COLLEGE OF THE BAHAMAS

SCHOOL OF CHEMISTRY, ENVIRONMENTAL & LIFE SCIENCES

COLLEGE PREPARATORY CHEMISTRY

(CHEMISTRY 071)

PRACTICAL HANDBOOK

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Cover Picture: *A crystal of antimony through the scanning-tunnelling microscope. Individual atoms can be seen, though their shapes are distorted.*

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RULES FOR THE PROTECTION AND SAFETY OF STUDENTS IN THE CHEMISTRY LABORATORY

The following rules are not intended to make life difficult for you. Rather they are for your protection. If you follow them carefully the laboratory will be a safe place in which to work.

The chemistry laboratory is not a play-room. Many of the chemicals you will encounter are flammable, or poisonous, or could cause burns. The apparatus, too, can be hazardous at times. If apparatus and chemicals are not properly used they could cause serious injury either to yourself or to others. For these reasons you should read, and think carefully about, the following regulations.

1) You are not allowed to work in the laboratory unless a supervisor is present.

2) You must wear safety glasses in the laboratory. You are expected to buy your own. They are available from Chapter One and other local stores.

3) You must wear suitable clothing: no loose or hanging sleeves or hair or other items of clothing. Shoes must be of the closed type – no slippers.

4) Always follow your supervisor's instructions carefully.

5) Treat chemicals as dangerous until you know better.

6) Never taste or smell chemicals unless instructed to do so by your supervisor. They may be poisonous.

7) Try not to get chemicals on your hands or skin generally. Some of them are caustic (they cause burns). Immediately after handling corrosive or poisonous substances, even if you only touched the bottle, always rinse your hands under the tap.

8) Fuming or volatile chemicals are best handled in the fume cupboards - or failing this, near a window.

9) Never eat or drink in the laboratory. You do not know what you may have picked up on your fingers. By the same token always wash your hands before you leave the laboratory.

10) There is a shower in the laboratory which you can dive under if you spill a lot of something dangerous (e.g. concentrated sulfuric acid) on yourself. Make sure you know where it is and how to work it. Cool, calm action can avoid injury from even the largest spills.

11) There is an eye-bath in the laboratory. You can use it to rinse things out of your eyes. Make sure you know where it is and how it works.

12) There are fire extinguishers in the laboratory. Make sure you know where they are and how to use them. Acquaint yourself with the type of fire that each is to be used with.

13) Stay at your bench unless there is good reason to move. Do not wander aimlessly around the laboratory. NEVER run in the laboratory!

14) Do not sit down to do experimental work (except titrations). You may need to move quickly to avoid danger!

15) Do not clutter the floor with bags or stools. When you need to move quickly you don't want to trip up!

16) Do not work with a cluttered bench. Keep things in order and keep books and so on to a minimum. Cluttered benches lead to accidents.

17) Do not reach right over the bench. Go round the bench to the sink, for example.

18) Never heat substances in sealed test tubes or other containers. They may explode.

19) When doing work with test tubes, especially heating them, make sure that the tube is pointed away

from yourself and others. Things can sometimes shoot out of the end.

20) Clean up spills and broken glass-ware immediately. They can be dangerous to others (and you!) if left.

21) Always work with small quantities of substances. This will make any danger smaller, too.

22) When handling glass-ware (for example pushing glass tubes through stoppers etc.) make sure that you find out the proper way to do it. People have been known to push broken-off tubes through their hands! The golden rule is to keep your hands as close together as possible and cover the glass with a cloth.

We should be grateful if you would also observe the following rules which are not directly connected with safety.

1) Do not throw solid matter (e.g.. filter paper) into the sinks. They easily get blocked up. Put solid waste into the trash can. Always leave your sink completely empty.

2) Report all breakages to the supervisor. We need to know what we are losing so that we can restock.

3) Please do not write on the benches. It is difficult to clean writing off.

4) Leave your bench as you would like to find it, even if it wasn't too good when you arrived!

5) Some laboratory apparatus is coated with plastic e.g.. clamps and test-tube racks. Make sure you keep clamps well away from heat and don't put very hot test-tubes in racks. You CAN put them on the bench, since the bench is more heat-resistant, unless they are extremely hot. In the latter case rest them on a gauze to cool.

ADVICE TO STUDENTS

1) Students must present themselves for practicals on time, properly dressed, and equipped with their safety glasses. Your lecturer/supervisor will give you detailed guidance. **Failure to comply will result in exclusion from practical and the loss of any credit pertaining to the practical.**

2) Attendance at practicals is compulsory. If you do not attend, your mark will automatically be zero, unless you present a medical certificate. (Medical certificates may be obtained from the campus nurse at no charge.) In some cases, *if you act in good time,* it may be possible to attend another section of the practical.

3) Time is very limited during practicals. In order to make the best possible use of your time in the laboratory, study the introduction and method sections carefully before coming to the practical. Consult other sources when you encounter difficult material.

4) Always complete as much as possible of the calculation before clearing up your apparatus. The calculations may indicate that you need to repeat some parts of your experiment. Your lecturer can advise you on this.

5) Always discuss your results with your lecturer or laboratory supervisor before leaving.

6) Most practicals will be evaluated by means of a short quiz at the beginning of the next practical. In order to obtain the best grade you must complete all the practical work and answer the questions in the problem section at the end of the section. You must also think carefully about the reasoning behind the procedures you are asked to carry out.

PRACTICAL CLASS NO. 1
AN INVESTIGATION OF SOME
PHYSICAL & CHEMICAL CHANGES

INTRODUCTION

Physical Properties and Chemical Properties

Each pure substance has its own properties which distinguish it from other substances. These are classified as either *physical* or *chemical* properties.

Physical properties are those that can be observed without changing the nature of the substance. They include the melting point and the boiling point of a substance, its solubility, texture, density and so on.

Chemical properties, on the other hand, can only be observed when a substance undergoes a change in its essential nature. Chemical properties include the "ability" of iron to rust, silver to tarnish, paper to burn, and sodium to react violently with water. Note the striking change in nature that the substance undergoes in each case: rust, for example is completely different from iron. This change in nature is known as a *chemical reaction* or *chemical change*.

Physical Changes and Chemical Changes

Closely connected with physical and chemical properties are physical and chemical *changes*. Physical changes are those changes that occur without any alteration in the underlying nature of the substance. These include *changes of state* such as boiling, melting and freezing. In each case a simple alteration in temperature may reverse the change completely. Chemical changes, on the other hand, bring about a fundamental change in the nature of the substance involved. Burning and rusting are just two of the numerous possible examples.

Four distinguishing features of physical and chemical changes are as follows:

QUESTION	PHYSICAL CHANGE	CHEMICAL CHANGE
Is a new substance formed?	No	Yes
Is a heat change involved?	No	Yes
Is the process *easily* reversible?	Yes	No
Is there any apparent change in mass?	No	Yes

The above differences between physical and chemical changes can only serve as a general guide. There are many exceptions. The distinction between a chemical and a physical change is not always clear cut. For example, condensation (the change of a gas into a liquid e.g. steam into water) is a physical change, but it involves the release of a great deal of heat. It is, however, easy to reverse (water can easily be changed back into steam). Again, breaking a priceless ornament is a physical change, but is very difficult to reverse! On the other hand there are some chemical changes which are easy to reverse. When an acid (such as vinegar) is added to blue litmus (a vegetable dye: you could try it at home using the juice from

beets) it changes to red. When an alkali is added (baking soda or some types of drain cleaner will do) it changes back to blue again.

The following experiments will introduce you to the essential differences between physical and chemical changes. In each case you should record your observations carefully on these sheets in the table provided for the purpose. Since extra sheets will not normally be available we suggest the use of a soft pencil. Note particularly whether any changes that occur are reversed later in the experiment, or are *easily reversible*, or whether they are *difficult to reverse*, and lead to a fundamental change in nature of the substance involved. You cannot draw any conclusions about changes in mass (unless a substance disappears completely), since we do not weigh anything in this practical.

In each case try to decide whether the change occurring is physical or chemical. This will form your conclusion in each case.

METHOD

A. Dissolution of Sodium Metal and Sodium Chloride in Water

The dissolution (i.e. "dissolving") of sodium metal in water is rather violent and can on occasion be dangerous. This will therefore be demonstrated to you by your lecturer. The observations have already been included in the chart to help you understand exactly what is required in other cases. Everyone has seen sodium chloride (common salt) dissolve in water. You may also be aware that the salt can be recovered unchanged by evaporating off the water. And yet it is true that common salt contains sodium. The properties of the two substances are very different. Record in the space provided what you observe when you dissolve common salt in water. You may try it if you like by shaking a little salt with a small amount of water in a test tube.

B. Heating Metals in Air

You are supplied with three different metals: lead, magnesium and nickel. Carefully carry out the following procedures and record your observations in the chart.

1) Grip a piece of lead in a pair of tongs in one hand and hold it well over the bench. Take a lit Bunsen burner in the other. Direct the Bunsen flame at an angle onto the lead. Do not allow anything to fall into the Bunsen burner. Note carefully any changes that the lead undergoes. Would you call them reversible or irreversible? Does the material left at the end differ very much from the material at the beginning?

2) Grip a piece of magnesium ribbon a few inches long in a pair of tongs and push the tip of it into the hottest part of a Bunsen flame. As soon as it catches fire remove it from the flame. Do you think any heat is being given out at this stage? Examine the ash produced and compare it with the original magnesium.

3) Hold the nickel wire in a pair of tongs and heat it in the hottest part of the Bunsen flame until it glows orange or white. Allow it to cool and then repeat the procedure two or three times. Consider carefully whether changes are fundamental or only superficial.

C. Heating Sulfur in the Absence or Presence of Air

1) Fill the end of a spatula with powdered sulfur ("flowers of sulfur") and place the sulfur in the bottom of a long narrow test tube. This will tend to prevent the air from reaching the sulfur in the bottom of the tube. Grip the test tube in a test-tube holder and heat the lower end gently and carefully in the Bunsen flame. Allow the material in the tube to bubble for a moment or two and then remove the tube from the flame and allow it to cool. Compare the contents of the tube with the starting material. In particular, scrape the surface of the solid lump in the tube with a spatula and look at the powder

produced. Probably some yellow powder will have formed in the cool parts of the tube. Examine this carefully.

2)   Take a deflagrating spoon and remove any lead or other material it may contain. Put a little sulfur in the spoon and heat carefully in a Bunsen flame. A gas will be liberated. Note its smell. Test the gas by placing a piece of damp blue litmus paper in it. This will tell you whether the gas is acidic or not.

D. Heating Lead(II) Nitrate

Put a very few crystals of lead(II) nitrate into a clean deflagrating spoon and heat it carefully in the Bunsen flame. Test the brown gas evolved with damp blue litmus paper. Continue to heat the crystals until there is no further change. Be sure to examine the solid residue. Note that it melts easily and changes colour when heated. Your lecturer may have a demonstration ready to show how the gas evolved can be tested for oxygen, and how the brown gas can be condensed and collected as liquid.

RESULTS AND OBSERVATIONS

A   DISSOLUTION OF SODIUM AND SODIUM CHLORIDE IN WATER

SUBSTANCE	OBSERVATIONS	CONCLUSIONS
SODIUM METAL	Sodium is a soft silvery solid metal. As soon as the sodium touched the water, bubbles of gas were evolved. The sodium melted to form a small, round ball and raced about the surface giving off white fumes. A hissing noise was also heard. The ball of sodium grew smaller until it eventually disappeared with a small explosion[1]. When tested with red litmus paper, the water in the trail of the sodium turned the litmus blue. This showed the presence of an alkali.	The nature of the sodium changed completely during the experiment. A new kind of matter was formed with evolution of heat and light. The mass of the original sodium decreased to nothing This showed that a chemical change had occurred.

1If too large a piece of sodium is used a very violent explosion may result. Since lecturers are considered more expendable than students, this procedure is demonstrated.

DISSOLUTION OF SODIUM AND SODIUM CHLORIDE IN WATER (CONTINUED)

SUBSTANCE	OBSERVATIONS	CONCLUSIONS
SODIUM		
CHLORIDE		

B   HEATING METALS IN AIR

METAL	OBSERVATIONS	CONCLUSIONS
LEAD		
MAGNESIUM		

HEATING METALS IN AIR (CONTINUED)

METAL	OBSERVATIONS	CONCLUSIONS
NICKEL		

C HEATING SULFUR

CON-DITIONS	OBSERVATIONS	CONCLUSIONS
IN A LONG TEST TUBE		
IN A DEFLAGRATING SPOON		

D   HEATING LEAD(II) NITRATE

COMPOUND	OBSERVATIONS	CONCLUSIONS
LEAD(II) NITRATE		

GENERAL CONCLUSIONS

Using your own words, summarize the differences between physical and chemical change in the table below.

NUMBER	PHYSICAL CHANGES	CHEMICAL CHANGES
1		
2		
3		

PROBLEMS

Answer the following questions in your own time. Try to be brief and to the point, but remember that some explanation is always necessary. You will not be required to hand these questions in for marking. You should discuss your answers with your lecturer. You will be given a test on the practical and these questions will form part of it. The mark will be your assessment for this practical.

1) a) Why does sulfur behave differently when it is heated in a long test tube as compared with heating it in the open air?

   b) After heating the sulfur in the long test tube you probably noticed some yellow powder forming towards the top of the tube. What was it? How did it get there?

2) When magnesium is burned it turns into a white ash.

   a) What happened to the original magnesium? What does the ash consist of? Do you think that the ash is lighter or heavier than the original magnesium?

   b) You probably noticed a white smoke. What did this consist of?

   c) When the ribbon stopped burning there was probably some unburnt magnesium left between the jaws of the tongs. Why did this not burn?

3) Name three physical properties which distinguish lead and nickel.

4) Name *three* new substances formed when lead(II) nitrate is heated.

5) Classify each of the following as a *physical* or *chemical* property.

   a) Copper combines with oxygen slowly when heated to 200EC to form a black powder.

   b) Gold is _

   c) Pure iron is bluish-white.

   d) Hydrogen reacts violently with chlorine if the two are mixed.

   e) Carbon monoxide is poisonous.

   f) Carbon reacts with nitric acid at high temperatures to form a gas.

   g) The melting point of a certain substance is 303.5°C.

   h) Sodium is easily cut with a knife.

6) Distinguish clearly between a physical *property* and a physical *change* in not more than 50 words.

7) Salt is found underground in large deposits. The salt may be extracted by pumping water into one hole and pumping salt water out of another. Upon what property of the salt does this depend? Is it physical or chemical?

8) Salt is produced in Inagua. Briefly describe the process used.

9) In what ways does pure solid salt differ from salt dissolved in water? Is dissolution a physical or a chemical change? Can you identify different types of dissolution?

10) Describe three examples of *chemical changes* which have not been encountered in this practical. In each case describe the starting material(s) (the *reactant(s)*) and the material(s) formed (the *products*). Give the names of these materials.

11) Describe one physical and one chemical change which occur in the human body.

PRACTICAL CLASS NO. 2
TECHNIQUES FOR THE
SEPARATION OF MIXTURES

INTRODUCTION

Mixtures are relatively easy to separate. Simple *physical methods* are used. The separation of compounds into their constituents may be achieved, but much more vigorous *chemical methods* must be used. Physical methods of separation depend on using a difference in the *physical properties* of the different substances making up a mixture. The difference could, for example, be a difference in melting point, a difference in boiling point, or perhaps a difference in solubility in water. Each of the following operations uses a different physical property to allow the separation of a mixture into its components.

PART A: The Separation of a Mixture of Sodium Chloride and Silica
INTRODUCTION

Sodium chloride is a compound of sodium (a reactive metal) and chlorine (a greenish-yellow poisonous gas), commonly encountered as "salt". Note how different this compound is from its constituent elements. Silica is a compound of silicon (an element like carbon) and oxygen, and is the main component of sand (though Bahamian beach sand actually contains very little silica, being mainly calcium carbonate). You are provided with a mixture of these two substances, and will separate them using a method known as *filtration*. Since this is a physical method it can separate the silica from the sodium chloride but cannot separate either of these two compounds into their constituent elements.

METHOD

Fill the end of a spatula with a little of the mixture (about 10g), put it into a 50cm^3 beaker and add distilled water until the beaker is about 1/3 full. Place the beaker on a gauze over a Bunsen and heat it until the water just boils. Place a filter funnel in the top of a conical flask. Fold a filter paper and put it in the funnel. Damp it with a little distilled water. Whilst the mixture in the beaker is still hot, pour all the liquid and solid into the filter paper. Guide the liquid with a glass rod and then use the same glass rod to scrape the solid material into the paper. Use a wash bottle to rinse the last grains of silica into the filter paper.

Wait until all the liquid has passed through the paper into the conical flask. This *filtrate* is a solution of sodium chloride. Note whether it is *clear* and whether it is *colourless*. The silica in the filter paper should be washed three times with a little water from the wash bottle and then put aside to dry. The liquid in the conical flask should be transferred to an evaporating dish. Place the evaporating dish on a gauze over a Bunsen and boil away most of the water, but *do not allow the dish to dry out completely*. Use the glass rod to keep the salt in the water in the middle of the evaporating dish.

When the salt is nearly dry, put a little water in the bottom of a larger beaker to make a steam bath. Heat this beaker of water and place the evaporating dish on top of it. Continue heating the evaporating dish indirectly in this way until the salt is dry. Keep stirring the drying salt to hasten evaporation.

When you have finished this separation, the sodium chloride and silica should be re-mixed and put into the bottle provided for the purpose.

PART B: The Separation of a Mixture of Iodine and Sodium chloride

INTRODUCTION

Most solids become liquid when they are heated, that is they melt. Not all do this however. A few, such as naphthalene (moth balls) and aluminium chloride, change directly into a gas (otherwise known as a vapour). This process is known as sublimation. Iodine is a borderline case: you may see a little liquid iodine existing for a moment if you heat it in a test tube. In an open container, however, no liquid is observed. When the vapour cools again it condenses directly to a solid. You will use this property of iodine to separate it from sodium chloride.

METHOD

Fill the end of a spatula with a little iodine and sodium chloride mixture and place it in a test tube. The use of too much of the mixture will lead to a poor separation. Hold the test tube in your hand at the top and heat only the very bottom of the test tube little by little in a Bunsen flame. Hold the test tube at an angle so that the top of the tube is not heated. If you burn your fingers you are doing it wrong! Control the heating by moving the tube in and out of the flame. By heating a little higher up the tube you can improve the separation until it is almost perfect. The iodine should finish up near the top of the tube and the sodium chloride should remain near the bottom.

PROBLEMS

Answer the following problems by referring to your own results. Give your answers in full but do not submit them for marking. Instead you will be given a short test on the material during class time.

1) What difference in physical properties was used to achieve the separation of sodium chloride and silica.?

2) Find another pair of substances that could be separated from their mixture using the same technique as sodium chloride and silica – i.e. shaking with water followed by filtration.

3) Explain how the filter paper allows the passage of a liquid through it but prevents the passage of the silica.

4) Give two reasons why the solution is heated before being poured into the filter paper.

5) Could sodium chloride and sugar be separated by the same technique that you used for sodium chloride and silica (i.e. using water and filtration)? Explain.

6) How does stirring the drying salt in the evaporating dish allow the salt to dry more quickly?

7) Do you think that the sodium chloride produced by the separation of sodium chloride and iodine was pure? How could you tell?

8) Iodine dissolves easily in alcohol. This solution is known as ***tincture of iodine***. What is tincture of iodine used for?

9) When chemically combined with other substances ***iodine*** is more correctly referred to as ***iodide***, as in the substances potassium iodide, or sodium iodide, or hydrogen iodide. What household substance often contains iodide (as opposed to iodine)? What is the purpose of the iodide in this household substance? The iodide is present as *potassium iodide*. Mention one difference in physical properties between *iodine* and *potassium iodide* which is apparent from an examination of the household substance.

10) Could the same separation be achieved with sodium chloride and silica if gasoline were substituted for water? Explain.

11) Why was the silica in the filter paper washed three times with distilled water?

12) Why is distilled water, rather than tap water, used in this and other experiments in chemistry?

13) Is "table salt" a *pure* substance? Does it contain anything apart from sodium chloride? If so what else does it contain?

14) Mention two pure (or nearly pure) substances that you often encounter. Why do you think that they are pure?

PRACTICAL CLASS NO. 3
FURTHER TECHNIQUES FOR THE
# SEPARATION OF MIXTURES

PART A: THE SEPARATION OF TWO LIQUIDS BY DISTILLATION
INTRODUCTION

All liquids boil when heated. They turn into gases. These are often referred to as *vapours*. Each liquid boils at a characteristic temperature known as its *boiling point*. The boiling point of water is 100°C. Other liquids boil at different temperatures. Gasoline boils at a lower temperature than water. Even at room temperature (around 20°C to 30°C) it evaporates very rapidly. For this reason it is known as a *volatile* liquid.

Methanol is another volatile liquid. It is very like the *ethanol* found in alcoholic drinks, but it is poisonous and does not smell so pleasant. Methanol boils at 65°C. When a mixture of methanol and water is heated gradually the methanol tends to boil off first. However the boiling point of such a mixture is always higher than that of pure methanol. The more water such a mixture contains, the higher will be the boiling point. The vapour, too, is a mixture, but it always contains more methanol than the liquid it springs from. If this vapour is condensed back to a liquid it can be collected. This new liquid, called the *distillate*, may be almost pure methanol if the procedure has been carried out carefully. Methanol is *flammable*. That is: it will burn. A simple check to see if the distillate burns when lit will tell us if it is mostly methanol or mostly water.

METHOD
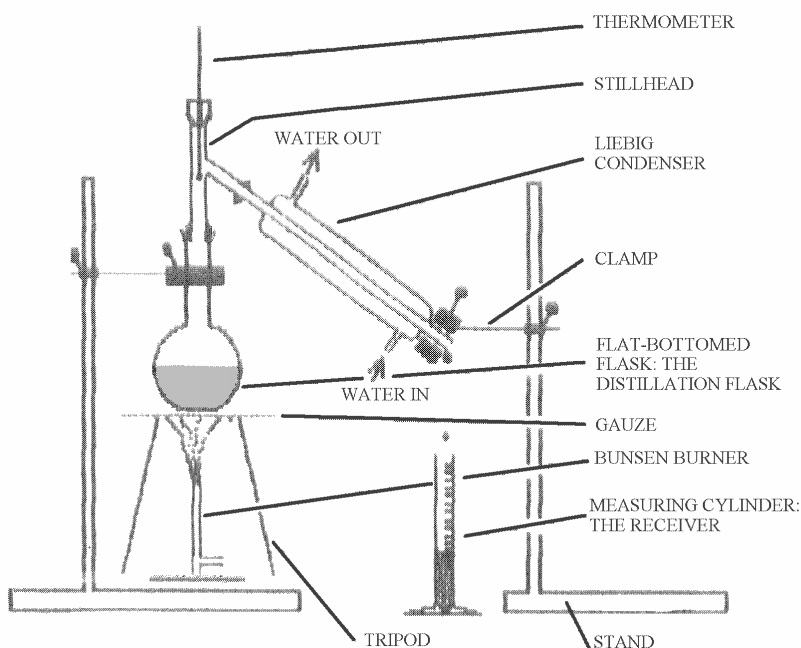
Probably this experiment will be given to you as a demonstration. If not, proceed as follows. Assemble your apparatus as shown in the diagram. Put ground-glass joints together gently. When you tighten clamps make sure that bare metal does not come into contact with glass. Make sure that two or more clamps are not all pulling in different directions: glass does not bend!

Measure $50cm^3$ of the methanol/water mixture provided into the distillation flask and heat the flask carefully until boiling starts. Collect the liquid that condenses (the *distillate*) in a measuring cylinder. Note the temperature on the thermometer as distillation starts (that is: when the first drop of distillate is collected). Record the temperature again as $2cm^3$, $4cm^3$, $6cm^3$, $8cm^3$ etc. of distillate are collected. Continue in this fashion until the total volume of distillate is about $40cm^3$.

DO NOT ALLOW THE DISTILLATION FLASK TO BOIL DRY. During distillation the heat should be controlled to maintain a steady drip into the measuring cylinder. From time to time withdraw a drop of the distillate with a teat-pipette and put it on a watch glass. Try to light it and make a note of the results, including the length of time it burns for. Also make a note of the appearance and smell of the distillate at regular intervals as the distillation proceeds. Record your observations in the table overleaf.

RESULTS

Volume of distillate / cm^3	Temperature / °C	Other observations	Volume of distillate / cm^3	Temperature / °C	Other observations
0				22	
2				24	
4				26	
6				28	
8				30	
10				32	
12				34	
14				36	
16				38	
18				40	
20					

Plot a graph of temperature (vertical axis) against volume (horizontal axis) for your results. Indicate on your graph (i) the region which corresponds to a high proportion of methanol, and (ii) the region which corresponds to a high proportion of water in the distillate. A good graph incorporates a title, fully labelled axes (including units of quantities plotted) and consistent and even scales. All experimental points should be plotted, regardless of whether they are "right" or not.

PART B   THE SEPARATION OF A MIXTURE OF SALTS BY CHROMATOGRAPHY

INTRODUCTION

Chromatography is one of the most versatile of modern methods of separating chemical compounds from one another. It can be used to separate mixtures of several compounds in solution though it is not limited to this. It is particularly useful where only very small amounts of material are available. The separations achieved by chromatography are based on two main principles: *adsorption* and *partition*.

Adsorption is a process by which molecules stick to the surface of solid particles, or on to the inside surfaces of pores in the solid. (It is very different from the familiar process of *absorption* by which a sponge takes up water.)

Partition is a process by which a substance splits itself between two different environments. Sometimes it can depend on a difference in solubility between two different solvents. For example, iodine, the purple substance that you met in a previous experiment is soluble in both water and oil. It is only slightly soluble in water but it is very soluble in oil. If iodine is shaken with water and oil together it partitions itself between the oil and the water. Most goes into the oil, since that is where it is most soluble, but some goes into the water. As a result, the water is only slightly coloured, but the oil is highly coloured.

There are many types of chromatography. These differ in the materials which are used for the stationary and the mobile phases.

In paper chromatography, which we are studying today, the stationary phase (not stationery phase) is the paper, because it doesn't move. The mobile phase is a solvent (sometimes water) which soaks up through the paper. The mobile phase moves through the stationary phase.

The substances to be separated are carried along by the mobile phase, but each different substance moves at a different rate, and so they are separated. This happens because each substance is partitioned between the stationary phase (on which it is adsorbed) and the mobile phase (in which it is soluble). Some substances are adsorbed weakly by the stationary phase and so their molecules spend most of their time in the mobile phase. As a result they are carried along at almost the same rate as the mobile phase is moving. On the other hand, some substances are adsorbed strongly by the stationary phase and their molecules spend very little of their time in the mobile phase. As a result they move much more slowly than the mobile phase. It is important to realise that molecules of the substances are continually moving between the mobile and the stationary phases, but spend more time in the one they have the strongest attraction for.

In today's practical you will try to separate a mixture of salts (as chemists call most compounds of metals). These will probably be iron(III) chloride ($FeCl_3$, nickel(II) chloride ($NiCl_2$), and copper(II) chloride ($CuCl_2$). They will probably be presented as a ready made-up solution but, if not, a mixture may be made up by dissolving 0.1g of each salt in 5cm^3 of water. Likewise the solvent which forms the mobile phase will probably be provided ready-mixed, but if not it can be made by mixing 90% propanone, 5% water and 5% dilute hydrochloric acid by volume. You will make these more visible on your chromatogram (the piece of paper after the chromatographic separation has been carried out on it) by spraying with a solution of dimethylglyoxime in dilute ammonia. Alternatively the paper may be dipped into this solution.

METHOD

1) Pour the mobile phase into a 100 cm^3 beaker to a depth of about 0.5 cm (about 5 to 10 cm^3). Cover the beaker immediately with aluminium foil to impede evaporation of the mobile phase. A rubber band may be helpful to secure the foil. Do not move the beaker after this.

2) Draw a pencil line across a strip of chromatography paper about two centimetres from one end.

3) If necessary clean a glass capillary tube by filling it with distilled water and then allowing the water to drain out completely on to a paper tissue. The tube must be emptied completely. Dip it into the *mixture of salts* (see introduction) so that the liquid passes up the tube by capillary action.

4) Touch the end of the tube to the left of the pencil line near the edge of the paper and let the liquid drain out on to the paper to form a *small* spot on the line. Dry the spot using a hair-dryer or similar apparatus and then repeat the procedure several times, spotting in exactly the same place with the same mixture each time. Keep the spot as small and concentrated as possible. Make sure that the spot is thoroughly dry before proceeding.

5) Make a second spot of one of the solutions of the pure salts (copper(II) chloride, iron(II) chloride or nickel(II) chloride) to the right of the centre of the line in the same way as for the mixture.

6) Repeat the procedure with a second piece of chromatography paper and the remaining two solutions of metal salts.

7) Using a razor blade, or a very sharp knife, cut two very narrow slits, just long enough to admit the strip of paper, in the top surface of the foil covering the beaker. The slits must support the strips of paper in the middle of the beaker in such a way that they do not touch each other nor the sides of the beaker.

8) Push the paper into the beaker with the pencil mark downwards. The paper should dip into the liquid

in the bottom, *but the spot should be completely clear of the liquid*.

9) Let the liquid soak up through the pieces of chromatography paper until it reaches the aluminium foil. Withdraw the paper from the beaker and dry it with the dryer.

10) Circle all spots which are visible with pencil.

11) Dip the pieces of paper in the dimethylglyoxime/sodium hydroxide solution to render the spots clearly visible. (If a spray is available, spray the pieces of paper with the solution in a fume cupboard). The different solutions will show different colours. Circle with pencil any spots which were not circled before.

12) Examine the developed *chromatograms* carefully and note how the various substances have been separated. See if you can find the pure substances you spotted amongst the substances in the mixture.

13) Attach the chromatogram to your booklet and keep it for future reference. You may be asked to submit it for marking either immediately after the practical or the following week.

PROBLEMS

Answer the following questions in the spaces provided below. Always remember that you must refer to your own results wherever possible.

1) Do you think that your separation was complete in the distillation experiment? Explain your opinion.

2) Distillation can be used to separate dissolved solids from liquids. Distilled water is made by distilling tap water. Give the names of two dissolved solids which can be removed from Bahamian tap water by distillation.

3) Distillation is used extensively in several industries based in the Bahamas. Briefly describe how distillation is used in one of them.

4) Based on your results you should have identified (at least) one of the metal ions in the mixture. How did you do this? Try to write your answer as clearly and concisely as possible.

5) Why was the solution of metal ions spotted several times in one place rather than making a single spot, or spotting in different places?

6) Why do you think it is important to cover the beaker whilst the liquid is soaking up through the chromatography paper?

7) Which of the metal ions that were present in the mixture binds most strongly to the stationary phase? Give reasons.

8) Why is it important to prevent the original spot on the chromatography paper from dipping into the solvent in the beaker?

9) Write a description of your chromatogram as if for someone who is not able to see it.

10) Bring your chromatograms and graphs to the next practical. You may be given a mark for them.

PRACTICAL CLASS NO. 4
ATOMS AND MOLECULES

INTRODUCTION

Matter may be defined as anything which has weight and takes up space. Unfortunately this definition gives little insight into its fundamental nature. Any theory which is to account for the observed properties of matter must say a lot more than this. Logically, there are two possible points of view: matter is either *continuous* or *particulate* in nature. If matter is continuous it can be divided into smaller and smaller pieces without limit - in the imagination, at least. If, on the other hand it is particulate, then sooner or later, as we continue to divide any piece of matter into smaller pieces, we shall arrive at the smallest possible piece which is still the same substance that we started with. (It might be that further division is still possible, but that the result is something entirely different from what we started with.) For example, you might visualise a piece of iron on the sub-microscopic level as being either like a lump of jelly or a handful of pebbles.

The ancient Greeks, who thought deeply about such things over two thousand years ago, coined the word *atom*. In Greek this means *uncut*. These days we also refer to *molecules*. Molecules are (usually) small clusters of atoms joined together. Today the existence of atoms and molecules is widely accepted because they make sense of such a wide variety of experimental observations.

It is most important for the student to understand clearly the distinction between an atom and a molecule. Let's try to make this distinction clear. Pure substances are either *elements* or *compounds*. A compound is made up of two or more elements chemically combined together. An atom is defined as the smallest particle of an element which can no longer be divided without losing its identity. A molecule, on the other hand, is the smallest particle of a substance *which can exist on its own*. This usually means a group of atoms strongly bonded together though we must also include the case of an atom on its own, since some types of atom are able to exist alone. Generally, however, single atoms very quickly join up with other atoms in some way. In other words they are *not* able to exist on their own.

According to these definitions both compounds and elements are made up of molecules. If we divide up a molecule of a compound then the result is something entirely different from the original compound. For example a molecule of the compound carbon monoxide (a poisonous gas) is made up of a carbon atom and an oxygen atom. Splitting this up can only give either an oxygen atom or a carbon atom, which are each entirely different from the original carbon monoxide. With an element, since the molecules consist of only one type of atom, the result is still that same element. As an example, take the element sulfur. The smallest particle of any sample of sulfur is one atom of sulfur. However, one atom of sulfur is not capable of separate existence. Sulfur atoms gather together in groups of eight. This group of eight sulfur atoms is called a molecule of sulfur.

HOW DO WE KNOW ATOMS AND MOLECULES EXIST?

These days we can actually see atoms and molecules using a very special kind of microscope known as a *scanning-tunnelling microscope*. Long before such microscopes were available, a great deal of indirect evidence made it very difficult *not* to believe in them. This indirect evidence is essentially that they make so many things easier to understand. This is one of the most important principals in science: when theories work, we believe in them. The principal is illustrated in the following experiments. They are simple experiments, but unless you use the ideas of atoms and molecules, the results are very difficult to understand.

PART A: The Diffusion of a Crystal of Potassium Permanganate
METHOD

Half-fill a beaker with tap water. Allow all movement of the water to cease. Take a single crystal of potassium permanganate on the end of a spatula and drop it into the beaker. Observe the beaker carefully for a few minutes, paying particular attention to the way the colour spreads away from the crystal.

PART B: An Investigation to Determine the Approximate Size of Atoms and Molecules
INTRODUCTION

When a drop of oil is placed on water it spreads out. As it spreads it gets thinner. You have probably seen the colours (described as *iridescent*) produced when oil is allowed to spread on the surface of a body of water. At first, the layer of oil is many molecules thick but as it spreads out it becomes thinner and thinner.

When it becomes only one molecule thick it cannot spread any more, since the molecules of oil stick together, and so it stops spreading[2]. The situation can be pictured as shown below:

Figure 4.1 How certain oils spread on the surface of water. Although the molecules are shown as round, they are really elongated, with one end that is attracted to the water surface, and another end that is repelled by it.

Since molecules are so small, any appreciable quantity of oil will cover a very large area indeed. In order that the layer of oil can be of a reasonable size for this experiment the amount of oil must be minute. This can be accomplished by using only a tiny amount of a very dilute solution of the oil in a liquid which evaporates very easily. *Petroleum ether* is such a liquid. When a single drop of a solution of the oil in petroleum ether is placed on water the solvent evaporates leaving only the oil on the surface of the water. The oil spreads with sufficient force to push back a fine powder such as *talc*[3]. The area of the clear patch so formed can then be estimated. If the volume of the oil itself is known, the thickness can be found. The thickness of the oil layer is the thickness (or diameter) of a single molecule of oil. Since a molecule of oil contains many atoms, each atom must be quite a lot smaller than this.

Note that this experiment is rather tricky. You will probably not be able to do it properly the first time. You should be ready to have several tries at it before being satisfied with the results.

METHOD

1) Fill a Petri dish with tap water. Put a little talcum powder (baby powder) on the back of your hand and blow it gently onto the surface of the water. A very thin, even layer of powder is required. Their

---

[2] This is not true of all oils: some are made of molecules that do not attract each another very strongly and so the molecules do not stop spreading when the layer is one molecule thick. Instead the molecules part company so that the layer of oil goes on spreading for ever.

[3] Talc is a naturally-occurring mineral which is very soft. In its powdered form it is used as "baby powder".

must be no large lumps. If the layer is too thick you will not be able to obtain any results. The layer should be barely visible.

2) Take up some of the oil solution provided in a *teat pipette*. Draw the solution in and out several times until the contents no longer tend to run out when the pipette is removed from the solution. Poise the pipette over the middle of the Petri dish, close to the water surface, (a steady hand is required for this) and allow *a single drop* to fall onto the surface of the water.

3) If the coating of powder on the surface is not too thick, a circular patch will be cleared as the oil pushes back the powder. Measure the diameter of this clears patch and hence its *radius*. Calculate its area from πr^2, where r is the *radius* of the circle.   Record the area in the results table with your working.

4) Determine the volume of a single drop of the *oil solution* as follows. Count how many drops of the solution are required to fill a measuring cylinder to the 1 cm^3 mark. Record this number. Divide 1cm^3 by this number to determine the volume of a single drop. For example, if 98 drops of the solution were required, then the volume of one drop would be given by:

$$Volume\ of\ 1\ drop\ =\ \frac{1\ cm^3}{98} = 0.0102\ cm^3$$

5) Find the volume of the oil itself in one drop of the solution. Remember that the solution is mainly the solvent, petroleum ether. This can be accomplished using two conversion factors. The solution contains 0.1cm^3 of oil in 1dm^3 of solution and this can be expressed as a conversion factor:

$$\frac{0.1\,cm^3 \text{of oil}}{1\,dm^3 \text{of solution}}$$

There are 1000 cm^3 in 1dm^3, and this can also be expressed as a conversion factor:

$$\frac{1\,dm^3}{1000\,cm^3}$$

For example, using the figure from the above calculation (0.0102 cm^3) and the second conversion factor, the *cm^3* of oil is changed to *dm^3*:

$$Volume\ of\ sol^n = 0.0102\,\cancel{cm^3} \times \frac{1\,dm^3}{1000\,\cancel{cm^3}} = 0.0000102\ dm^3$$

The next step is to change from dm^3 of solution to cm^3 of oil using the first conversion factor:

$$Volume\ of\ actual\ oil = 0.0000102\ \cancel{dm^3\ \text{of solution}} \times \frac{0.1\,cm^3 \text{ of oil}}{\cancel{1\,dm^3\ \text{of solution}}} = 0.00000102\ cm^3 \text{of oil}$$

You should show your own working in the space provided below in the results section.

6) From the volume of the oil, and its area, find the thickness of the layer and therefore the size of a molecule of oil, given that:

$$Volume = area \times thickness \text{ or } V = A \times T$$

$$\therefore \text{Thickness, } T = \frac{V}{A}$$

This is the diameter, or size, of an oil molecule.

RESULTS

REMEMBER TO INCLUDE THE UNITS OF ANY MEASUREMENTS.

Diameter of the clear patch =   _____

∴ Radius of the clear patch =   _____

∴ Area of the clear patch (A) =   _____

Number of drops of the oil solution in 1 cm^3 of it =   _____

∴ Volume of one drop of the oil solution =   _____
∴ Volume of the oil itself (V) in one drop of solution is given by:

Hence the thickness (or size) of an oil molecule (T) =

PROBLEMS

1) What process did you observe when the crystal of potassium permanganate was dropped into the beaker of water? Describe and explain this process with reference to small particles and their motion.

2) If you dropped a crystal of sugar into a beaker of water would you *see* the same process as with potassium permanganate? Would it actually occur? Explain.

3) The oil used for PART B is known as **oleic acid**. Go to the library and find the chemical formula from a dictionary or handbook of chemistry such as *Lange's Handbook of Chemistry*. Look in a section devoted to "organic" compounds. Write down the formula and state how many atoms there are in one molecule.

4) Why was the surface of the water coated with the fine powder before the oil was dropped onto it?

5) Explain why an oil slick does not continue spreading forever when a drop of oil is placed on a water surface.

6) Could the thickness of a molecule of sugar be estimated in the same way as a molecule of oil i.e. by dropping a dilute solution of sugar onto a water surface? Explain your answer.

7) Mention three different physical properties of petroleum ether that make it particularly suitable as a solvent for the oil in PART B.

8) The process of diffusion and the spreading of an oil layer lend support to the atomic theory. Briefly describe three other pieces of evidence for the atomic theory.

9) Using your value for the size of an oil molecule, calculate how many molecules of oil are necessary to reach across your fingernail when placed end to end. (You will need to measure the width of your fingernail.)

PRACTICAL CLASS NO. 5
PREPARATION OF VARIOUS
SAMPLES OF COPPER(II) OXIDE

INTRODUCTION

In this experiment you are asked to prepare copper(II) oxide (CuO) in two different ways. The samples are to be analysed in practical class no. 6 in order to show that each contains the same proportions of copper and oxygen. It is therefore essential that you store the samples you make in this class in the *desiccators*[4] provided so that you can recover them easily for the next one.

METHOD OF PREPARATION
SAMPLE A: STARTING WITH COPPER

Place about 1g of clean pure copper foil in a large crucible in a ***fume-cupboard***. Carefully measure about 5 cm^3 of concentrated nitric acid into a measuring cylinder. (If it's a little over it doesn't matter). Pour the contents of the measuring cylinder on to the copper. The mixture will froth up as the copper and the nitric acid ***react*** together. This is a typical example of a vigorous chemical reaction.
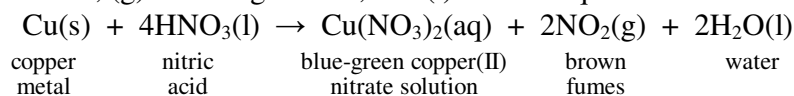
Brown fumes of nitrogen dioxide are ***given off***. Nitrogen dioxide is poisonous so you should avoid breathing it in. Test the ***fumes*** with blue litmus paper. This will show whether the fumes are acidic or not. A blue-green copper(II) nitrate solution is formed. This will also contain some unreacted nitric acid since this was added ***in excess*** to ensure that all the copper would dissolve. If some copper remains unreacted you should consult the lecturer before proceeding.

Place the crucible containing the solution in a pipe-clay triangle on top of a tripod. Cover the crucible with a lid. Heat carefully but directly (no gauze) with a Bunsen so that the contents boil gently without spilling. Eye protection should be worn during this operation. Watch the crucible constantly and control the amount of heat. ***DO NOT LEAVE YOUR CRUCIBLE UNATTENDED WHILST HEATING***.
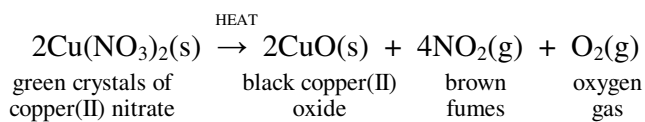
Continue heating carefully until the contents have evaporated to dryness. Further heat the green or blue solid (copper(II) nitrate) left in the crucible until it turns completely black. Remember to heat the material adhering to the sides of the crucible as well. This is best done by directing the Bunsen flame at the side of the crucible. You will note a green coloration of the flame. This is characteristic of any material containing copper. Eventually not a speck of green should remain.

The black solid produced is the first sample of copper(II) oxide. Allow the crucible to cool and then scrape the black powder onto a piece of aluminium foil, label it carefully with the names of your group members and its origin ("A" here), and store it in a desiccator ready to be used in experiment 6.

The chemical equations below show the chemistry you have just done. Note that: (s) means "solid", (aq) means "dissolved in water", (g) means "gaseous", and (l) means "liquid".

$$Cu(s) \ + \ 4HNO_3(l) \ \rightarrow \ Cu(NO_3)_2(aq) \ + \ 2NO_2(g) \ + \ 2H_2O(l)$$

copper	nitric	blue-green copper(II)	brown	water
metal	acid	nitrate solution	fumes	

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[4] A desiccator is a sealed piece of apparatus which maintains a dry atmosphere for storage. The desiccator normally includes a substance which absorbs water (a *desiccant*) to keep this atmosphere dry.

$$2Cu(NO_3)_2(s) \xrightarrow{\text{HEAT}} 2CuO(s) + 4NO_2(g) + O_2(g)$$

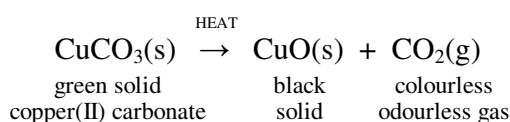| green crystals of copper(II) nitrate | black copper(II) oxide | brown fumes | oxygen gas |

## SAMPLE B: STARTING FROM COPPER(II) CARBONATE

Place about 4g copper(II) carbonate in a dry crucible and heat it directly with a Bunsen. Carbon dioxide is given off. You should test for it as soon as you start heating. You can do this by holding a glass rod with a drop of lime water on the end *near* to the contents of the crucible for a few moments. A milky appearance developing in the drop shows the presence of carbon dioxide. You might try this test again after it appears that your sample is completely black throughout.

The copper(II) carbonate *decomposes*, turning from green to black, whilst carbon dioxide is given off. After cooling and labelling, store the black material in a desiccator.

The chemistry is as follows:

$$CuCO_3(s) \xrightarrow{\text{HEAT}} CuO(s) + CO_2(g)$$

| green solid copper(II) carbonate | black solid | colourless odourless gas |

Limewater is a solution of calcium hydroxide dissolved in water which reacts with carbon dioxide as follows.

$$Ca(OH)_2(aq) + CO_2(g) \rightarrow CaCO_3(s) + H_2O(l)$$

| calcium hydroxide solution | white precipitate of chalk |
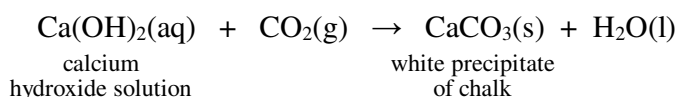
PROBLEMS

4) When evaporating the copper(II) nitrate solution copious *white* fumes are usually liberated before the brown nitrogen dioxide is seen. What do you think might be responsible for these fumes?

5) When preparing sample A, why is it important to remove all traces of green or blue colour?

6) The desiccator is intended to keep your samples dry. How does it do this?

7) When a substance dries it gets lighter. Suggest a method, involving weighing, by which you could make sure that no more water could be removed from sample A by further heating.

8) Would you expect sample B to continue to evolve carbon dioxide whilst being heated *even after it had turned completely black*? Explain.

9) Why does limewater turn milky when carbon dioxide is passed through it? What happens if one continues to pass carbon dioxide through limewater after it has turned milky?

10) When your samples were heated strongly you may have noted a green coloration of the Bunsen flame. What element caused this? The coloration of a flame can be used to identify several elements in their compounds. Such tests are known as *flame tests*. For example sodium colours flames an intense yellow. See if you can find which elements colour a flame red, white, and lilac.
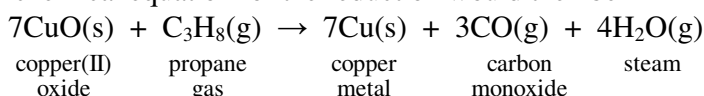
PRACTICAL CLASS NO. 6
ANALYSIS OF SAMPLES OF
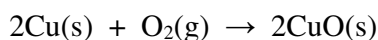## COPPER(II) OXIDE

INTRODUCTION

This is an experiment to demonstrate the ***Law of Definite Proportions***. You will attempt to show that three samples of copper(II) oxide contain the same percentages of copper and oxygen even though the samples were prepared in different ways. In this way you will demonstrate the truth of the law.

It will be necessary to remove the oxygen from the copper(II) oxide leaving only the copper. This process is known as ***reduction***. It is quite easy to reduce copper(II) oxide by heating it in a stream of hydrogen gas or "lab" gas as used for fuelling the Bunsen burners. For simplicity we assume that the lab gas is pure propane, C_3H_8. A chemical equation for the reduction would then be

$$7CuO(s) \; + \; C_3H_8(g) \; \rightarrow \; 7Cu(s) \; + \; 3CO(g) \; + \; 4H_2O(g)$$

copper(II)	propane	copper	carbon	steam
oxide	gas	metal	monoxide	

The reaction is accompanied by a visible colour change since the original copper(II) oxide is black whilst the copper metal produced is a pink colour. The water produced in the reaction is in the form of steam since the reaction occurs at a temperature of 300°C to 400°C. Precautions must be taken to ensure that if the steam condenses in the cooler parts of the apparatus it does not run back into a hot part and crack the glass.

The opposite process to reduction is ***oxidation***. Copper can be oxidised simply by heating it in a stream of oxygen or air.

$$2Cu(s) \; + \; O_2(g) \; \rightarrow \; 2CuO(s)$$

METHOD

1) Weigh a clean dry porcelain (or metal) ***boat*** and record its mass in the results table.

2) Fill the boat to the brim with copper(II) oxide. Each group of students will use one of the samples of copper(II) oxide prepared in the previous practical class. Your lecturer will instruct you which one to use. Your experiment will be more accurate the more copper(II) oxide you use - provided you don't spill any after the second weighing. When you have filled the boat remove any excess so that it will not spill easily. Note the origin of the sample in the results table i.e. A or B.

3) Weigh your boat, now full of copper(II) oxide, and record the mass in the results table.

4) If you use a combustion tube open at both ends, mount it between two clamps. The clamps should be positioned at each end of the tube and the tube should slope down towards the outlet. If you use a large test tube with a small hole in the bottom, mount it in one clamp so that it is more or less level, and so that the clamp is as close to the stoppered end of the test tube as possible. You are going to heat part of your tube strongly and since the clamps contain plastic parts, heat will damage them. For this reason:

    IT IS ESSENTIAL THAT ALL CLAMPS ARE WELL AWAY FROM THE HEAT.

5) Once the tube is firmly clamped, push the boat inside gently until it is well away from the clamp(s). Stopper the tube with a stopper (or stoppers) fitted with glass and plastic tubes so that it can be connected to the gas supply. If you spill any of the contents of your boat at this stage you will have to go back to step (3).

6) Make sure that your apparatus will not leak and then connect it to the laboratory gas supply. Turn on

the gas tap *CAREFULLY* to establish a gentle flow of gas. One way to test this is by wetting the end of the outlet hole so that it blows bubbles. Then, when you are sure that all the air has been driven from the apparatus, light the gas coming from the outlet. Adjust the gas to produce a small steady flame.

7) Heat the tube, directly under the boat, with a Bunsen burner. Heat gently at first and then more strongly. If the heat is too strong to begin with the tube may crack. Continue heating until the black copper(II) oxide is completely converted to pink copper metal. Note that sometimes a dark coloured film develops on the surface of the copper when propane is used for the reduction. If this happens do not be concerned. It will probably be necessary to move the Bunsen from time to time to ensure complete conversion. You may see the water produced in the chemical reaction condensing in the cooler parts of the tube. Do not allow it to run back onto a hot part of the tube.

8) Allow the boat to cool in the stream of gas. When it is cool and can be touched comfortably the gas should be turned off and the boat removed.

9) Weigh the boat and its contents and record the mass in the results table. Note: if the boat is still hot it will not be possible to weigh it accurately.

10) Calculate the percentages of copper and oxygen in the copper(II) oxide as:

$$\frac{\text{mass of copper}}{\text{mass of copper(II)oxide}} \times 100\% \text{ and,}$$

$$\frac{\text{mass of oxygen}}{\text{mass of copper(II) oxide}} \times 100\%$$

RESULTS

Sample used (A or B) _____

Mass of the empty boat _____ g

Mass of the boat full of <u>copper</u>(II) oxide _____ g

∴ Mass of the copper(II) <u>oxide</u> _____ g

Mass of the boat + copper <u>metal</u> _____ g

∴ Mass of the copper metal produced _____ g

And mass of oxygen removed _____ g

∴ Percentage of copper metal in the
copper(II) oxide _____ %

And percentage of oxygen in the copper(II) oxide _____ %

Total of the two percentages _____ %

If your percentages do not add up to 100%, go back and check your calculations.

Write the results for the other sample below. If, for example, you have analysed sample B, you should collect results for samples A from another group.

Sample origin (A or B)   _____

% copper   _____

% oxygen   _____

PROBLEMS

1) Do you think that your results are in support of the law of definite proportions? Explain your answer.

2) Where does the oxygen go when the copper(II) oxide is reduced?

3) Why must the porcelain boats be both clean and dry when they are weighed?

4) Why is it necessary to maintain the flow of gas over the freshly prepared copper metal whilst it cools?

5) Do you think that the presence of, say, copper(II) nitrate (the blue material) in one of your samples might affect your results? Explain.

6) Why is it important not to let condensation run back onto a hot part of the reduction tube? What can be done to prevent this from happening?

7) Why is it important to heat the tube only near the bottom end and to clamp the tube right at the top end?

8) Why is it important to make sure all the air has been flushed out of the tube before attempting to light the propane gas?

9) Why is it important to allow a boat to cool properly before it is weighed?

10) A porcelain boat was first weighed empty. Its mass was found to be 3.1246 g. It was then filled with a certain oxide of iron and re-weighed. Now the mass was 4.3245 g. The boat was placed in a combustion tube and heated in a stream of hydrogen. After cooling the mass of the boat and its contents was found to be 3.9929 g. Calculate the percentages of oxygen and of iron in the sample.

PRACTICAL CLASS NO. 7
DETERMINATION OF THE MELTING
POINT OF A SOLID

INTRODUCTION

Most solids have their particles (atoms, molecules or ions) packed in a highly ordered arrangement known as a ***crystal lattice***. The particles are closely packed together. They are only able to oscillate about their mean positions in the lattice, and cannot move past one another. When the solid is heated, enough energy may be supplied to overcome the forces of attraction between the particles and the lattice is destroyed. If this happens the particles become more mobile with the freedom to slide over one another. Thus the substance melts and turns into a liquid. The temperature at which this happens is called the melting point of the solid. The melting point may be considered as a measure of the strength of the forces of attraction between the particles. The stronger the forces of attraction, the higher the melting point. The melting point of a solid has a fixed value which changes only very slightly with pressure (unlike the boiling point). This means that the determination of the melting point is useful in helping to identify a substance. Mixtures generally melt at a lower temperature than the major component in the mixture. They also melt over a range of temperature rather than at a single temperature. For this reason the determination of the melting point can also be used to see whether a substance is pure or not.

In principle, a pure solid melts at an exact, single value of the temperature. That is, whilst the solid is actually melting, and both solid and liquid are present, the thermometer reading remains constant. In practice a thermometer shows a narrow ***range of temperature*** as the substance melts. For example, the ***melting point range*** of benzoic acid may be recorded as 122.4 to 123.0°C. The first figure, 122.4°C is the temperature at which liquid first appears as the solid is heated. This is the best estimate of the melting point of benzoic acid. The second figure, 123.0°C, is the temperature at which solid is no longer visible. If an impurity is added to benzoic acid, or occurs there as a result of poor purification, the melting point (the temperature at which melting commences) is lowered and the melting point range is broadened.

These effects of impurities on the melting point of a solid help in both identifying unknown solids as well as deciding on their state of purity. For example, if you suspect that a certain solid may not be the one you think it is, you could confirm your suspicions by adding some of the pure solid from a different source to your suspect sample. If the melting point drops and the melting range broadens, you have confirmed your suspicions.

In this experiment you will determine the melting point of a pure solid and then observe the effect of an added impurity on its melting behaviour.

METHOD

Your lecturer will provide you with a pure solid, X and an impure solid Y. Sample Y is a mixture of sample X and the substance thymol.

1) Close one end of a melting point tube in a Bunsen flame and pack the bottom (closed end) with solid X to a depth of approximately 5mm.

2) Attach the melting point tube with a small rubber band to a thermometer, making sure that the solid in the tube is directly opposite the mercury bulb of the thermometer.

3) Set up your melting point apparatus as demonstrated to you by your lecturer and heat gently with continual stirring. Make a first trial run to find the ***approximate*** melting point of X. This is the reading on the thermometer when the sample ***begins*** to melt.

4) Add some cold water to your beaker so as to reduce the temperature to about 5°C below the melting point of the sample. If the sample does not solidify remove it from the water for a few moments until it does. Try a second time to find the melting point. This time try to do your determination as accurately as possible. Increase the temperature very slowly until the sample melts. The temperature at which melting **BEGINS** is the melting point.

5) Repeat the procedure on sample Y, but this time try to measure the temperature both when melting begins and when it finishes. These two temperatures constitute the melting point range of Y.

6) Record your results in the results table below.

RESULTS

Melting point of solid X _____

Melting point range of solid Y _____ to _____

PROBLEMS

You may need to refer to reference texts such as *Lange's Handbook of Chemistry* in the reference section of the library for melting points and other information. The substances used in this practical are *organic compounds*.

1) The melting points of various compounds are given below. **From your results** deduce the identity of sample X.

acetamide	81.0°C	lauric acid	44.1°C
benzoic acid	122.4°C	palmitic acid	62.8°C
thymol	49.6°C	naphthalene	80.2°C
capric acid	31.4°C		

Probable identity of sample X _____

2) Why is water a suitable liquid to use for your heating bath?

3) Look up the boiling point of glycerol. Under what circumstances would it be useful to use glycerol instead of water in the determination of a melting point? Give the name of a compound whose melting point determination would have to be carried out with glycerol rather than water.

4) What two effects would the presence of an impurity have on the melting point of benzoic acid?

5) Why is it important to seal the lower end of the melting point tube thoroughly?

6) Three tubes (A, B and C) each contain a white crystalline solid. The solids all melt at 149°C. A 50/50 mixture of the solids from tubes A and B melts over the range 130 to 139°C, but a 50/50 mixture of the solids from A and C melts at 149°C. Now answer the following questions:

   a) What can you say about the identities of the solids in A, B and C? i.e. how does that from A compare with B and C, and how does B compare with C?

   b) In what range would a 50/50 mixture of the solids from B and C melt?

7) Explain the meaning of the term **melting point range**.

8) Explain carefully the meaning of the statement: "The melting point of a pure solid is the same as its

freezing point."

9) By referring to the kinetic theory of matter explain and describe the process of melting at the molecular level. What effect does the strength of the forces operating between the particles (whether atoms, ions or molecules) have on the melting point?

10) Look up the melting points of the following substances: hydrogen, iron and sodium chloride. You will find them under *inorganic compounds* in *Lange*, although the first two are elements. Explain, with reference to the nature of the particles of which they are composed (atoms, molecules, or ions), and the forces acting between them, why the melting points are high or low.
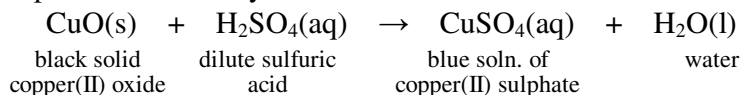
PRACTICAL CLASS NO. 8
THE PREPARATION OF HYDRATED COPPER(II) SULPHATE CRYSTALS FROM COPPER(II) OXIDE

INTRODUCTION

There are three main classes of simple chemical compound. These are (i) acids, (ii) bases and (iii) salts. Acids and bases are chemically opposite in nature. They are distinguished in many ways from each other. For example acids turn blue litmus paper "red" and bases turn red litmus paper blue. They react easily and quickly together and when they do so they produce salts, together with water. By and large the salts produced are much less reactive than the acids and bases they spring from. Acids and bases are generally *corrosive* and may cause burns when they come into contact with the skin. Generally salts are not nearly so corrosive, and do not burn the skin.

In this practical you will react the base copper(II) oxide (which is insoluble in water) with a dilute solution of sulfuric acid in water: "dilute sulfuric acid". Acids are usually used in a dilute form, since they are much safer to handle like this. Even when dilute, sulfuric acid will gradually make holes in your clothes if you spill it on them, and will sting painfully if you get it in a cut or graze, or in your eyes. Since copper(II) oxide is insoluble in water it is not at all dangerous, and may be handled quite safely.

The reaction may be expressed chemically as follows:

$$CuO(s) \quad + \quad H_2SO_4(aq) \quad \rightarrow \quad CuSO_4(aq) \quad + \quad H_2O(l)$$

black solid	dilute sulfuric	blue soln. of	water
copper(II) oxide	acid	copper(II) sulphate	

As the black copper(II) oxide and the colourless solution of sulfuric acid react together you will see the black solid gradually disappear and the solution turn blue due to the formation of copper(II) sulphate. It is only necessary to evaporate off the water to obtain crystals of this salt. If the evaporation is carried out carefully the hydrated salt: $CuSO_4.5H_2O$ (copper(II) sulphate 5-water) is produced[5]. Five water molecules are associated with every pair of copper and sulphate ions.

$$CuSO_4(aq) \quad + \quad 5H_2O(l) \quad \rightarrow \quad CuSO_4.5H_2O(s)$$

copper(II) sulphate	copper(II) sulphate
in solution	crystals
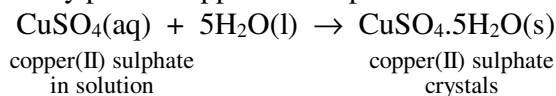
Note that the dot in the formula for copper(II) sulphate 5-water is not a decimal point: it simply separates the two parts of the formula.

METHOD

Using a measuring cylinder, place 25cm^3 of dilute sulfuric acid (2 mole dm^{-3}) in a 250cm^3 beaker and add 25cm^3 of distilled water. Place the beaker on a gauze over a Bunsen burner and warm it gently. Add a little copper(II) oxide to the hot acid. Stir with a glass rod until all the material dissolves. The solution should become a clear blue colour. Add further portions of copper(II) oxide until the copper(II) oxide no longer dissolves. At this stage the solution should be persistently dark and cloudy. At this stage all the acid has been used up and no further copper(II) oxide will dissolve - it is said to be in *excess*.

If the solution turns a cloudy pale green, add further acid, 1 cm^3 at a time with prolonged stirring, until the solution goes clear blue again.

Most substances containing the blue Cu^{2+} ion are blue or green.

As soon as this stage is reached put a filter paper and funnel in the top of a conical flask and, using the glass rod to guide the liquid into the filter paper, filter off the remaining solid, collecting the filtrate in the conical flask.

There will probably be too much water present for crystals of copper(II) sulphate 5-water to separate at this stage, so some of it must be removed. Pour the solution into an evaporating dish and heat gently. At intervals, dip the end of a clean dry glass rod into the evaporating liquid, withdraw it with a drop of liquid on the end, and then allow it to cool. If the main solution is ready to crystallize the drop of liquid on the end of the glass rod will turn almost completely to crystals.

When the solution is ready, turn off the Bunsen burner and allow the evaporating dish to cool. When the crystals have had time to form fully, scrape them into a fresh filter paper with the glass rod and filter off any liquid. Wash the crystals with a little distilled water from a wash bottle. This can be accomplished simply by squirting a jet of water over the crystals.

Place the crystals on a piece of tissue to dry. Ask your lecturer if the crystals themselves are going to be assessed. If so, label the filter paper on which they stand with the names of your group members since they will be inspected by the lecturer and a mark awarded. Good crystals are large and regular in shape and of an even colour. When dissolved in water the resulting solution should not contain any sulfuric acid.

PROBLEMS

1) Why is it desirable to use an excess of the copper(II) oxide in the reaction i.e. why is it arranged that some copper(II) oxide remains unreacted?

2) Why are the crystals washed with water?

3) What is the chemical name and the formula of
   a) the acid used in the experiment?
   b) the base used in the experiment?
   c) the blue crystals which were prepared in the experiment?

4) What is meant by the term "salt" in chemistry?

5) The word "anhydrous" means "without water". What is the formula for anhydrous copper(II) sulphate?

6) Give three properties of acids and bases which serve to distinguish one from the other.

7) What is meant by the term "alkali"? How does an alkali differ in its physical properties from a base such as copper(II) oxide? Give the names and formulae of two alkalis.

8) How do you know when the solution containing the copper(II) sulphate is ready to crystallize?

9) Calculate the relative formula masses of: anhydrous copper(II) sulfate and copper(II) sulfate 5-water. (Ans. 160 and 250 respectively, to nearest whole number.)

10) What element (when combined with other elements) forms compounds which are usually bluish in colour?

PRACTICAL CLASS NO. 9

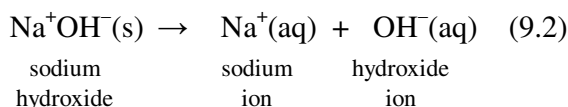## PART A: ACIDITY, BASICITY AND pH

THEORETICAL BACKGROUND

Acids and bases can be distinguished in many ways, amongst them their effect on litmus paper. Acids turn "blue" litmus paper "red" and bases turn red litmus paper blue. (Red litmus paper is really more of a pink colour, and blue litmus paper is rather purplish.) A solution which does not affect litmus paper is said to be neutral.

Acids and bases owe their properties to their effects on hydrogen ions. The molecules of an acid split up in water. They release hydrogen ions.

$$H_2SO_4(l) \rightarrow 2H^+(aq) + SO_4^{2-}(aq) \quad (9.1)$$

sulfuric hydrogen sulfate
acid ion ion
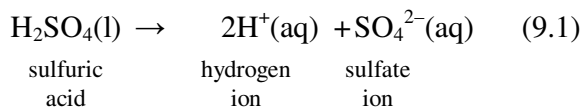
Bases release hydroxide ions when they dissolve in water.

$$Na^+OH^-(s) \rightarrow Na^+(aq) + OH^-(aq) \quad (9.2)$$

sodium sodium hydroxide
hydroxide ion ion

Hydroxide ions react readily with hydrogen ions to form water molecules.

$$H^+(aq) + OH^-(aq) \rightleftharpoons H_2O(l) \quad (9.3)$$

hydrogen hydroxide water
ion ion molecule
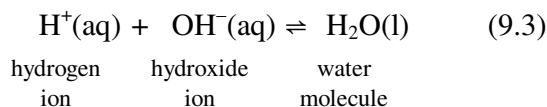
For this reason hydroxide ions tend to reduce the concentration of hydrogen ions in a solution. The "$\rightleftharpoons$" sign in the equation above means that the reaction is reversible. Hydrogen and hydroxide ions rapidly, and almost completely, join together to form water molecules when they meet each other, but also water molecules have a slight tendency to split up into hydrogen and hydroxide ions. For this reason even pure water contains hydrogen ions in a very low concentration[6]). Pure water is by definition neutral (that is, neither acidic nor basic). Solutions with a higher concentration of hydrogen ions than pure water are acidic.

There is a simple relationship between the concentration of hydrogen ions and the concentration of hydroxide ions. It is like a see-saw:

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[6] 0.000 0001 mol dm^{-3} in pure water or any neutral aqueous solution, that is a solution where the solvent is water.
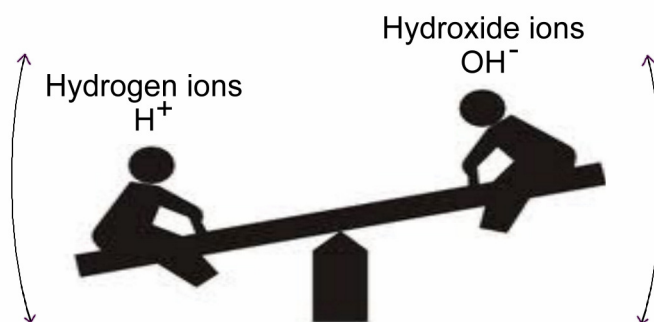
Figure 9.1 The hydrogen ion/hydroxide ion seesaw. As hydrogen ions go up, hydroxide ions go down, and vice versa. In pure water the concentrations are equal.

The concentration of hydrogen ions in pure water may be increased by adding an acid. If the concentration of hydrogen ions is high, the solution is ***strongly acidic***. If the concentration of hydrogen ions is low but still higher than in pure water, the solution is ***weakly acidic***.

Bases increase the concentration of hydroxide ions in solution, and so decrease the concentration of hydrogen ions because they react with them according to equation 9.3. If the concentration of hydroxide ions is much higher than in pure water, the concentration of hydrogen ions will be very low (see figure 9.1), and the solution is ***strongly basic*** (we also say "strongly alkaline"). If the concentration of hydroxide ions is just slightly higher than in pure water, the concentration of hydrogen ions is not quite so low and the solution is ***weakly basic***.

The concentration of hydrogen ions in a solution is conveniently represented by the pH value. A neutral solution has pH 7. Contrary to what one would expect, a low pH (below 7) represents a high concentration of hydrogen ions and so an acidic solution. A high pH (above 7) represents a basic (or alkaline) solution. This is summarized below.

pH VALUE

The concentration of hydrogen ions in a solution is conveniently represented by its pH value[7]. This is summarized below. Note that fractional values are possible, for example 13.2 is a strongly basic pH between 13 and 14.

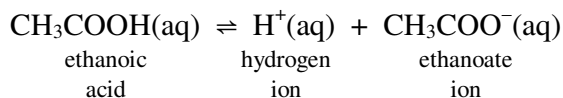| pH VALUE	DESCRIPTION
14	Very strongly basic
13	Strongly basic
12	Strongly basic
11	Weakly basic
10	Weakly basic
9	Weakly basic

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[7] Technically the pH is given by pH = -log(concentration of hydrogen ions). You will find the log function on a scientific calculator.

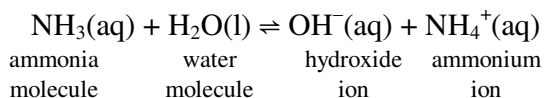|	
8	Very weakly basic
7	Neutral
6	Very weakly acidic
5	Weakly acidic
4	Weakly acidic
3	Weakly acidic
2	Strongly acidic
1	Strongly acidic
0 (or negative)	Very strongly acidic

STRONG AND WEAK ACIDS AND BASES

Some acids, such as hydrochloric acid, nitric acid and sulfuric acid are called ***strong acids***. This is because they split up completely into ions in solution - hydrogen ions and negative ions. They are said to ***dissociate*** completely in solution. For example, sulphuric acid behaves like this. Its dissociation is given by equation 9.1 above.

Most acids are weak acids. They only release a few of their hydrogen atoms in solution. Examples include carbonic acid and ethanoic acid. They are said to dissociate only partially and so the reversible sign, $\rightleftharpoons$, is used. For ethanoic acid (CH_3COOH - found in vinegar) this is written:

$$CH_3COOH(aq) \rightleftharpoons H^+(aq) + CH_3COO^-(aq)$$

      ethanoic   hydrogen   ethanoate
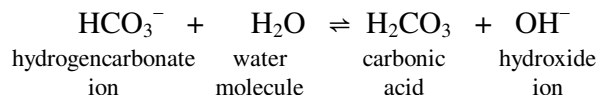   acid   ion   ion

Something similar also applies to bases. A strong base dissociates completely into hydroxide ions and positive ions in water. Sodium hydroxide is a good example, as shown in equation 9.2 above.

A base which releases only a few hydroxide ions in solution is known as a *weak* base. Ammonia is a weak base, as is the hydrogencarbonate ion (HCO_3^-). These bases react with water molecules to release hydroxide ions.

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

     ammonia   water   hydroxide   ammonium
     molecule   molecule   ion   ion

(For this reason solutions of ammonia are often referred to as "ammonium hydroxide". This, however, is not quite correct.)

$$HCO_3^- + H_2O \rightleftharpoons H_2CO_3 + OH^-$$

hydrogencarbonate   water   carbonic   hydroxide
        ion   molecule   acid   ion

Weak bases only lower the concentration of hydrogen ions in solution slightly.

NEUTRAL SUBSTANCES

Substances (or ions) which do not have any tendency to release hydrogen ions into solution, nor to remove them from solution, may be described as neutral.

INDICATORS

Litmus is a dye whose colour varies depending on whether it is placed in acidic or basic solution. Such dyes are known as *acid/base indicators*, or just indicators. Apart from litmus, there are many dyes which function as acid/base indicators. Two very useful ones are *methyl orange* and *phenolphthalein*. Litmus changes colour at around pH 7. Other indicators change colour at different pH values: methyl orange around pH 4 and phenolphthalein around pH 9. Litmus is usually used absorbed on paper (hence "litmus paper") whereas the other two are used as solutions.

INDICATOR	pH AROUND WHICH COLOUR CHANGES	COLOUR AT LOWER pH	COLOUR AT HIGHER pH
METHYL ORANGE	3.5	RED	ORANGE
LITMUS	7	RED	BLUE
PHENOLPHTHAL-EIN	9	COLOURLESS	PURPLE

UNIVERSAL INDICATOR

By mixing several different indicators together we can produce a mixture that shows a range of colours, the actual colour depending on the pH. Such a mixture is known as a *universal indicator*. It is possible to measure the pH of a solution by dipping a piece of paper containing the universal indicator into the solution and comparing the colour of the paper with a colour chart.

PRACTICAL EXERCISE

In today's laboratory exercise you will see the use of several indicators, as well as Universal Indicator paper You will test various solutions with the indicators and record the colours developed. Using the universal indicator paper you will measure the pH of each of the solutions and so classify them as strongly acidic, weakly basic, and so on.

APPARATUS & MATERIALS

Solutions of the following indicators: methyl orange and phenolphthalein.

The following indicator papers: litmus (red and blue) and universal.

About half a dozen small test tubes, a test tube rack, a white tile, and several small beakers.

Solutions of sulfuric acid, ethanoic acid, sodium hydroxide, and ammonia, all in 0.1M concentration[8].

METHOD

1) Measure 1 cm^3 of the first solution (this could be any) into a measuring cylinder and add water to the 10 cm^3 mark. Be sure you use the 0.1M solutions not the 1M solutions which are used for the second experiment. Tip the contents into a test tube to mix them.

2) Test the solution by placing a drop on a small piece of litmus paper using a glass rod. Use red litmus paper for testing bases, and blue litmus paper for testing acids. Record the new colour of the litmus

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[8] "M", pronounced "molar", is a unit of concentration. It tells us how much of a substance (measured in "moles") is present in a cubic decimetre (dm^3) of solution. A cubic decimetre is also known as a "Litre". It is roughly equal to a US quart.

paper.

3) Place a drop of the solution on a piece of universal indicator paper. Compare the new colour with the colour chart supplied with the indicator paper. Read the pH off the chart and record this in the results table.

4) Split the remaining solution into two parts. Place one drop of methyl orange indicator solution in one part and one drop of phenolphthalein indicator solution in the other. Record the colour of the solution in the results table. (If there is no colour record: "colourless".) The colours are best viewed against a white background.

RESULTS & CONCLUSIONS

Complete the table below and decide whether each of the solutions is weakly acidic, strongly basic etc. Record your decisions in the column headed "classification".

SOLUTION OF:	INDICATOR: RECORD FINAL COLOUR AND pH			pH from UNIVERSAL INDICATOR	CLASSI-FICATION
	METHYL ORANGE	PHENOL-PHTHALEIN	LITMUS PAPER		
0.1M SULFURIC ACID (H_2SO_4)					
0.1M ETHANOIC ACID ($C_2H_4O_2$)					
0.1M SODIUM HYDROX-IDE (NaOH)					
0.1M AMMONIA (NH_3)					

PART B: ENDOTHERMIC AND EXOTHERMIC PROCESSES

Note: your supervisor will tell you which parts of this practical to perform, based on time and the availability of materials.

THEORETICAL BACKGROUND

Chemical changes, and some physical changes, involve heat. Sometimes the heat is used up *i.e.* converted into chemical energy. In this case the change is referred to as ***endothermic***. If a thermometer is dipped into a solution in which such a change is occurring, it will show a decrease in temperature. Sometimes we say that heat is "taken in" during endothermic processes.

Sometimes, on the other hand, and this is more common, heat is released during a chemical (or sometimes a physical) process. In other words chemical energy is converted into heat. Such processes are known as ***exothermic***. They are associated with a ***rise*** in temperature.

PRACTICAL EXERCISE

During this exercise you will investigate some processes to see whether they are exothermic or endothermic.

APPARATUS AND MATERIALS

50 cm^3 or 100 cm^3 beaker, thermometer, 25 cm^3 measuring cylinder, glass rod, 1M sodium hydroxide solution, 1M hydrochloric acid solution. Sodium thiosulfate 5-water crystals.

METHOD

I) NEUTRALIZATION OF 1 M SODIUM HYDROXIDE SOLUTION

(Principally a chemical change.)

1) Measure 25 cm^3 of 1M sodium hydroxide solution into a 50 cm^3 or 100 cm^3 beaker. (Do not confuse it with the more dilute 0.1M sodium hydroxide solution that you used for the previous experiment.)

2) Measure the temperature of the sodium hydroxide solution with a thermometer. Record the temperature in the results table.

3) Place 25 cm^3 of 1M hydrochloric acid solution in a measuring cylinder.

4) Rapidly pour the hydrochloric acid solution into the beaker of sodium hydroxide solution. Stir vigorously with the glass rod to mix the solutions and immediately measure the temperature of the mixture. Record the highest temperature in the results table.

5) Decide whether the process was endothermic or exothermic and record your decision in the results table.

6) Write a balanced chemical equation, with states, to show the reaction.

II) DISSOLUTION OF SODIUM THIOSULFATE CRYSTALS

(Principally a physical change.)

1) Fill a test tube about one quarter full with distilled water.

2) Measure the temperature of the water with the thermometer.

3) Drop in a few crystals of hydrated sodium thiosulfate crystals.

4) Stir gently with the thermometer and record the lowest temperature reading. If the reading on the thermometer does not change at all, add more crystals of sodium thiosulfate until it does.

5) Decide whether the process was endothermic or exothermic and record your decision in the results table.

III) DISSOLUTION OF CONCENTRATED SULFURIC ACID

(Both physical and chemical changes are involved.)

This experiment will be carried out as demonstration, since if it is done carelessly it can be dangerous!

1) Place about 100 cm^3 of distilled water in a 250 cm^3 beaker.

2) Measure the temperature of the water with a thermometer. (The thermometer must read to above 100°C.)

3) Carefully, a little at a time, with continual stirring, add 25 cm^3 of concentrated sulphuric acid from a measuring cylinder.

4) Record the highest temperature reading.

5) Decide whether the process was endothermic or exothermic and record your decision in the results table.

IV) REACTION OF BARIUM HYDROXIDE AND AMMONIUM NITRATE CRYSTALS

(Principally a chemical change.)

If this experiment is carried out by students, quantities should be divided by 10.

1) Place about 32 g of barium hydroxide 8-water ($Ba(OH)_2.8H_2O$) in a 250 cm^3 beaker.

2) Measure the temperature of the contents with a thermometer. (The thermometer must read to well below 0°C.)

3) Add 18 g of ammonium nitrate and stir gently with the thermometer.

4) Use a piece of moist red litmus to test any gas evolved. Avoid inhaling any gases or vapours given off. What is the gas?

5) Record the lowest temperature reading.

6) Decide whether the process was endothermic or exothermic and record your decision in the results

7) Write a balanced chemical equation, with states, to show the reaction. Barium nitrate and water are among the products.

RESULTS

PROCESS	INITIAL TEMPERA-TURE	FINAL TEMPERA-TURE	TEMPERA-TURE CHANGE (ΔT)[9]	EXOTHERMIC or ENDO-THERMIC
NEUTRALI-ZATION OF SODIUM HYDROXIDE				
DISSOLU-TION OF SODIUM THIOSUL-FATE				
DILUTION OF CONCEN-TRATED SULFURIC ACID				

PROBLEMS

1) Write down chemical equations to represent the dissociation of each of the acids or bases used in this practical (parts A & B), as well as that of water itself. Make sure you include state symbols.

9Put "+" for an increase and "-" for a decrease eg.-2.5°C means a decrease of 2.5°C.

2) Write down chemical equations to represent the reactions you encountered in part B of the practical. Make sure you include state symbols.

3) Explain why, when diluting concentrated sulphuric acid, you should always add acid to water, never the other way round.

PRACTICAL CLASS NO. 10
FURTHER SALT PREPARATIONS

## PART A: PREPARATION OF AN ACID SALT BY TITRATION
INTRODUCTION

This method of preparing salts is necessary when all the reactants and products are soluble in water. In this situation it is very difficult to judge when the right amounts of reactants have been mixed, so that the result is the pure salt and not a mixture of the salt and unused reactants. With the preparation of copper(II) sulfate it was easy: we just added a little bit more than enough of the copper(II) oxide to use up the sulfuric acid - the extra was easily filtered off since it is insoluble in water. In preparation B below the situation is even simpler - the salt itself is insoluble and can be separated from any unused reactants or other products by filtration or centrifugation.

The situation is most difficult when we want to make an acid salt. This is a salt in which not all of the replaceable hydrogen of an acid has been replaced with a metal. For example, in potassium sulfate (K_2SO_4) all the hydrogen of sulfuric acid (H_2SO_4) has been replaced with potassium. Potassium sulfate is known as a normal salt. The corresponding acid salt is potassium hydrogensulfate ($KHSO_4$). Here only half of the replaceable hydrogen has been replaced by potassium. Potassium hydrogensulfate can be made by reacting potassium hydroxide solution with dilute sulfuric acid:

$$KOH(aq) + H_2SO_4(aq) \rightarrow KHSO_4(aq) + H_2O(l)$$
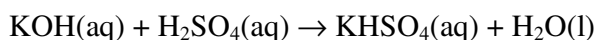
but if too much potassium hydroxide is added the normal salt will be formed:

$$H_2SO_4(aq) + 2KOH(aq) \rightarrow K_2SO_4(aq) + 2H_2O(l)$$
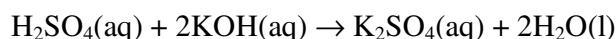
It is important to mix exactly the right quantities of acid and alkali if a pure product is to be obtained. This is achieved by titration. Here acid is added to alkali carefully until all the alkali has been used up. At this stage the solution turns from alkaline to acidic and contains the normal salt and the amount of acid added may be measured.

$$2KOH(aq) + H_2SO_4(aq) \rightarrow K_2SO_4(aq) + 2H_2O(l)$$
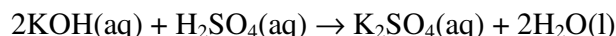
This change can be spotted by testing the solution with an indicator. If the same amount of acid is then added again the normal salt is turned into the acid salt:

$$K_2SO_4(aq) + H_2SO_4(aq) \rightarrow 2KHSO_4(aq)$$
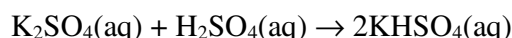
You will also be asked to measure temperatures in order to investigate any heat changes during the reaction.

METHOD AND RESULTS

1) Using a measuring cylinder, carefully measure 15 cm^3 of the potassium hydroxide solution in a conical flask. Add 1 or 2 (literally) drops of litmus solution using a teat pipette. Note the colour of the solution and then measure and record its temperature.

   Temp = _____ °C   Colour: _____

2) Mount a burette vertically in a burette clamp. Fill it with dilute sulfuric acid nearly to the brim. Open the tap wide to run some acid into a beaker underneath and expel air bubbles from the tap. Run acid out of the burette until the level is at or below the zero mark. Note this initial reading of the burette.

   Initial burette reading = _____ cm^3.

3) Run about 10.0 cm^3 of acid from the burette into the flask fairly rapidly, swirling and checking for any colour change as you go. If you note a colour change stop adding acid and immediately and call

for advice. Record the temperature of the solution again.

Temp = _____ °C

4) If the solution is still alkaline (it should be) continue adding acid from the burette, 0.5 cm^3 at a time, swirling and checking the colour after each 0.5 cm^3 addition, until the solution is neutral (i.e. the colour is intermediate between blue and red) or JUST acidic (i.e. just turned red). Record this final burette reading and calculate what volume of acid has been added.

Final burette reading = _____ cm^3

Volume of acid added = final burette reading - initial burette reading = _____ cm^3.

5) Discard the solution in the conical flask, rinse it with distilled water and carefully measure a second portion of 15 cm^3 of potassium hydroxide into it. Do not add any indicator.

6) Add double the volume of acid added in the first run. For example, if you first added a total of 16.5 cm^3, add a total of 33.0 cm^3.

7) Transfer the solution to an evaporating dish and evaporate vigorously until the volume has been reduced to about 5½ to 6 cm^3. (Measure the volume from time to time with a measuring cylinder.)

8) Pour the remaining solution into a test tube and allow to cool. Filter off any crystals which form.

9) Examine the crystals with a hand lens and compare their appearance with a sample of the normal salt (potassium sulfate) provided[10].

10) Dissolve a little of your salt and the potassium sulfate separately in water. Test each solution with universal indicator paper and record any difference in pH.

## PART B: PREPARATION OF AN INSOLUBLE SALT BY PRECIPITATION

INTRODUCTION

The reaction between an acid and a base (such as you saw in practical class no. 8 and experiment A above) is not the only method of preparing a salt. If the salt is insoluble in water it is usually possible to prepare it by reacting two soluble substances in a reaction known as partner exchange, or less accurately "double decomposition". Salts usually consist of two parts: a metal (or a metallic group) and a non-metal (or a non-metallic group). The metal and non-metal are present in their combined forms which are completely different from the free elements. What makes the difference is their charge. The metal atoms carry a positive charge and are known as positive ions, or "cations"; and the non-metal atoms carry a negative charge and are known as negative ions or "anions". The positive and negative charges are always present in equal numbers in a salt and so the salt as a whole has no charge.

Zinc nitrate and sodium carbonate are no exceptions. Zinc nitrate may be written simply as $Zn(NO_3)_2$ or alternatively as $Zn^{2+}(NO_3^-)_2$, showing that each zinc ion carries two positive charges and each nitrate ion (NO_3^-) carries one negative charge. (That's why there are two nitrate ions to each zinc ion: to make the total charge zero.)

Sodium carbonate may be written simply as Na_2CO_3 or to show the charges: $(Na^+)_2CO_3^{2-}$. The sodium ions carry only one positive charge each and the carbonate ions carry two negative charges each. That is why there are two sodium ions to each carbonate ion.

In a partner exchange reaction the ions simply swap partners, for example:

$$(Na^+)_2CO_3^{2-}(aq) + Zn^{2+}(NO_3^-)_2(aq) \rightarrow Na^+NO_3^-(aq) + Zn^{2+}CO_3^{2-}(s)$$
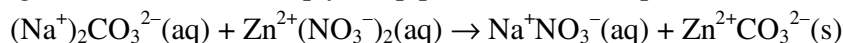
10If the crystals provided have no clear shape, make fresh ones by the following method. Place potassium sulfate in a test tube to a depth of about ½ cm. Add distilled water to about double this depth. Heat to nearly boiling whilst mixing. Pour off the supernatant into another tube and allow this to cool. Crystals should form. Filter them off.

Since the reaction can only work if the ions are free to move it has to be carried out in solution. The zinc carbonate is insoluble and so it precipitates. This provides the driving force for the reaction.

In fact, in solution, the ions are really completely independent of one another, and so the reaction may more simply be represented as:

$$Zn^{2+}(aq) + CO_3^{2-}(aq) \rightarrow Zn^{2+}CO_3^{2-}(s)$$
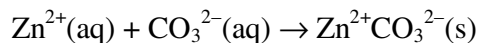
(In a solid the ions are firmly stuck together.)

Any solutions containing the required partners may be mixed and the precipitate formed is then removed by filtering or centrifuging. The solid must be washed to remove unwanted impurities and then dried in an oven.

In practice sodium carbonate solution is not used in this preparation of zinc carbonate, since it is too alkaline and other reactions tend to occur which lead to the precipitation of basic zinc carbonate ($ZnCO_3.Zn(OH)_2$). Since the basic salt is not wanted, the rather more acidic sodium hydrogencarbonate ($NaHCO_3$) is used as the source of carbonate ions. The hydrogencarbonate ions tend to split into hydrogen ions and carbonate ions in solution:

$$HCO_3^-(aq) \rightarrow H^+(aq) + CO_3^{2-}(aq)$$
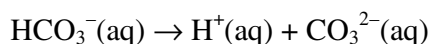
and the carbonate ion reacts with zinc ions as above.

METHOD

1) Add about 10 cm^3 of zinc chloride solution (1M) to an equal volume of sodium hydrogencarbonate solution (2M). If effervescence occurs test with litmus paper. If the litmus turns red, add further sodium hydrogencarbonate solution a drop at a time with a teat pipette until a test with litmus paper reveals that the solution is no longer acidic.

2) Split the mixture equally between two *identical* centrifuge tubes and place them in the centrifuge directly opposite each other. Spin for about half a minute to precipitate the zinc carbonate.

3) Decant off the "supernatant" (the liquid on top), add a few cm^3 of distilled water, stir thoroughly and then spin again as before.

4) Finally, pour off the water and transfer the precipitate to a filter paper to dry. Depending on time and availability you may place the filter paper in a cool oven to hasten drying.

PROBLEMS

1) What salt was prepared in experiment A?

2) Suggest three salts other than the one prepared in experiment A that could be prepared by reaction of an acid with an alkali. You will need to look up the solubilities of various substances in your text-book.

3) For each of the salts of question 2 suggest a pair of solutions that could be used as starting materials.

4) Write chemical equation for the preparation of one of the salts you suggested for question 2.

5) What was being removed when the precipitate in experiment A was being washed with water?

6) Why was no indicator placed in the solution the second time the acid was added to the alkali in experiment A?

7) During the preparation of the acid salt in experiment A, acid was added to alkali. What experimental evidence was there to suggest that a chemical reaction occurred? Was the reaction exothermic or exothermic? Give a reason for your answer.

8) Explain any differences you observed when you tested the solutions of potassium hydrogensulfate

and potassium sulfate with universal indicator paper.

9) What salt was prepared in experiment B?

10) Do this question only if you have covered ionic equations in class. The chemical equations in experiment B are given as full equations. Rewrite them as ionic equations, omitting spectator ions in the normal way.

11) Why must two identical tubes be placed opposite each other in the centrifuge?

12) Suggest three salts other than the one prepared in experiment B that could be prepared by a partner exchange reaction. You will need to look up the solubilities of various substances in your textbook.

13) For each of the salts of question 12 suggest a pair of solutions that could be used as starting materials.

14) Write a chemical equation for the preparation of one of the salts you suggested for question 12.