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9.1.1 FLAME TESTS

Flame Tests

- Metal ions produce a **colour** if heated strongly in a flame
- Ions from different metals produce different colours
- The flame test is thus used to identify metal ions by the colour of the flame they produce
- Dip the loop of an **unreactive** metal wire such as nichrome or platinum in dilute acid, and then hold it in the blue flame of a Bunsen burner until there is no colour change
- This cleans the wire loop and avoids contamination
 - This is an important step as the test will only work if there is just **one type** of ion present
 - $^\circ\,$ Two or more ions means the colours will mix, making identification erroneous
- Dip the loop into the solid sample and place it in the edge of the **blue** Bunsen flame
- Avoid letting the wire get so hot that it glows red otherwise this can be confused with a flame colour





Diagram showing the technique for carrying out a flame test

• The colour of the flame is observed and used to identify the metal ion present

Cation	Colour of Flame
Li ⁺	Red
Na+	Yellow
K+	Lilac
Ca ²⁺	Orange-red
Cu ²⁺	Blue-green



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Diagram showing the colours formed in the flame test for metal ions

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Exam Tip

The sample needs to be heated strongly, so the Bunsen burner flame should be on a blue flame.

9.1.2 TESTS FOR CATIONS

Tests for Cations

- Metal cations in aqueous solution can be identified by the **colour** of the precipitate they form on addition of **sodium hydroxide**
- If only a small amount of NaOH is used then normally the **metal hydroxide** precipitates
- In excess NaOH some of the precipitates may dissolve
- For this reason just a few drops of NaOH are added at first and very slowly
- If it is added too quickly and the precipitate is soluble in excess, then you run the risk of missing the formation of the initial precipitate which dissolves as quickly as it forms if excess solution is added
- A small amount is thus added, very gradually and any colour changes or precipitates formed are noted
- Then the NaOH is added in excess and the reaction is observed again
- Ca2+ ions can be distinguished from A*l3+* as calcium hydroxide **does not dissolve** in excess NaOH but aluminium hydroxide does
- Most transition metals produce hydroxides with distinctive colours

Metal Cation	Effect of adding NaOH	
Aluminium (Al ³⁺)	White precipitate, dissolves in excess NaOH to form a colourless solution	
Calcium (Ca ²⁺)	White precipitate, insoluble so remains in excess NaOH	
Copper (II) (Cu ²⁺)	Light blue precipitate, insoluble in excess	
Iron (11) (Fe ²⁺)	Green precipitate, insoluble in excess	
Iron (III) (Fe ³⁺)	Red-brown precipitate, insoluble in excess	







Testing for ammonium ions, NH 4

- Ammonium ions are also tested with sodium hydroxide, but not by a precipitation reaction
- To test for the ammonium ion, gentle heating is required after adding the NaOH solution
- If the ammonium ion is present, **ammonia gas** is produced which can be tested with red litmus paper:

NH₄⁺ (aq) + OH– (aq)→ NH3 (g) + H2O (l)



Exam Tip

Be sure to distinguish between the term "colourless" and "clear". A solution that loses its colour has become colourless. A clear solution is one that you can see through such as water. Solutions can be clear and have colour e.g: dilute copper sulphate.





9.1.3 TESTING FOR AMMONIA

Testing for Ammonia

- Ammonia is a gas with a **pungent** smell that turns damp **red** litmus paper **blue**
- Hold the litmus paper near the mouth of the test tube, but be careful to avoid touch the sides of the test tube
- If you are testing for ammonia produced from ammonium ions and sodium hydroxide, avoiding touching the sides prevents traces of sodium hydroxide from also turning the red litmus paper blue



Diagram showing how to perform the test for ammonia gas using damp red litmus paper

- Another test for ammonia is to react the gas with HCl
- A white smoke of ammonium chloride is formed if ammonia gas is present:

$\mathsf{NH3} + \mathsf{HC}l \to \mathsf{NH4C}l$





Exam Tip

Make sure you understand the difference between ammonium and ammonia. Ammonium

refers to the aqueous cation, NH+4, whereas ammonia is the gas, NH3.

9.1.4 TESTS FOR ANIONS

Tests for Anions

Carbonate Ion

- Add dilute acid and test the gas released.
- Effervescence should be seen and the gas produced is CO2 which forms a **white** precipitate of calcium carbonate when bubbled through limewater:



Limewater turns milky in the presence of CO2 caused by formation of insoluble calcium carbonate



Sulfate Ion

- Acidify with dilute hydrochloric acid and add aqueous barium chloride.
- A white precipitate of barium sulfate is formed:



 $Ba^{2+}(aq) + SO_{4}^{2-}(aq) \rightarrow BaSO4(s)$





Halide Ions

- Acidify with dilute nitric acid (HNO3) followed by the addition of silver nitrate solution (AgNO3).
- This forms a silver halide precipitate:



Testing for halide ions

- Depending on the halide present, a **different coloured precipitate** is formed, allowing for identification of the halide ion
- Silver chloride is **white**, silver bromide is **cream** and silver iodide is **yellow**



Each silver halide produces a precipitate of a different colour







Exam Tip

When it comes to qualitative inorganic analysis, always remember that there will be a test for the metal **cation** part of the molecule and another test for the **anion** part.



9.1.5 CORE PRACTICAL: IDENTIFYING IONS

Core Practical: Identifying Ions

Aim:

To use chemical tests to identify the ions in unknown binary ionic compounds

Materials:

- Bunsen burner
- Test tubes and test tube rack
- Teat pipette
- Nichrome wire mounted in handle or cork
- Limewater
- 0.4 mol dm-3 dilute hydrochloric acid
- 0.1 mol dm-3 barium chloride solution
- 0.4 mol dm-3 dilute nitric acid
- 0.05 mol dm-3 silver nitrate solution
- Various samples of salts and salt solutions

Practical Tip:

Key to this practical is your level of organisation. You will have many containers, solutions and samples so your work space and results table must be neat and tidy

Method:

- 1. There are a number of strategies you could choose in order to identify the ions in unknown salts
- 2.Common analysis strategies include flame tests, and tests for sulphate, carbonate and halide ions
- 3. They can be carried out in any particular order, and you will probably not need to carry them all out on any one sample
- 4.Only small amounts of each sample and reagent are needed
- 5.You may need to dissolve a sample of salt in a little distilled water if the salt you are given is in the solid state
- 6.Record your observations carefully in a table of results as you work through the tests
- 7.Repeat any tests that do not provide a clear result i.e. a colour change that was difficult to identify



Results:

Record your results for each test carefully in a suitable table

Evaluation:

Once you have tabulated the results from the tests you performed, use them to infer the identity of the ions that the salt contains

Conclusion:

You can then identify the salt from the cation and anion present. When inferring the formulae and names of unknown salts, make sure that you balance the charges on the ions in the formula. E.g. the

test for B was positive for the Fe2+ cation, therefore the anion must be an ion with a -2 charge or two ions each with a single -1 charge

Worked Example

Example

A student was given two salts labelled A and B. The following set of results were obtained from a series of qualitative tests performed by the student on the samples. Use the results to identity the salts.

Salt	Test	Result
A	Flame test	Red flame - must contain Li*
A	Dilute nitric acid and silver nitrate solution added	Cream precipitate – must contain Br-
В	Flame test	No change to flame colour – could be Fe or Al
В	NaOH added	Green precipitate formed - must contain Fe ²⁺
В	Dilute nitric acid and silver nitrate solution added	No reaction – must not contain a halide ion
В	Acidified with dilute hydrochloric acid and aqueous barium chloride added	White precipitate formed – must contain SO_4^{2-}

Conclusion:

- Salt A contains lithium and bromide ions, so it must be lithium bromide, LiBr
- Salt B contains iron(II) and sulfate ions, so it must be FeSO4



9.1.6 INSTRUMENTAL METHODS OF ANALYSIS

Advantages of Instrumental Methods

- Advancements in technology and computing have allowed for the development of instruments designed to analyse chemical substances
- Methods of analysis include X-ray, Infra-Red and Mass Spectroscopy, Gas Chromatography and Flame Photometry
- These analytical techniques require modern day instruments which are a vital part of chemistry laboratories
- The advantage of using these instruments over more traditional methods include:
 - They provide greater **accuracy**
 - $_{\circ}~$ They are **faster** and **easier** to use
 - $_{\circ}\,$ They are **automated** and can perform **multiple simultaneous** sampling and testing
 - $_{\circ}\,$ Modern instruments are very **sensitive** and can work with very small **sample sizes**



Exam Tip

You need to discuss the advantages of instruments over chemical testing in terms of **sensitivity, speed and accuracy**.

Flame Photometry

- This technique is used to analyse metal ions in solution
- When substances are heated they often emit energy in the form of light
- This is due to electrons falling back to their original **energy levels** after becoming **excited** which causes them to **jump up** one or more energy levels
- Flame emission spectroscopy works by exposing the sample to a **very hot flame** and then measuring the **intensity** and **wavelength** of the light emitted
- The output is an **emission spectrum** in which different elements produce lines in different parts of the spectrum





Diagram of an emission spectrum for mercury obtained from flame photometry

- The emission spectrum consists of brightly coloured thin lines on a dark background and each element ion produces a **unique** spectrum
- Flame emission spectroscopy also works for mixtures of ions
- This is a major advantage over flame testing which can only analyze one ion at a time
- The intensity of the light produced is proportional to the number of ions vaporised, so the technique can be used to determine the concentration of metal ions in a solution by reference to a standard solution of known concentration



A calibration curve for solutions containing calcium ions. Different standard solutions have their intensity measured and plotted on a graph against concentration. This linear relationship allows the intensity of an unknown solution to be measured and its concentration read off the graph.



Reference Data

- Ions in **unknown samples** can be identified by comparing the sample spectrum to reference spectra
- This is particularly useful if the sample contains a number of different ions
- The following flame spectrum for example was obtained for solution containing an unknown metal:

Spectrum for an unknown element

• When compared to the reference spectra below we can see that the solution must contain **sodium** ions:





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Reference spectra for elements