

# E5 IDENTIFICATION OF INORGANIC COMPOUNDS

## THE TASK

To identify simple ionic compounds unambiguously.

## THE SKILLS

By the end of the experiment you should be able to:

- identify the presence of common cations and anions in inorganic compounds,
- design systematic experimental procedures to identify unknowns,
- follow a flow chart.

## OTHER OUTCOMES

- You will develop generic scientific skills including semi-micro techniques, qualitative analysis and observing and accurately recording results.
- You will practise writing balanced chemical and ionic equations.

## INTRODUCTION

Any process that can provide a qualitative determination of the ions present in a simple inorganic compound is based upon knowledge of acid/base chemistry, redox chemistry and solubility. In this regard, the identification of a single pure compound is therefore very much simpler than the identification of a mixture. This experiment deals only with the identification of simple compounds, *ie* those that contain only one cation and one anion.

## Systematic Identification

In most instances a specific reaction is needed in order to identify a particular ion. A general procedure involving all of these reactions is dealt with in this experiment. This procedure has three fundamental steps, as outlined below:

- Observation of colour of solid
- Solubility in water and colour of solution (if applicable)
- Tests for cations and anions.

### *Observations of colour and solubility*

Table E5-1 on the following page lists the colours of the common form of each substance in this course. For example anhydrous  $\text{CoCl}_2$  is actually blue, but the table refers to the more common hydrated form,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , which is red. Compounds which are unstable or do not exist are indicated with a “—”.

A list of common colours for ions in solution can be found in Table E5-2. The solubilities of the salts are given in Table E1-3 (page E1-13) and the procedures used in E1 should be applied in testing the solubility in water. Colour and solubility are generally insufficient to identify a compound with certainty, rather they provide supporting evidence for the ion tests.

## E5-2

Table E5-1 Colours of Solid Compounds

	Cl <sup>-</sup>	Br <sup>-</sup>	I <sup>-</sup>	OH <sup>-</sup>	O <sup>2-</sup>	SO <sub>4</sub> <sup>2-</sup>	NO <sub>3</sub> <sup>-</sup>	CO <sub>3</sub> <sup>2-</sup>	
Ag <sup>+</sup>	white	yellow	yellow	brown	brown	white	white	white	
Cu <sup>2+</sup>	green	black	–	blue	black	blue	blue	green	
Pb <sup>2+</sup>	white	white	yellow	white	yellow	white	white	white	
Sn <sup>2+</sup>	white	yellow	orange	white	black	white	white	–	
Ni <sup>2+</sup>	green	green	black	green	black	green	green	green	
Co <sup>2+</sup>	red	red	black	pink	black	red	red	red	
Fe <sup>3+</sup>	brown	red	–	brown	red	yellow	grey	–	
Fe <sup>2+</sup>	green	grn-yel	grey	green	black	green	green	green	
Cr <sup>3+</sup>	violet	green	black	blue/grn	black	violet	violet	–	
Cd <sup>2+</sup>	white	yellow	grn/yell	white	brown	white	white	white	
Zn <sup>2+</sup>	white								
Mn <sup>2+</sup>	pink	pink	pink	pink	grey	pink	pink	pink	
Al <sup>3+</sup>	white								–
Mg <sup>2+</sup>									
Ca <sup>2+</sup>									
Sr <sup>2+</sup>									
Ba <sup>2+</sup>									
Na <sup>+</sup>									
K <sup>+</sup>									
NH <sub>4</sub> <sup>+</sup>				–	–				

Table E5-2 Colours of Ions in Solution.

Cation	Colour of ion in aqueous solution
Cr <sup>3+</sup> (aq)	green or blue or grey
Mn <sup>2+</sup> (aq)	colourless to very pale pink
Fe <sup>2+</sup> (aq)	pale green (colourless in very dilute solution)
Fe <sup>3+</sup> (aq)	pale violet, but yellow in the presence of most anions
Co <sup>2+</sup> (aq)	pink
Ni <sup>2+</sup> (aq)	green
Cu <sup>2+</sup> (aq)	blue or green

All other common cations and all anions under consideration are colourless.

## Anion Tests

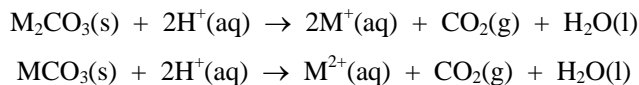
The anion tests are as follows:

- A1 Test for carbonates
- A2 Tests for halides
- A3 Test for sulfates
- A4 Test for nitrates

If no anion has been identified, then the anion is assumed to be either oxide or hydroxide.

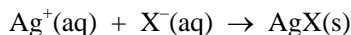
### *A1: Test for carbonates*

A sample of the solid is tested with nitric acid solution. The evolution of an odourless gas, as evidenced by bubbling or fizzing of the solid, indicates an acid/base reaction of a carbonate. Although many carbonates are insoluble in water, all react in this way with dilute nitric acid.

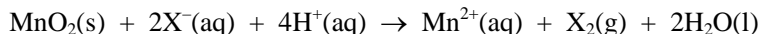


### *A2: Test for halides*

The solid is dissolved in nitric acid and tested with silver nitrate solution. If a white or creamish coloured precipitate forms, the presence of a halide is indicated.

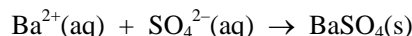


To determine which halide is present a further test must be undertaken. The identity of the halide is distinguished by oxidation of the halide ion to free halogen, which is easily recognised by its colour. To accomplish this, the solid unknown is oxidised with manganese dioxide in the presence of sulfuric acid. Evolution of a pale green to colourless gas ( $\text{Cl}_2$ ) indicates chloride, a brown gas ( $\text{Br}_2$ ) indicates bromide and a violet gas ( $\text{I}_2$ ) indicates iodide.



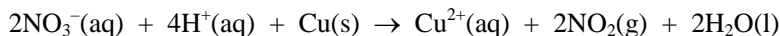
### *A3: Test for sulfates*

The solid is dissolved in nitric acid and a few drops of barium nitrate solution added. A white precipitate indicates the presence of sulfate ions.



### *A4: Test for nitrates*

The unknown solid and some copper turnings are moistened with water, gently heated, and then treated with 18 M  $\text{H}_2\text{SO}_4$ . The evolution of  $\text{NO}_2(\text{g})$ , a brown and acrid gas, and the appearance of a blue colour in the solution due to  $\text{Cu}^{2+}$  ions indicates the presence of nitrate ions. This is due to the redox reaction involving copper and nitrate ion that occurs.



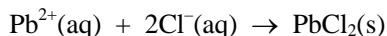
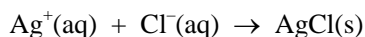
## Cation Tests

The cation tests are as follows:

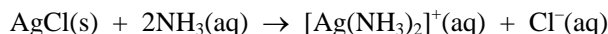
- C1 Test for  $\text{Ag}^+$  and  $\text{Pb}^{2+}$  as insoluble chlorides
- C2 Tests for  $\text{Cd}^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Sn}^{2+}$  as insoluble sulfides (acidic conditions)
- C3 Test for  $\text{Fe}^{3+}$ ,  $\text{Cr}^{3+}$  and  $\text{Al}^{3+}$  as insoluble hydroxides
- C4 Test for  $\text{Co}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Zn}^{2+}$  as insoluble sulfides (basic conditions)
- C5 Test for  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Ba}^{2+}$  as insoluble carbonates
- C6 Test for  $\text{NH}_4^+$
- C7 Flame tests for  $\text{Na}^+$  and  $\text{K}^+$

### *C1: Test for $\text{Ag}^+$ and $\text{Pb}^{2+}$*

The solid is dissolved in nitric acid and hydrochloric acid added. The presence of a white precipitate indicates the presence of  $\text{Pb}^{2+}$  or  $\text{Ag}^+$ .

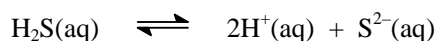


To identify which cation is present, just leave the precipitate to stand for a few minutes in direct sunlight.  $\text{AgCl}$  will go a distinct purple colour as it decomposes to metallic silver (this was the basis of the photographic industry before digital cameras), whilst  $\text{PbCl}_2$  will undergo no change. Your identification is confirmed by isolating the precipitate from the supernatant liquid using a centrifuge and applying one further test.  $\text{PbCl}_2$  will dissolve in hot water,  $\text{AgCl}$  does not.  $\text{AgCl}$  will dissolve in  $\text{NH}_3$ ,  $\text{PbCl}_2$  does not.

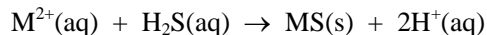


### *C2: Test for $\text{Cd}^{2+}$ , $\text{Cu}^{2+}$ and $\text{Sn}^{2+}$*

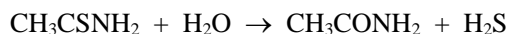
The concentration of  $\text{S}^{2-}$  ions in a solution of  $\text{H}_2\text{S}$  can be reduced to extremely low levels by making the solution acidic. The high concentration of  $\text{H}^+$  ions forces the equilibrium



to move even further to the left. Under these conditions of extremely low  $\text{S}^{2-}$  concentration, only the most insoluble metal sulfides will precipitate.



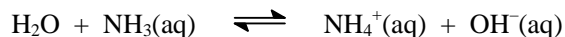
The aqueous  $\text{H}_2\text{S}$  is generated *in situ* from thioacetamide, which is hydrolysed under the acidic conditions.



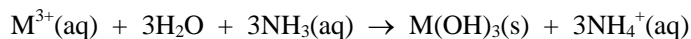
From the colours of the precipitate and initial solution, it is possible to distinguish the above cations. If a precipitate forms, then either  $\text{Cd}^{2+}$  (bright yellow),  $\text{Sn}^{2+}$  (brown) or  $\text{Cu}^{2+}$  (black from a blue or green solution).

*C3: Test for Fe<sup>3+</sup>, Cr<sup>3+</sup> and Al<sup>3+</sup>*

These cations have the most insoluble hydroxides. The concentration of OH<sup>-</sup> must be very low in order to avoid precipitating hydroxides of other cations. An NH<sub>4</sub>Cl/NH<sub>3</sub> buffer solution is used to achieve the necessary conditions. Ammonia, NH<sub>3</sub>, is a weak base that hydrolyses only slightly. In the presence of ammonium ion, NH<sub>4</sub><sup>+</sup>, this hydrolysis is even more suppressed, and the resultant [OH<sup>-</sup>] is in the order of 10<sup>-5</sup> M.



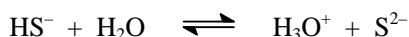
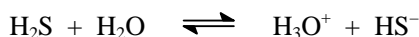
The sample is dissolved in nitric acid and heated for several minutes. This not only dissolves the solid, but also oxidises any Fe<sup>2+</sup> present to Fe<sup>3+</sup>. A buffer solution of solid NH<sub>4</sub>Cl and 16 M NH<sub>3</sub> is then added. A brown precipitate indicates the presence of Fe<sup>3+</sup> in the solution (although it may have initially been Fe<sup>2+</sup> or Fe<sup>3+</sup> in the solid), a grey or green precipitate indicates Cr<sup>3+</sup> and a white precipitate indicates Al<sup>3+</sup>.



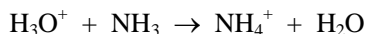
If iron is detected, a further test is required to determine its oxidation state in the original compound.

*C4: Test for Co<sup>2+</sup>, Mn<sup>2+</sup>, Ni<sup>2+</sup> and Zn<sup>2+</sup>*

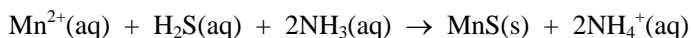
Although all have insoluble sulfides and hydroxides, precipitation of these ions in C2 and C3 was avoided because of the very low [S<sup>2-</sup>] and [OH<sup>-</sup>] respectively. They are precipitated as sulfides in this test under slightly basic conditions because the [S<sup>2-</sup>] is considerably higher.



Both these equilibria are pushed to the right in the presence of an NH<sub>4</sub>Cl/NH<sub>3</sub> buffer solution. The NH<sub>3</sub> combines with the H<sub>3</sub>O<sup>+</sup> ions, creating a greater [S<sup>2-</sup>] than is possible under acidic or neutral conditions.

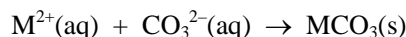


The unknown is dissolved in hydrochloric acid and the buffer of solid NH<sub>4</sub>Cl and 16 M NH<sub>3</sub> is added. The presence of the ammonia converts Co<sup>2+</sup>, Ni<sup>2+</sup> and Zn<sup>2+</sup> ions into their ammine complexes. This was a factor in preventing these ions from precipitating as insoluble hydroxides in test C3, as the concentration of free M<sup>2+</sup>(aq) ions in the presence of ammonia is very small. Aqueous H<sub>2</sub>S is again generated *in situ* from thioacetamide. If a precipitate forms, then either Ni<sup>2+</sup> (black from a blue-violet solution), Co<sup>2+</sup> (black from a brown solution), Zn<sup>2+</sup> (white), or Mn<sup>2+</sup> (peach, orange-pink) is present.



*C5: Test for Ca<sup>2+</sup>, Ba<sup>2+</sup>, Sr<sup>2+</sup> and Mg<sup>2+</sup>*

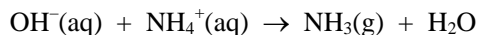
The unknown is dissolved in water or acid and sodium carbonate solution added. A white precipitate indicates the presence of Mg<sup>2+</sup>, Ca<sup>2+</sup>, Sr<sup>2+</sup>, or Ba<sup>2+</sup>.



To identify the specific cation present, a flame test is performed. No colour (or yellow due to Na<sup>+</sup> contaminant) indicates Mg<sup>2+</sup>, dull (brick) red indicates Ca<sup>2+</sup>, crimson indicates Sr<sup>2+</sup> and pale green indicates Ba<sup>2+</sup>.

*C6: Test for NH<sub>4</sub><sup>+</sup>*

The presence of NH<sub>4</sub><sup>+</sup> is confirmed through the evolution of ammonia, NH<sub>3</sub>, from the acid/base reaction that occurs when the compound is treated with a solution of the strong base, OH<sup>-</sup>.



The ammonia is detected by smell and by using pink litmus paper, which turns blue in the presence of the weak base NH<sub>3</sub>.

*C7: Flame tests for Na<sup>+</sup> and K<sup>+</sup>*

A brilliant, persistent yellow colour indicates Na<sup>+</sup>, while a lilac (pale purple) flame indicates K<sup>+</sup>.

## LAB-WORK

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## Anion Tests

*Method for A1: Test for carbonates*

Place the solid (0.1 g) in a semi-micro tube and then add 4 M HNO<sub>3</sub> (0.5 mL). If an odourless gas is evolved this indicates CO<sub>3</sub><sup>2-</sup>.

Test Na<sub>2</sub>CO<sub>3</sub> in the above way. Record your observations and conclusions.

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Give the net ionic equation for any observed reaction.

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Although most carbonates are insoluble in water, all react in this way with acids. Any insoluble metallic oxide or hydroxide likewise reacts with excess 4 M HNO<sub>3</sub> to give a clear solution. (Note: A clear solution is one that contains no precipitate; it may or may not be coloured.) Many oxides are contaminated with carbonate (due to reaction with CO<sub>2</sub> in the air) and may release a small amount of CO<sub>2</sub>(g).

Any unknown that does not give a clear solution with excess 4 M HNO<sub>3</sub> is an insoluble salt of a strong acid, and is specifically excluded from this course. Therefore, the compounds of Table E5-1 specifically omitted are:

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*Method for A2a: Test for halides*

If the solution from A1 does not evolve  $\text{CO}_2$ , place another portion of the solid (10 mg) in a semi-micro tube, add 4 M  $\text{HNO}_3$  (0.5 mL) and heat the solution until clear (but not necessarily colourless). [If you observe a brown solution, a dark precipitate or a brown gas, take a new sample of the solid (10 mg) and dissolve in water (0.5 mL), then add 4 M  $\text{HNO}_3$  (1 drop).] In either case, continue the test by adding 0.1 M  $\text{AgNO}_3$  (1 drop). A whitish precipitate indicates  $\text{Cl}^-$  or  $\text{Br}^-$ , while a yellowish precipitate indicates  $\text{Br}^-$  or  $\text{I}^-$ . A blank test should always be carried out on deionised water to check this test. Proceed to A2b if and only if a halide is found to be present.

*Method for A2b: Test for specific identification of halide*

If a halide is present, identify it by the following procedure. In a dry semi-micro tube place the solid (10 mg) and  $\text{MnO}_2$  (10 mg). Support the tube in a holder and carefully add 18 M  $\text{H}_2\text{SO}_4$  (3 drops). Heat the tube gently **in a water bath**. Evolution of a colourless or light green gas ( $\text{Cl}_2$ ) indicates  $\text{Cl}^-$ , a brown gas ( $\text{Br}_2$ ) indicates  $\text{Br}^-$  or a violet gas ( $\text{I}_2$ ) indicates  $\text{I}^-$ . The presence of chlorine, if suspected, must be confirmed by a test with moist litmus paper. If the gas bleaches the litmus paper,  $\text{Cl}_2$  is present.

Test NaCl in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of AgCl.

Give the net ionic equation for the oxidation of  $\text{Cl}^-$  to  $\text{Cl}_2$  by  $\text{MnO}_2/\text{H}^+$ .

Test KBr in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of AgBr.

Give the net ionic equation for the oxidation of  $\text{Br}^-$  to  $\text{Br}_2$  by  $\text{MnO}_2/\text{H}^+$ .

Test KI in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of AgI.

Give the net ionic equation for the oxidation of  $\Gamma^-$  to  $I_2$  by  $MnO_2/H^+$ .

*Method for A3: Test for sulfates*

Place the solid (0.1 g) in a semi-micro tube, add 4 M  $HNO_3$  (0.5 mL) and heat until the solution is clear. Add 0.1 M  $Ba(NO_3)_2$  (5 drops) and let the solution stand for 1 minute. A white precipitate indicates the presence of  $SO_4^{2-}$ .

Test  $CoSO_4$  in the above way. Record your observations and conclusions.

Give the net ionic equations for any observed reaction.

*Method for A4: Test for nitrates*

Place the solid (10 mg) and Cu turnings (10 mg) in a semi-micro tube, then add water (2 drops). Heat and then add 18 M  $H_2SO_4$  (2 drops). Brown fumes indicate the presence of  $NO_3^-$ .

Test  $NaNO_3$  in the above way. Record your observations and conclusions.

Give the net ionic equations for any observed reaction.

If no anion is detected in the previous tests, the unknown must be an oxide or hydroxide. The decision between these two can often be made simply on the basis of appearance once the cation is known. In other cases distinguishing between them may require quantitative analysis or even a crystal structure determination, since the distinction between the members of the series; oxide, hydrated oxide, hydroxide; may be very fine. Where the identification is difficult for a particular unknown, both alternatives (*ie* oxide or hydroxide) should be specified.



## Cation Tests

### *Method for Cl<sup>-</sup>: Test for Ag<sup>+</sup> and Pb<sup>2+</sup>*

In a semi-micro tube dissolve the unknown solid (10 mg) in 4 M HNO<sub>3</sub> (0.5 mL). Warm if necessary, then cool. Add 4 M HCl (1 drop). A white precipitate indicates Ag<sup>+</sup> or Pb<sup>2+</sup>. Allow the precipitate to stand for a few minutes. If the precipitate is AgCl, it will take on a definite purple tinge. If no change is detected, the precipitate is PbCl<sub>2</sub>. Use a centrifuge to isolate the precipitate and confirm your identification as follows.

If Pb<sup>2+</sup> is suspected, add water (1.0 mL) and heat in a water bath. Dissolution indicates Pb<sup>2+</sup>. Stirring of the mixture may be necessary to get all of the precipitate to dissolve. PbCl<sub>2</sub> will reprecipitate as a white solid if the test tube is allowed to cool.

If Ag<sup>+</sup> is suspected, add 4 M NH<sub>3</sub> (0.5 mL) to the precipitate and stir. Dissolution (probably incomplete) occurs. To the solution add 15 M HNO<sub>3</sub> by drops until a white precipitate is formed. This confirms Ag<sup>+</sup>.

Test Pb(NO<sub>3</sub>)<sub>2</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of lead(II) chloride.

Test AgNO<sub>3</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of silver chloride.

Give the net ionic equation for the dissolution of silver chloride in ammonia solution.

## E5-10

*Method for C2: Test for Cd<sup>2+</sup>, Cu<sup>2+</sup> and Sn<sup>2+</sup>*

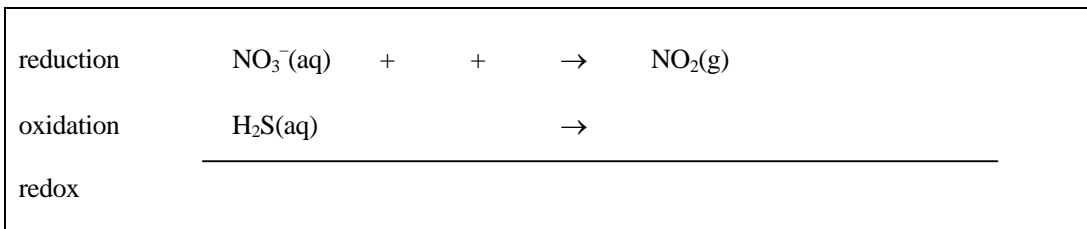
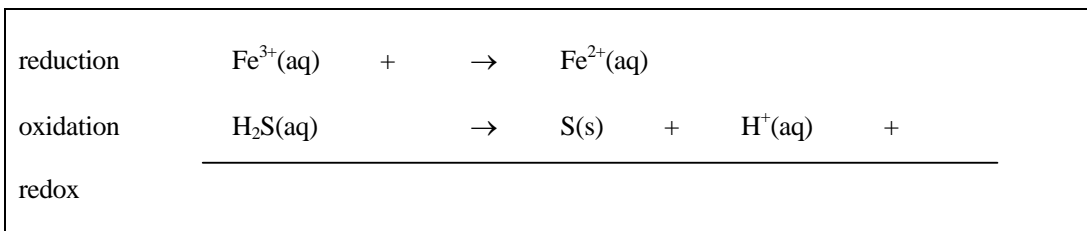
In a fume hood, place the solid (10 mg) in a semi-micro tube and add 0.4 M HCl (0.5 mL). If the anion was found to be CO<sub>3</sub><sup>2-</sup>, O<sup>2-</sup>, or OH<sup>-</sup> add further HCl (0.5 mL). Heat on a water bath, add thioacetamide solution (10 drops, to produce H<sub>2</sub>S) and stir the resulting mixture. Further heating for several minutes may be required to form a precipitate. The appropriate conclusion to be drawn from the formation of a particular precipitate is:

Bright yellow indicates Cd<sup>2+</sup>.

Brown indicates Sn<sup>2+</sup>.

Black, possibly tinged with brown, (from a blue or green solution) indicates Cu<sup>2+</sup>.

A very fine, pale yellow or off-white precipitate of S should be ignored. This can occur when testing an unknown if it contains either Fe<sup>3+</sup> ions or NO<sub>3</sub><sup>-</sup> ions. Both of these ions can oxidise H<sub>2</sub>S to elemental sulfur. Complete the following redox equations.



Test CdSO<sub>4</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of cadmium sulfide.

Test  $\text{SnCl}_2$  in the above way. Record your observations and conclusions.

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Give the net ionic equation for the precipitation of tin(II) sulfide.

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Test  $\text{CuSO}_4$  in the above way. Record your observations and conclusions.

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Give the net ionic equation for the precipitation of copper(II) sulfide.

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<b>Demonstrator's Initials</b>	
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**E5-12***Method for C3: Test for Fe<sup>3+</sup>, Cr<sup>3+</sup> and Al<sup>3+</sup>*

Place the solid unknown (10 mg) in a semi-micro tube, add 4 M HNO<sub>3</sub> (0.5 mL), heat for 3 minutes, then cool. Add NH<sub>4</sub>Cl (0.5 g) and 16 M NH<sub>3</sub> (0.5 mL). Ignore a trace of brownish precipitate, probably hydrated MnO<sub>2</sub>.

A brown precipitate of Fe(OH)<sub>3</sub> indicates Fe<sup>2+</sup> or Fe<sup>3+</sup> in the original unknown.

To distinguish between Fe<sup>2+</sup> and Fe<sup>3+</sup>, dissolve the original unknown solid (10 mg) in 4 M HCl (0.5 mL) and place two drops separately on a white tile.

To one add 0.1 M 2,2'-bipyridine in ethanol (1 drop). A pink colour indicates Fe<sup>2+</sup>.

To the other add 0.1 M KSCN (1 drop). A blood red colour indicates Fe<sup>3+</sup>.

A green, grey-green or blue-violet precipitate indicates Cr<sup>3+</sup>.

A white, grey or blue-grey precipitate indicates Al<sup>3+</sup>.

The Cr(OH)<sub>3</sub> and Al(OH)<sub>3</sub> precipitates may be semi-transparent and gelatinous and only obvious after centrifuging.

Test FeSO<sub>4</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the oxidation of Fe<sup>2+</sup> to Fe<sup>3+</sup> with nitric acid.

reduction	$\text{NO}_3^-(\text{aq})$	+	+	→	$\text{NO}_2(\text{g})$	
oxidation	$\text{Fe}^{2+}(\text{aq})$			→	$\text{Fe}^{3+}(\text{aq})$	+
redox						

Give the net ionic equation for the precipitation of iron(III) hydroxide.

Test Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of iron(III) hydroxide.

Test  $\text{Cr}(\text{NO}_3)_3$  in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of chromium(III) hydroxide.

Test  $\text{Al}_2(\text{SO}_4)_3$  in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of aluminium hydroxide.

*Method for C4: Test for  $\text{Co}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Ni}^{2+}$  and  $\text{Zn}^{2+}$*

Place the solid (10 mg) in a large test-tube and add 0.4 M HCl (0.5 mL). If the anion was found to be  $\text{CO}_3^{2-}$ ,  $\text{O}^{2-}$ , or  $\text{OH}^-$  add further HCl (0.5 mL). If the anion is  $\text{CO}_3^{2-}$ , boil the solution for 5 minutes to remove all  $\text{CO}_2$ ; otherwise use only sufficient heat to dissolve the solid. Cool the solution, add solid  $\text{NH}_4\text{Cl}$  (0.1 g) and 16 M  $\text{NH}_3$  (0.5 mL), and stir well. Heat in a water-bath and add thioacetamide solution (15 drops). The generation of  $\text{H}_2\text{S}$  can be slow under these conditions and further heating for several minutes may be required before a precipitate forms.

A black precipitate (NiS) from a blue-violet solution indicates  $\text{Ni}^{2+}$ .

A black precipitate (CoS) from a brown solution indicates  $\text{Co}^{2+}$ .

A white precipitate (ZnS) indicates  $\text{Zn}^{2+}$ .

A pink precipitate (MnS) indicates  $\text{Mn}^{2+}$ .

Test  $\text{NiCO}_3$  in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of nickel(II) sulfide.

Test  $\text{CoSO}_4$  in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of cobalt(II) sulfide.

Test  $\text{ZnO}$  in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of zinc sulfide.

Test  $\text{MnSO}_4$  in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of manganese(II) sulfide.

*Method for C5: Test for  $\text{Ca}^{2+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Mg}^{2+}$*

Dissolve the solid (10 mg) in water (0.5 mL) or, if necessary, in 0.4 M HCl (0.5 mL). Add 1 M  $\text{Na}_2\text{CO}_3$  (5 drops). A white precipitate indicates  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$  or  $\text{Ba}^{2+}$ . To distinguish among these, place the solid (10 mg) on a watch-glass, moisten with 10 M HCl and perform a flame test on the mixture.

Dull red indicates  $\text{Ca}^{2+}$ .

Crimson indicates  $\text{Sr}^{2+}$ .

Pale green indicates  $\text{Ba}^{2+}$ .

No colour (other than the ever-present yellow of sodium) indicates  $\text{Mg}^{2+}$ .

If it is found difficult to distinguish between  $\text{Ca}^{2+}$  and  $\text{Sr}^{2+}$ , carry out comparison tests on known substances.

Test MgO in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of magnesium carbonate.

Test CaCO<sub>3</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of calcium carbonate.

Test Sr(NO<sub>3</sub>)<sub>2</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of strontium carbonate.

Test BaCl<sub>2</sub> in the above way. Record your observations and conclusions.

Give the net ionic equation for the precipitation of barium carbonate.

**E5-16**

*Method for C6: Test for  $\text{NH}_4^+$*

Place the solid (0.5 g) in a 100 mL beaker with 4 M NaOH (5 mL). Warm gently if necessary. An odour of  $\text{NH}_3$  indicates  $\text{NH}_4^+$ . Evolution of  $\text{NH}_3$  can be confirmed by holding a piece of moist pink litmus paper placed over (but not touching) the mouth of the beaker. If the paper turns blue, then  $\text{NH}_3$  is confirmed.

Test  $\text{NH}_4\text{Cl}$  in the above way. Record your observations and conclusions.

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Give the net ionic equation for the evolution of ammonia.

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*Method for C7: Flame tests for  $\text{Na}^+$  and  $\text{K}^+$*

Place the unknown (10 mg) on a watch-glass, add 10 M HCl (1 drop), or more until reaction ceases. Perform a flame test on the mixture.

A brilliant, persistent, yellow flame indicates  $\text{Na}^+$ .

A lilac flame, which is often masked by the yellow  $\text{Na}^+$  flame but is distinct when viewed through Co-blue glass, indicates  $\text{K}^+$ .

Test NaCl in the above way. Record your observations and conclusions.

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Test KBr in the above way. Record your observations and conclusions.

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Demonstrator's Initials	
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## Flow charts

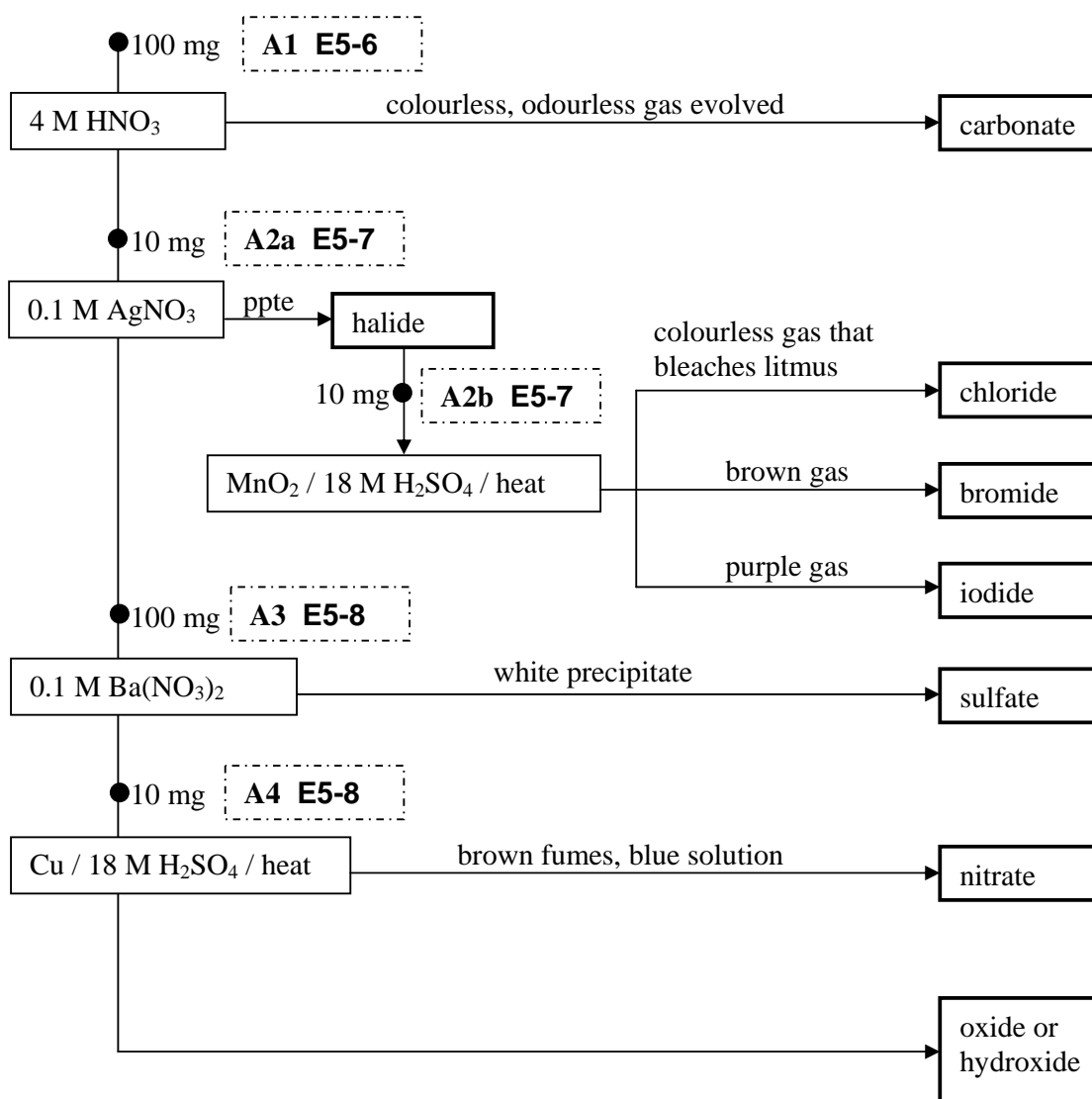
The tests A1 - A4 and C1 - C7 described in the first part of the lab-work have been carried out on compounds specifically chosen to give positive results. If conducted on other ions, the results obtained may be either positive or negative. However, if the tests are applied in the correct sequence, they can be used to identify the ions in an unknown specimen. The entire procedure is known as qualitative inorganic analysis and is summarised in the following flow charts.

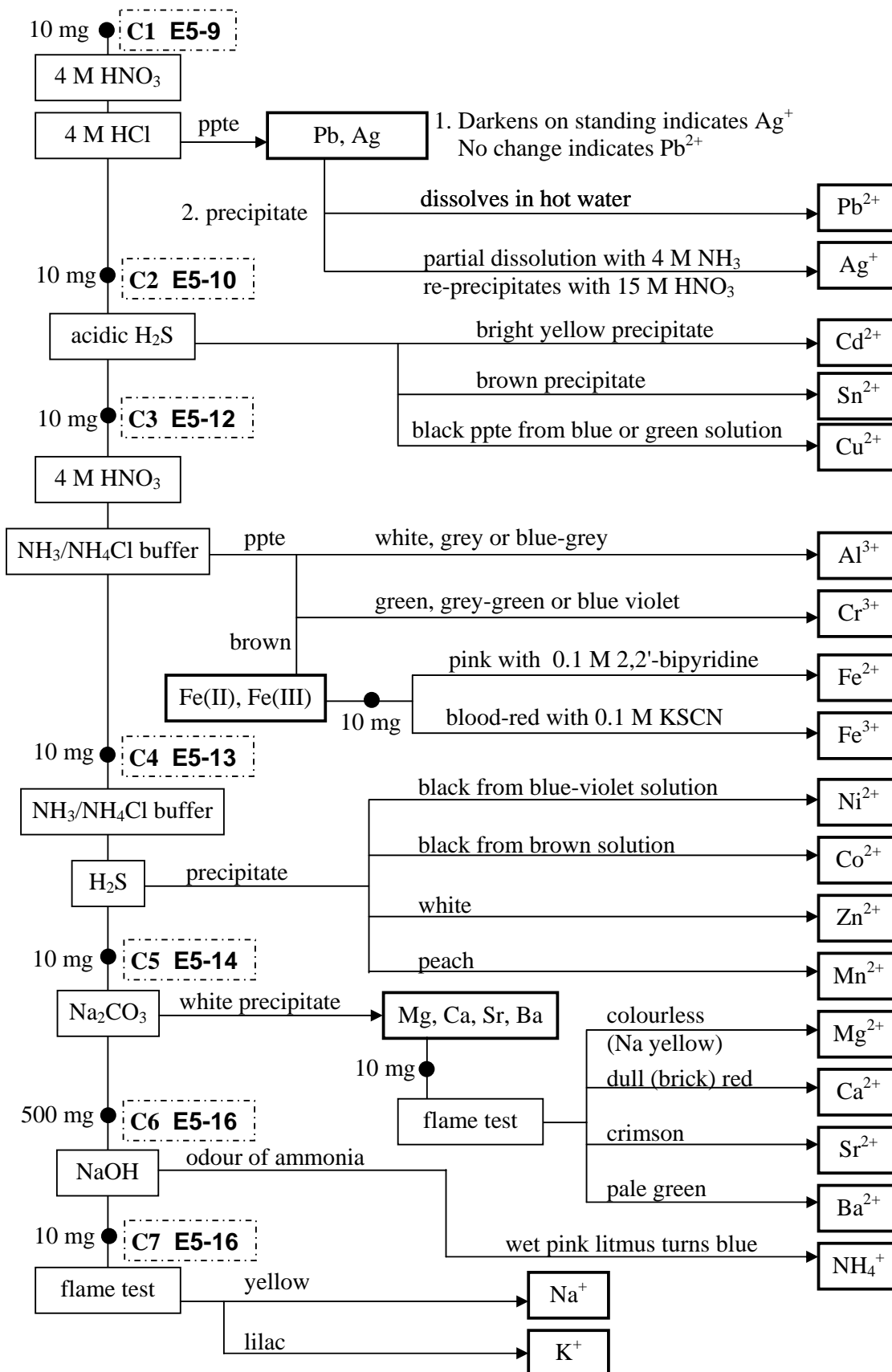
● = Take a new sample

⋮ = Test name and page number for full description

□ = reagent

□ = deduction





## Unknowns

When testing an unknown compound, it is important to perform all the tests in the specified order shown in the flow charts. A fresh sample of the unknown should be used for each test and the tests performed sequentially until a positive result is obtained.

Report your findings on each unknown in the E5 result sheets on page E5-20.

Identify at least three of the unknowns, E5A to E5P. Select any unknown (not necessarily E5A) and after identifying it, continue with the next two in alphabetical order.

Obtain a sufficient quantity of unknown for all tests by transferring, with your clean metal spatula, about 1 g of unknown to a specimen tube.

Record the colour of the solid salt. This will allow a confirmation of the suspected anion/cation pair at the conclusion of the cation identification. (Table E5-1)

Test the solubility in water and observe the colour of the solution. (See table E5-2.)

*It is important to carry out these tests in the order indicated in the flow charts.* The later tests assume that particular ions have been eliminated in the earlier tests, so positive results can only be relied upon if earlier ions have been eliminated. For example, a colourless solution gives a white precipitate when treated with  $\text{Na}_2\text{CO}_3$ . (Test C5). The ions that give this result are  $\text{Pb}^{2+}$ ,  $\text{Sn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Ba}^{2+}$ . This result is used to identify initially  $\text{Mg}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$  or  $\text{Ba}^{2+}$ , but this deduction can only be made if Tests C1, C2, C3 and C4 have all given negative results, thus showing that the cation is not  $\text{Pb}^{2+}$ ,  $\text{Sn}^{2+}$ ,  $\text{Al}^{3+}$  or  $\text{Zn}^{2+}$ .

*The anion identification is to be performed first* as the procedures used in a particular cation identification test may differ according to the type of anion. Since the unknowns used in this practical exercise will contain only one cation, it is unnecessary to continue with the cation tests once the cation has been identified.

*Remember to take the indicated quantities of chemicals.* Excess is not always better and may confuse the result.

If the anion has been identified as “oxide or hydroxide” then the colour of the compound should be used to distinguish them, if possible, once the identity of the cation is known.





