PRACTICAL NUMBER 1
TESTS FOR METAL IONS IN SOLUTION

INTRODUCTION

Most common metals have insoluble hydroxides. They can be precipitated from solution by the addition of either ammonia solution (often referred to as "ammonium hydroxide") or sodium hydroxide solution. The properties of the precipitate can be used to identify many metal ions. The exceptions are the alkali metals (Li, Na, & K) and calcium (whose hydroxide is sparingly soluble) but these metals can be identified by flame tests.

In the this practical you will meet two main types of reaction:

- precipitation reactions, where the metal ion (positive) comes into contact with a hydroxide ion (negative) to form the insoluble metal hydroxide, which is precipitated. A precipitate is marked by a cloudiness or milkiness appearing in a formerly clear solution. A precipitate may or may not settle on standing.
- redissolution reactions, where the metal hydroxide precipitated in the first step redissolves on the addition of excess sodium hydroxide or ammonia solution, or both. (By *excess* is meant *beyond that necessary to produce complete precipitation of the metal hydroxide.)*

Many of these reactions depend upon the presence of hydroxide ions in solution. Both sodium hydroxide and ammonia solutions contain hydroxide ions, but sodium hydroxide solution contains a much higher concentration of them under comparable conditions. The reason for this is that compounds of metals are *ionic*, that is they are composed of separate ions: cations (positive) and anions (negative). In solution these ions act and react independently. Thus sodium hydroxide solution contains independent sodium ions and hydroxide ions, $Na^+(aq)$ and $OH^-(aq)$ in equal concentration.

However, ammonia solution contains hydroxide ions because ammonia molecules react reversibly with water molecules to form them:

$$NH_3(aq) + H_2O(l) \rightleftharpoons NH_4^+(aq) + OH^-(aq) \qquad (1.1)$$

Only a few of the ammonia molecules in solution react with water molecules at any instant, so the concentration of hydroxide ions is much lower than the concentration of ammonia molecules. This is shown in the table:

Substance	Overall Concentration	Concentration of OH^- ions		
NaOH(aq)	1 M	1 M		
NH_3(aq)	1 M	0.004 M		

Nevertheless, the presence of hydroxide ions means that both ammonia solution and sodium hydroxide solution give precipitates of metal hydroxides where these hydroxides are insoluble – see below for examples.

i) Precipitation reactions

The addition of either ammonia solution or sodium hydroxide solution to a solution of a metal compound gives a precipitate of the metal hydroxide if that hydroxide is insoluble. Note that the hydroxides of sodium, potassium and lithium are soluble, so that no precipitate results with these ions. Calcium hydroxide is sparingly soluble, so that concentrated solutions give a precipitate whilst dilute ones do not.

For example zinc salts give a white gelatinous precipitate of zinc hydroxide:

$$Zn^{2+}(aq) + 2OH^{-}(aq) \rightarrow Zn(OH)_2(s) \qquad (1.2)$$

and iron(III) salts give a reddish-brown precipitate of iron(III) hydroxide:

$$Fe^{3+}(aq) + 3OH^{-}(aq) \rightarrow Fe(OH)_3(s) \qquad (1.3)$$

Silver salts sometimes give a white precipitate of silver hydroxide:

$$Ag^{+}(aq) + OH^{-}(aq) \rightarrow AgOH(s) \qquad (1.4)$$

but silver hydroxide is very unstable and the precipitate usually turns brown very rapidly (the white colour may not even be noticed) as it decomposes to silver oxide:

$$2AgOH(s) \rightarrow Ag_2O(s) + H_2O(l) \qquad (1.5)$$

Iron(II) salts give a pale green precipitate:

$$Fe^{2+}(aq) + 2OH^{-}(aq) \rightarrow Fe(OH)_2(s) \qquad (1.6)$$

which rapidly darkens and eventually turns brown due to atmospheric oxidation:

$$4Fe(OH)_2(s) + O_2(aq) + 2H_2O(l) \rightarrow 4Fe(OH)_3(s) \qquad (1.7)$$

Many metals may be identified simply by the colour of the precipitate they give.

Ammonia solution does not usually produce a precipitate with calcium ions because the concentration of hydroxide ions is too low and calcium hydroxide is slightly soluble in water.

Magnesium hydroxide, though insoluble, is sometimes very difficult to see, and may be missed.

Ammonium compounds do not give any precipitate with ammonia or sodium hydroxide solutions. The ammonium ion behaves like a metal ion in many reactions and is generally classed with them but, unlike a metal, ammonia is a weak base and may be displaced from its salts by a stronger one. If a mixture of sodium hydroxide solution and an ammonium salt is heated, ammonia gas is liberated. Equation (1) above is driven into reverse. It's presence may then be recognised by its smell, and confirmed by its ability to turn moist red litmus blue (it is the only common alkaline gas) and by the white smoke produced with hydrogen chloride:

$$NH_3(g) + HCl(g) \rightarrow NH_4Cl(s) \qquad (1.8)$$

Hydrogen ions give no precipitate either. (What reaction occurs here?)

ii) Flame tests

The only common metal ions which do not give precipitates with sodium hydroxide or ammonia solutions are potassium, sodium, and lithium. This is because their hydroxides are soluble. For these compounds flame tests must be used to identify them. A relatively volatile chloride of the metal is produced and heated strongly in a Bunsen flame. The vapour produced turns the Bunsen flame a colour characteristic of the metal. Sodium gives an orange-yellow, potassium a lilac and lithium a scarlet flame colour. Many other metals also produce colours. Since the colour of sodium is very intense it often obscures the colours of other metals, especially potassium, but it can be blocked out by viewing the flame through blue glass. Almost everything is contaminated by sodium in small amounts, and so only an intense and persistent coloration of the flame should be taken as positive for sodium.

iii) Redissolution reactions

The precipitation reactions above occur if a small amount of either ammonia solution or sodium hydroxide solution is added. If more is added than required to precipitate the metal completely (excess) then, in some cases, the precipitate redissolves. Whether it does or not depends both on the metal ion involved and whether sodium hydroxide or ammonia solution is used, since a different type of reaction occurs with each reagent.

For example, copper(II) salts, which form blue (or sometimes green) solutions in water, give a pale blue precipitate of copper(II) hydroxide on addition of either sodium hydroxide or ammonia solution. If excess sodium hydroxide is added to this precipitate there is no further reaction. Excess ammonia solution, on the other hand, at first causes the precipitate to darken, and then to redissolve and form a very deep blue solution. This colour is caused by the formation of a *complex ion*: the tetramminecopper(II) ion, $[Cu(NH_3)_4]^{2+}$, which is soluble in water. Complex ions are common in chemistry. For example, the carbonate and sulfate ions are complex ions.

A zinc salt reacts rather differently. Addition of either sodium hydroxide or ammonia solution results in the precipitation of white zinc hydroxide. If further reagent (either sodium hydroxide or ammonia) is added, the precipitate redissolves. Again the redissolution is due to the formation of soluble complex ions. If excess sodium hydroxide is added the soluble tetrahydroxozincate ion (sometimes referred to as the zincate ion) is formed, and a colourless solution results:

$$Zn(OH)_2(s) + 2OH^-(aq) \rightarrow \underset{\text{tetrahydroxozincate ion}}{[Zn(OH)_4]^{2-}(aq)} \qquad (1.9)$$

If excess ammonia solution is added then another colourless, soluble ion, the tetramminezinc ion, is formed:

$$Zn(OH)_2(s) + 4NH_3(aq) \rightarrow \underset{\text{tetra-amminezinc ion}}{[Zn(NH_3)_4]^{2+}(aq)} + 2OH^-(aq) \qquad (1.10)$$

Another type of behaviour is seen with iron(III). If excess of either sodium hydroxide or ammonia is added to a precipitate of iron(III) hydroxide nothing happens. No complex ions are formed in either case and so no redissolution occurs.

In the case of copper(II) ions, a hydroxo-complex is not formed but an ammine complex is. This means that copper(II) hydroxide does not redissolve in excess sodium hydroxide but does redissolve in excess ammonia solution. Contrast this behaviour with that of zinc where soluble complex ions are formed with both reagents.

Silver is similar to copper in that an ammine complex is formed but a hydroxo-complex is not. The formula of the ammine complex, however, is $[Ag(NH_3)_2]^+$.

Aluminium forms a soluble hydroxo-complex ($[Al(OH)_4]^-$) but no ammine complex and so redissolution is seen in sodium hydroxide solution but not in ammonia.

To sum up, various different things may happen when the two reagents are added in turn to samples of solutions containing metal ions. Such is the variety of different reactions that they can be used to identify most of the common metals. The possible observations with some common metal ions are summarised in the tables below.

A) ACTION OF SODIUM HYDROXIDE SOLUTION

Sodium hydroxide is added drop-wise (ie. one drop at a time) to the solution. If a precipitate is detected more sodium hydroxide solution is added to see if the precipitate redissolves. If there is no precipitate the mixture is warmed gently and the test for ammonia performed.

OBSERVATION	INFERENCE (cation present)
White ppt., soluble in excess	Lead(II), zinc, or aluminium ion[1]
White ppt., insoluble in excess.	calcium or magnesium ion
Blue ppt., insoluble in excess	copper(II) ion
Green ppt., rapidly turning brown, insoluble in excess	iron(II) ion
Brown ppt., insoluble in excess.	iron(III) or silver[2] ion
No ppt., but ammonia liberated on warming	ammonium ion
No ppt., no ammonia liberated on warming	potassium, sodium, lithium, dilute calcium, hydrogen or no ion
No ppt., solution turns from orange to yellow.	An effect due to the *anion,* the dichromate ion. (See Expt. 2,)

B) ACTION OF AMMONIA SOLUTION

Dilute ammonia solution is added drop-wise to the solution until it is present in excess in the same fashion as for sodium hydroxide.

OBSERVATION	INFERENCE (cation present)
White ppt., soluble in excess	zinc or silver ion[2]
White ppt., insoluble in excess.	Calcium (conc.)[3], magnesium, lead(II), or aluminium ion
Blue ppt., soluble in excess to form deep blue solution.	copper(II) ion
Green ppt., rapidly turning brown, insoluble in excess	iron(II) ion
Brown ppt., insoluble in excess.	iron(III) ion
Brown ppt., soluble in excess	silver[2] ion
No ppt.	potassium, sodium, lithium, calcium[3], hydrogen or no ion

C) FLAME TESTS

Perform a flame test on the solution only if necessary to identify the cation present.

FLAME COLOURATION	FLAME COLOUR SEEN THROUGH BLUE GLASS	INFERENCE (cation present)
Intense, persistent yellow or orange	No colour	Na^+
Lilac (a kind of purple red)	Crimson	K^+

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[1] The white precipitates produced by lead and aluminium are easily distinguishable. Aluminium gives pale gelatinous precipitates which settle very slowly. Lead gives dense, very white precipitates which settle very rapidly.

[2] Precipitates produced by silver may initially be white, grey, or brown, but if they are white they usually go grey or brown quite rapidly.

[3] Calcium ion does not usually give a precipitate with ammonia solution, but may do if the calcium salt is concentrated.

FLAME COLOURATION	FLAME COLOUR SEEN THROUGH BLUE GLASS	INFERENCE (cation present)
Carmine red	Violet	Li^+
"Brick red": orange red	Greenish	Ca^{2+}
"Apple green": yellowish green	Bluish green	Ba^{2+}
Blue green or azure	No colour	Cu^{2+}
Blue white	No colour	Pb^{2+}

METHOD

Consult the flow sheet on page 12 for more help with the procedure.

1) You will be provided with several substances either as solids or in solution. Each may contain one of the following cations: NH_4^+, Li^+, Na^+, K^+, Mg^{2+}, Al^{3+}, Pb^{2+}, Ca^{2+}, Fe^{2+}, Fe^{3+}, Cu^{2+}, Zn^{2+}, Ag^+. Your samples may be different from your neighbours, so be sure to identify them thoroughly when handing in work for marking (to the letter given in the table add the number you see on the container). If you are provided with solids you must dissolve them before carrying out the tests (except for flame tests). Use about 0.1 to 1 g in 5 to 10 cm^3 of water – a tiny bit on the end of a spatula in about an inch of water in the test tube. If the solution is not clear it may be allowed to settle (if necessary it may be filtered) and the clear liquid used for tests. If the material is insoluble in water it may be dissolved in dilute acid.

   Students usually make up their solutions too concentrated. This tends to produce too much precipitate and makes it difficult to redissolve the precipitate.

2) Test each of the solutions (A to F) with each reagent in turn and record your results in the results table. Do not use too much of the solutions or your results will suffer. A depth of about 1 cm in a test tube, or a volume of about 1 cm^3, is appropriate. This allows easy mixing.

3) Add 1 drop of the first reagent (dilute sodium hydroxide solution) to a sample of a solution. Mix thoroughly. Continue adding the reagent drop by drop until a precipitate, which persists on shaking, is seen. If no precipitate is seen then warm the mixture. Carefully smell

How to Carry Out a Flame Test

- Light a Bunsen with the gas full on and the air vent fully open. Find the hottest part of the flame by moving the flame-test wire in it until it glows brightest and whitest.
- Carry two small portions (about 2 cm^3 each) of concentrated hydrochloric acid from the fume cupboard to your bench in small (say 25 cm^3) beakers. Cover each with a watch glass to keep down fumes. Keep one portion for cleaning the flame-test wire and one for moistening samples.
- Clean the flame test wire until it no longer colours the Bunsen flame. This is done by repeatedly dipping the wire into concentrated hydrochloric acid and heating it as strongly as possible in the flame.
- Place a little of your solid on a watch glass and dampen it with concentrated hydrochloric acid. Dip the wire into it so as to pick up a little on the end.
- Heat the sample in the hottest part of the flame and note the colour.
- If you suspect the presence of potassium, or if the flame is orange, view the flame through blue glass to check. (Blue glass is particularly useful when potassium is present with sodium, though it may be useful with other ions too.)

the solution. If the presence of ammonia is suspected confirm by testing with moist red litmus and by placing the mouth of the tube near an open bottle of hydrochloric acid. Hydrogen chloride reacts with ammonia to produce a white smoke.

4) When the precipitate has been examined, discard most of your solution and precipitate to leave only ¼ to ½ an inch in the bottom of the test tube. Pour in the reagent to double the depth (or more if necessary) to see if the precipitate redissolves in excess. Record your observations very briefly in the results table in the *observations* column, e.g. "white ppt. redissolved in excess".
5) Proceed similarly with another portion of the same solution and the other reagent (ammonia solution). (Of course you should not do the test for ammonia - why not?) Again record your observations.
6) If there is no precipitate with either reagent, and ammonia is not detected, perform a flame test on a further portion of the solution or, if available, the solid. (You may also choose to do the flame test in other cases, for example, where calcium is suspected.)
7) On the basis of these results, and by comparison with the tables in the introduction, attempt to decide which cation is present in each case. Record the formula of the ion under "inferences"[4].
8) Write an ionic equation (or equations) to represent each and every chemical change you observed. Put these in the inferences column. (You may number your observations and equations to show which equation corresponds to which observation.)
   - There should be an equation for each observation, though one equation will often suffice for two observations.
   - If an equation is written without the observation to which it applies it cannot earn marks.
   - No equation should be written if no chemical reaction occurs.
   - No equation is necessary for a flame test.
   - **Equations which are not balanced, do not show correct charges, or do not include correct states are WRONG**.

At the end of the practical hand in your work. You will be given a mark on the basis of this.

RESULTS

If you find the following results table too small, a larger version is available at http://fbanks.info/Chem135/Pracs/Practical_number_1_results_table.pdf. It is intended to be printed on legal sized paper.

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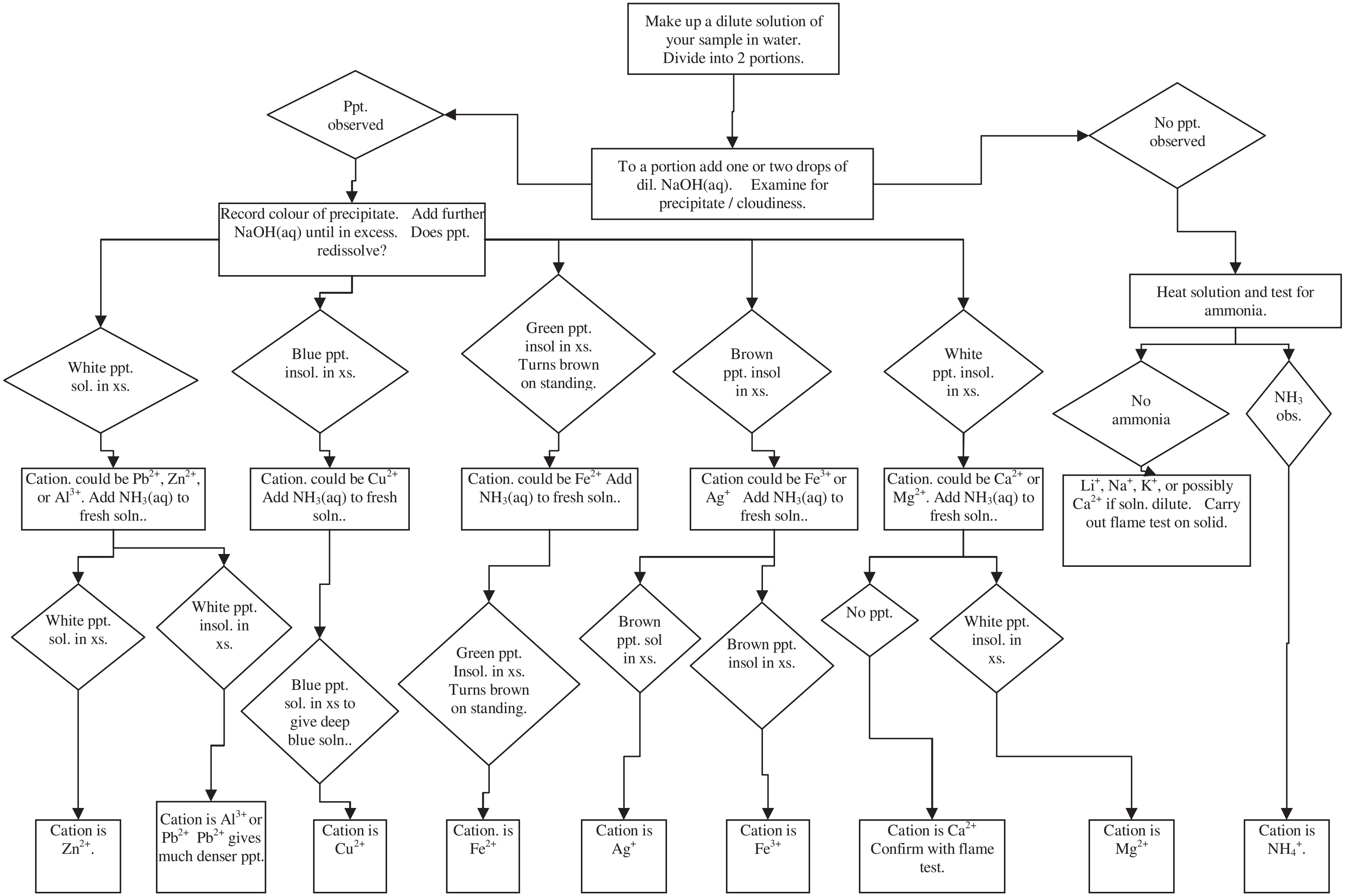
[4] Solutions should contain a compound in 0.1 M to 1 M concentration. Each sample contains one of the following ions: potassium, sodium, lithium, calcium, magnesium, aluminium, zinc, iron(II), iron(III), lead(II), copper(II), silver, ammonium. No compound contains more than one of these ions.

Flow Chart for Chem 135 Practical 1



SAMPLE TESTED (Put the number of your sample in the brackets.)	**OBSERVATION WITH NaOH(aq)**	**OBSERVATION WITH NH_3(aq)**	**FLAME COLOUR**	**INFERENCE (cation present) and ionic equation(s)** (One equation for each chemical change.)
A( )				
B( )				
C( )				
D( )				
E( )				
F( )				

PRACTICAL NUMBER 2
TESTS FOR THE IDENTIFICATION OF COMMON ANIONS IN SOLUTION

INTRODUCTION

All the more common anions (except the nitrate ion) found in solution may be identified by the precipitation of a salt containing that anion. Since all nitrates are soluble, the nitrate ion is identified by another test such as the ***Devarda's Alloy Test*** or the ***Brown Ring Test***, which do not involve precipitation. In this practical only the *Devarda's Alloy Test* is used.

a) TESTS FOR THE SULPHATE, SULPHITE, CARBONATE, HYDROGENCARBONATE, CHROMATE AND DICHROMATE IONS

Theses ions are identified by adding a solution containing barium ions (barium nitrate or barium chloride[5] solution) to the test solution. A precipitate of the appropriate barium salt forms in each case:

$Ba^{2+}(aq) + SO_4^{2}\text{-}(aq) \rightarrow BaSO_4(s)$ (2.1)

$Ba^{2+}(aq) + SO_3^{2}\text{-}(aq) \rightarrow BaSO_3(s)$ (2.2)

$Ba^{2+}(aq) + CO_3^{2-}(aq) \rightarrow BaCO_3(s)$ (2.3)

$Ba^{2+}(aq) + 2HCO_3^{-}(aq) \rightarrow BaCO_3(s) + H_2O(l) + CO_2(g)$ (2.4)

$Ba^{2+}(aq) + CrO_4^{2-}(aq) \rightarrow BaCrO_4(s)$ (2.5)

$2Ba^{2+}(aq) + Cr_2O_7^{2-}(aq) + H_2O(l) \rightarrow 2BaCrO_4(s) + 2H^{+}(aq)$ (2.6)

All the precipitates are white except for barium chromate formed in equations (2.5) and (2.6). Barium chromate is bright yellow.

Barium sulfate does not redissolve in dilute nitric or hydrochloric acid but the others do, since they are salts of weak acids:

$BaSO_3(s) + 2H^{+}(aq) \rightarrow SO_2(g) + H_2O(l) + Ba^{2+}(aq)$ (2.7)

$BaCO_3(s) + 2H^{+}(aq) \rightarrow CO_2(g) + H_2O(l) + Ba^{2+}(aq)$ (2.8)

$BaCrO_4(s) + 2H^{+}(aq) \rightarrow Ba^{2+}(aq) + H_2CrO_4(aq)$ (2.9)

All the resulting solutions are colourless, except that formed from barium chromate, which is orange. Since gases are produced with sulphites, carbonates and hydrogencarbonates, effervescence is expected, but this is not always seen, especially with sulfur dioxide, since it is very soluble in water. It has a strong, characteristic, smell, and this should be apparent even if no effervescence is seen. The gas may always be driven off by warming the solution, since gases are less soluble at higher temperatures. Confirmatory tests for the gases should always be carried out: carbon dioxide, of course, turns lime water milky.

$CO_2(g) + Ca^{2+}(aq) + 2OH^{-}(aq) \rightarrow CaCO_3(s) + H_2O(l)$ (2.10)

Most students do not realise that the complete test requires that bubbling of carbon dioxide must continue until the precipitate redissolves:

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[5] If the solution being tested contains silver or lead ions, or another metal with an insoluble chloride, addition of barium chloride will give a precipitate of that metal chloride. In such a case barium nitrate should be used.

$$CaCO_3(s) + CO_2(g) + H_2O(l) \rightarrow Ca^{2+}(aq) + 2HCO_3^-(aq) \tag{2.11}$$

Sulphur dioxide turns acidified potassium dichromate solution green[6]:

$$3SO_2(g) + 2H^+(aq) + \underset{\text{orange}}{Cr_2O_7^{2-}(aq)} \rightarrow 3SO_4^{2-}(aq) + \underset{\text{green}}{2Cr^{3+}(aq)} + H_2O(l) \tag{2.12}$$

Sulfur dioxide also give a precipitate with lime water:

$$SO_2(g) + Ca^{2+}(aq) + 2OH^-(aq) \rightarrow CaSO_3(s) + H_2O(l) \tag{2.13}$$

but this does not redissolve in excess in the same way as with carbon dioxide. Confusion between sulfur dioxide and carbon dioxide is unlikely because of the strong smell of the former.

Note that sulfites are usually contaminated with sulfate, since sulfites are gradually oxidised to sulfates by oxygen from the air. This means that the white precipitate produced with barium chloride solution may not redissolve completely on addition of dilute acid. If you suspect sulfate, always check for sulfite also.

Carbonate and hydrogencarbonate behave similarly with barium ions, producing a precipitate of barium carbonate, but differently with magnesium ions: only carbonate gives a precipitate in the cold[7]; hydrogencarbonate needs to be heated in the presence of magnesium ions in order to produce the same precipitate:

$$Mg^{2+}(aq) + CO_3^{2-}(aq) \rightarrow MgCO_3(s) \tag{2.14}$$

$$Mg^{2+}(aq) + 2HCO_3^-(aq) \xrightarrow{\text{heat}} MgCO_3(s) + H_2O(l) + CO_2(g) \tag{2.15}$$

This serves to distinguish these ions.

Chromate and dichromate can be distinguished by their colours, chromate being yellow and dichromate orange. In fact the two are in equilibrium in solution:

$$2CrO_4^{2-}(aq) + 2H^+(aq) \rightleftharpoons Cr_2O_7^{2-}(aq) + H_2O(aq) \tag{2.16}$$

The position of the equilibrium depends on the pH. Orange dichromate is turned yellow by the addition of base and *vice versa*.

b) TESTS FOR THE CHLORIDE, BROMIDE AND IODIDE IONS

These are precipitated as their silver salts. Since the ions dealt with in the last section also give precipitates with silver ions, the solution must be free of sulfate, sulfite, carbonate, hydrogencarbonate, chromate, and dichromate. For this reason the test with barium ions (as in (a) above) must be performed before this test. Silver nitrate solution is added to a ***fresh sample*** of the original solution. If a precipitate is observed it is the corresponding silver halide:

$$Ag^+(aq) + Cl^-(aq) \rightarrow \underset{\text{white}}{AgCl(s)} \tag{2.17}$$

$$Ag^+(aq) + Br^-(aq) \rightarrow \underset{\text{very pale yellow or cream}}{AgBr(s)} \tag{2.18}$$

$$Ag^+(aq) + I^-(aq) \rightarrow \underset{\text{pale yellow}}{AgI(s)} \tag{2.19}$$

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[6] The green may be too pale to see. The solution may appear to be colourless.

[7] Magnesium chloride solution may give a precipitate with hydrogencarbonate in the cold if the hydrogencarbonate solution is too concentrated.

The colours indicated are the colours of the fresh precipitates. Silver halides decompose rapidly in the presence of light (the reaction used in photographic film) and so the colours change rapidly.

The colours of the fresh precipitates are rather similar. It is essential to perform a confirmatory test to be sure which anion is present. Silver chloride is soluble in dilute ammonia solution (as well as concentrated ammonia solution), silver bromide is only soluble in concentrated ammonia solution (though rather slowly), whilst silver iodide is not soluble in either. The formation of the soluble diamminesilver ion ($[Ag(NH_3)_2]^+$) is responsible in both cases:

$$AgCl(s) + 2NH_3(aq) \rightarrow [Ag(NH_3)_2]^+(aq) + Cl^-(aq) \qquad (2.20)$$

$$AgBr(s) + 2NH_3(aq) \rightarrow [Ag(NH_3)_2]^+(aq) + Br^-(aq) \qquad (2.21)$$

This ion was also encountered in the test for silver ions that you did in practical #1. It is responsible for the redissolution in excess of the precipitate formed by mixing a solution of a silver salt with dilute ammonia solution.

c) TEST FOR THE NITRATE AND NITRITE IONS

If the solution has been tested with both barium ion and silver ion solutions, and no precipitate has been produced, it is possible that the solution contains nitrate ion. Since all common nitrates are soluble, nitrate ions are not usually detected by means of a precipitation reaction. One possible test is the *Devarda's Alloy Test.* In this latter, the nitrate ion is reduced to ammonia with Devarda's alloy or equivalent.

$$3NO_3^-(aq) + 18H_2O(l) + 8Al(s) + 5OH^-(aq) \rightarrow 3NH_3(aq) + 8[Al(OH)_4]^-(aq) \qquad (2.22)$$

(Nitrite ions react similarly but will not be included amongst your unknowns.)

Devarda's alloy is a mixture of aluminium, copper and zinc. It is used in the presence of concentrated sodium (or potassium) hydroxide. Aluminium foil may be used in place of Devarda's alloy. The aluminium also reacts with the hydroxide ion (very exothermically) to produce hydrogen gas, so effervescence is seen whether or not the nitrate ion is present:

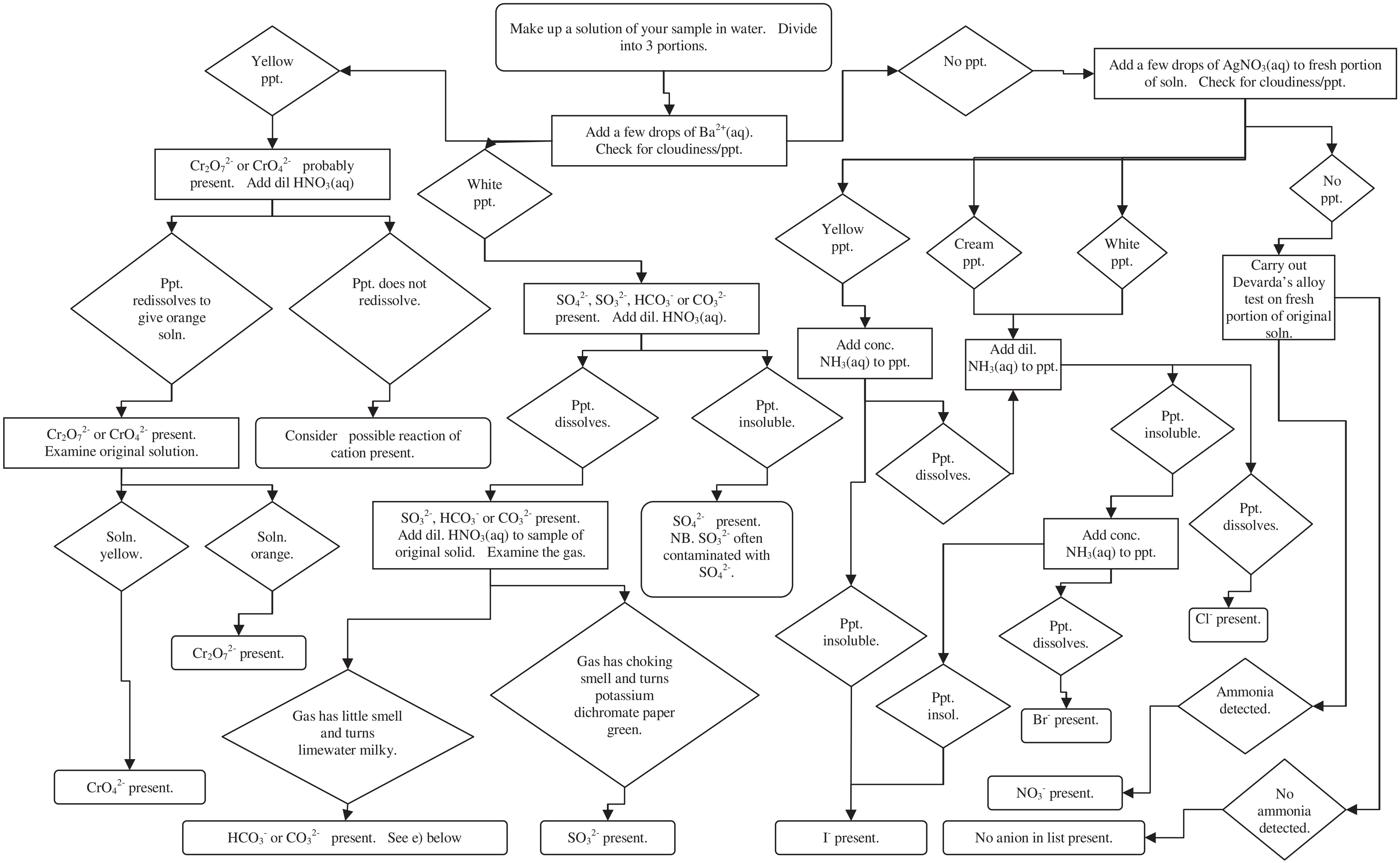
$$2Al(s) + 2OH^-(aq) + 6H_2O(l) \rightarrow 2[Al(OH)_4]^-(aq) + 3H_2(g) \qquad (2.24)$$

Since ammonium salts evolve ammonia when warmed in the presence of an alkali, the presence of ammonium ion must be eliminated before the test is carried out.

METHOD

1) You are provided with six samples. They may be solids or solutions. If they are solids you should dissolve them as in the previous experiment. Bear in mind that if you use an acid you also introduce an anion. Any effervescence should be treated as an important observation and any gas evolved should be thoroughly tested. Note also that your samples may not be same as your neighbour's so that it is very important to identify samples completely in any work submitted for marking.
2) Carry out the following tests, as appropriate, on separate 1 or 2 cm^3 samples (¼ to ½" depth) (Use a measuring cylinder the first time and then judge by eye.) Each solution contains only one of the following anions: nitrate, sulfate, sulfite, chloride, bromide, iodide, carbonate, and hydrogencarbonate. However a particular anion may be found in more than one solution.
3) Do not do every test on every solution. On a given sample, carry out the tests in order until the anion is identified. Then go on to the next sample.
4) Refer to the flowchart on page 17 for more guidance.

Flowchart for Chem 135 Practical 2



1) TEST WITH BARIUM CHLORIDE (OR BARIUM NITRATE) SOLUTION[8]

a) Add ***barium chloride*** solution dropwise to a 1 or 2 cm^3 sample of a solution.

b) If a precipitate forms the identity of the anion may be determined at this stage. If the solution is coloured, make sure that all the anion is precipitated by adding excess Ba^{2+}. If the colour persists, it is probably due to the cation. Pour off the supernatant and wash the precipitate before proceeding. If no precipitate forms go on to test with silver nitrate solution.

c) Add dilute nitric or dilute hydrochloric acid to see if the precipitate dissolves in acid. Look for effervescence and smell the solution carefully. Warming the solution helps to expel any gas. See the table below for further details.

d) If a carbonate, hydrogen carbonate or sulfite is suspected, produce more gas by adding the acid to a sample of the original solid. and test it appropriately:

   i) If carbon dioxide is suspected, (smell of Alka Seltzer®) test for the gas by bubbling it through lime water.

   ii) If sulfur dioxide is suspected, test the gas with a piece of paper dipped in a mixture of potassium dichromate solution and dilute sulphuric acid (potassium dichromate paper.)

e) To distinguish between carbonate and hydrogencarbonate, add magnesium chloride solution to a fresh sample of your solution. Carbonate gives an immediate precipitate in the cold. Hydrogencarbonate requires warming.

f) If chromate or dichromate is suspected, examine the original solution.

Ion suspected	**Colour of ppt., and its behaviour with dilute strong acid.**	**Comments**	**Confirmatory tests**
SO_4^{2-}	White, insoluble	No reaction	None
SO_3^{2-}	White, soluble	Probably no effervescence, sharp smell of $SO_2(g)$ especially on warming.	Gas turns moist blue litmus paper red and decolourises potassium dichromate paper or turns it green.
CO_3^{2-} or HCO_3^-	White, soluble	Probable effervescence. Faint Alka Seltzer® smell.	Add dilute acid to fresh solid. Bubble gas through limewater. Limewater turns milky and then clear. Add $MgCl_2(aq)$ to soln. of sample: gives immediate ppt. in cold with CO_3^{2-} but only on warming[7] with HCO_3^-.
CrO_4^{2-} or $Cr_2O_7^{2-}$	Yellow, soluble to give orange solution.	No gas evolved.	Examine original solid or solution. Orange indicates $Cr_2O_7^{2-}$, yellow indicates CrO_4^{2-}. (Note that this assumes a colorless cation.)

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[8] Some metals have insoluble chlorides (e.g lead, silver and mercury). A white precipitate of the metal chloride will result when barium chloride solution is added. Barium nitrate solution should be used instead.