2014 international year of crystallography

United Nations Educational, Scientific and Cultural Organization

International Union of Crystallography

Partners for the International Year of Crystallography 2014

Global Experiment 2014

The Art of Crystallisation

ROYAL SOCIETY OF CHEMISTRY

Registered charity number 207890

Royal Society of Chemistry

Global Experiment 2014

The Art of Crystallisation

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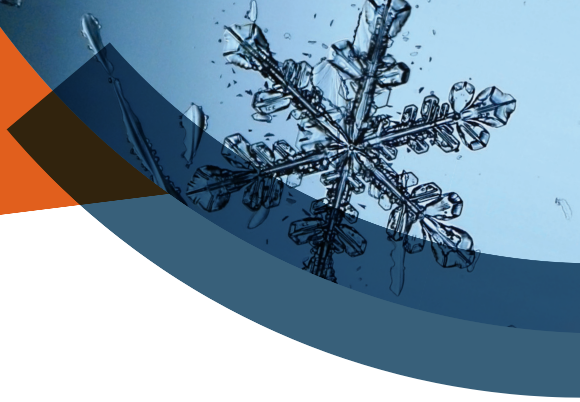

What are the best conditions for growing the biggest crystals?

Introduction

There are four simple parts to the Global Experiment. These instructions will guide you through them.

- **Part A:** Dissolving and saturating your samples (Practical)
- **Part B:** Growing crystals of your samples (Practical)
- **Part C:** Sharing your data – post your results to our global interactive map (Results)
- **Part D:** Analysing the website to discover the best conditions (Conclusions)

We have included some background information on the importance of crystals in everyday life and teacher/technician notes for further advice at the end of these instructions.

Chemicals

Samples	Health and Safety	Availability
Table Salt (sodium chloride, halite)	**LOW HAZARD** (do not consume, risk of contamination)	Readily available in supermarkets
Granulated Sugar (sucrose)	**LOW HAZARD** (do not consume, risk of contamination)	Readily available in supermarkets
Epsom Salts (magnesium sulfate, can be used as bath salts)	**LOW HAZARD**	Readily available in supermarkets, pharmacies, chemical supply companies and online retailers (eg Amazon)
Potassium Nitrate (saltpetre, nitre)	**OXIDISING** (contact with combustible material may cause fire)	Readily available from chemical supply companies and online retailers (eg Amazon)
Alum (Aluminium potassium sulfate, potash alum)	**LOW HAZARD**	Readily available in supermarkets, pharmacies, chemical supply companies and online retailers (eg Amazon)

If you are planning on taking part in this experiment, we advise you to acquire all these samples.

It is advisable to wear safety glasses with side shields for this experiment.
Eyes: Wear eye protection.

For more information, please refer to the general guidance on health and safety at the end of these instructions.

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Background on crystals and their importance in everyday life

Crystals are all around us in everyday life. They range from common and inexpensive items such as salt and sugar through to expensive items such as diamonds and other jewels.

Almost anything can be made into a crystal through the process of crystallisation. Most commonly, crystallisation is the (natural or artificial) process for the formation of solid crystals from a solution. It is possible to grow crystals in other ways too, such as allowing metals to solidify from their melted states. The electronics industry relies on growing single crystals of silicon in this way.

Crystallisation is also a useful chemical technique to separate or purify a solid. This is done by dissolving the sample in a hot liquid, making a saturated solution. Anything that does not dissolve in the hot liquid can be removed by filtration and what remains can then grow pure crystals which can be collected and dried (**figure 1**).

Definition

A crystalline solid is made up of atoms or molecules which are arranged in a repeating pattern and stacked over and over again, very much like a three-dimensional brick wall (or several layers of stacked marbles on top of one another). In many ways, looking at a crystal is the closest the human eye will ever get to observing the order of atoms and molecules.

History

Just over a hundred years ago, father-and-son team William Henry and William Lawrence Bragg first showed that X-rays can be used to map the positions of atoms within a crystalline solid and determine its three-dimensional structure. This process is called crystallography and, to help celebrate this discovery, 2014 is the International Year of Crystallography.
The two Braggs were awarded the Nobel Prize for this discovery in 1915 and, at the age of 25, Lawrence Bragg is still the youngest ever winner. Since this discovery, nearly 30 Nobel Prizes have been awarded which have used crystallography.

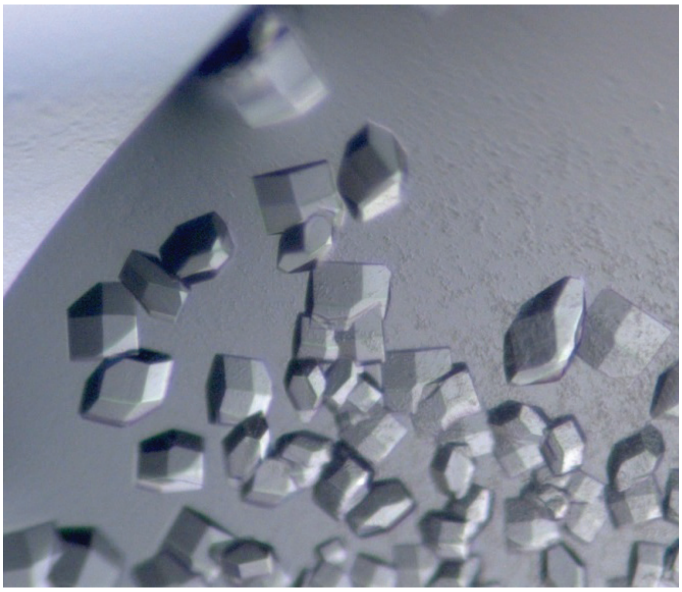

Figure 1
Single crystals of lysozyme, a protein found in hen egg whites

Examples

Some of the largest crystals ever discovered can be found in the *Cueva de los Cristales* in Mexico; some are taller than three human adults! The conditions in which a crystal is grown can affect its size and shape and this can affect the sample's overall properties. The same atoms or molecules can be arranged in different ways within a sample (known as allotropes or polymorphs) and these also affect the sample's properties. One of the best known examples of this is diamond and graphite - both of which are made from carbon (**figure 2**). The structure of carbon in diamond prevents it from conducting electricity but it is well known for its hardness; this is determined by the chemical bonds between the carbon atoms. Graphite, on the other hand, has a different carbon atom arrangement which does allow it to conduct electricity and also means that it is much softer than diamond. In fact, because of its softness, graphite is often used in pencils for drawing as the layers of its atoms can easily slide over each other.

Crystals are important in society today because they are used in many everyday products including washing powders, medicines and electronics to name but a few.

The industries that produce crystalline materials and ingredients must ensure a consistent behaviour from their products. All of the variables in the process of crystallisation must be carefully monitored and controlled so that it can be reproduced.

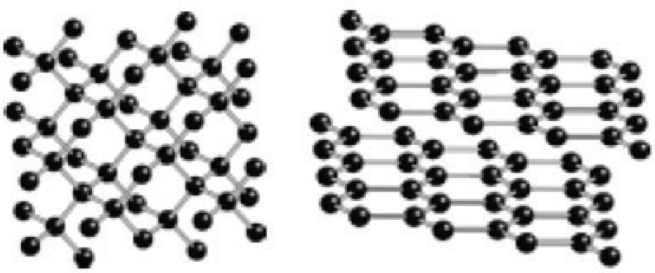

Figure 2
Two of the allotropes of carbon; diamond (left) and graphite (right)

For example:

- Aspirin is absorbed differently by our bodies in its different crystal forms
- Ice cream has to have a consistent crystal size and shape to ensure the right texture, flavour and shelf life. Small crystals make a smoother ice cream. If you leave ice cream in your freezer for a long time, the crystals grow large and it doesn't taste as good!
- The electrical properties of single silicon crystals are useful for semiconductors and computer chips in our phones, televisions and computers
- The pearl-like sheen of many cosmetics is dependent on the right crystal form

In the Global Experiment 2014, you will be performing the process of crystallisation on many readily available samples at home or in schools. You will see that the samples behave very differently and will be able to compare your results with students around the world to see the global picture.

Part A: Dissolving and saturating your samples

In this experiment, you will find out that each sample has its own unique properties. You have a choice of five different samples (table salt, sugar, Epsom salts, alum and potassium nitrate) to make into a saturated solution. Once you have chosen your sample, you will need to carry out the experiment three times to obtain an average and record your observations.

A saturated solution is one which will not allow any more of the sample to dissolve at a particular temperature.

- **If you are taking part in the experiment on your own you will need to test all five samples**

- **Within a class, working in pairs you can experiment with one sample and report your observations back to the class. Collaborate to find out other people's results**

Please post your data to our website.

Materials

- Clear plastic disposable cups (or similar, eg glass beakers)
- The five samples: Table salt, sugar, Epsom salts, alum and potassium nitrate (½ a cup of each sample is plenty)
- Teaspoon (or spatula)
- Cold tap water
- Small measure (measuring 40 cm^3 is required, eg. a measuring cylinder, beaker or clean medicine syringe)
- Balance or kitchen scales
- Thermometer (widely available from online retailers)

Procedure

1. Accurately measure 40 cm^3 of cold tap water into a clear plastic disposable cup and record its mass (record this in the student worksheet provided **[A]**).
2. Carefully add ¼ of a teaspoon of your sample (table salt, sugar, Epsom salts, alum or potassium nitrate) to the cup of water and stir for 30 seconds. Once dissolved, continue adding ¼ teaspoon measures followed by stirring until no more of the sample will dissolve.
3. Measure the temperature of this saturated solution (record in the table **[B]**).
4. Record the mass of the cup and saturated solution which should clearly have approx. ¼ of a teaspoon quantity of solid sample sitting at the bottom (record in the table **[C]**).
5. Calculate the mass of the sample required to saturate 40 cm^3 of your local tap water (record in the table **[D]**).
6. To ensure your data are consistent, repeat steps 1 to 5 twice more or compare with class colleagues.
7. Using the student worksheet, calculate the average temperature of the saturated solution during the experiment **[E]** and record this for posting to the website.
8. Calculate the average mass of your sample needed to saturate 40 cm^3 of your local tap water **[F]** and record this for posting to the website.
9. Gather the data for all five samples and record this in the overall conclusion.

Part A: Dissolving and saturating your samples

Student worksheet

Name ....

What is the definition of a crystal? ....
....
....

The sample I am testing is ....

	Result 1	Result 2	Result 3
Mass of disposable cup and 40 cm^3 of cold local tap water (g) **[A]**			
Temperature of the saturated solution (°C) **[B]**			
Mass of saturated solution and cup (g) **[C]**			
Mass of my sample needed to saturate 40 cm^3 of cold local tap water (g) **[C - A = D]**			
Average Data			
Average temperature of the saturated solution **(°C) [(B1+B2+B3) / 3 = E]**			
Average mass of my sample needed to saturate 40 cm^3 of cold local tap water (g) **[(D1+D2+D3) / 3 = F]**			

Overall conclusion for posting to the website

	Table Salt	Sugar	Epsom Salts	Alum	Potassium Nitrate
The average temperature of the saturated solution during the experiment for each sample (°C) **[E]**					
The average mass to saturate 40 cm^3 of local tap water for each sample (g) **[F]**					

Could you tell the difference between the samples if they were not labelled?
....
....
....

If an unknown sample was used to prepare a saturated solution with average properties **[E]** 8 °C and **[F]** 9.5 g, which of the five samples do you think it could be?
....
....
....

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Part B: Growing crystals of your samples

You have completed 'Part A: Dissolving and saturating your samples' and will have seen that the five samples have very different properties. In this experiment you will be making a saturated solution at a higher temperature and then cooling it down so that some of the dissolved material comes out of solution in a crystalline form.

All five samples should be tested by the class (or individuals).

- **Do you think the amount of sample that dissolves in hot water will be different from cold? Why?**

- **Can you predict which sample will grow the biggest crystal?**

After a week of crystal growth you should post your most successful result from each sample to our website (in the unlikely event that you get no crystals, please post this data also).

Materials

- Kettle (hot water required, needs adult supervision) [Potential burns/spill/slip hazards]
- Thermometer (widely available from online retailers)
- Container/cup (eg glass beakers, styrofoam cup or clear plastic disposable cup)
- The five samples: Table salt, sugar, Epsom salts, alum or potassium nitrate (½ a cup of each sample is plenty)
- Teaspoon (or spatula)
- Small measure (measuring 40 cm^3 is required, eg. a measuring cylinder, beaker or clean medicine syringe)
- Filter paper (or paper towel/kitchen roll/coffee filters)
- A thin wooden food skewer (contamination: do not re-use)[Potential sharp stick injury]
- Clothes pegs (or alternative way to suspend the skewer in the saturated solution)
- A magnifying glass to see your crystals more clearly

Procedure

1. Ask an adult to boil tap water.
2. Into a clean container/cup add four full teaspoon measures of your sample (table salt, sugar, Epsom salts, alum or potassium nitrate).
3. Ask an adult to measure 40 cm^3 of the hot water (the temperature needs to be at 70 °C or above) and transfer this to your container/cup with your sample inside. [Potential burns/spill/cup melting hazards] – [Safety Tip: you could use secondary containment to prevent burns or spills].
4. Stir for 30 seconds and – if required – add more sample repeatedly until your sample will no longer dissolve (larger amounts than in Part A can be added to get to saturation).
5. Fold a square filter paper into a triangle making two folds and open it making a cone shape (see **figure 3**).
6. Pour your warm saturated sample through the cone-shaped filter paper into a clean, empty plastic disposable cup (this process removes undissolved material).
7. Using a wooden skewer and clothes pegs, suspend the tip of the skewer just below the surface of the solution (see **figure 4**).
8. Leave the cup for a week for crystal growth. After a few hours, crystals can often be seen in the bottom of the cup but the slower-growing crystals will grow on the stick. [Tip: if after one day you do not get any crystals add a few grains of solid sample to encourage crystal growth].
9. After a week, record the temperature of the remaining liquid on your results table **[G]**.
10. Remove the stick and identify the best (biggest) single crystal from your cup (see **figure 5**).
11. Match your crystal to our 'size and shape charts' below and record your data in the results table **[H]** and **[I]**. Collaborate with others to get results for all the samples.

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Part B: Growing crystals of your samples

Experiment Setup

Figure 3
The folded cone-shaped paper towel which can be used to filter the solution

Figure 4
Suspend the stick so that the tip sits just below the surface of the solution

Figure 5
An example of a crystal (end point): take a photo and post your data to us

Crystal observations

The stick may have grown one large crystal (**figure 5**) or grown multiple smaller crystals. You will also have crystals in the bottom of the cup; please choose the biggest.

The samples you have crystallised typically fit the following crystal systems: table salt (cubic, **X**), sugar (monoclinic, **U**), Epsom salts (orthorhombic, **V**), alum (cubic, **X**) and potassium nitrate (orthorhombic, **V**).

Look at your crystals and see if you agree with this classification, make your own choice and record your observations on your student worksheet.

Now collaborate with your class (if working alone compare all your results) to obtain the best result for each sample and post this data to our website.

- The temperature of the remaining solution for each sample (°C) **[G]**
- The biggest single crystal from each sample (range from 8-28) **[H]**
- The most likely crystal shape for each sample (range from T-Z) **[I]**

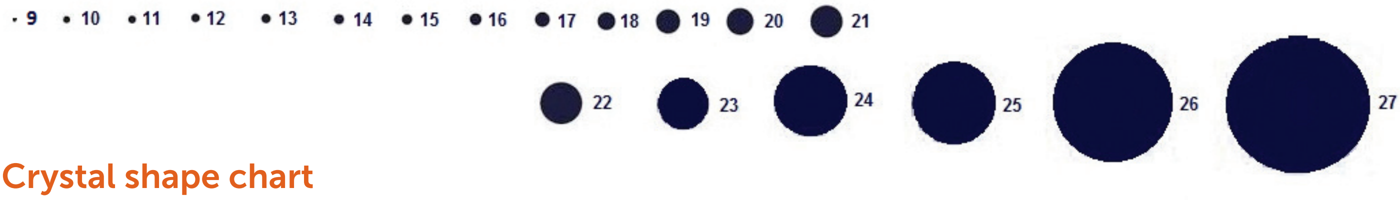
Tip: A magnifying glass will help you see your crystal system more clearly.

Part B: The crystal size and shape charts

Please print and share for class use

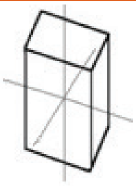
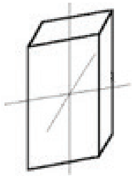
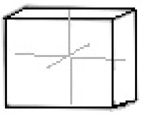
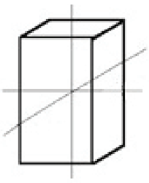
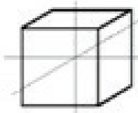
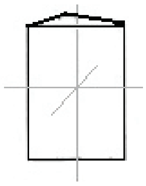
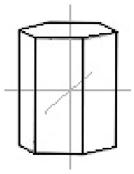
Crystal size chart

Choose a number closest to your sample (8 = smaller, 28 = bigger) **[H]**.
If you have needle-shaped crystals, fit the length to the circle's diameter below.



Crystal shape chart

Choose a letter of the system closest to your sample **[I]**.

						
Triclinic (T)	**Monoclinic (U)**	**Orthorhombic (V)**	**Tetragonal (W)**	**Cubic (X)**	**Trigonal (Y)**	**Hexagonal (Z)**
Like a packet of cereal squashed in two directions	Like a packet of cereal squashed on one side (it can be hard to tell T and U apart – U is a thin crystal)	Like a packet of cereal (or matchbox) crystals can also be very long and 'needle-like' (fairly common)	Like two cubes stuck together	Like a cube (fairly common) sometimes the crystals do not have pointed corners so can appear 'dia-mond-like'	Like a 'Toblerone™' crystals can be needle-like in appearance (quite rare but distinctive)	Like an unsharpened pencil, crystals can be 'needle-like' (quite rare but distinctive)
Crystal has six faces	Crystal has six faces	Crystal has six faces	Crystal has six faces	Crystal has six faces	Crystal has five faces	Crystal has eight faces
Lengths: from one corner the three sides are different lengths	Lengths: from one corner the three sides are different lengths	Lengths: from one corner the three sides are different lengths	Lengths: from one corner two sides are of the same length and the other is approx. double	Lengths: from one corner all the sides are of the same length	Lengths: from one corner two sides are of the same length and the other is longer	Lengths: from one corner two sides are of the same length and the other is longer
Corners: none are at 90°	Corners: some are 90° but not all	Corners: all are at 90° (often a thin crystal)	Corners: all are at 90°	Corners: all are at 90°	Corners: some are at 90° and others are not	Corners: some are at 90° and others are not

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Part B: Growing crystals of your samples

Student worksheet

Name ....

Do you think the amount of sample that dissolves in hot water will be different from cold? Why?

....

....

....

....

Can you predict which sample will grow the biggest crystal? Why?

....

....

....

....

The sample I am using to grow crystals is. ....

Temperature of the remaining solution (your local room temperature) (°C) **[G]**	The size of the biggest single crystal (range from 8-28) **[H]**	The shape of the biggest single crystal (range from T-Z) **[I]**

The collaborative best results for each sample for posting to the website

	Table Salt	Sugar	Epsom Salts	Alum	Potassium Nitrate
Temperature of the remaining solution (your local room temperature) (°C) **[G]**					
The size of the biggest single crystal (range from 8-28) **[H]**					
The shape of the biggest single crystal (range from T-Z) **[I]**					

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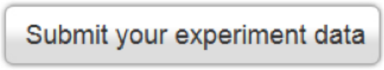
Part C: Sharing your data – Post your results to our global interactive map

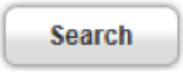
Post all your data and pictures to our Global Experiment website: http://rsc.li/ge2014

1) **Follow the link to post your data and click 'Submit your experiment data'**

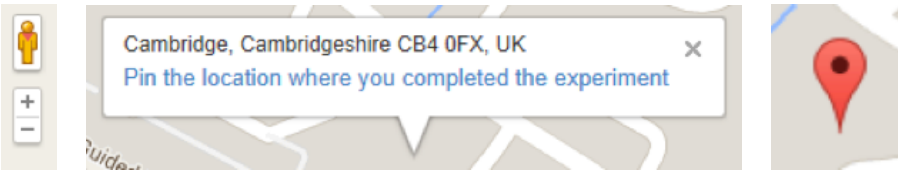


2) **Location Specifics:** Enter a Name, valid Email address and 'class or team name'

3) **Location Details:** Enter a country and post/zip code and click 'search'



Use the interactive map to find your location. Once found, click on the map to 'pin the location'



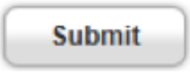
Then click on the blue text 'Pin the location where you completed the experiment'.
This will add a 'red marker' to your location

4) **Experiment Data:** You can now enter all your experiment data on the table provided

(If you have not completed all of the samples just enter what you have)

5) **Finally, upload your experiment images to feature on our website**

You can also tweet about the Global Experiment using #globalexperiment



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Part D: Analysing the website to discover the best conditions

Analysis on the website

After completing the experiment and posting your data why not analyse the data available to identify trends?

The Global Experiment website offers an interactive map so you can search and find specific data, just by clicking on the pins. You can also access all of the data by using the 'Export data' button, which provides a spreadsheet.

The graphs on the website provide the weighted average data.

- **Where was the biggest crystal grown?**
- **What were the conditions? (Temperature)**

Other useful research

There are many websites which provide local data on tap water - (below are some examples)

http://www.ecowater.co.uk/why-a-water-softener/test-your-water-water-in-the-uk/post-code-checker/ (UK)

http://www.ewg.org/tap-water/whats-in-yourwater.php (US)

A UK example would be to compare the results between London and Edinburgh. In London the tap water is 'hard' (the water contains a high concentration of calcium and magnesium mineral salts) but in Edinburgh the water is 'soft'. Are the results between London and Edinburgh very different?

Extension activities

Try conducting your own research.

The hardness/softness, pH, salinity (saltiness) and additives (fluoride) in tap water varies around the world.

Can you find out anything about the local water at the location of the biggest crystal data?

What are the environmental considerations for this location?

- **Humidity (could high levels of humidity affect the growth of crystals?)**
- **Dustiness of the air quality (could a dusty room grow bigger crystals?)**

Teacher/Technician notes

The experiment has been designed as a class practical for age range 7-14. You will need to assess the timings of this experiment to fit the experience of your students.

You can use the data on the Global Experiment website to compare data from students around the world to discover something new (errors should be discussed as part of this).

It is recommended that you use all five samples when taking part in the experiment but you can take part and submit data on less.

Also, it is best to post data from Part A and B at the same time (after a week of crystal growth) but it is possible to submit Part A and B separately if you would like.

The experiments

500 g-1 kg bags of each sample will be plenty for a class experiment.

You do not need to be very accurate for this experiment; it can be done using kitchen scales and has been designed so you can take part at home or in school.

Part A: additional ideas

Dissolving and saturating your samples: With your class you could number half a cup of each of the samples and – in pairs – the students could try to identify their unknown sample by comparing their results to the Global Experiment results page (http://rsc.li/ge2014).

If you run the experiment this way, please ensure you know what the samples really are so you can post your data onto our website.

Part A: Questions and Answers

Q: What is the definition of a crystal?

A: A crystalline solid is made up of atoms or molecules which are arranged in a repeating pattern and stacked over and over again.

Q: In future, could you tell the difference between the samples if they were not labelled?

A: Yes, they all have different properties.

Q: If an unknown sample was used to prepare a saturated solution with average properties [E] 8 °C and [F] 9.5 g which of the five samples do you think it could be?

A: Potassium nitrate.

Part B: additional help with crystal growing

Not all of the samples grow crystals equally well so here are some tips to help.

1) Run a few in parallel
2) Sugar crystals are the most difficult to grow
3) If after a day no crystals appear, add a few grains of the same solid sample to encourage crystal growth (when we tried this for sugar it worked well – this is called 'seeding')

Part B: coloured crystals

At the end of step 6 you could add a few drops of food colouring (or the ink from a highlighter pen) to grow coloured crystals. We don't know what effect this will have on the crystal growth for this experiment but it certainly makes it more fun!

Part B: instant crystals

At the end of step 7 you can rapidly cool the disposable cups containing the solutions in icy water which – for some of the samples – causes instant crystals. This fast crystallisation will not give very big crystals but this difference in crystal size can be discussed with the class once the bigger crystals have developed after a week.

Part B: Questions and Answers

Q: Do you think the amount of sample that dissolves in hot water will be different from cold? Why?

A: Yes, because in hot water the particles move about more and so more can dissolve.

Q: Can you predict which sample will grow the biggest crystal?

A: Just a prediction – but students might feel that sugar and Epsom salts might grow the biggest crystals because these dissolve the most in water (the experiment will reveal the real answer).

Make full use of **Part D: Analysing the website to discover the best conditions.**

Standard health and safety guidance for schools

Health and safety in practical chemistry in schools and colleges affects all concerned: teachers, lecturers and technicians, employers and students, parents or guardians, as well as authors and publishers.

These guidelines refer to procedures in the United Kingdom. If you are working in another country you may need to make alternative provisions.

As part of the reviewing process, the Global Experiment has been checked for health and safety.

We have attempted to ensure that:

- All common recognised hazards have been identified
- Suitable precautions are suggested

It is assumed that:

- Safe work practices are followed when chemicals are handled
- Eye protection is worn whenever risk assessments require it
- Great care and adult supervision is required when transferring hot water
- When carried out in schools - practical work is conducted with teacher supervision and in a properly equipped area
- When carried out in schools - mains-operated equipment is regularly inspected, properly maintained and appropriate records are kept
- When carried out in schools - first aid facilities and a trained first aider are available

Teachers' and their employers' responsibilities

Under the COSHH Regulations, the Management of Health and Safety at Work Regulations, and other regulations, UK employers are responsible for making a risk assessment before hazardous procedures are undertaken or hazardous chemicals and materials are used. Teachers are required to co-operate with their employers by complying with such risk assessments. However, teachers should be aware that mistakes can be made and, in any case, different employers adopt different standards.

Reference material

Model risk assessments have been taken from, or are compatible with:

CLEAPSS Hazcards (see **CLEAPSS website**)
CLEAPSS Laboratory handbook (see **CLEAPSS website**)
CLEAPSS Recipe cards (see **CLEAPSS website**)
ASE Revised Topics in Safety: key updated revisions are in the website (see **ASE website**)

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