

# Section B: Some Essential Background Chemistry

## Chapter 10: Making Salts

### Soluble and insoluble salts

#### The importance of knowing whether a salt is soluble or insoluble in water

You will remember that acids react with carbonates to give a salt, carbon dioxide and water. In the case of calcium carbonate (for example, marble chips) reacting with dilute hydrochloric acid, calcium chloride solution is produced.



If you try the reaction between calcium carbonate and dilute sulfuric acid, nothing much seems to happen if you use large marble chips. You will get a few bubbles when you first add the acid, but the reaction soon stops.

*This chapter looks at some of the practical problems in making pure samples of salts in the lab. Remember that a salt is what is formed when the hydrogen in an acid is replaced by a metal. For example, sulfates come from sulfuric acid, chlorides from hydrochloric acid, and nitrates from nitric acid.*



Figure 10.1 Calcium carbonate reacting with dilute hydrochloric acid.

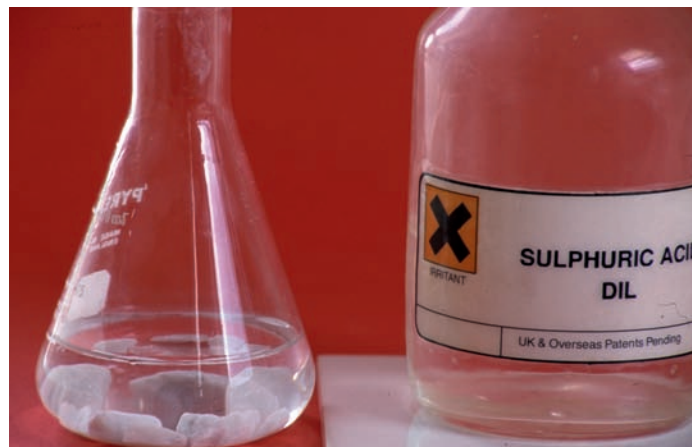


Figure 10.2 Calcium carbonate not reacting with dilute sulfuric acid.

The problem is that the calcium sulfate produced in the reaction is almost insoluble in water. As soon as the reaction starts, a layer of calcium sulfate is formed around the calcium carbonate, stopping any further reaction.

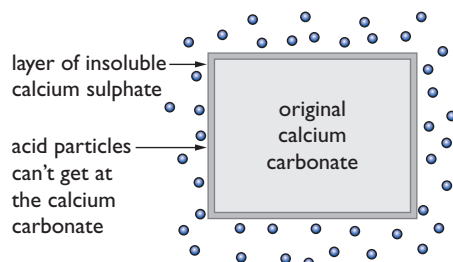


Figure 10.3 The reaction between calcium carbonate and sulfuric acid.

Any attempt to produce an insoluble salt from the reaction between a solid and a liquid will fail for this reason.

You may have noticed that the bottle in the right-hand photograph is labelled as 'sulphuric acid' rather than 'sulfuric acid'. You must expect to come across both versions of this name. 'Sulphuric' is the traditional English spelling.

How quickly the reaction stops may well depend on the size of the marble chips (because that affects the surface area), the concentration of the acid, the volume of acid added, and the amount the flask is shaken. This would make an interesting investigation – particularly if you could then explain any pattern in your results.

**Notes on the table**

To keep the table simple, it includes one or two compounds (like aluminium carbonate, for example) which don't actually exist. Don't worry about these. The problem won't arise at GCSE.

Hydroxides have been included for the sake of completeness, although they are not salts. The list is in reactivity series order, apart from the ammonium group. Ammonium compounds often have similarities with sodium and potassium compounds, and so are included near them.

There is no clear cut-off between 'insoluble' and 'almost insoluble' compounds. The ones picked out as 'almost insoluble' include the more common ones that you might need to know about elsewhere in the course.

**Solubility patterns**

	nitrate	chloride	sulfate	carbonate	hydroxide
ammonium	soluble	soluble	soluble	soluble	soluble
potassium	soluble	soluble	soluble	soluble	soluble
sodium	soluble	soluble	soluble	soluble	soluble
barium	soluble	soluble	insoluble	insoluble	almost insoluble
calcium	soluble	soluble	almost insoluble	insoluble	almost insoluble
magnesium	soluble	soluble	soluble	insoluble	insoluble
aluminium	soluble	soluble	soluble	insoluble	insoluble
zinc	soluble	soluble	soluble	insoluble	insoluble
iron	soluble	soluble	soluble	insoluble	insoluble
lead	soluble	insoluble	insoluble	insoluble	insoluble
copper	soluble	soluble	soluble	insoluble	insoluble
silver	soluble	insoluble	soluble	insoluble	insoluble

key



soluble



insoluble

almost insoluble  
(slightly soluble)

Figure 10.4 Solubility patterns.

Note that:

- all sodium, potassium and ammonium compounds are *soluble*
- all nitrates are *soluble*
- most common chlorides are *soluble*, except lead(II) chloride and silver chloride
- most common sulfates are *soluble*, except lead(II) sulfate, barium sulfate and calcium sulfate
- most common carbonates are *insoluble*, except sodium, potassium and ammonium carbonates
- most metal hydroxides are *insoluble* (or *almost insoluble*), except sodium, potassium and ammonium hydroxides.

It can seem a bit daunting to have to remember all this, but it isn't as hard as it looks at first sight.

Except for the carbonates and hydroxides, most of these compounds are soluble. Learn the exceptions in the sulfates and chlorides. The reason for the exceptions in the carbonates and hydroxides is that all sodium, potassium and ammonium compounds are soluble.

## Making soluble salts (except sodium, potassium and ammonium salts)

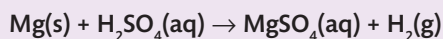
These all involve reacting a solid with an acid. You can use any of the following mixtures:

- **acid + metal** (but only for the moderately reactive metals from magnesium to iron in the reactivity series)
- **acid + metal oxide or hydroxide**
- **acid + carbonate.**

Whatever mixture you use, the method is essentially the same.

### Making magnesium sulfate crystals

Enough magnesium is added to some dilute sulfuric acid so that there is some left over when the reaction stops bubbling. This is to make sure there is no acid left in the final mixture.



The unused magnesium is then filtered off, and the magnesium sulfate solution is concentrated by boiling it until crystals will form when it is cooled. You can test for this by cooling a small sample of the solution quickly. If the sample crystallises, so will the whole solution.

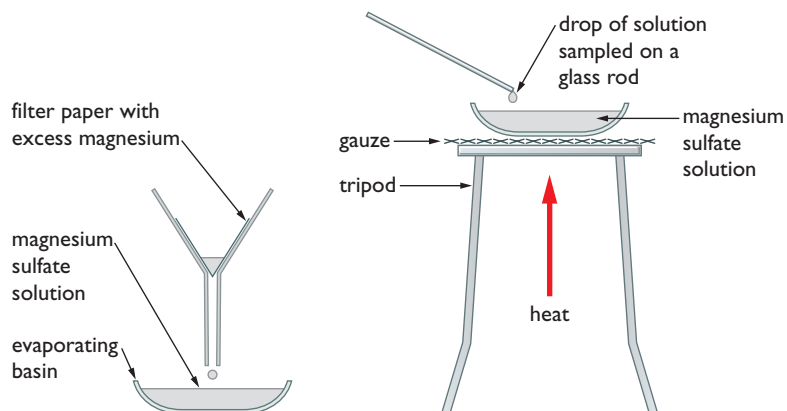


Figure 10.6 Making magnesium sulfate crystals.

In the diagram, a small drop on the end of a glass rod is cooled rapidly in the air to see whether crystals form.

Finally, the solution is left to form colourless magnesium sulfate crystals. Any uncrystallised solution can be poured off the crystals, and the crystals can be blotted dry with paper tissue.

### Why not just evaporate the solution to dryness? Water of crystallisation

It would seem much easier to just boil off all the water rather than crystallising the solution slowly, but evaporating to dryness wouldn't give you magnesium sulfate crystals. Instead, you would produce a white powder of **anhydrous** magnesium sulfate.

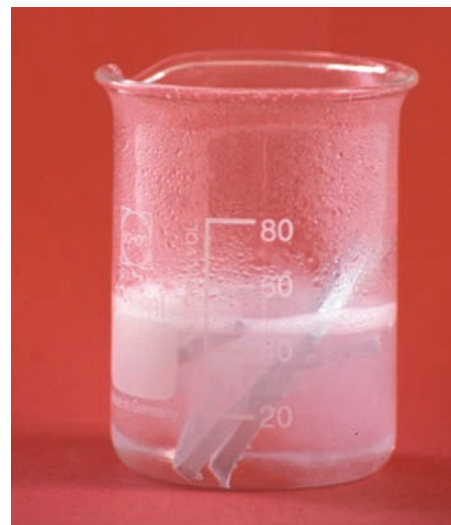


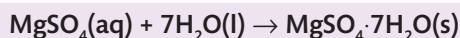
Figure 10.5 Dilute sulfuric acid with excess magnesium.

In the formula for the crystals, you might find either a dot or a comma between the  $\text{MgSO}_4$  and the  $7\text{H}_2\text{O}$ .



Figure 10.7 Copper(II) sulfate crystals.

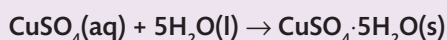
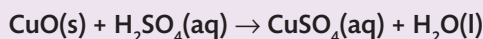
'Anhydrous' means 'without water'. When many salts form their crystals, water from the solution becomes chemically bound up with the salt. This is called **water of crystallisation**. A salt which contains water of crystallisation is said to be **hydrated**.



The extra water in the equation comes from the water in the solution.

### Making copper(II) sulfate crystals from copper(II) oxide

The method is identical, except that you add an excess of black copper(II) oxide to hot dilute sulfuric acid. You can easily see when you have an excess because you are left with some unreacted black solid.



### How do you know whether you need to heat the mixture?

Carbonates react with dilute acids in the cold, and so does magnesium. Most other things that you are likely to come across need to be heated.

cold	hot
carbonates magnesium	most other substances

Figure 10.8 Do you need to heat the mixture?

## Making sodium, potassium and ammonium salts

### The need for a different method

In the method we've just been looking at, you add an excess of a solid to an acid, and then filter off the unreacted solid. You do this to make sure all the acid is used up.

The problem is that all sodium, potassium and ammonium compounds are soluble in water. The solid you added to the acid would not only react with the acid, but any excess would just dissolve in the water present. You wouldn't have any visible excess to filter off. There's no simple way of seeing when you have added just enough of the solid to neutralise the acid.

### Solving the problem by doing a titration

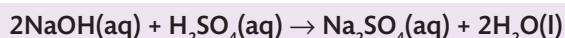
You normally make these salts from sodium or potassium hydroxide or ammonia solution, but you can also use the carbonates. Fortunately, solutions of all these are alkaline. That means you can find out when you have a neutral solution by using an indicator.

The method of finding out exactly how much of two solutions you need to neutralise each other is called a **titration**. The point at which an indicator changes colour during the titration is called the **end point** of the titration.

Before you go on, it would be a good idea to read pages 74–75 in Chapter 9 for the background to these reactions.

Having found out how much acid and alkali are needed, you can make a pure solution of the salt by mixing those same volumes again, but without the indicator.

## Making sodium sulfate crystals



25 cm<sup>3</sup> of sodium hydroxide solution is transferred to a conical flask using a pipette, and a few drops of methyl orange are added as the indicator.

Dilute sulfuric acid is run in from the burette until the indicator turns from yellow to orange.

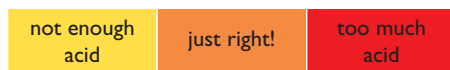
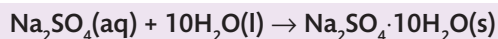
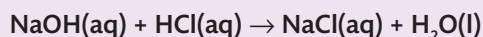


Figure 10.10 Colour changes for methyl orange.

The volume of acid needed is noted, and the same volumes of acid and alkali are mixed together in a clean flask without any indicator. The solution can be crystallised by evaporating it to the point that crystals will form on cooling, and then leaving it for the crystals to form. The crystals are finally separated from any remaining solution and allowed to dry.

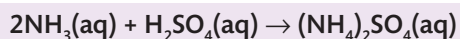


## Making sodium chloride crystals



You would need to do the titration using dilute hydrochloric acid rather than dilute sulfuric acid. However, once you have re-mixed the acid and the alkali without the indicator, you can then evaporate the sodium chloride solution to dryness rather than crystallising it slowly. Sodium chloride crystals don't contain any water of crystallisation, so you can save time by evaporating all the water off in one go. The disadvantage is that you end up with either a powder or very tiny crystals.

## Making ammonium sulfate crystals



Using ammonia solution rather than sodium hydroxide solution makes no difference to the method. Although simple ammonium salts don't have water of crystallisation, you would still crystallise them slowly rather than evaporating them to dryness. Heating dry ammonium salts tends to break them up.

## Making insoluble salts

### Precipitation reactions

To make an insoluble salt, you do a **precipitation reaction**. A **precipitate** is a fine solid that is formed by a chemical reaction involving liquids or gases. A precipitation reaction is simply a reaction that produces a precipitate.



Figure 10.9 Apparatus for carrying out a titration.

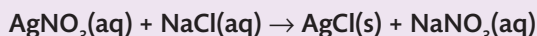


Figure 10.11 A precipitate of silver chloride.

The water molecules in the solutions have been left out to avoid cluttering the diagram.

For example, if silver chloride is produced from a reaction involving solutions, you get a white precipitate formed, because silver chloride won't dissolve in water – and so is seen as a fine white solid.

The photograph shows the results of this reaction:



### Explaining what's happening

Silver nitrate solution contains silver ions and nitrate ions in solution. The positive and negative ions are attracted to each other, but the attractions aren't strong enough to make them stick together. Similarly, sodium chloride solution contains sodium ions and chloride ions – again, the attractions aren't strong enough for them to stick together.

When you mix the two solutions, the various ions meet each other. When silver ions meet chloride ions, the attractions are so strong that the ions clump together and form a solid. The sodium and nitrate ions remain in solution because they aren't sufficiently attracted to each other.

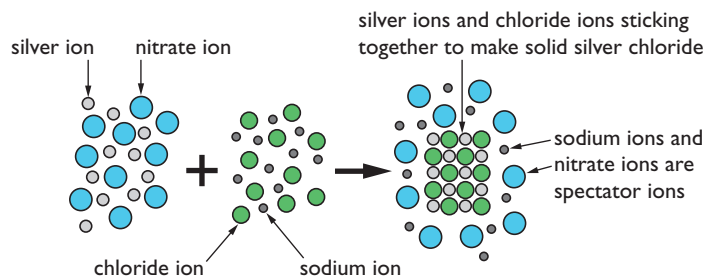
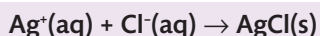


Figure 10.12 Precipitation of silver chloride.

Ionic equations for precipitation reactions are simple to write. Write down the formula for the precipitate on the right-hand side of the equation. Write down the formulae for the ions that have clumped together to produce it on the left-hand side. Don't forget the state symbols.

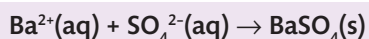
### Writing ionic equations for precipitation reactions

The ionic equation for a precipitation reaction is much easier to write than the full equation. All that is happening in one of these reactions is that the ions of an insoluble salt are clumping together to form the solid. The ionic equation simply shows that happening. You don't need to worry at all about the spectator ions – they aren't doing anything.



This means that if you mix any solution containing silver ions with any solution containing chloride ions, you will get the same white precipitate of silver chloride.

### Making pure barium sulfate



You can mix solutions of any soluble barium compound (for example, barium chloride or barium nitrate) with any soluble sulfate. The sulfate doesn't necessarily have to be a salt. Dilute sulfuric acid contains sulfate ions, so you can perfectly well use that.

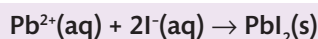
Suppose you decide to use barium chloride solution and dilute sulfuric acid.

You would mix the solutions to get a white precipitate of barium sulfate. The hydrogen ions from the sulfuric acid and the chloride ions are just spectator ions and aren't involved at all.

The mixture is filtered to get the precipitate. The solid barium sulfate is impure because of the presence of the spectator ions and any excess barium chloride solution or sulfuric acid. It is washed with pure water while it is still on the filter paper and then left to dry.

### Making pure lead(II) iodide

It doesn't matter if the salt is unfamiliar to you, as long as you are told that it is insoluble in water. For example, to make lead(II) iodide you would have to mix a solution containing lead(II) ions with one containing iodide ions.



The most common soluble lead(II) salt is lead(II) nitrate. A simple source of iodide ions would be sodium or potassium iodide solution, because all sodium and potassium salts are soluble.

The photograph shows the yellow precipitate of lead(II) iodide. This can now be filtered, washed and dried.

### Summarising the methods of making salts

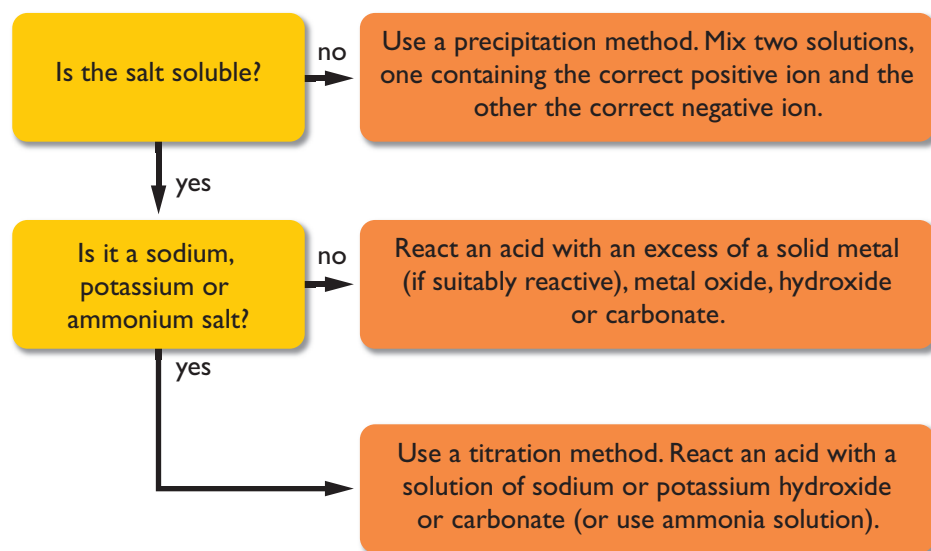


Figure 10.14 Methods of making salts.

In an exam, simply use the words 'filter, wash and dry the precipitate'.



Figure 10.13 A precipitate of lead(II) iodide.

# End of Chapter Checklist

## You should now be able to:

- ✓ know which salts are soluble and which are insoluble in water
- ✓ give practical details of how you would make a pure, dry sample of a salt from the reaction between a suitable solid (metal, metal oxide, metal hydroxide or metal carbonate) and an acid
- ✓ know which salts can be made by this method
- ✓ describe how to make pure, dry samples of sodium, potassium or ammonium salts using a titration method
- ✓ describe how to make pure, dry samples of insoluble salts using precipitation reactions
- ✓ write ionic equations for precipitation reactions.

## Questions

- 1** Sort the following compounds into two lists – those that are soluble in water, and those that are insoluble.  
sodium chloride, lead(II) sulfate, zinc nitrate, calcium carbonate, iron(III) sulfate, lead(II) chloride, potassium sulfate, copper(II) carbonate, silver chloride, aluminium nitrate, barium sulfate, ammonium chloride, magnesium nitrate, calcium sulfate, sodium phosphate, nickel(II) carbonate, chromium(III) hydroxide, potassium dichromate(VI)
- 2** **a)** Describe in detail the preparation of a pure, dry sample of copper(II) sulfate crystals,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , starting from copper(II) oxide.  
**b)** Write full equations for **(i)** the reaction producing copper(II) sulfate solution, **(ii)** the crystallisation reaction.
- 3** **a)** Read the following description of a method for making sodium sulfate crystals,  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ , and then explain the reasons for each of the underlined phrases or sentences.  
  
25 cm<sup>3</sup> of sodium carbonate solution was transferred to a conical flask using a pipette, and a few drops of methyl orange were added. Dilute sulfuric acid was run in from a burette until the solution became orange. The volume of acid added was noted. That same volume of dilute sulfuric acid was added to a fresh 25 cm<sup>3</sup> sample of sodium carbonate solution in a clean flask, but without the methyl orange. The mixture was evaporated until a sample taken on the end of a glass rod crystallised on cooling in the air. The solution was left to cool. The crystals formed were separated from the remaining solution and dried.  
**b)** Write equations for **(i)** the reaction producing sodium sulfate solution, **(ii)** the crystallisation reaction.
- 4** Suggest solutions that could be mixed together to make each of the following insoluble salts. In each case, write the ionic equation for the reaction you choose.  
**a)** silver chloride  
**b)** calcium carbonate  
**c)** lead(II) sulfate  
**d)** lead(II) chloride
- 5** Describe in detail the preparation of a pure, dry sample of barium carbonate. Write the ionic equation for the reaction you use.
- 6** There are three main methods of making salts:  
A reacting an acid with an excess of a suitable solid  
B using a titration  
C using a precipitation reaction.  
  
For each of the following salts, write down the letter of the appropriate method, and name the substances you would react together. You should state whether they are used as solids or solutions. Write an equation (full or ionic as appropriate) for each reaction.  
**a)** zinc sulfate  
**b)** barium sulfate  
**c)** potassium nitrate (nitric acid is  $\text{HNO}_3$ )  
**d)** copper(II) nitrate  
**e)** lead(II) chromate(VI) (a bright yellow insoluble solid; chromate(VI) ions have the formula  $\text{CrO}_4^{2-}$ ).