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| Chemical Investigations |
| Analysing Cations |
| Teacher / Technician Guide |



Introduction

Identifying substances is a very important aspect of chemistry.

To test for many cations, it is common to use flame tests, which are covered in a different document. Here we look at a variety of simple tests that will allow for the identification of a variety of cations in solution: aluminium, ammonium, calcium, copper, iron II, iron III, lead and zinc.

Each group will need

|  |  |
| --- | --- |
| Your samples | Test tubes & rack |
| Sodium hydroxide (0.4 mol l-1) | Ammonia (1 mol l-1 ) |
| Potassium hexacyanoferrate III (0.2 mol l-1) | Potassium thiocyanate (0.2 mol l-1) |
| Potassium iodide (0.2 mol l-1) | Alizarin S (0.1%) |

The concentrations are not critical here as the tests are qualitative.

Safety

0.4 mol l-1 sodium hydroxide is an irritant. Wear eye protection.

To do

The first set of tests involves adding sodium hydroxide and ammonia to two different samples of your unknown.

**Part 1 – adding sodium hydroxide**

1. Add a few cm3 of your unknown solution to a test tube.
2. Add a few drops of sodium hydroxide – Is a precipitate formed?
3. Now add more sodium hydroxide – until the test tube is about ¾ full. Stir and see if the precipitate dissolves.

**Part 2 - Results and further actions**

1. No precipitate – in this case it is probably one of the following – ammonium, potassium sodium, lithium or barium.

Hold a piece of moist red litmus over the top of the mixture with sodium hydroxide. If it goes blue, ammonia is being released so it was an **ammonium** ion

If there is no change, you need a flame test to distinguish the ions.

1. Blue precipitate – probably copper

To confirm – to another sample, add excess ammonia. If a blue precipitate forms and then dissolves to a deep blue solution, it is **copper**

1. Green precipitate – probably iron II but possibly Chromium

To confirm, to another sample add a few drops of potassium hexacyanoferrate III (ferricyanide) a deep blue colour shows it is **iron II**

1. Brown precipitate – red/brown is iron III but a white precipitate that goes brown could be manganese II

To confirm, to another sample add a few drops of potassium thiocyanate a deep red colour shows it is **iron III**

1. White precipitate that does not dissolve in excess hydroxide – **calcium or magnesium**

These can be distinguished by a flame test.

1. White precipitate that does dissolve in excess hydroxide – aluminium, zinc or lead

Add ammonia in excess. If the white precipitate formed dissolves in excess, it is **zinc**.

If not, Add a few drops of potassium iodide solution. A brilliant yellow colour means it is **lead**

No colour means it is **aluminium**

To confirm aluminium:

* To 5 cm3 of the (neutral or acid) test solution, add 1 cm3 of alizarin and then ammonia until the solution is alkaline – as shown by it going purple.
* Boil for a few moments and allow to cool.
* Add dilute ethanoic acid. A red colour or precipitate remaining is evidence of aluminium.

Extensions / Additions

**Testing for Cobalt 2+ ions**

Add ammonia solution

Small quantities give a precipitate of a basic salt of complex structure (blue) or Cobalt hydroxide Co(OH)2 (rose-red).

In excess this dissolves to form the orange/yellow complex hexaamminecobalt(II) ion:

Co2+ + 6NH3 🡨🡪[Co(NH3)6]2+

This darkens in air to a deep red solution. (You can speed up the process by adding hydrogen peroxide)

**Testing for Nickel 2+ ions.**

Not generally used in schools but may be encountered at Higher / Advanced Higher.

Add dimethylglyoxime solution. Then add a bit of ammonia to make solution basic. Nickel will give a red precipitate.

**Testing for Chromium ions**

Avoid chromates and dichromates.

Chromium III ions give a green precipitate with sodium hydroxide. It dissolves in excess to give a green solution.

Confirmatory test

Add aqueous ammonium sulphide and Chromium III ions form a black precipitate of chromium sulphide (Cr2S3) precipitate. It quickly hydrolyses to chromium III hydroxide Cr(OH)3 which is a green precipitate.

**Testing for Manganese II ions**

With NaOH solution the Mn2+ ion, an off white / beige precipitate of manganese hydroxide Mn(OH)2  forms. It is insoluble in excess

On standing, it darkens to give manganese dioxide which is a [black precipitate](https://www.chemistryscl.com/chemistry_theories/precipitate/main.html).

Some additional tests

**Confirmatory test for lead**

You can use dithizone – though potassium iodide is easier.

**Test for Ammonium/potassium ions**

Sodium hexanitritocobaltate(III) (sodium cobaltinitrite) is used to test for the presence of ammonium and potassium ions. Though the emission of ammonia gas or a lilac flame test might be an easier option.

**Testing for cobalt II, copper II and Nickel II**

You can use Thiooxamide (rubeanic acid) for this – but easier methods are described above.

Technician’s Guide

Each group will need

|  |  |
| --- | --- |
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| Sodium hydroxide (0.4 mol l-1) | Ammonia (1 mol l-1 ) |
| Potassium hexacyanoferrate III (0.2 mol l-1) | Potassium thiocyanate (0.2 mol l-1) |
| Potassium iodide (0.2 mol l-1) | Alizarin S (0.1%) |

Preparing solutions

The alkalis and the potassium salts are familiar enough to need no details.

There are, though, some less common reagents that may be used.

**Alizarin solution**

The working solution is approximately 0.1% (m/v) solution

Alizarin itself is not soluble in water. You have two alternatives:

1. Use you need the water-soluble salt. It will be listed as ‘Alizarin S’ or ‘Alizarin Red S’ (CAS Nor 130-22-3)

Dissolve 0.1 g of Alizarin S/Alizarin Red S in 100 cm3 of distilled/deionised water.

1. Alizarin (CAS No 72-48-0) itself, though, is soluble in propanone

Dissolve 0.1 g of Alizarin (1,2-dihydroxyanthraquinone) in 100 cm3 of propanone.

Alternatively, you can prepare a 1% ‘stock’ solution of either (1 g per 100 cm3) and dilute as required.

**Dimethyl Glyoxime**

Wear eye protection. Work away from any sources of ignition..

To prepare a 1% (m/v) solution, dissolve 1 g of dimethylglyoxime in 100 cm3 of ethanol/IDA.

Transfer the prepared solution to a suitable labelled bottle.

**Dithizone (diphenylthiocarbazone)**

Wear eye protection and gloves. Work in a fume cupboard. Keep away from any sources of ignition.

Add roughly 0.01 g of dithizone to 100 cm3 of propanone (acetone). Stir to dissolve.

*The concentration of the reagent is not critical as it forms a very strongly coloured solution*.

Transfer the prepared solution to a suitable labelled bottle.

**Sodium hexanitritocobaltate(III) (sodium cobaltinitrite)**

Wear eye protection. Avoid skin contact.

Add 4 g of the solid to 100 cm3 of distilled water. Stir to dissolve as much as possible.

Filter the solution into a suitable labelled bottle.

NB it has a maximum shelf-life of only around 3 weeks.

**Thiooxamide (rubeanic acid)**

Wear eye protection. Keep away from any sources of ignition

To prepare a 0.5% (m/v) solution, add 0.5 g of thiooxamide to 100 cm3 of ethanol/IDA. Stir to dissolve.

Transfer the prepared solution to a suitable labelled bottle.