PROJECT M

**1000 samples,
100 schools,
one great big experiment**

Teacher guide

The experiment should take place across two 45 minute lessons, at least a week apart. Students can work in groups or individually. There are enough resources in the pack for 10 groups. We would recommend a maximum group size of 3 students.

We hope to publish the results of this work in a scientific journal, with all the participants listed as authors. In order to do this we need a clear record of the full names of all the participating students and teachers. If you wish to be included as an author on the paper it is important that you email us a list of all participants and the full school address when you complete the experiment. Please refer to term 13 in the terms and conditions, http://www.diamond.ac.uk/ProjectM/Terms-and-Conditions.html

---

Lesson 1 – Producing Calcium Carbonate – 45 minutes

In this first practical, the students will be synthesising calcium carbonate from calcium chloride dihydrate and sodium carbonate. Each group has an individual sample sheet which lists the additive and water they should use – your school will all use the same additive but each group has a different mass of the additive. One group will be making a control sample without an additive and one group should use tap water instead of distilled or deionised water.

Accompanying resources:

- **Equipment sheet:** Items for day one. All the equipment needed for the first part of the practical is listed here.
- **Method sheet:** Calcium Carbonate Synthesis. The method gives a step-by-step guide to the experiment which the students should be able to follow
- **Video:** 'Producing Calcium Carbonate' (15 minutes). In this video, Dr Claire Murray and Dr Julia Parker demonstrate the experiment. If there's enough lesson time, this can be watched either before the practical or along with the lesson
- **Student Worksheet:** Questions 1 – 7 are practical questions which must be answered during the experiment and uploaded onto our website afterwards. Questions 8 – 14 are optional for after the practical.

Lesson 2 – Loading Your Sample – 45 minutes

In the second practical, the students will load their calcium carbonate samples into Kapton capillary tubes. It's really important that there are no gaps in the capillary tubes, as this will impact the quality of the data we collect. Two Kapton capillaries should be filled with each sample. Once filled, the barcodes should be used to label the samples. Please make sure that the samples are labeled correctly – we will not be able to use them if they are mislabeled. Please see instructions below on how to label your sample and send it back to Diamond. The remaining powder sample should be put into a vial, securely sealed and the vial placed into the large resealable bag along with the small resealable bag containing the two capillaries and the completed worksheet.

PROJECT M

**1000 samples,
100 schools,
one great big experiment**

Accompanying resources:

- **Equipment sheet:** Items for day two. All the equipment needed for the second part of the practical is listed here.
- **Method sheet:** Loading your sample. The method gives a step-by-step guide to the experiment which the students should be able to follow
- **Video 2:** 'Loading Your Sample' (5 minutes). Claire and Julia demonstrate how to load your calcium carbonate into the Kapton capillary tubes.

Additional resources

- **Calcium carbonate fact sheet:** Background information and fun facts about calcium carbonate, may be suitable for younger students
- **Video 3:** 'Project M & Diamond Light Source' discusses the science behind the project, gives an overview of Diamond and shows how the samples will be analysed on Beamline 11. This will be available on the website.
- **Teacher overview:** similar scope to video 3. May be useful in leading further discussions with the students and can be distributed to them as necessary.

Uploading your answers to our system

Each group will have a unique sample ID, sample number and password. They can log into www.diamond.ac.uk/ProjectM/Results and upload the answers from their worksheets to our system. Teachers should have sample IDs, sample numbers and passwords for all of their schools' samples. Please check the sample information has been uploaded correctly. Please also keep hold of these sheets as you will need this information to login and view your data after we have run the experiment.

Packaging and labeling your sample

Each group should have two capillaries in a small plastic bag, correctly labeled with their barcode sticker. The sample vial containing the rest of the product and labeled with the sample number (e.g. 20-1) should also be returned, in case it is needed for further analysis. All of these items should be placed together with the worksheet into a larger plastic resealable bag. All ten plastic bags (one per sample) should be sent back, along with the safety information sheets for the product.

In total, you should be sending 10 worksheets, 10 sample vials and 20 capillaries to Diamond – two Kapton capillaries of each sample per small labeled bag. Please use the stamped addressed envelope provided. If you and your students would like to be included on the paper, please email us a full list of participants.

All samples should be returned to us by 20th March. If you have any questions, please let us know on **projectm@diamond.ac.uk** diamond

PROJECT M

1000 samples,
100 schools,
one great big experiment

Equipment list – per group

Day 1

Provided in the pack

Calcium chloride dihydrate
Sodium carbonate
Funnel
Filter paper
Additive (if using)
Worksheet

Additional

Access to a balance minimum 2 decimal places
3 x weighboat
2 x conical flasks 100 cm^3
Measuring cylinder 25 cm^3
Deionised water wash bottle
2 x 100 cm^3 volumetric flasks (with stoppers)
2 x pipettes
2 x 500 cm^3 conical flask
Timer
50 cm^3 measuring cylinder
Petri dish
Thermometer

Day 2

Provided in the pack

Kapton capillary tubes
Vials
Glue (cyanoacrylate)
Toothbrush/ Tweezers
Small resealable plastic bag
Large resealable plastic bag
Worksheet
Barcodes

Additional

Pestle and mortar (if possible)
Ruler
Small piece of blutac
Scissors
Spatula
Suitable container for glue, e.g. weighboat

diamond

PROJECT M

1000 samples,
100 schools,
one great big experiment

Project M - 1000 samples, 100 schools, one great big experiment

Overview of Project M

Project M was devised by Dr Claire Murray and Dr Julia Parker, beamline scientists at Diamond Light Source. Claire and Julia use X-rays to look at the structure of different materials, a technique known as X-ray crystallography. Researchers working on crystallography have been awarded the Nobel Prize a total of 29 times – it's considered a very important field.

Project M involves students from 100 schools across the county who will synthesise a total of 1,000 samples of calcium carbonate, made with specially selected additivities (including amino acids). These will then be packaged up and sent back to Julia and Claire at Diamond to analyse in their laboratory, beamline I11. They want to analyse all of the samples in a 24 hour period, which will also test the capability of their beamline.

Calcium carbonate has three arrangements of atoms in crystalline form, known as polymorphs: calcite, aragonite and vaterite. Amino acids, as part of proteins, are known to produce a biological scaffold which influences the formation of the different polymorphs. Different amino acids can direct the crystallisation of the different polymorphs, but we don't understand which amino acids produce which polymorph or how. This is what we hope to find out from Project M.

What do we expect?

We are expecting to see a mix of vaterite and calcite as aragonite is usually only formed at high temperatures, or under high pressure. Once we have completed the analysis on Beamline I11, the students can log into our website, see the diffraction pattern from their individual sample and work out which of the polymorphs are in their sample. From this, we can determine the influence of each additive on the crystal structure.

What is Diamond?

Diamond Light Source is the UK's synchrotron, located in Oxfordshire. Diamond works like a super microscope, using the power of electrons to produce intense beams of light which are used to investigate the structure and properties of a variety of materials. Diamond is used by researchers from all sorts of disciplines - archaeology to engineering to chemistry.

Thousands of exciting discoveries have been made at Diamond. In 2007, researchers from Cardiff University used the synchrotron to discover hidden content of ancient documents. By illuminating the documents, but not opening them, they could penetrate the layers of parchment to see inside. In 2010, scientists from Imperial College London used the synchrotron to discover how HIV infects human cells – these findings will inform new treatments.

How does the synchrotron work?

Electrons are fired from the electron gun into the machine, where they are sped up through a series of three particle accelerators, called the linac, the booster and the storage ring. During this, the electrons reach almost light speed. Once they enter the storage ring, the electrons are moving so fast that they could travel around the entire world 7.5 times in a single second. The storage ring is what gives Diamond its donut shape. But it's not actually a circle; it's a 561.6 m polygon, which contains bending magnets at the corners. As the electrons pass through the magnets, they suddenly change direction which causes them to emit a bright beam of light. The wavelengths of light range from X-ray to far infrared, and these are used in the experiments.

Diffraction

Many materials in the world around us are crystalline. Crystals are made up of regular arrays of atoms. At Diamond, we use our x-ray beam to carry out many diffraction experiments in crystals. These allow us to work out where the atoms are - known as the crystal structure. Bragg's law defines the relationship between the wavelength of the X-ray beam we are using and how it diffracts off the planes of atoms in the crystal.

But what if we don't have just one crystal and instead have many tiny, randomly oriented crystallites? These materials are powders and the pattern of one of these tiny crystallites is too small for us to see, but together they produce diffraction rings. By taking a slice down through the rings in any direction, we can get a diffraction pattern. The diffraction pattern peaks tell us about the 3D size and shape of the lattice and intensities of the peaks tell us where atoms are located in the 3D lattice.

We'll be using X-ray diffraction in Project M to work out the structure of the calcium carbonate and determine which polymorph has been produced.

Loading samples on to the beamline and results

It's important that all of the individual samples of calcium carbonate are barcoded so we can keep track of them – we have 1000 to deal with! We use a brass pin to load each samples onto the carousel. The carousel can hold 300 samples, which means we'll have to load it four times during the 24 hours to run all of the samples. The robot picks up each sample individually and places it at the centre of the diffractometer, a large circular instrument. Detectors around the sample identify the diffraction pattern. When we look at each pattern, the different polymorphs of calcium carbonate will be identified by looking for peaks in specific regions. This will help us work out the impact of the different amino acids and other additives that you are using.

Method - Calcium Carbonate Synthesis

Before you start

Make sure that you have reviewed your risk assessments.

Have your Student Worksheet ready to fill in.

Keep your worksheet safe as you will need this to upload your information to the website and to send back to us.

Please use deionised or tap water according to your individual instructions. You can substitute distilled water for deionised if deionised isn't available, but please remember to report this on the website.

Check that you have all of your equipment ready to go, you will need:

Day 1

- Balance
- 3x weighboats (minimum of 2 decimal places)
- 2x 100 cm^3 conical flasks
- 25 cm^3 measuring cylinder
- Deionised water wash bottle
- 2x 100 cm^3 volumetric flasks
- Funnel
- 2x pipettes
- 2x 500 cm^3 conical flask
- Filter paper
- 50 cm^3 measuring cylinder
- Petri dish
- Calcium chloride dihydrate
- Sodium carbonate
- Additive (if using)
- Timer
- Thermometer

Synthesis of your sample

1. Use a thermometer to record the air temperature and note it on your worksheet.

2. Weigh calcium chloride dihydrate ($CaCl_2.2H_2O$, 1.47 g).

a. Place the weighboat onto the balance, and tare – set to zero.

b. Using the weighboat, measure out as close to 1.47 g of $CaCl_2.H_2O$ as you can. Please record the exact weight on your worksheet.

3. Using a new weighboat, measure out as close to 1.06 g of sodium carbonate (Na_2CO_3) as you can. Please record the exact weight on your worksheet.

4. Preparing a volumetric solution.

a. Into a clean conical flask, dissolve your sodium carbonate sample with ≈25 cm^3 of deionised or tap water. Swirl until the solute if fully dissolved. Please record which water you used on your worksheet.

b. Using a funnel, transfer this solution into a 100 cm^3 volumetric flask.

c. Wash out the conical flask by swirling ≈5 cm^3 of deionised or tap water into the volumetric flask. Repeat once, then rinse your funnel into the flask to ensure that all washings are collected with your solution.

d. Finally, use a pipette to fill your volumetric flask up to the meniscus line.

e. Holding on tightly to the lid, invert and shake the flask for approximately 1 minute.

5. Repeat step 3, using your calcium chloride dihydrate in place of sodium carbonate. If you are using an additive, weigh it out and add it now to the calcium chloride dihydrate solution, according to the instructions

6. Mixing the two solutions.

a. Prepare your filter paper. Fold it in half, then half again. Take one of the four sides and pull it back to make a cone. Weigh the filter paper, then place into the funnel on a clean conical flask.

b. Set your timer for 2 minutes. Pour both prepared solutions into a 500 cm^3 conical flask. Swirl the conical flask for 2 minutes.

c. Filter the contents into the clean conical flask using the filter paper and funnel.

d. Wash the used conical flask with 50 cm^3 of deionised water, and filter the washings into the second conical flask using the filter paper. NB. Regularly swirl the flask as you wash it out to ensure that all of the sediment is removed.

e. Through the funnel, slowly add 50 cm^3 acetone.

7. Carefully, unwrap the filter paper and leave to dry for at least a week in a petri dish, if possible with a lid. It will help to put it somewhere warm, like a window or by a radiator.

Method - Loading your capillary tubes

Day 2

- Pestle and mortar (if possible)
- Ruler
- Small piece of blutac
- Capillary tube
- Glue (cyanoacrylate)
- Scissors
- Spatula
- Toothbrush/ Tweezers
- Vials
- Small resealable plastic bag
- Large resealable plastic bag
- Barcodes
- Suitable container for glue, e.g. weighboat

8. Loading your capillaries

a. Measure and cut your capillary to 50 mm in length.

b. Place a small dab of glue into a suitable container, e.g. weighboat, and dab one end of the capillary into the glue to seal one end. Allow the capillary to dry by resting it on a piece of blutac.

c. If you have a pestle and mortar, grind up your sample using a pestle and mortar. This may take a couple of minutes.

d. Carefully, scoop up the powder into the capillary. Pack it in tightly to make sure that the capillary is full, and that there are no air gaps. It may help to vibrate the capillary tube along a toothbrush, tweezer edge or to tap the tube with the end of a spatula.

e. Once full, seal the second end.

f. You should now have a filled 50 mm long capillary tube, without any air gaps, and sealed with glue on both ends.

g. Repeat a-f to fill a second capillary.

h. These two capillaries should be placed in the small resealable plastic bag and labeled with your barcode.

i. Load the remainder of your powder into the vial and close by screwing the lid on tightly. Label the vial with your sample number, e.g. 20-2.

j. Ensure all of the information for your experiment is recorded on the worksheet you have sent us and is also uploaded to the website www.diamond.ac.uk/ProjectM. Remember to keep the sheet with your sample ID and password safe as you will need this information to log back into the website to see your diffraction pattern when we collect it.

k. Place the small resealable plastic bag barcoded with your sample number, your worksheet and your sample vial into the large resealable plastic bag. Your teacher will send this back to Diamond together with your classmates' samples.

Student Worksheet

School name	
Sample ID	Sample Number

Practical questions – please fill out on the webpage and send this version back with your sample

1. What is the air temperature?	
2. How many grams of calcium chloride dihydrate did you use? Please use the reading from your scales.	
3. How many grams of sodium carbonate did you use? Please use the reading from your scales.	
4. Did you use deionised, distilled or tap water? ☐ Deionised ☐ Distilled ☐ Tap	
5. Which additive did you use? How much?	
6. What do you see when you mix the calcium chloride and sodium carbonate solutions?	
7. Additional Comments? (Any extra information that you think might be important for us to know!)	

Optional extra questions for after the practical
8. Calculate the number of moles in 1.47 g of calcium chloride dihydrate ($CaCl_2 . 2H_2O$)
9. Calculate the number of moles in 1.06 g of sodium carbonate (Na_2CO_3)
10. Why is it important to use deionised water when making up solutions in the lab?
11. What is the purpose of using a volumetric flask? How is it different to a conical flask?
12. Calculate the molar concentrations of both volumetric flasks
13. Give the equation for the reaction which occurs as the two solutions are mixed
14. Why might it be important to ensure that once the two solutions are mixed, the flask is swirled for 2 minutes?

PROJECT M

1000 samples,
100 schools,
one great big experiment

Calcium carbonate $CaCO_3$

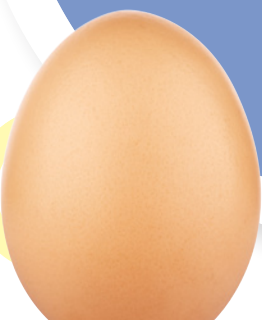
The earths crust contains more then 4% calcium carbonate, making it one of nature's most abundant raw materials.

What is it?

- Calcium carbonate is a chemical compound.
- It is made up of three elements: carbon, oxygen and calcium.
- There are three polymorphs (forms) of calcium carbonate: calcite, vaterite and aragonite.

Limestone is a type of rock, mainly composed of calcium carbonate. Limestone is quarried (dug out of the ground) and used as a building material. It is also used in the manufacture of cement, mortar and concrete.

Calcium carbonate is the main component of…



Did you know…?

The oldest building in the world is made of Calcium carbonate. It's called Khufu's Pyramid and can be found just outside Giza, Egypt.