

Core practical 15: Analyse organic and inorganic unknowns

Objectives

- To research the tests for ions and organic compounds
- To successfully test unknown substances to establish their identity

Safety

- Wear goggles.
- Concentrated hydrochloric acid, sodium hydroxide and concentrated sulfuric acid are corrosive.
- Nitric acid, hydrochloric acid and limewater are irritants.
- Barium chloride and bromine water are toxic and irritants.
- Ethanol is flammable – do not use it near a lit Bunsen burner.
- Pay attention to the hazard warnings on the tubs and bottles of unknown substances. In particular, if anything is marked flammable, do not use anywhere near a lit Bunsen burner. If heating a flammable substance is required then it *must* be heated by standing it in a beaker of hot water.

Specification links

- Practical techniques 2, 4, 11
- CPAC 2a, 2b, 2c, 2d, 3a, 3b, 4a, 4b, 5a, 5b

Procedure**Identifying inorganic ions**

1. Look up how to carry out the flame test to identify metal cations.
2. Write a plan for the safe conduct of a flame-test experiment. You should describe in detail how you would carry out the experiment and the results you would expect.
3. Look up how to use sodium hydroxide to identify metal cations.
4. Write a plan for the safe conduct of this sodium hydroxide experiment. You should describe in detail how you would carry out the experiment and the results you would expect.
5. Look up how to use silver nitrate to identify halide ions.
6. Write a plan for the safe conduct of this silver nitrate experiment. You should describe in detail how you would carry out the experiment and the results you would expect.
7. Look up how to test for sulfate ions.
8. Write a plan for the safe conduct of this experiment to test for sulfate ions. You should describe in detail how you would carry out the experiment and the results you would expect.
9. Look up how to test for carbonate ions.
10. Write a plan for the safe conduct of this experiment to test for carbonate ions. You should describe in detail how you would carry out the experiment and the results you would expect.
11. Carry out your experiments to identify substances A–D. Pay particular attention to the management of safety.

Organic analysis

1. Look up how to test for alkenes.
2. Write a plan for the safe conduct of this experiment to test for an alkene. You should describe in detail how you would carry out the experiment and the results you would expect.
3. Look up how to test for an aldehyde.
4. Write a plan for the safe conduct of this experiment to test for an aldehyde. You should describe in detail how you would carry out the experiment and the results you would expect.
5. Look up how to test for carboxylic acids.
6. Write a plan for the safe conduct of this experiment to test for carboxylic acids. You should describe in detail how you would carry out the experiment and the results you would expect.
7. Carry out your experiments to identify substances X–Z. Take appropriate safety precautions.

Notes on procedure

- Students research tests for inorganic anions and cations and some organic substances. This work is revision.
- Students should be given four unknown inorganic substances labelled A–D and three unknown organic substances labelled X–Z. Their task is to identify these substances.
- Note that this practical will take more than one lesson to complete.

The tests that students should research and plan detailed methods for are given below.

Flame tests

1. Use a clean nichrome wire to mix a sample of a solid compound with a few drops of concentrated hydrochloric acid.
2. Hold the wire at the edge of a hot Bunsen burner flame and observe the colour.

Flame colour	Metal cation
yellow	sodium
lilac	potassium
yellow-red	calcium
pale green	barium
red	lithium, strontium
red-violet	rubidium

Using sodium hydroxide to identify cations

1. Dissolve the unknown substance in water.
2. Add aqueous sodium hydroxide, drop-by-drop, until there is no further change.

Metal ion solution	Observation on adding aqueous NaOH	Observation on adding excess aqueous NaOH
chromium(III) $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$	green precipitate	precipitate dissolves to a green solution
iron(II) $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$	green precipitate turning brown on exposure to air	precipitate is insoluble
iron(III) $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$	red-brown precipitate	precipitate is insoluble
cobalt(II) $[\text{Co}(\text{H}_2\text{O})_6]^{2+}$	blue precipitate turning pink on standing	precipitate is insoluble
copper(II) $[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$	blue precipitate	precipitate is insoluble
Group 2 cations: $\text{Mg}^{2+}(\text{aq})$, $\text{Ca}^{2+}(\text{aq})$ $\text{Ba}^{2+}(\text{aq})$	white precipitate	precipitate is insoluble
Group 1 cations: $\text{Na}^+(\text{aq})$, $\text{K}^+(\text{aq})$	none	—

Testing for halide ions

1. Dissolve the unknown substance in distilled water.
2. Add a few drops of dilute nitric acid to the solution.
3. Add a few drops of silver nitrate solution.
4. Record the colour of any precipitate formed.
5. The identity of the halide can be confirmed by adding aqueous ammonia.

Anion	Precipitate		Addition of aqueous ammonia	
	Colour	Formula	Dilute	Concentrated
chloride Cl ⁻	white	AgCl	soluble	—
bromide Br ⁻	cream	AgBr	insoluble	soluble
iodide I ⁻	pale yellow	AgI	insoluble	insoluble

Testing for sulfate ions

1. Dissolve the unknown substance in distilled water.
2. Add a few drops of dilute hydrochloric acid to the solution.
3. Add a few drops of barium chloride solution.
4. The formation of a white precipitate of barium sulfate confirms the presence of sulfate ions in the solution.

Testing for carbonate ions

1. Add dilute hydrochloric acid to a small amount of the solid substance in a test tube.
2. Bubble any gas evolved through limewater.
3. The formation of a white precipitate confirms that carbonate ions were originally present in step 1.

Testing for alkenes

1. Add approximately 1 cm³ of bromine water to the unknown organic substance in a test tube.
2. Stopper and gently shake.
3. If an alkene is present, the colour will change from orange-brown to colourless.

Testing for an aldehyde

1. Prepare some Tollens' reagent by pouring 1 cm³ of silver nitrate solution into a test tube. Add one drop of sodium hydroxide solution to the silver nitrate solution. Dissolve the precipitate formed by adding ammonia solution drop-wise until the precipitate just dissolves.
2. Add 10 drops of the unknown organic substance to the test tube.
3. Stand the tube in a beaker of just boiled water and leave for 5 minutes.
4. Record your observations. If the aldehyde group is present, a silver mirror will form on the inside of the test tube.

Testing for a carboxylic acid group

1. Mix 1 cm³ of the unknown substance with an equal amount of ethanol in a test tube. Add a few drops of concentrated sulfuric acid.
2. Heat the mixture by standing the test tube in a beaker of hot water.
3. Pour the reaction mixture into aqueous sodium carbonate in a boiling tube.
If the unknown substance is a carboxylic acid, there will be an ester smell, like glue or pear-drops.

Answers to questions

1. It removes anions, such as carbonate ions, that would form a precipitate with silver nitrate.
2. Add dilute hydrochloric acid; sulfate(IV) ions will dissolve but sulfate(VI) ions will not.
3. Heat with Fehling's or Benedict's solution; the formation of a red precipitate indicates the presence of an aldehyde group.

Sample data

A: copper(II) sulfate

B: sodium carbonate

C: calcium bromide

D: iron(II) chloride

X: cyclohexene

Y: propanal

Z: ethanoic acid

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Equipment

- | | |
|--|-------------------------------------|
| • concentrated hydrochloric acid | • three bottles labelled X, Y and Z |
| • 1 mol dm ⁻³ sodium hydroxide | • nichrome wire |
| • 1 mol dm ⁻³ nitric acid | • delivery tube |
| • 0.1 mol dm ⁻³ silver nitrate solution | • Bunsen burner and safety mat |
| • 1 mol dm ⁻³ ammonia solution | • test tubes and bungs |
| • 1 mol dm ⁻³ dilute hydrochloric acid | • distilled water |
| • barium chloride solution | • dropping pipette |
| • limewater | • spatula |
| • bromine water | • boiling tube |
| • sodium carbonate solution | • 250 cm ³ beaker |
| • ethanol | • 100 cm ³ beaker |
| • concentrated sulfuric acid | • access to a kettle |
| • four tubs labelled A, B, C and D | |

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Organic analysis

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4. Write a plan for the safe conduct of this experiment to test for an aldehyde. You should describe in detail how you would carry out the experiment and the results you would expect.
5. Look up how to test for carboxylic acids.
6. Write a plan for the safe conduct of this experiment to test for carboxylic acids. You should describe in detail how you would carry out the experiment and the results that you would expect.
7. Carry out your experiments to identify substances X–Z. Take appropriate safety precautions.

Analysis of results

- Use the results of your tests to identify unknown substances A–D and X–Z.

Questions

1. When testing an inorganic compound for the presence of halide ions, why is nitric acid added before adding silver nitrate?
2. What further test could you do to distinguish between sulfate(VI) ions and sulfate(IV) ions?
3. Outline a further test that you could use to identify the presence of an aldehyde group.

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Safety

- Wear goggles.
- Consult CLEAPSS Hazcards® 6, 10A, 15B, 40A, 47A, 67, 87, 91, 95A and 98A. Perform a risk assessment using up-to-date information before this practical is carried out.

Disposal:

- All the ammoniacal silver nitrate solution should be washed down the foulwater drain with plenty of water. It must not be stored – silver filaments form, which have been known to explode.

Equipment per student/group	Notes on equipment
concentrated hydrochloric acid	Corrosive Irritant Must be kept in the fume cupboard
1 mol dm ⁻³ sodium hydroxide	Corrosive
1 mol dm ⁻³ nitric acid	Irritant
0.1 mol dm ⁻³ silver nitrate solution	Low hazard
1 mol dm ⁻³ ammonia solution	Low hazard
1 mol dm ⁻³ dilute hydrochloric acid	Irritant
barium chloride solution	Toxic and irritant
limewater	Irritant
bromine water	Toxic and irritant
sodium carbonate solution	Low hazard
ethanol	Flammable – do not use anywhere near a lit Bunsen burner.
concentrated sulfuric acid	Corrosive Must be kept in the fume cupboard.
copper(II) sulfate in a tub labelled A	Corrosive and harmful to the environment.
sodium carbonate in a tub labelled B	Irritant
calcium bromide in a tub labelled C	Low hazard
iron(II) chloride in a tub labelled D	Harmful and irritant
cyclohexene in a bottle labelled X	Flammable and harmful
propanal in a bottle labelled Y	Highly flammable
ethanoic acid; 4 mol dm ⁻³ solution in a bottle labelled Z	Corrosive
nichrome wire	
delivery tube	

Bunsen burner and safety mat	
test tubes and bungs	
distilled water	
dropping pipette	
spatula	
boiling tube	
250 cm ³ beaker	
100 cm ³ beaker	
access to a kettle	

Notes