School of Chemical Sciences



ACID-BASE TITRATION AND CATION TEST HANDBOOK

http://chem.usm.my

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LIST OF AWARDS

RECEIVED GOLD AWARDS FOR:





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INTRODUCTION

This handbook is prepared in order to integrate the microscale approach into the chemistry curriculum of Malaysian secondary schools taking into consideration the limited funding in terms of chemicals, glassware and laboratory facilities.

The programme aims at reaching out to students as well as teachers to become involved in chemistry as a subject in order to increase their interest in science besides promoting science in a smaller scale. In our part, miniaturized or microscale chemistry is part of our attempts to solve problems related to limited chemicals. Besides attending lectures on selected topics, teachers will also be involved in conducting new experiments using USM new glass-based miniaturized microscale kits developed by our staff.

COMPONENTS	IN	THE	KIT
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Components	Quantity
Acetate sheet	1
Aluminium micro stand	1
Combo plate (24 well)	1
Micro burette (2mL)	2
Nail and clip	2
Propette	4
Syringe (3mL)	2
Toothpick	10

COMPONENTS	IN	THE	KIT
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Chemicals	Volume (mL)
1 mol dm ⁻³ Al(NO ₃) ₂	5
1 mol dm ⁻³ Zn(NO ₃) ₂	5
1 mol dm ⁻³ Pb(NO ₃) ₂ ,	5
1 mol dm ⁻³ CuSO ₄	5
1 mol dm ⁻³ NaOH	5
2 mol dm ⁻³ NH3	5
1 mol dm ⁻³ KI	5
0.100 mol dm ⁻³ HCl	10
0.1 mol dm- ³ NaOH	10
Phenolphthalein solution	5

Exp 1: Acid-Base Titration

Determining the end point of the titration between hydrochloric acid (HCl) and sodium hydroxide (NaOH) solution using an acid-base indicator.

Objective:

To determine the end point of the titration between HCl and NaOH solution using an acid-base indicator

Engagement:

In order to attract the students' attention regarding this topic, the teacher will give an activity on identifying acids and bases as follows:

- In front of the classroom, the teacher will hold two cups, one filled with vinegar and the other with detergent. The teacher will say, "I have a cup of vinegar and a cup of detergent.
- After that, the teacher will ask the following questions to the students:
 - Is vinegar considered an acid or a base?
 - Is detergent considered an acid or a base?
 - What happened when vinegar and detergent combined?

- To determine if the students' guesses are correct, the teacher will test the vinegar and detergent with litmus papers (blue and red).
- The blue litmus paper turned red signifying that the vinegar is acidic and the detergent turned the red litmus paper into blue signifying that it is basic (alkaline).
- **Conclusion:** Vinegar is acidic and detergent is basic. Example of acid is hydrochloric acid, HCl and example of a base is hydroxide, NaOH. When acid and base combined, the acidity of an acid is neutralized by an alkali and vice versa. This is known as neutralization reaction.

Explore:

The students are asked to set up the experiment.

Apparatus:

1 comboplate, 1 microstand, 2 microburette, 1 syringe, 1 microspatula, 2 propettes, 2 set of nail and clips and 5 toothpick.

Chemicals:

0.100 mol dm⁻³ hydrochloric acid, HCl, 0.1 mol dm⁻³ sodium hydroxide, NaOH, phenol-phthalein solution.

Microscale chemistry kit set-up:



Procedure:

- Assemble the microburette as shown in figure above. Push the microstand into a well of the comboplate. Orient the pairs of arms on the central stem of the microstand so that one arm of each pair is directly above well. Clip the assembled microburette into each arm of the microstand above well.
- 2. Put the comboplate on a piece of white paper.
- 3. Rinse the 2 cm³ microburette with the sodium hydroxide solution in the following way:
 - i. Set up the microburette by attaching the plastic syringe to the top of the 2 cm³ microburette.
 - Rinse the microburette with sodium hydroxide. Use the syringe to fill up the microburette. Repeat the rinsing process twice.
- 4. Fill the rinsed microburette with exactly 1.00 cm³ of the sodium hydroxide solution. Dispense all this solution into a well (E1) of the comboplate. Repeat this step twice, dispensing the 1.00 cm³ aliquots of sodium hydroxide into two other different wells (E2 and E3).
- 5. Empty the microburette, rinse it with water.

- 6. Rinse the microburette at least three times with the hydrochloric acid, HCl.
- Fill the microburette with the HCl to the 0.00 cm³ level (or there about). Read the level of the meniscus and record this as the initial volume of HCl (titration 1).
- 8. Use the thin stemmed propette to add one drop of phenolphthalein solution to the NaOH in the E1.
- 9. Position the microburette above the comboplate so that the tip of the microburette is above E1.

Note Do not place the microburette too close to well as the microspatula may knock against it during stirring of the solution in the well. This may cause drops of the solution to splash out of the well.

10. Push down gently on the syringe plunger and add one drop of HCl into E1. Stir the solution in E1 with a toothpick. Be careful not to spill any solution out of the well. Leave the microspatula in the well during the titration.

- 11. Continue to add HCl from the microburette one drop at a time until the indicator changes color from pink to colorless. Stir the solution in E1 after each drop is added.
- 12. Observe the volume of HCl in the microburette and record this as the final volume. Calculate the volume of HCl dispensed.
- 13. Refill the microburette with the HCl and record the initial volume (Titration 2).
- 14. Add one drop of phenolphthalein indicator to the NaOH in E2. Position the microburette above E2 by moving the microstand.
- 15. Now, that you know the approximate volume of the HCl required to titrate the NaOH, you can add the HCl a little more quickly than before until about 0.04 cm³ before the expected end point. Stir thoroughly.
- 16. Add the HCl slowly, one drop at a time with stirring, until the indicator changes from pink to colorless. Record the final volume (Titration 3).
- 17. Repeat the titration in E3.
- 18. Reject any results where the end point has been overshot.

Data and observations:

Titration No.	1	2	3
	Volume	e of HC	l/cm ³
Final burette reading (cm ³)			
Initial burette reading (cm ³)			
Volume of hydrochloric			
acid, HCl needed (cm ³)			

Average volume of HCl used:

Conclusions: Questions:

- 1. a) What is the average volume of HCl needed to neutralize 25.0 cm^3 of NaOH?
 - b) Write the equation for the neutralization reaction.

2. a) How is the end point of the titration determined?

b) How else could you determine the end point?

- 3. Explain the following
 - a) A clean burette must be rinsed with a little acid before filling it up.
 - b) There must be no air bubbles in the tip of the burette.
 - c) Burette readings must be taken at eye level.
 - d) There is no need to rinse the conical flask with NaOH.

Explain:

The teacher explains to the students the followings points:

The reaction of an acid and a base is called a neutralisation reaction. In this reaction, the acidity of an acid is neutralised by an alkali. At the same time, the alkalinity of the alkali is neutralised by the acid. A salt and water are the only products of a neutralisation reaction.

It is a general term for a method in a quantitative chemical analysis in which the amount of a substance is determined by the measurement of the volume that the substance occupies. It is commonly used to determine the unknown concentration of a known reactant. Volumetric analysis is often referred to as titration, a laboratory technique in which one substance of known concentration and volume is used to react with another substance of unknown concentration.

Acid-base titration

Titration is a very useful laboratory technique in which one solution is used to analyse another solution. One of the solutions is a standard solution of known concentration and is delivered from a burette. The technique involves determining accurately the volume of the standard solution needed to react exactly with a known volume of another solution contained in a conical flask in a reaction for which the equation (stoichiometry) is known. The completion of the reaction occurs when sufficient standard solution is added from the burette to react exactly with another solution in a flask.

End point

The point at which the reaction is observed to be completed is the end point. The end point in the volumetric method of analysis is the signal that tells the analyst to stop adding reagent and make the final reading on the burette. The endpoint is observed with the help of indicators such as phenolphthalein, methyl orange, litmus solution or universal indicator solution. Example: Neutralization reaction between sulphuric acid, H₂SO₄ and sodium hydroxide, NaOH.

a) H⁺ ion from sulphuric acid neutralises the OH⁻ ion from sodium hydroxide

$H^+ + OH^- \rightarrow H_2O$

- b) As more H⁺ ion is added, the neutralization reaction decreases the concentration of OH⁻ ion in the flask. Hence, the color of the indicator becomes paler.
- c) At the end point, all OH⁻ ions in the conical flask are neutralised by H⁺ ions. The indicator turns colorless.
- d) The products of neutralisation are sodium sulphate and water. The flask contains aqueous sodium sulphate. Mobile ions, Na^+ and SO_4^{2-} are present in the solution.

The burette is rinsed with a small amount of sulphuric acid to ensure and prevent water from diluting the acid as it is poured into the burette. The burette reading should be taken with an eye placed at the same level as the meniscus to ensure the accurate volume is recorded.

Elaboration:

The teacher will use dialogue with the students:

Now we know that neutralization is a type of chemical reaction in which a strong acid and strong base react with each other to form water and salt. Have you ever been unlucky enough to be stung by a wasp or a bee? Bee stings are acidic in nature, which is why a household remedy for a bee sting is baking soda or sodium bicarbonate, which is a basic substance. A wasp sting, on the other hand, is mildly basic, so a household remedy for this will be vinegar, also known as acetic acid. These simple treatments ease these painful stings by a process called neutralization.

Heartburn, as well as an acidic stomach due to eating too much spicy food, can be relieved by taking an antacid. The antacid is alkaline/basic and helps neutralize the stomach's acidic environment. You may have used medicine to ease your heartburn via the process of neutralization.

By applying this format to that of a neutralization reaction, it will look like:

$$AB + CD \rightarrow AD + CB$$

 $HF + KOH \rightarrow H(OH) + KF$

 $HF + \ KOH \rightarrow \ H_2O + \ KF$

Example:

Write an equation to show the neutralization reaction between strong acid solution and strong basic solution.

- 1. HCl and NaOH
- 2. HBr and KOH

Evaluate:

Teacher shows a video regarding acid-base titration. https://youtu.be/dLNsPqDGzms

Now, it is time, to sum up, our lesson. What we can conclude from the experiment is that when strong acid solution combines with a strongly basic solution it will produce salt and water through a chemical reaction called Neutralization. For gathering students' understanding regarding acidbase titration, the teacher gives simple quizzes to the students.

1. Which of the following equations best represents a neutralization reaction?

A.
$$Ba(SO_4)_2(aq) + Na_2SO_4(aq) \rightarrow BaSO_4(s) + 2NaOH(aq)$$

- B. $CH_3COOH(aq) + NH_3(aq) \rightarrow CH_3COO^-(aq) + NH_4(aq)$
- C. $Cl_2(g) + H_2O(l) \rightarrow HOCl(aq) + HCl(aq)$
- D. $P_4(s) + 3OH^{-}(aq) + 3H_2O(l) \rightarrow PH_3(g) + 3H_2(g) + 3H_2PO_2^{-}$
- 2. Identify the salt in the following reaction:

 $KOH_{(aq)} + HBr_{(aq)} \rightarrow H_2O_{(l)} + KBr_{(aq)}$

- A. KOH
- B. HBr
- C. H₂O
- D. KBr

- 3. An indicator
- A. changes color over a pH range
- B. is usually a weak acid that changes color when it loses a proton
- C. should be chosen so that its color changes over a pH range that includes the pH at the equivalence point of the titration
- D. all of the above is true.
- 4. The end point in a titration occurs
- A. when the acid and base present completely react with each other
- B. when the solution has a pH equals to 7
- C. when the indicator changes colour
- D. when the pH of the solution no longer changes

Exp 2: Cations Test

Objectives:

- 1. To conduct a reaction between cation in aqueous solution with sodium hydroxide and ammonia
- 2. To conduct cations test for Al^{3+} , Zn^{2+} , Cu^{2+} , Pb^{2+} cations

Engage:

A teacher shows several compounds such as table salt, fertilizer and asks students:

- 1. What is a chemical name for table salt and fertilizer?
- 2. Please identify the cation and anion for table salt and fertilizer?
- 3. How can you identify that table salt contained sodium (Na⁺) ion and ammonium (NH₄⁺) ion?
- Na^+ and NH_4^+ ions can be identified through a cation test.

Explore:

Apparatus:

Acetate paper, toothpick

Chemicals:

1 mol dm⁻³ Al(NO₃)₂, 1 mol dm⁻³ Zn(NO₃)₂, 1 mol dm⁻³ Pb(NO₃)₂, 1 mol dm⁻³ CuSO₄, 1 mol dm⁻³ NaOH, 2 mol dm⁻³ NH₃, and 1 mol dm⁻³ KI.

Procedure:

- 1. Use a marker to write down the cations and reagents that will be used for the tests.
- Put 1-2 drops of cation solutions (Al³⁺, Zn²⁺, Cu²⁺, Pb²⁺) on an acetate (plastic) sheet (in appropriate boxes).
- **3.** Add 1-2 drops of reagents (NaOH, NH₃, and Potassium Iodide) and stir with a toothpick.
- 4. If a precipitate forms, record its color.
- 5. Add another 2 drops of reagent and stir with the same toothpick.
- 6. Does the precipitate dissolve in excess reagent? Record your observation.
- 7. Repeat steps 1 to 5 using different cation solutions.

Data and observations:

Conclusions:

Explain:

The teacher explains to the students:

Cations can be identified by their reactions with aqueous sodium hydroxide and aqueous ammonia.

Sodium hydroxide and aqueous ammonia produce hydroxide ion which will react with most anions to form a precipitate.

$$NaOH + H_2O \rightarrow Na^{\scriptscriptstyle +} + 2OH^{\scriptscriptstyle -} + H^{\scriptscriptstyle +}$$

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^-$$

Different cations like aluminium Al³⁺, calcium Ca²⁺, copper(II) Cu²⁺, iron(II) Fe²⁺, iron(III) Fe³⁺, lead(II)

Pb²⁺, zinc Zn²⁺ produce different colored precipitates, which may or may not dissolve in excess alkali. Zn(OH)₂, Al(OH)₃ and Pb(OH)₃ dissolve in excess NaOH solution, this is because Zn(OH)₂, Al(OH)₃ and Pb(OH)₃ are amphoteric, they can react with NaOH to form salt and water ans the reactions are described by the equations below:

 $Zn(OH)_2 + 2NaOH \rightarrow Na_2ZnO_2 + 2H_2O$ $Al_2(OH)_3 + 3NaOH \rightarrow Na_3Al_2O_3 + 3H_2O$ $Pb(OH)_2 + 2NaOH \rightarrow Na_2PbO_2 + 2H_2O$

The table below shows the summary of the precipitate form by a different cation.

	NaOH(aq)	NH ₃ (aq)	
Na ⁺	-	-	
Ca ²⁺	White precipitate.	-	
Mg^{2+}	White precipitate.	White precipitate.	
Al ³⁺	White precipitate.		
	Dissolve in	White precipitate	
	excess NaOH	white precipitate.	
	solution.		

	NaOH(aq)	NH ₃ (aq)
	White	
	precipitate.	
Al ³⁺	Dissolve in	White precipitate.
	excess NaOH	
	solution.	
	White	
	precipitate.	White precipitate.
\mathbf{Zn}^{2+}	Dissolve in	Dissolve in
	excess NaOH	excess NH3 solution.
	solution.	
	White	
	precipitate.	
Pb ²⁺	Dissolve in	White precipitate.
	excess NaOH	
	solution.	
Cu ²⁺		Blue precipitate.
		Dissolve in
	Blue precipitate.	excess NH ₃ solution
		and form a blue
		solution.

Elaborate:

How do you differentiate the presence of Cu^{2+} in $CuCl_2$ and Pb^{2+} in $PbSO_4$?

Evaluate

You have salt X, salt Y, salt Z in your home. Salts X and Z are white in color while salt Y is dark green. Salts X and Y are useful in sewage and waste watertreatment. Salt Z is useful to grow plants. When salt X and Y dissolved in NaOH, salt X formed dirty green precipitate and salt Y formed a red-brown precipitate. Salt Z dissolved in NaOH. What is your observation when salt X, Y and Z dissolved in ammonia? Which salt consist $NH_4^+Fe^{2+}$ and $Fe^{3+?}$

SAFETY PRECAUTIONS

1 mol dm⁻³ Al(NO₃)₂

Precautions: Wash face, hands and any exposed skin thoroughly after handling Wear protective gloves/protective clothing/eye protection/face protection. Keep away from heat/sparks/open flames/hot surfaces. - No smoking Keep/Store away from clothing/ other combustible materials. Take any precaution to avoid mixing with combustibles Skin: Wash with plenty of soap and water If skin irritation occurs: Get medical advice/attention Take off contaminated clothing and wash before reuse Eyes: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing, If eye irritation persists: Get medical advice/attention.

Storage: Keep containers tightly in a dry, cool and well-ventilated place. Do not store near combustible materials.

Personal protection: Wear personal protective equipment. Ensure adequate ventilation. Avoid dust formation. Do not get in eyes, on skin, or on clothing. Avoid ingestion and inhalation. Keep away from clothing and other combustible materials.

1 mol dm⁻³ Zn(NO₃)₂

Precautions: If medical advice is needed, have product container or label in hand. Keep out reach of children. Read label before use. Keep away from heat/sparcks/open flames/hot surfaces. No smoking. Keep/Store away from clothing/combustible materials. Take any precaution to avoid mixing with combustible materials. Skin: rinse cautiously with water for several minutes. Remove contact lenses if present. Continue rinsing and if eye irritation persists, get medical advice/attention. Inhaled: Remove victim to fresh air and keep at rest in a position comfortable for breathing. Call Poison Centre or doctor/physician, if you feel unwell.

Storage: Store in a well ventilated place. Keep container tightly closed.

Personal protection: Wear protective gloves/protective clothing/eye protection/face protection. Wash skin thoroughly after handling. Wear protective gloves/protective clothing/eye protection/face protection. Avoid breathing dust/fume/gas/mist/vapours/spray. Use only outdoor or in a well-ventilated area.

1 mol dm⁻³ Pb(NO₃)₂

Precautions: If medical advice is needed, have product container or label in hand. Keep out reach of children. Read label before use. Keep away from heat/sparks/open flames/hot surfaces. No smoking. Keep/Store away from clothing/combustible materials. Take any precaution to avoid mixing with combustible materials. Wash skin thoroughly after handling. Use only in outdoors or in a well-ventilated area.

Storage: Store in a well ventilated place. Keep container tightly closed.

Personal protection: Use personal protective equipment. Ensure adequate ventilation. Avoid dust formation. Avoid contact with skin, eyes and clothing.

1 mol dm⁻³ CuSO₄

Precautions: Wash face, hands and any exposed skin thoroughly after handling Do not eat, drink or smoke when using this product Wear protective gloves/protective clothing/eye protection/face protection Skin IF ON SKIN: Wash with plenty of soap and water If skin irritation occurs: Get medical advice/attention Take off contaminated clothing and wash before reuse Eves IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing If eye irritation persists: Get medical advice/attention Ingestion IF SWALLOWED: Call а POISON CENTER or doctor/physician if you feel unwell Rinse mouth.

Storage: Keep containers tightly closed in a dry, cool and well-ventilated place. Store under an inert atmosphere.

Personal protection: Use personal protective equipment. Ensure adequate ventilation. Avoid dust formation. Avoid contact with skin, eyes and clothing.

0.1 mol dm⁻³ / 1 mol dm⁻³ NaOH

Precautions: If medical advice is needed, have product container or label in hand. Keep out reach of children. Read label before use. Do not breathe dust/fume/gas/mist/vapours/spray. Wash thoroughly after handling. Wear protective gloves/protective clothing/eye protection/face protection. Absorb spillage to prevent material damage.

Storage: Protect form freezing and physical damage. Provide ventilation for containers. Avoid storage near extreme heat and incompatible materials. Store in original container. Do not store in metallic containers. Keep containers tightly closed and upright.

Personal protection: Wear safety glasses with side shields or googles and a face shield. Wear appropriate chemical resistant clothing (with long sleeves) and appropriate chemical resistant gloves.

2 mol dm⁻³ NH₃

Precautions: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking. Do not breathe gas and vapours. Wash hands thoroughly after handling. Avoid release to the environment.

Storage: Store in well ventilated place and keep container tightly closed. Store locked up.

Personal protection: Wear protective clothing, eye protection, face protection.

1 mol dm⁻³ KI

Precautions: Do not breathe mists. Do not ingest. Do not get in eyes, on skin or on clothing. Wash well after use. Do not allow smoking and food consumption while handling. Sweep up immediately to eliminate slippery hazard.

Storage: Store in a cool, dry and well-ventilated area. Keep container tightly closed. Keep away from heat, sparks and flame. Protect against physical damage. Store away from incompatible materials (attack metals in presence of moisture).

Personal protection: Wear protective clothing, eye protection, face protection.

0.1 mol dm⁻³ HCl

Precautions: Wash hands and other exposed areas with mild soap and water before eating. Provide good ventilation in process area to prevent formation of vapour. Do not breathe mist, vapours and spray.

Storage: Store in a cool, dry, ventilated area away from incompatible materials. Store in original container. Keep containers tightly closed and upright. Keep away from food and drink. Keep out of the reach of children.

Personal protection: Wear safety glasses with side shields or googles and a face shield. Wear appropriate chemical resistant clothing (with long sleeves) and appropriate chemical resistant gloves.

Phenolphthalein solution

Precautions: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking. Keep away from food and drink. Do not breathe gas and vapours. Use only non-sparking tools. Take precautionary action against static discharge. Wash hands thoroughly after handling.

Storage: Keep only in original container in a cool, well ventilated place away from heat sources, ignition sources and incompatible materials. Keep container tightly closed.

Personal protection: Wear safety glasses with side shields or googles and a face shield. Wear appropriate chemical resistant clothing (with long sleeves) and appropriate chemical resistant gloves.

Chemical Safety Data Sheet (SDS)

No.	Chemicals	Link for SDS
1.	$1 \text{ mol dm}^{-3} \text{Al}(\text{NO}_3)_2$	https://bit.ly/2DVFdrp
2.	1 mol dm ⁻³ Zn(NO ₃) ₂	https://bit.ly/2SZvFoZ
3.	$1 \text{ mol dm}^{-3} Pb(NO_3)_2$	https://bit.ly/2SOeLdC
4.	1 mol dm ⁻³ CuSO ₄	https://bit.ly/2Pou9HO
5.	1 mol dm ⁻³ NaOH	https://bit.ly/2G8F8Eh
6.	$2 \text{ mol dm}^{-3} \text{NH}_3$	https://bit.ly/2tdR5QN
7.	1 mol dm ⁻³ KI	https://bit.ly/2hMCedv
8.	0.100 mol dm ⁻³ HCl	https://bit.ly/2mc4wiN
9.	0.1 mol dm ⁻³ NaOH	https://bit.ly/2G8F8Eh
10.	Phenolphthalein solution	https://bit.ly/2GPk05u

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