_4^{-2} \rightleftharpoons Ag_2CrO_{4(s)}$

- indicators : trial → potassium chromal " colour → reddish-brown"
- Mass of $NaCl_{(s)}$ = 0.6163g Using Analytical balance.
  - uncertainty = ∓0.0001
- volume of NaCl solution = 10.00 ml ∓ 0.05 ↗ $\frac{1}{2}$ * least division = $\frac{1}{5}$ x0.1 = ±0.0
- volume of $AgNO_3$ solution = 10.10 ml ∓ 0.07 ↗ $\sqrt{(0.05)^2+(0.05)^2} = 0.07$

- $n = \frac{m}{m.m} = \frac{0.6163\,g}{58.44\,g/mol} = 0.0105\,mol \rightarrow M = \frac{n}{V_{(L)}} = \frac{0.0105}{100 \times 10^{-3}} = 0.105\,mol/L$ (1)

$M V_{(NaCl)} = M V_{AgNO_3} \rightarrow 0.105 * 10.00 = M_{AgNO_3} * 10.10$

$M_{(AgNO_3)} = 0.1039\,mol/L$ (1)

$M.m_{(AgNO_3)} = 0.1039\,\frac{mol}{L} * 169.91\,\frac{g}{mol} = 17.65\,g/L$

→

"PART B" Standardization of $AgNO_3$ by Fajan's method.

Equation: $NaCl + AgNO_3 \rightleftharpoons AgCl_{(s)} + NaNO_3$

Indicator : 5 drops of dichlorofluorescine "colour → pink"

Mass NaCl : 0.6163 ∓0.0001, Mm : 58.44 g/mol, V_{NaCl} = 10.00ml ∓ 0.05

$V_{AgNO_3} = 10.30_{ml} \mp 0.07$

$M V_{NaCl} = M V_{AgNO_3}$

$0.105 * 10.00 = M \cdot 10.30 \Rightarrow M_{AgNO_3} = 0.1019\,mol/L$

molar mass of $AgNO_3$ = 169.91 g/mol

$0.1019\,\frac{mol}{L} * 169.91\,\frac{g}{mol} = 17.31\,g/L$ (1 molarity (g/L))

* The mean value : molarity = $(0.1039 + 0.1019)/2 = 0.1029\,mol/L$

$(17.65 + 17.31)/2 = 17.48\,\frac{g}{L}$

→

"PART C" Standardization of potassium Thiocynate by volhard's method.

$Ag^+ + SCN^- \rightleftharpoons AgSCN_{(s)} \Rightarrow$ White.

$Fe^{+2} + SCN^- \rightleftharpoons [FeSCN]$ "red brown"

indicator → ferric indicator "1ml" → colour "red brown colour" → permanent for 1min
→ 2.5 ml of $MHNO_3$

$V_{(AgNO_3)}$ → 10.00ml ∓0.05 "using pipet"

uncertainty : $\frac{1}{2}$ * least division $\frac{1}{2} * 0.1 = 0.05$

Volume of KSCN : 10.50 ∓ 0.07 ↗ $\sqrt{(0.05)^2+(0.05)^2} \mp 0.07$

From Part A & B → $M_{(AgNO_3)} = 0.1039\ M$ • $V_{AgNO_3} = 10.00\ ml$

$\bar{V}_{KSCN} = 10.40\ mL$ [(10.50 + 10.30/2) = 10.40 ml ± 0.07]

$(M_1V_1)_{Ag} = (M_2V_2)_{KSCN} \Rightarrow M_{KSCN} = \frac{M_1V_1}{M_2} = \frac{10.00\ ml \times 0.1029\ M}{10.40\ ml} = 0.0999\ M$

$Mm_{KSCN} = 97.0\ g/mol$

$97.0\ \frac{g}{mol} \times \frac{0.0999\ mol}{L} = 9.69\ g/L$

(1)

• PART D "I Determination of unknown mixture of Halides (NaCl + KCl)
- unknown number 22

I : According to Mohr's method :-

Volume of unknown : 10.00 mL ± 0.05

Volume of $AgNO_3$ $\bar{V}$: 10.85 ± 0.07 ((10.80 + 10.90)/2) = 10.85 mL)

indicator : Mixed indicators.

Calculations :

mass of the unknown $x + y = 0.065$

Molarity $(AgNO_3) = 0.1029\ M$ avg.

$n_{(AgNO_3)} = M * \bar{V} = 0.1029\ \frac{mol}{L} \times 10.85\ mL \times 10^{-3} = 0.00112 = 11.2 \times 10^{-4}\ mol$

$= 0.00111$

(1)

$(M_1V_1)_{(unknown)} = (M_2V_2)_{AgNO_3}$

moles of unknown $\Rightarrow \frac{x}{M_{mNaCl}} + \frac{y}{M_{mKCl}} \Rightarrow \frac{x}{58.5} + \frac{y}{74.5} = M\ V_{AgNO_3}$ ...(2)

$x + y = 0.065 \rightarrow y = 0.065 - x$ ...[1]

Substitute (1) in (2)

$\frac{x}{58.5} + \frac{(0.065 - x)}{74.5} = 10.85 \times 10^{-3} \times 0.1039\ M$ ok

$74.5x + 58.5x + 3.80 = 4.913$ 4.880.

$16x = 1.113 \Rightarrow x = 0.0696\ g$

$x = \frac{0.0696}{10 \times 10^{-3}\ L} = 6.96\ g/L$ X 1.080 0.0675

$y = 0.0046 \rightarrow 0.46\ g/L$ X

Determining their weight per L volume to mL

Source of errors:

1. errors in NaCl
2. uncleaned glassware.
3. uncalibrated glass wares.
4. Not observing the end point in titration (adding more drops)
5. errors in preparing NaCl solution (adding more volume of distille water, not cleaning the glass rod of funnel that may have partic of NaCl on its surface.
6. Air bubbles in Buret or pipet will make errors in volume.

— Conclusion

in precipitation titration we use $AgNO_3$ as titrant, and this's kind of titration is used to titrate halides, cyanid and also isocynate and to a less extent for phosphate sulfid.

There are 3 general methods:-

1. mohr's method (direct) 2. Fajan's method 3. Volhard's metho "indirect"

in the first part and second one we calculate the molarity of $AgNO_3$ & used it in the following parts → so each part of this experiement depends on it's previous parts.

we used several types of indicators & determine the mass of the component of the known.

→ Purpose

Determine the concentration of Cl^- by forming a precipitate in the three diffrent methods.

• Question 11

(1) Because if we use a basic solution $Ag \xrightarrow{OH^-} AgOH_{(s)}$ which will change to Ag_2O so we will need more volume of $AgNO_3$ and there will be a large positive error, on the other hand if we use an acidic solution $CrO_4^{-2} \xrightarrow{H^+} Cr_2O_7^{-2}$ and there will be $Ag_2Cr_2O_7$. ($Ksp(Ag_2Cr_2O_7) > Ksp\ Ag_2CrO_4$) so we will need an extra $AgNO_3$ to have the end point (there will be positive error)

(2) In an acidic solution your indicator (Fluoresceine) will react with H^+, so our indicator will be the anion of weak acid, it forms protonated species that will not function as an indicator, so there will be extra volume of $AgNO_3$ (positive determination error)

(3) $Ksp\ Ag_2Cr_2O_7 > Ksp\ Ag_2CrO_4$ so we need extra volume of $AgNO_3$ to reach the end point (positive error)

(4) Mohr's & fajan's method, as we said above --- $Ag^+ \xrightarrow{OH^-} Ag(OH)_{(s)}$ there will be Ag_2O ppt → large positive error.

→ Volhard's method $Fe^{+3} \xrightarrow{OH^-} Fe(OH)_{3(s)}$ --- there will be Fe_2O_3 (it will be more volume of $AgNO_3$ → positive erro)

(5) Nitro benzene will cover the surface of AgCl ppt also it will prevent the below reaction from occuring (the reaction is between AgCl and the titrant SCN^- to form AgSCN

$$AgCl + SCN^- \rightleftharpoons AgSCN + Cl^-$$ & $Ksp\ AgCl > Ksp\ AgSCN$

(in order to prevent the negative error as a result of less volume of the titrant SCN^-)

Name :-

Reg. No :-

Locker No :- 553

Instructor :- Dr. safwan Al-frehat

Date :- 10 - April - 2023

section :- 9

Exp. No :- 5

Title of the Experiment :- Redox Titrations (Dichromate titrations)

9.5

Experiment 5
Data Sheet and Calculations

Name: Section:
Reg. No.: Date:

Unknown ID No.: 49	
(A) Determination of Fe^{2+} in a mixture	
Vol. of unknown solution	Vol. of $K_2Cr_2O_7$
1) 10,00 ± 0,05 2) 10,00 ± 0,05 3)	3,60 ± 0,07 3,30 ± 0,07
(B) Determination of Fe^{3+} after reduction	
Vol. of unknown solution	Vol. of $K_2Cr_2O_7$
1) 10,00 ± 0,05 2)	4,60 ± 0,07

Wt. of Fe^{2+} per liter =
Wt. of Fe^{3+} per liter =

Instructor's signature: .....

Instructor's Name: .....

- calculation of the results and their precition :-

Part A :- Determination of Fe^{+2} in the mixture (unknown No:- 49

- avarage volume of $K_2Cr_2O_7$ = $\frac{3,60 + 3,30}{2} = 3,45 \pm 0,09$

$$6(M*V)_{K_2Cr_2O_7} = (MV)_{Fe^{+2}}$$

$$6 * 0,02 * 3,45 = 10,00 * M$$

$$M_{Fe^{+2}} = 0,0414\ M$$

- molar mass for Fe = 55,845 $\frac{g}{mol}$

$$0,0414\ \frac{mol}{L} * 55,845\ \frac{g}{mol} = 2,311\ \frac{g}{L}\ Fe^{+2}$$ (before reduction)

Part B :- Determination of Fe^{+2} after reduction

Fe^{+3} + john's reductor → Fe^{+2}

- Volume of $K_2Cr_2O_7$ = 4,60 ± 0,07

$$6(M*V)_{K_2Cr_2O_7} = (MV)_{Fe^{+2}} \rightarrow 6 * 0,02 * 4,60 = M\ (10,00)$$

$$M_{Fe^{+2}} = 0,0552\ M$$

$$0,0552\ \frac{mol}{L} * 55,845\ \frac{g}{mol} = 3,0829\ \frac{g}{mol}\ Fe^{+2}$$

Fe^{+3} (g/L) = 3,082 − 2,311 = 0,7716 $\frac{g}{L}$ Fe^{+3}

-0.8

What is the role of H_3PO_4 in determination of Fe^{+2} by titration with K_2Cr_2O

H_3PO_4 reacts with yellow Fe^{+3} (resulting for titration) to form $[Fe(HPO_4)]^+$ which is colorless so that the end point of reduction is more clearly visible Moreover, H_3PO_4 lowers the reduction potential of Fe^{+3} - Fe^{+2} couple by complexation (Fe^{+3} is reduced) and hence tends to increase the reducing power of Fe^{+2}

2] Can you detemine Iron in ores using $K_2Cr_2O_7$?

Yes, I can, Because the iron ores contains Fe_2O_3 and Fe_3O_4 which can be dissolved in an acidic solution in order to have Fe^{+3} and reduced it as the equation $Fe^{+3} + e^- \rightarrow Fe^{+2}$ then titrate it with titrant $Cr_2O_7^{-2}$ with the use of H_3PO_4 and diphenylamine sulfuric acid indicator

3] How could you cheak that Fe^{+3} has been completely reduced to Fe^{+2}?

When the yellow color has disappeared entirely; this confirms that all Fe^{+3} has been reduced to Fe^{+2}. Also, to cheak the complete reduction of Fe^{+3}, one drop of the solution is added to a drop of NH_4SCN on a Porcelain tile, if no blood-red-color formation appear, this means that there is a complete reduction of Fe^{+3} to Fe^{+2}

Name :

- Reg No :

- Exp No : 7

The title of Experiement : complexometric Titration Using EDTA .

Instractor: Dr - safwan Freihat.

locker No: 163

Course : Experiemental Analytical lab.

Date: 7th oct.

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Experiment 7
Data Sheet and Calculations

Name: Section: 9

Reg. No.: Date: 5-Dec

(A) Standardization of EDTA				
Mass of $ZnSO_4.7H_2O$	0.3358			
Data	Trial 1	Trial 2	Trial 3	Average
Volume of $ZnSO_4.7H_2O$ Solution	10.00 ±0.05			
Volume of EDTA solution	12.40 ±0.07			
Molarity of EDTA solution	0.0094 mol/L			
(B) Determination of Water Hardness				
Data	Trial 1	Trial 2	Trial 3	Average
1- Vol. of tap water	50.00 ±0.05			
2- Vol. of EDTA solution (V_1) (corresponding to Ca and Mg)	15.80 ±0.07			
3- Vol. of tap water	50.00 ±0.05			
4- Vol. of EDTA (V_2) (corresponding to Ca)	6.60 ±0.07			

Calculations.

A standarization of EDTA with standard zinc sulfate.

- wt of $ZnSO_4 \cdot 7H_2O$ = 0.3358 g ± 0.0001
- Molar mass of $ZnSO_4 \cdot 7H_2O$ = 287.36 g/mol
- V of EDTA = 12.40 ± 0.07 mL (one Trial)

$$n_{\text{for } ZnSO_4 \cdot 7H_2O} = 0.3358\ g \times \frac{1\ mol}{287.369\ g} = 1.1678 \times 10^{-3}$$

$$M \text{ of } ZnSO_4 \cdot 7H_2O = \frac{1.168 \times 10^{-3}}{100 \times 10^{-3}} = \boxed{0.012\ M}$$

number of mol of $ZnSO_4 \cdot 7H_2O$ = M * V

$$0.012 * 10 * 10^{-3} = 1.168 \times 10^{-4}$$

$n_{\text{for EDTA}} = 1.168 \times 10^{-4}$

$$M \text{ of EDTA} = \frac{\text{moles}}{V \text{ in L}} = \frac{1.168 \times 10^{-4}}{12.40 * 10^{-3}} = \boxed{0.0094\ M}$$

or 9.42×10^{-3}

B determination of water hardness.

$$Mg^{+2} + H_2y^{-2} \rightleftharpoons MgY^{-2} + 2H^+$$

$$Ca^{+2} + H_2y^{-2} \rightleftharpoons CaY^{-2} + 2H^+$$

② V_1 of EDTA = 15.80 ± 0.07 mL (one Trial)

V_2 of EDTA = 6.60 ± 0.07 (one Trial)

mwt Ca = 40 g/mol

mwt Mg = 24.3 g/mol

number of moles EDTA = M * V_1

$$0.0094 * 15.80 \times 10^{-3} = \boxed{1.48 \times 10^{-4}\ mol}$$

follow

mole of EDTA = $n_{Ca^{+2}} + n_{Mg^{+2}} = 1.48 \times 10^{-4}$ mol

$M_{Ca^{+2}+Mg^{+2}} = \frac{1.48 \times 10^{-4}}{50.000 \times 10^{-3}} = \boxed{2.97 \times 10^{-3} M}$

number of mole of EDTA = $M_{EDTA} V_2 = 0.0094 \times 6.60 \times 10^{-3}$

$= \boxed{6.204 \times 10^{-5} \text{ mol}}$

$M_{Ca^{+2}} = \frac{n = n_{EDTA}}{V} = \frac{6.204 \times 10^{-5}}{50.00 \times 10^{-3}} = \boxed{1.241 \times 10^{-3} M}$

$ppm\ Ca^{2+} = 1.241 \times 10^{-3} \frac{mol}{L} \times \frac{40 g}{mol} \times 10^{3} \frac{mg}{g}$

$= \boxed{49.64 \text{ ppm}}$

number of mole of Mg^{+2} = $(1.48 \times 10^{-4}) - (6.204 \times 10^{-5})$

$= 8.588 \times 10^{-5}$

$\boxed{8.6 \times 10^{-5} \text{ mol}}$

$M_{Mg^{+2}} = \frac{8.6 \times 10^{-5}}{50.00 \times 10^{-3}} = \boxed{1.72 \times 10^{-3} M}$

$ppm = 1.72 \times 10^{-3} \frac{mol}{L} \times \frac{100 g}{1 mol} \times \frac{1000}{1 g} = \boxed{172 ppm}$

(CaCO3 Hardness)

molar mass of $CaCO_3 = 100 g/mol$,

Volume of EDTA = $V_1 - V_2 = 9.20$

$15.80 - 6.60 = 9.20$

$\pm 0.07 \quad \pm 0.07$

Practical Analytical Chemistry

- Name :-

- Reg No :-

- locker No :-

- Course :- Experimental Analytical Chemistry "lab" (0303216)

- Section No :- "9"

- Instructor :- Dr- Safwan Freihat.

- Date : :- 9-12-2023

- Exp No :- Exp 8

- Title of the experiement : Determination of calcium by quantitative precipitation & titration.

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Experiment 8
Data Sheet and Calculations

Name: Section: 9

Reg. No.: Date: 4-12-2023

Unknown ID No.: 31

(A) Standardization of $KMnO_4$

Mass of Sodium oxalate 2.614.6 ± 0.0001 g per 100 mL

Molarity of sodium oxalate .....

Vol. of $Na_2C_2O_4$ solution	Vol. of $KMnO_4$
1) 10.00 ± 0.05	① 9.20 ± 0.07
2) 10.00 ± 0.05	② 9.40 ± 0.07
3)	

Molarity of $KMnO_4$ solution 0.021 M

Concentration of $KMnO_4$ in g/L: 3.329/L

(B) Precipitation and Titration of Ca^{2+}

Vol. of Ca^{2+} solution	Vol. of $KMnO_4$
1)	14.60 ± 0.07
2) 10.00 ± 0.05	
3)	

Concentration of Ca^{2+} solution in g/L 3.09 g/L

Instructor's Signature: .....

Instructor's Name: .....

- The equation for the principle reaction in the analysis :-

(1) $5C_2O_4^{-2} + 2MnO_4^- + 16H^+ \longrightarrow 10CO_2 + 2Mn^{+2} + 8H_2O$

Standarization of KMnO4

(2) $Ca^{+2} + C_2O_4^{-2} \rightleftharpoons CaC_2O_4(s)$ oxalate preciptation agent.

(3) $CaC_2O_4 + H_2SO_4 \rightarrow CaSO_4 + H_2C_2O_4$ → dissolving of CaC_2O_4 by H_2SO_4

- Equations that show the calculation employed in computing the results :-

$n = M \times V(L)$, $n = \frac{m}{\text{molar mass}}$

- Calculation of the results & their precision :-

* [A] Standarization of 0.1 M KMnO4 :-

- $m_{Na_2S_2O_4} = 0.6106g \mp 0.0001$ ← Digital balance error.
- $M_m(Na_2C_2O_4) = 133.998\ g/mol$
- $(n_{Na_2S_2O_4}) = \frac{0.6106}{133.998} = 4.490 \times 10^{-3}$ mole $Na_2C_2O_4$
- $M_{Na_2C_2O_4} = n / V = 4.490 \times 10^{-3} / 0.100 = 0.0490$ M
- $\bar{V}\ Na_2C_2O_4 = (10.00 + 10.00)/2 = 10.00ml \mp 0.07$

$\bar{V}\ KMnO_4 = (9.20 \mp 0.07 + 9.40 \mp 0.02)/2 = 9.30 \mp 0.10$

$2\ \frac{n}{M.V}C_2O_4^{-2} = 5\ \frac{n}{M \times V}MnO_4^-$

$2 \times 0.0490 \times 10 \times 10^{-3} = 5 \times M(MnO_4^-) \times 9.30 \times 10^{-3}$

$\Rightarrow M(MnO_4) = 0.021\ M$

- Concentration of MnO_4^- in (g/L)

$M.m = 158.032\ g/mol\ (KMnO_4)$

$0.021 \frac{mol}{L} \times 158.032 \frac{g}{mol} = 3.32\ g/L$

* [B] preciptation of Calcium as Calcium oxalate & titration with standard KMnO4 solution :- Unknown No :-

$n_{Ca^{+2}} = n_{C_2O_4^{-2}} = \frac{5}{2} n_{MnO_4^-} \Rightarrow 2MV_{Ca^{+2}} = 5MV_{MnO_4^-}$

$\Rightarrow 2 \times 10.00 \times 10^{-3} \times M_{Ca^{+2}} = 5 \times 14.6 \times 0.021 \times 10^{-3} \Rightarrow M_{Ca^{+2}} = 0.077\ M$

$M.m = 40.078\ g\ Ca/mol$

Concentration of Ca^{+2} in (g/L)

$0.077 \frac{mol}{L} \times 40.078 \frac{g}{mol} = 3.09\ g/L$

* Sources of error :-

1. Error while detecting the initial & final volume of the burette as a reason of the wrong position of eye.
2. Error while detecting the color change as the end point of titration.
3. The precipitate wasn't dissolved completely with hot water & H_2SO_4.
4. Error when weighing on balance

* Conclusion :-

Metal ions such as Ca^{+2}, Cu^{+2}, Pb^{+2} & Zn^{+2} give sparingly soluble oxalates & could be quantitavity determined by dissolving the washed precipitate in dilute sulfuric acid and titrate the librated oxalic acid with standard $KMnO_4$ solution. This method is widely used for determination of calcium.

Calcium is precipitated as the oxalate by addition of ammonium oxalate solution to a dilute HCl solution of Ca^{+2}. followed by neutralization of acid with dilute ammonia solution. The precipitat is washed, dissolved in dilute H_2SO_4 & the oxalic acid obtained is titrated with standard $KMnO_4$ solution.

*Questions:-

[1] Why methyle red is used?

- Because this indicator can detect the end point in our titration because methyl red indicator has a pH range from 4.4 to 6.3 and it as the same as pH when we reach the endpoint, the solution will be neutral slightly basic.

[2] Why the preciptate must be washed?

1- to get rid of Cl^- because it reacts with Kmno4 to give Cl_2 and it will cause to have an error (positive error)

2- to get rid of $C_2O_4^{-2}$ (Oxalate) because it reacts with H_2SO_4 to give $H_2C_2O_4$, which will consume more volume of the titrant (Kmno4) and be the cause of having ± error

[3] what are the possible sources of error in carrying out this experiement?

1. incomplete dissolving CaC_2O_4 by H_2SO_4.
2. Incomplete washing
3. incomplete filtration
4. incomplete precipitation of the solution
5. There was free chloride(Cl^-) of free oxalate ($C_2O_4^{-2}$)

Name:

Reg No:

locker No: 163

Course: Analytical Chemistry lab

Section No: 9

Date: 13-12-2023

exp No: 10

Title of experiement: Gravimetric Determination of Nickel as Bis(Dimethylglyoximato) Nickel(II)

Instructor: Dr-safwan Freihat

Unknown No: 30

9.5

Experiment 10
Data Sheet and Calculations

Name: Section: 9

Reg. No.: Date: 19-12-2023

Unknown ID No.: 39	
Volume of solution used	1000 ± 0.05
Mass of empty crucible	42.7429 ± 0.0001
Mass of empty crucible +Ni(DMG)$_2$	43.5848 ± 0.0001
Mass of Ni(DMG)$_2$ precipitate	0.8419 ± 0.0001
Mass of Ni	0.1710
Mass of Ni per liter solution (mg/L)	17100 mg/L

Instructor's Signature: .....

Instructor's Name: .....

12/12

Calculations:-

- Equations:

$Ni^{+2} + 2DMG \rightarrow Ni(DMG)_2 + 2H^+$

- Equations that show the calculation employed in computing the results:

mass of crucible + $Ni(DMG)_2$ − mass of empty crucible = mass of $Ni(DMG)_2$

$1mol\ Ni(DMG)_2 : 1mol\ Ni^{+2}$

$no\ of\ moles = \frac{mass}{molar\ mass}$

* Calculate of result and their Precision:

M.m of $Ni(DMG)_2 = 288.9146\ g/mol$

M.m of $Ni^{+2} = 58.6934\ g/mol$

$m_{Ni(DMG)_2} = 0.8419 \pm 0.0001$

$n_{Ni(DMG)_2} = \frac{0.8419}{288.9146} = 2.9 \times 10^{-3}\ mol$

$1mol\ Ni(DMG)_2 \rightarrow 1mol\ Ni^{+2}$

So: $2.9 \times 10^{-3}\ mol\ Ni(DMG)_2 \rightarrow 2.9 \times 10^{-3}\ mol\ Ni^{+2}$

mass of $Ni^{+2} = 2.9 \times 10^{-3} \times 58.6934\ (g/mol)\ Ni^{+2}$

$= 0.1710\ g\ Ni^{+2}$

- Concentration: $Ni^{+2}(mg/L) = \frac{0.171 \times 10^3\ mg}{10.00 \times 10^{-3}\ L} = 17100\ mg/L$ of Ni^{+2}

- Source of Error:

1. not completely filteration.
2. error in balance perhaps it wasn't calibrated well
3. the precipitate wasn't washed well.
4. the preciptate wasn't heated for an enough time.

- conclusion:

the gravimetric determination of a substance, the precipitating agent should be as specific as possible for that substance: DMG has to specify to precipitate only palladium from acidic solution & only Ni From weakly basic Soln.

In ammonical soln, Ni is precipitated quantitatively by DMG as bright strawberry colored complex ($Ni(DMG)_2$)

Problems:-

(1) Why precipitation is carried out in an ammonical solution?

Because in ammonical solution Ni^{+2} will be precipitated by DMG

$$Ni^{+2} + 2DMG \rightleftharpoons Ni(DMG)_2 + NH_4OH + 2H^+$$

The slow addition of aqueous amonia increases the PH to have a basic solution & slowly decreases in solubility of the complex & the precipitate is formed.

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

Because $Ni(DMG)_2$ is bulky & tends to creep up the walls of the container, if we add an extra amount of DMG we will need an excess amount of ethanol in order to remove it, this will be the reason to stablize the precipitate & decrease the yield.

(3) Suggest a way for transferring traces of $Ni(DMG)_2$ that stick to the sides of the beaker into the sintered glass crucible

- Use warm water & then use 30% ethanol.

(4) Why 30% ethanol is used for washing?

- (1) In order not to have peptization when washing the precipitate & ethanol is a volatile substance which is good for washing
- (2) to get rid of an excess DMG from the precipitate.

Experiment 11
Data Sheet and Calculations

Name: Section:
Reg. No.: Date:

Unknown ID No.: 42		
Trial	Volume of 0.02 M NaOH	Volume of unknown
1	19.10 ±0.05 mL	10.00 mL ±0.05 ml
2	mL	mL
3	mL	mL
	Average: ..... mL	

- Number of mmols of H^+: $0.02 \times 19.10 = 3.82 \times 10^{-4}$

- Number of mmols of displaced (exchanged) Ca^{2+} per liter of the unknown: 1.91×10^{-4}

- Concentration of Ca^{2+}: $\frac{1.91 \times 10^{-4} \times 40.078}{0.010}$ ppm: 0.77×10^3 $\frac{mg}{L}$

Instructor's Signature: .....

Instructor's Name: .....

Experiment 12

Data Sheet and Calculations

Name: Section:
Reg. No.: Date:

A. PURE COMPONENTS

Component	x_s	x_c	$x_c/x_s = R_f$
Cargo red	6.1	2.2	0.36
Phenyl red	6.1	2.0	0.33
Bromothymol blue	6.1	3.4	0.56
Methyl orange	6.1	3.0	0.49

x_s = Distance of the solvent front, x_c = Distance of the component

B. UNKNOWN SAMPLE

Distance of the solvent front _____ cm (x_s)

Spot Nr.	X_c	$R_f = X_c/X_s$	SpotIdentification
1	3.3	0.54	Bromothymol blue
2			
3			
4			

Instructor's Signature: .....

Instructor's Name: .....