Established in 1879

RAFFLES GIRLS' SCHOOL (SECONDARY)
RAFFLES PROGRAMME
YEAR 4 CHEMISTRY

Name : __________________________ (   ) Class : _______
Date : ______________

# QA Notes (I) – Introduction and Laboratory Techniques

Qualitative analysis (QA) is the process of identifying the **cations** and **anions** present in unknown substances using chemical reactions and test reagents. Unlike quantitative analysis, where the amount of a particular chemical is measured, a qualitative analysis scheme simply confirms the presence or absence of certain chemicals or ions. The chemical reactions used to identify the cations and anions mostly involve observable changes including:

- production of a **precipitate**,
- evolution of a **gas**, or
- visible **colour change**

## 1.1 Preliminary examination

Before carrying out any chemical reaction, a preliminary examination to note the appearance, colour and solubility of the unknown substance often gives clues to the identity of the unknown chemical or the element present in the substance.

### (a) Colour

- In general, compounds of **transition metals** have characteristic colours.
- Group I, II and III metals generally form white salts that dissolve to form colourless solutions.
- However, as can be seen from **Table 1**, there are several possibilities for a given appearance. The appearance is not conclusive and you should carry out further chemical tests to arrive at your final conclusion about the identity of the sample.

**Table 1**: Characteristic colours of some chemicals

| **Colour** | **Inferences** |
|---|---|
| Colourless | Dilute acids, alkalis and solutions of salts of Group I, II and III metals |
| White | Solid salts of $Na^+$, $K^+$, $NH_4^+$, $Ca^{2+}$, $Zn^{2+}$, $Pb^{2+}$, $Al^{3+}$ |
| Black | $CuO$, $CuS$, $CoO$, $FeO$, $FeS$, $PbS$, $MnO_2$, $I_2$ crystals |
| Grey | Metals e.g. Zn, Fe, Al in powder form |
| Dark green | Chromium salts |
| Light green | Iron(II) salts |
| Blue or bluish green | Hydrated copper(II) salts |
| Yellow or brown | Solutions of iron(III) salts ($Fe^{3+}$), $PbI_2$, $AgI$ |
| Pale pink | Manganese(II) salts ($Mn^{2+}$) |
| Purple | $KMnO_4$ |

- Crystalline salts sometimes show different colours when in hydrated and anhydrous states. For example, $CuSO_4.5H_2O$ is blue and anhydrous $CuSO_4$ is white in colour.

### (b) Solubility of ionic compounds

Below is a summary of the solubility of common substances in water.

| The following are **soluble in water:** | |
|---|---|
| 1. Salts of $NH_4^+$, $K^+$, $Na^+$ | |
| 2. Nitrates | |
| 3. Hydrogencarbonates | |
| 4. Sulfates | ***except*** $PbSO_4$, $BaSO_4$, $CaSO_4$ (sparingly soluble) |
| 5. Halides including<br>Chlorides<br>Bromides<br>Iodides | <br>***except*** $PbCl_2$, AgCl<br>***except*** $PbBr_2$, AgBr<br>***except*** $PbI_2$, AgI |

| The following are **insoluble in water:** | |
|---|---|
| 1. Carbonates | ***except*** those of $Na^+$, $K^+$, $NH_4^+$<br>CaO, $Ca(OH)_2$, CaS, $CaSO_3$ are slightly soluble. |
| 2. Hydroxides | |
| 3. Oxides | |
| 4. Sulfides ($S^{2-}$) | |
| 5. Sulfites ($SO_3^{2-}$) | |

## 1.2 Types of Reactions involved in QA

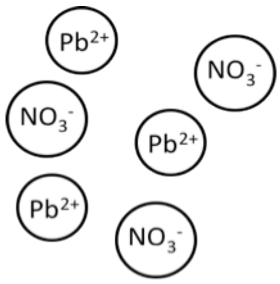
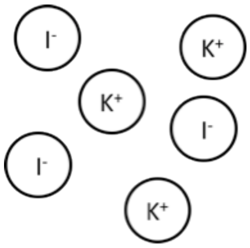
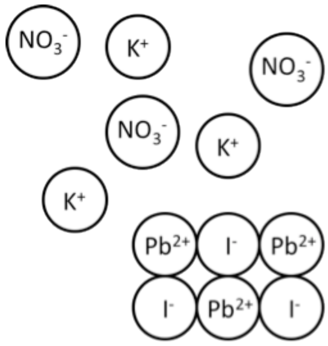
After a preliminary examination of the appearance and solubility of the unknown substance, chemical reactions such as precipitation reactions, acid-base reactions, thermal decomposition and redox reactions are carried out to identify the unknown chemical or ion(s) present in the substance. In a **confirmatory test**, each ion has a unique response to an added chemical such as a color change of the solution or the formation of a precipitate.

For example, if an acid is added to the solution and a gas that forms a white precipitate in limewater is evolved, we can conclude that the solution contains carbonate ions ($CO_3^{2-}$ ions). This test described is a confirmatory test for the presence of carbonate ions.

Hence, observations specific to certain types of chemical reactions allow us to infer and make conclusions about the identity of specific ions.

## (a) Precipitation Reaction

- Precipitation reaction is one that results in the formation of an insoluble product when two solutions are mixed together.
- The insoluble solid formed separates from the solution and is called a precipitate. For example, when we mix lead(II) nitrate solution and potassium iodide solution, a yellow precipitate of lead(II) iodide forms.
- In the boxes below, draw the changes in the arrangement of the ions when precipitation takes place between lead(II) nitrate and potassium iodide solutions.

| | | |
|---|---|---|
|  |  |  |
| *lead(II) nitrate solution* | *Potassium iodide solution* | *What happens when the two solutions are mixed together?* |

Chemical equation: $\mathbf{Pb(NO_3)_2\,(aq) + 2KI\,(aq) \rightarrow PbI_2\,(s) + 2KNO_3\,(aq)}$

Ionic equation: $\mathbf{Pb^{2+}\,(aq) + 2I^-\,(aq) \rightarrow PbI_2\,(s)}$

## (b) Acid-base neutralization reaction (Revision)

- An acid is a substance that dissociates in water to produce hydrogen ions. Common laboratory acids include dilute hydrochloric acid, dilute nitric acid and dilute sulfuric acid.

- A base is a substance that reacts with acids to produce a salt and water. Bases can be metal oxides or hydroxides. Examples of bases are iron(II) oxide, iron(II) hydroxide, zinc oxide, zinc hydroxide, copper(II) oxide and copper(II) hydroxides.

- An alkali is a substance that dissociates in water to produce hydroxide ions. Common alkalis in the laboratory are sodium hydroxide solution, potassium hydroxide solution and aqueous ammonia.

### (c) Thermal Decomposition

- Thermal decomposition is a reaction in which a single compound breaks up into two or more simpler compounds or elements when heated. The decomposition reaction is irreversible.

*Example 1* Copper(II) carbonate decomposes on heating to form copper(II) oxide and carbon dioxide:

$$\mathbf{CuCO_3\,(s) \rightarrow CuO\,(s) + CO_2\,(g)}$$

*Example 2* Zinc nitrate decomposes on heating to give zinc oxide, nitrogen dioxide and oxygen:

$$\mathbf{Zn(NO_3)_2\,(s) \rightarrow 2ZnO\,(s) + 4NO_2\,(g) + O_2\,(g)}$$

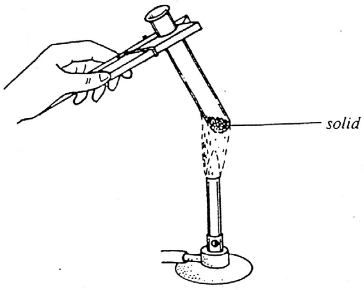
### (d) Redox reaction – to be covered in next topic

## 1.3 Laboratory Techniques

### (a) Quantity of Substances Used

There is a common misconception that using large quantities of chemicals for a reaction would produce better results. In fact, using excessive amounts of chemicals could lead to a waste of time as it takes a longer time for the reagents to react. It may even lead to wrong results e.g. if too much of salt is present, its solubility in excess reagent will not be observed.

Students are recommended to use the following guidelines, unless otherwise specified:



i. If tests are carried out with the unknown in its **solid** form, the amount used should be just enough to fill the **hemisphere** at the bottom of a test tube.

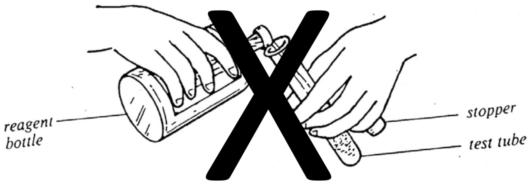
ii. If tests are carried out with a solution of the unknown, a stock solution can first be prepared. The **stock solution** may be prepared by filling the bottom hemisphere of a test tube with the solid and dissolving it in half a test tube of **distilled water**. For each test, fill the test tube to a depth of approximately **1 cm** with the stock solution.

***Why is tap water not used to prepare stock solution?***

**Tap water may contain ions such as $Ca^{2+}$, $Mg^{2+}$, $NO_3^-$, $SO_4^{2-}$ and $Cl^-$ and using it to prepare stock solution could lead to 'false-positive' results.**

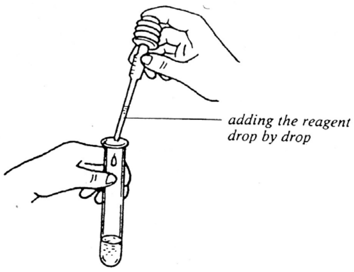
## (b) Handling of Reagents

- Use a **spatula** to transfer **solid** chemicals. Avoid spilling on your hands, clothes or table tops as some chemicals cause stains or burns.



- Before using a solution from a reagent bottle, **read the label to avoid using the wrong reagent**. Hold the bottle on the side of the label to avoid spilling the solution over the label. Then pour the solution carefully into the test tube. Always remember to replace the stopper after the solution has been used.

- It is essential that all reagents should be free from contamination. In transferring a liquid reagent from a dropper into the test tube containing the solution of the unknown, hold the dropper **just above** the mouth of the test tube and allow the reagent to drop into the test tube. **Do not leave the dropper on the bench or allow the dropper tip to touch anything except for the reagent itself.**

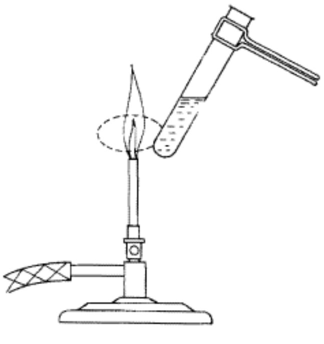


- The reagent should be added **dropwise**. If not, you may miss certain observations. As a result, you may draw the wrong conclusion.

## (c) Heating

- When heating an unknown in its **solid** form, do so **gently** at first and then more strongly. If not, you may miss certain observations.

- When a **solution or suspension** is heated, care should be taken to prevent spurting. Apply a small flame near the surface of the liquid and remove it once the solution starts boiling. The test tube should be moved in and out of the flame intermittently and shaken gently.

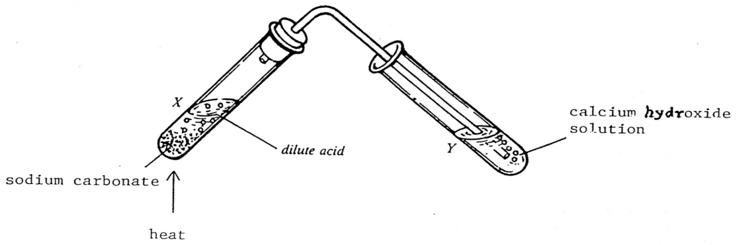


## (d) Testing for gases

- You should test for a gas **when you observe effervescence or when you heat a substance.** Although instructions may not be given to test for a gas, you are expected to carry out tests and write down your observations and conclusions when you suspect that a gas may be given off. You should record the colour and odour of the gas and any specific test that determine its identity, e.g. reaction with litmus, glowing or lighted splints, or limewater.

- When testing carbon dioxide with limewater (calcium hydroxide solution) or testing sulfur dioxide with acidified potassium manganate (VII) solution, a delivery tube may be used to deliver the gas into about 2 $cm^3$ of the test solution.

- Heat is used to generate the gas in the reactant test tube **X** which is then bubbled into the test reagent in test tube **Y**. It is important to remember that the **test reagent in test tube Y must be removed before removing reactant test tube X from the flame**. This is to ensure that suck back does not occur. Otherwise, if the flame is removed, the gases in the reactant test tube **X** would cool and contract and the test reagent would then get "suck back" into the hot reactant test tube.



## (e) General work ethics

- Always work in a tidy and systematic manner. Remember that a tidy bench is indicative of a methodical mind.
- Reagent bottles and their stoppers should not be left on the bench. They should be returned to their correct places upon the shelves immediately after use.
- When carrying out a test involving the formation of precipitate, make sure that both the solution to be tested and the reagent are absolutely free from suspended particles. Contamination often gives wrong observations and conclusions.
- Do not waste gas or chemicals. The size of the Bunsen flame should be no larger than absolutely necessary. It should be extinguished when no longer required. Avoid using unnecessary excess of reagents. Reagents should always be added portion-wise.
- It is important that students should not follow instructions blindly, but rather understand the purpose of the operation e.g. addition of reagents, being aware of the nature of the reagent used (whether it is acting as an oxidizing or reducing agent) and then make logical deductions from the observations noted.
- All results, whether positive or negative or inconclusive, must be recorded clearly and concisely at the time they are made. The writing up of experiments should not be postponed until after one has left the laboratory.

### (f) Safety Rules

- Always wear safety glasses when working in the laboratory.
- Long hair must be tied and long fringes pinned up while in the laboratory. PE shorts are not to be worn in the laboratory.
- Contact lenses should not be worn in the laboratory. Chemicals can get under the lens and damage the eye before the lens can be removed. It is often very difficult to remove the contact lens from the eye after a chemical splash.
- Close the air hole of the Bunsen burner when lighting the flame. Once the flame is on, adjust the air hole to give a non-luminous flame. Normal heating is usually done with a Bunsen flame in which the air hole of the burner is half opened.
- Whenever heating is done in a test tube, use a test tube holder and never point the mouth of the test tube at your classmate or yourself.
- Never look down into a test tube (even with safety glasses on – there is still a risk of hot solution suddenly being ejected into your face). Always view coloured products through the wall of the test tube.
- If a poisonous gas is liberated during an experiment, the rest of the experiment should be carried out in a fume cupboard. Never breathe too deeply when noting the odour of the gas evolved.
- Great care should be taken when handling concentrated acids.
- Report all burns, cuts and breakages to your teacher immediately.

## References

1. Kam, K. W., & Kwan, S. N. (2000). *Laboratory Manual: Qualitative Analysis (Inorganic and Organic) for 'A' Level* (5th ed.). Singapore: Federal Publications.

2. Svehla, G. (1990). *Vogel's Qualitative Inorganic Analysis* (Rev. 6th ed.). Harlow: Longman.