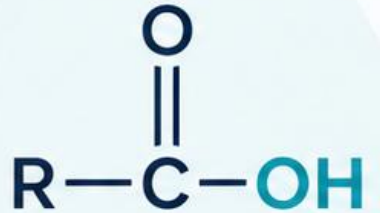
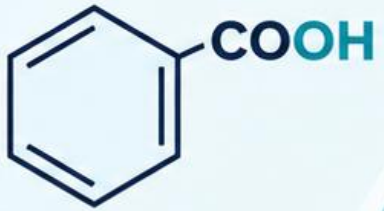
Organic Chemistry II Lab

Identification of Carboxylic Acids

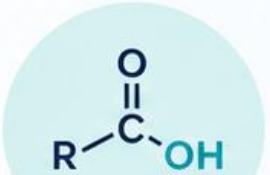
Aliphatic & Aromatic







From functional group → chemical evidence → confident identification



Functional group

→



Chemical evidence

→



Confident identification

2nd Stage

Asst. Lect. Gheith M. Abbas Alasadi
M.Sc. Pharmaceutical Chemistry

One integrated lecture

Opening Challenge: Two Unknown Acids

You have 60 seconds. Which test would you choose first — and what evidence would change your decision?

UNKNOWN A

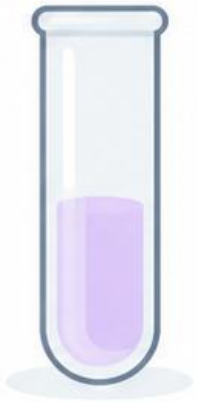


Clues

- Decolourizes alkaline $KMnO_4$
- Positive Tollens-type reducing behaviour
- $FeCl_3$ test: red solution

Your hypothesis: _____

UNKNOWN B



Clues

- $FeCl_3$ test: violet solution
- Aromatic –COOH + phenolic –OH
- Forms methyl salicylate on esterification

Your hypothesis: _____

Think → Pair → Share: name one observation that is more diagnostic than “it is acidic.”

General Objective and Learning Map

One lecture: structure → testing strategy → evidence-based identification.

General objective

By the end of this lab session, students will classify carboxylic acids, select appropriate identification tests, interpret visible evidence, and justify the identity of an unknown acid safely.

1. **Hook + Pre-test**
2. **Concept bridge**
3. **General test**
4. **Special tests**
5. **Group challenge**
6. **Post-test**

Engage → Explore → Explain → Practice → Evaluate

Teaching focus: decisions and evidence — not memorizing isolated reactions.

Behavioral Learning Objectives

By the end of this lab session, students will be able to…

Define
the carboxyl group and distinguish R–COOH from Ar–COOH.

Classify
common acids as aliphatic, aromatic, mono-, di-,or polycarboxylic.

Explain
why neutralization is required before the ferric chloride test.

Select
a suitable special test for formic, acetic, succinic, tartaric, oxalic, lactic, citric, or salicylic acid.

Interpret
colour change, precipitate, decolourization, fluorescence, and ester formation as chemical evidence.

Identify
an unknown carboxylic acid using at least two converging observations and safe laboratory practice.

Now, Let’s Move to Kahoot!

Pre-test activity — answer individually

1. Take out your phone or laptop
2. Scan the QR code or enter the game PIN
3. Answer all questions within the time limit



Ready? Let’s begin.

Concept Bridge: One Functional Group, Different Chemical Clues

Carboxylic acids share –COOH, but the rest of the molecule controls their diagnostic behaviour.

ALIPHATIC

R—COOH

R–COOH

Examples

Formic • Acetic • Succinic • Oxalic • Tartaric • Lactic • Citric

- May show redox, precipitation, fluorescence or iodoform behaviour.

AROMATIC



Ar–COOH

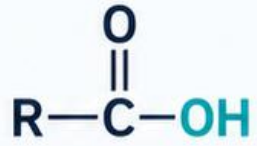
Examples

Benzoic acid • Salicylic acid

- The aromatic ring and extra substituents can create distinctive colour or complexation clues.
- Salicylic acid also contains a phenolic –OH group.

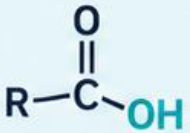
Diagnostic Strategy: Do Not Guess from One Test

Build an identification from a sequence of evidence.

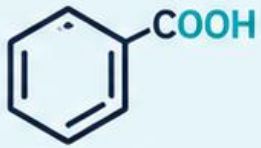


1 CLASSIFY

Is it aliphatic or aromatic?
How many –COOH / –OH groups?



Aliphatic
R–COOH



Aromatic
Ar–COOH

2 GENERAL TEST

Prepare the neutral solution, then add $FeCl_3$.



Neutral solution ($–COO^-$)

Add $FeCl_3$

3 SELECTIVE TEST

Choose a test suggested by structure and first observation.

- Reducing tests (Formic acid)
- Ester formation (Acetic or Salicylic acid)
- Succinic acid test (Fluorescence)
- Calcium chloride test (Tartaric acid)

4 CONFIRM

Require a second independent clue before naming the unknown.



Key point

One **colour** or one test alone is not sufficient for identification. Use a sequence of tests and a consistent pattern of observations.

General Test: Why Neutralize Before $FeCl_3$?

The test works best when the acid is converted to its carboxylate/ammonium salt and excess ammonia is removed.



1 DISSOLVE

Prepare the sample solution



Dissolve ~0.5 g of the acid in water.

2 NEUTRALIZE

Add dilute NH_3 dropwise



Add dilute NH_3 dropwise with shaking until the solution becomes basic (indicator changes colour).

3 REMOVE EXCESS NH_3

Heat gently



Heat gently until the excess ammonia is removed.
(Do not identify ammonia by smell.)

4 COOL

Return to room temperature



Cool the solution to room temperature before adding $FeCl_3$.

5 ADD $FeCl_3$

Add a few drops of $FeCl_3$ solution



Add a few drops of ferric chloride solution and observe the colour or precipitate.

Safety Update

- ✓ Use indicator paper/phenolphthalein to check neutrality.
- ✓ Do not identify ammonia by smelling.
- ✓ Perform heating only by the approved laboratory method.

Ferric Chloride Test: Read the Pattern

Use the observation to narrow the possibilities — then confirm with a special test.

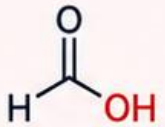
$FeCl_3$ (after neutralization)



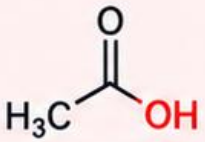
Formic / Acetic Acid

(Aliphatic, simple)





Formic acid
(HCOOH)



Acetic acid
(CH_3COOH)

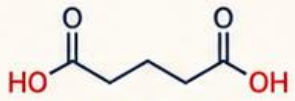
Red solution

Use as screening evidence (aliphatic acid).

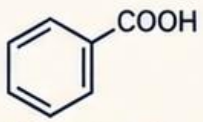
Succinic / Benzoic Acid

(Dicarboxylic / Aromatic)





Succinic acid
($HOOC–CH_2–CH_2–COOH$)



Benzoic acid
(C_6H_5COOH)

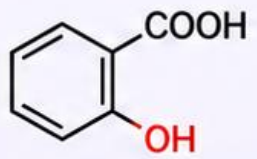
Light brown precipitate

Use as screening evidence.

Salicylic Acid

(Aromatic with phenolic –OH)





Salicylic acid
(o-Hydroxybenzoic acid)

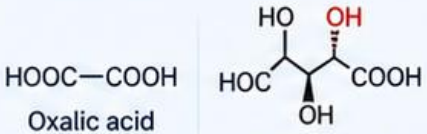
Violet solution

Highly distientive clue (phenolic –OH + aromatic).

Oxalic / Tartaric / Citric / Lactic Acid

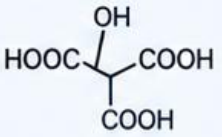
(Polycarboxylic or hydroxy acids)



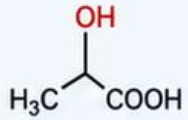


Oxalic acid

Tartaric acid



Citric acid



Lactic acid

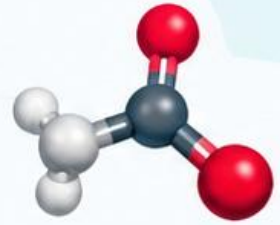
No special change

Use as screening evidence.

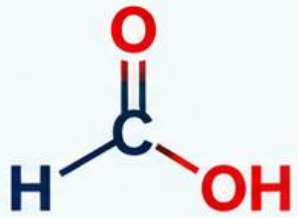
Key idea: $FeCl_3$ is a screening step. The identity becomes stronger when a second test agrees.

Formic Acid: The Reducing Carboxylic Acid

Structural clue: HCOOH can behave as a reducing agent because of the hydrogen attached to the carbonyl carbon.



STRUCTURE



Formic acid
(HCOOH)

Why is it a reducing acid?

The hydrogen attached to the carbonyl carbon can be easily oxidized, so HCOOH acts as a **reducing agent** with selected reagents.

1 $HgCl_2$ test



HCOOH + $HgCl_2$
(white precipitate)

Gray Hg
(metallic)

White Hg_2Cl_2 precipitate → gray Hg with excess acid/heating.

$$Hg_2Cl_2 + HCOOH \rightarrow 2Hg + 2CO_2 + 2HCl$$

2 Tollens' test



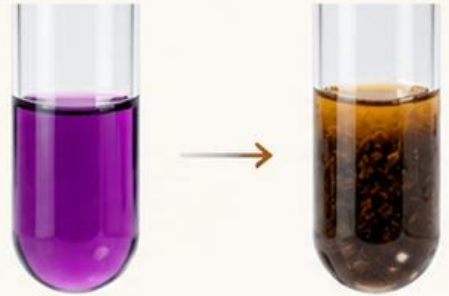
Tollens' reagent
($Ag(NH_3)_2^+$)
(colorless)

Silver mirror
(Ag^0)

Positive reducing response
(silver deposition/mirror).

$$HCOOH + 2[Ag(NH_3)_2]^+ + 2OH^- \rightarrow 2Ag + CO_2 + 4NH_3 + 2H_2O$$

3 Alkaline $KMnO_4$ test



Alkaline $KMnO_4$
(violet)

MnO_2 precipitate
(brown)

Violet colour disappears → brown MnO_2 precipitate.

$$5HCOOH + 2KMnO_4 + 3H_2SO_4 \rightarrow 5CO_2 + 2MnSO_4 + K_2SO_4 + 8H_2O$$

Diagnostic pattern: **$FeCl_3$ red + reducing behaviour → strongly supports formic acid.**

Acetic Acid: Confirm by Ester Formation

Acetic acid does not show the special reducing reactions of formic acid; ester formation becomes the key confirmation.

Esterification reaction

$$CH_3COOH + CH_3CH_2OH \xrightarrow[\text{warm (water bath)}]{\text{conc. } H_2SO_4} CH_3COOCH_2CH_3 + H_2O$$

Acetic acid (ethanoic acid) + **Ethanol** → **Ethyl acetate** (sweet, fruity smell)

Experimental steps

1 Mix reactants



- Add a small amount of acetic acid.
- Add ethanol.
- Add a few drops of conc. H_2SO_4 (catalyst).

2 Heat (water bath)



- Heat gently in a warm water bath for a few minutes.

3 Work up



- Add $NaHCO_3$ solution to neutralize the acid and remove excess H_2SO_4 (effervescence of CO_2).

4 Observation



- A pleasant, fruity odour **of ethyl acetate** is **produced.**
  (Do not smell directly.)

Why this test?

Formic acid: reducing tests positive (Tollens', alkaline $KMnO_4$, $HgCl_2$).

Acetic acid: those reducing tests are negative.

Ester formation: provides a different confirmation path for acetic acid.

Contrast, don't memorize. Use a different test to confirm the identity.

Safety note: Avoid direct smelling. | Use only a warm water bath. | Follow your instructor's SOP.

Succinic Acid: Fluorescence as a Visual Signature



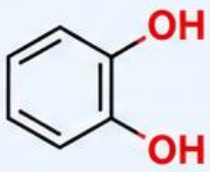
Resorcinol + acid conditions create a strongly coloured fluorescent product after alkalinization.

1 COMPOUNDS

$HOOC–CH_2–CH_2–COOH$

Succinic acid

+



Resorcinol

Key reagents

- Resorcinol
- Conc. H_2SO_4 (acid conditions)
- 10% NaOH (for alkalinization)

2 REACTION & HEATING



Mix substrate with resorcinol and conc. H_2SO_4.

Heat gently (using instructor-approved method).

3 ALKALINIZATION



Cool, then add excess 10% NaOH.

4 EXPECTED OBSERVATION

Red solution



After heating (in acid)

Green fluorescence



After adding NaOH (in base) under UV light

Diagnostic clue

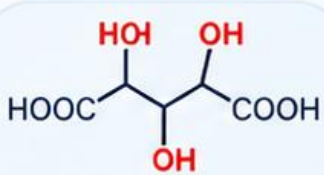
Red colour with green fluorescence after alkalinization.

Safety note

- ✔ Use only the instructor-approved heating method.
- ✔ Handle conc. H_2SO_4 and NaOH with care.

Tartaric Acid: A Three-Clue Diagnostic Pattern

Use multiple observations together — especially the Fenton and calcium tests.



Tartaric acid

1 $CaCl_2$ test

White calcium tartrate precipitate

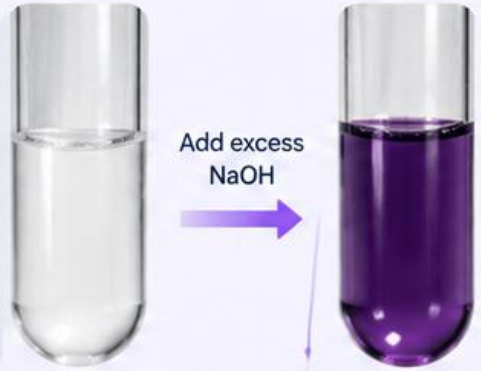


- Add $CaCl_2$ solution to the sample.
- A **white precipitate of calcium tartrate** forms.

Solubility test:
Precipitate dissolves in dilute HCl, not in dilute acetic acid.

2 Fenton's reagent (Fe^{2+}/H_2O_2)

Intense violet colour after excess NaOH



After Fe^{2+} + H_2O_2 (in acidic medium)

Intense violet colour (positive test)

- Add Fenton's reagent (Fe^{2+}/H_2O_2).
- Then add excess NaOH.
- A characteristic **intense violet colour** develops.

This is a positive and highly useful confirmation for tartaric acid.

3 Conc. H_2SO_4

Heavy charring with gas evolution



- Add a few drops of conc. H_2SO_4.
- Heat gently by the instructor-approved method.
- Heavy **charring** occurs with gas evolution (CO_2, H_2O).

Supportive test but less selective; follow safety SOP.

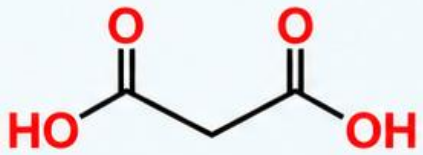
Best reasoning: Fenton positive + $CaCl_2$ precipitate with characteristic solubility pattern → tartaric acid.

Oxalic Acid: Acidic $KMnO_4$ Is the Key Discriminator

Compare its pattern with tartaric acid: similar $CaCl_2$ behaviour, but different redox/Fenton response.



Structure & key idea



Oxalic acid
(HOOC–COOH)

Acidic $KMnO_4$

Violet colour disappears on warming in acidic medium (reducing behaviour).

No corresponding reaction in alkaline $KMnO_4$ under this protocol.

Diagnostic tests for oxalic acid

1 $CaCl_2$ test

White precipitate; similar to tartaric acid



Oxalic acid solution (clear)

White calcium oxalate precipitate (CaC_2O_4)

Similar to tartaric acid ($CaC_4H_4O_6$ precipitate).

2 Fenton's reagent

Negative (no violet colour)



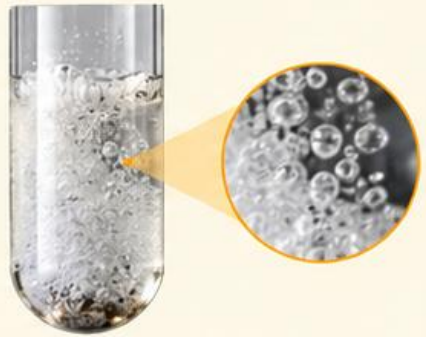
After Fe^{2+}/H_2O_2 (no violet colour)

Remains colourless (negative test)

Unlike tartaric acid, no intense violet colour develops.

3 Conc. H_2SO_4

Gas evolution with little darkening



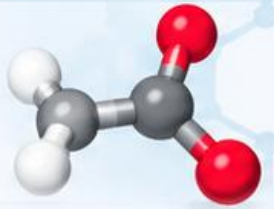
Effervescence (CO_2) with little darkening.

Supportive test but less selective; follow safety SOP.

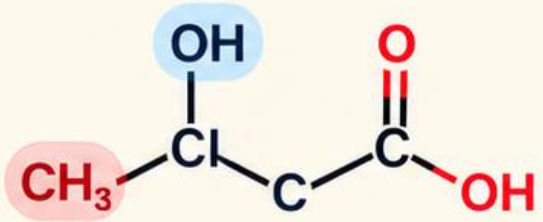
Decision clue: **Acidic $KMnO_4$ positive** (decolourization) + **Fenton negative** + **$CaCl_2$** precipitate → **oxalic acid.**

Lactic Acid: Iodoform Gives the Strongest Structural Clue

The α-hydroxy methyl pattern supports iodoform formation.



Structure and key feature



$CH_3–CH(OH)–COOH$

Lactic acid
(2-hydroxypropanoic acid)

Structural trigger for iodoform:

- Free methyl group ($CH_3–$)
- Hydrogen on the carbon bearing –OH (α-hydroxy methyl pattern)

This structural pattern supports iodoform formation.

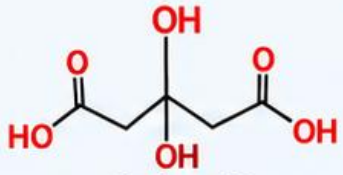
Evidence pattern (experimental tests)

1	**Iodoform test**	• Warm with I_2/NaOH. • Formation of yellow CHI_3 precipitate.	 Before	 Yellow CHI_3 precipitate	**Positive** → yellow CHI_3 precipitate
2	**$CaCl_2$ test**	• No white precipitate (does not form calcium lactate under these conditions).	 Before	 After (no ppt)	**Negative** No white precipitate
3	**Fenton's reagent**	• No intense violet colour (saturated α-hydroxy acid, not a reducing carbonyl).	 Before	 After (no colour)	**Negative** No violet colour develops
4	**Conc. H_2SO_4 test**	• Dehydration and charring with gas evolution (CO_2, H_2O).	 Before	 Charring with gas evolution	**Gas evolution** + considerable blackening

Key conclusion: Lactic acid gives a positive iodoform test (yellow CHI_3) and negative $CaCl_2$ and Fenton tests, with charring on conc. H_2SO_4 — consistent with the α-hydroxy methyl structure.

Citric Acid: Use the Sulfuric-Acid / Nitroprusside Sequence

The colour sequence helps distinguish citric acid from tartaric, oxalic, and lactic acids.



Citric acid

Experimental sequence

1 Conc. H_2SO_4



- Add a small amount of citric acid.
- Add conc. H_2SO_4 and warm gently (instructor-approved method).

2 Cool and dilute



- Cool the mixture.
- Dilute with water.
- Make alkaline with **NaOH** (pH ~ 8–10).

3 Add sodium nitroprusside



- Add sodium nitroprusside solution ($Na_2[Fe(CN)_5NO]$).
- An intense red colour develops.

4 Observation



Intense red colour (positive test)

Other clues (for comparison)

$CaCl_2$ test
Positive pattern (white calcium citrate precipitate).


White ppt.

Fenton's reagent
Negative (no violet colour).


No colour

Conc. H_2SO_4
Mixture turns yellow without charring; gas evolution occurs.


Yellow solution + gas evolution

Diagnostic end-point

Intense red colour after adding sodium nitroprusside (sulfuric-acid / nitroprusside sequence) → **citric acid.**

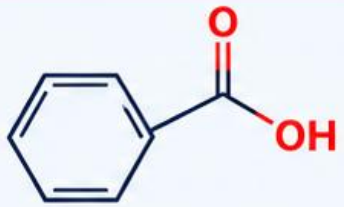
Safety note

- Use only the instructor-approved acid-heating method.
- Handle conc. H_2SO_4, NaOH, and sodium nitroprusside with care.

Aromatic Carboxylic Acids: Salicylic Acid Stands Out

Aromatic acids have –COOH directly attached to the aromatic ring; extra groups create additional diagnostic chemistry.

1 BENZOIC ACID



Benzoic acid
(C_6H_5COOH)

$FeCl_3$ outcome

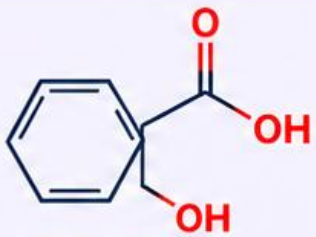


Light brown precipitate

- Forms a light brown precipitate with $FeCl_3$ (weak complex).
- No phenolic –OH group, so no intense violet colour.

Use additional evidence if the unknown must be confirmed.

2 SALICYLIC ACID



Salicylic acid
(o-hydroxybenzoic acid)

Key structural feature

- Aromatic –COOH directly attached to the ring.
- Additional phenolic –OH group (*ortho* position).
- This phenolic –OH gives characteristic reactions with $FeCl_3$.

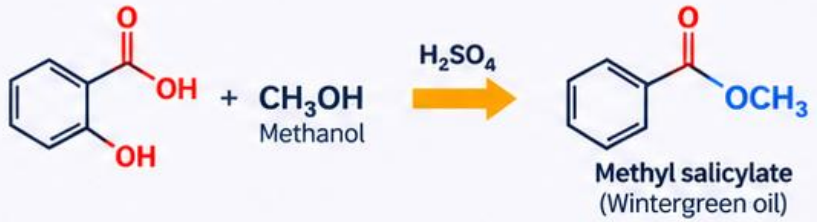
$FeCl_3$ test



Violet solution (positive)

- Phenolic –OH forms a violet complex with $FeCl_3$.
- Distinctive and useful for salicylic acid.

Esterification confirmation



Methyl salicylate
(Wintergreen oil)

Salicylic acid + methanol → methyl salicylate (ester formation confirms the identity).

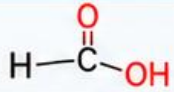
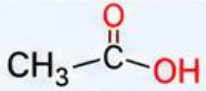
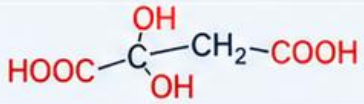
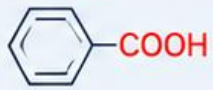
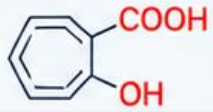
Safety note

- Methanol is toxic: never pipette by mouth.
- Avoid direct odour identification; use only the instructor-approved method.

From Observation to Identity: Compare the Evidence

The fastest route is to recognize patterns, not isolated facts.

Use at least **two observations** that agree to identify the acid.

Acid	Structure (R–COOH)	$FeCl_3$ clue	Best confirmation (key test)	Class
Formic acid		Red colour	**Reducing tests +** (e.g., Tollens / Fehling)	Aliphatic
Acetic acid		Red colour	**Esterification** (fruity odour)	Aliphatic
Succinic acid	$HOOC–CH_2–CH_2–COOH$	Light brown ppt	**Green fluorescence** (resorcinol + conc. H_2SO_4, then NaOH)	Aliphatic
Tartaric acid	$HOOC–CH(OH)–CH(OH)–COOH$	No special change	**Fenton violet + $CaCl_2$** (intense violet colour + white ppt)	Aliphatic
Oxalic acid	$HOOC–COOH$	No special change	**Acidic $KMnO_4$ + / Fenton –** (decolourizes $KMnO_4$; Fenton negative)	Aliphatic
Lactic acid	$CH_3–CH(OH)–COOH$	No special change	**Iodoform test +** (yellow CHI_3 precipitate)	Aliphatic
Citric acid		No special change	**Nitroprusside red / Fenton –** (after NaOH)	Aliphatic
Benzoic acid		Light brown ppt (weak complex)	**Aromatic –COOH** (characteristic aromatic behaviour)	Aromatic
Salicylic acid		Violet solution	**Methyl salicylate formation** (with methanol + H_2SO_4, **wintergreen oil**)	Aromatic

Rule: identify only when at least two observations point to the same candidate.

Student Practice: Build an Evidence Table

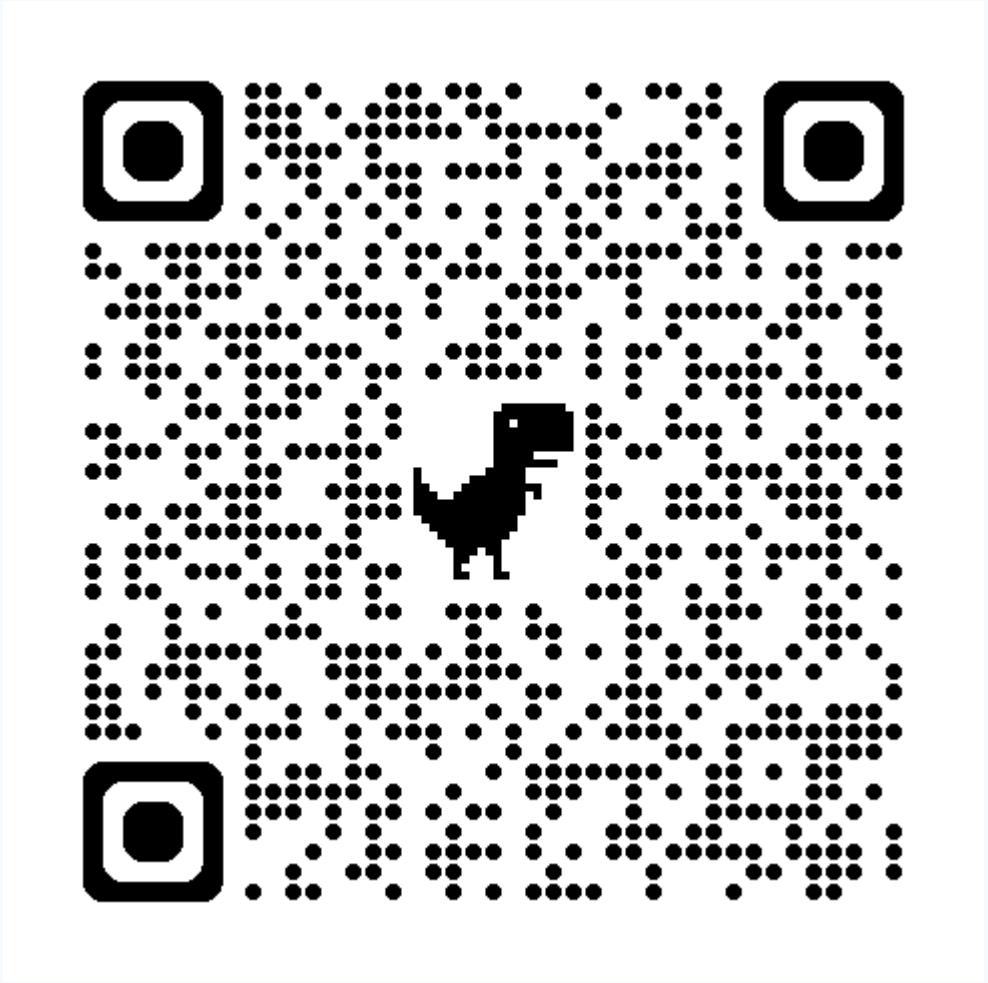
Students complete this during the experiment before writing the final identity.

Test / stage	Observation	Chemical meaning	Decision / next action
Initial classification			
$FeCl_3$ test			
Special test 1			
Special test 2			
Final identity			

Assessment rule: no final identity without process control + at least two converging observations.

Post-Test: Evidence of Learning

Students answer individually before leaving the lab.



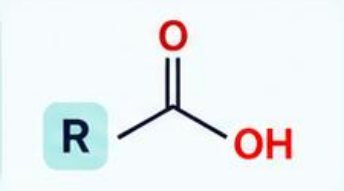
Lecture Summary: Three Things to Remember

Structure tells you what to test; evidence tells you what to conclude.

1 CLASSIFY

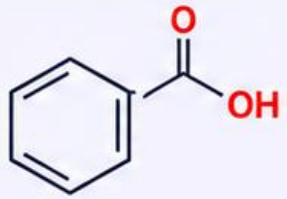
Use structure to predict tests.

Aliphatic (R–COOH)



- Use aliphatic acid tests
- Look for additional functional groups (–OH, C=O, etc.).

Aromatic (Ar–COOH)



- Aromatic ring present
- Use aromatic-specific evidence (e.g., $FeCl_3$, esterification).
- Check for extra groups (e.g., phenolic –OH).

2 TEST STRATEGICALLY

Use a logical sequence of tests.



- Start with general tests ($FeCl_3$).
- Use specific tests to discriminate between candidates.
- Consider colour changes, precipitates, and gas evolution.

3 JUSTIFY

Use multiple, converging observations before naming the unknown.



Exit ticket: Name one acid, its most useful special test, and the observation you would expect.

Thank You

Questions & Discussion



Think like an analytical chemist:

Move from evidence to a confident identification.

1	2	3	4	5
Classify	**Test**	**Observe**	**Confirm**	**Justify**
Use structure to predict likely tests.	Apply general and specific tests in order.	Record colour changes, precipitates and gas evolution.	Look for at least two converging results.	Combine the evidence to name the unknown.

Exit ticket:
Name one acid, its most useful special test, and the observation you would expect.

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