Synthetic chem tour

**Synthetic chemistry lab tour – teacher notes**

# Introduction

A number of applied school science courses recommend that students should make a site visit to some aspect of the chemical industry. Such visits are often difficult to organise and some areas are almost impossible to get access to. Major constraints on organising visits to laboratories, pilot plants or production sites are time, space, safety and compliance with Good Manufacturing Practice. Visitors are prohibited in some areas when active large scale chemistry is underway and therefore planning visits weeks in advance becomes impracticable.

To partly address the needs of applied science courses and some A-level chemistry courses, virtual visits of the chemistry laboratories and pilot plant on a pharmaceutical company’s sites has been created. Although students can navigate round them at leisure, there may be benefits from them being teacher led using a projected image from the web site. Questions are also provided to enhance the learning experience.

## On-screen text from the virtual tour of a synthetic chemistry laboratory.

The text that appears in the info box on screen is reproduced here. Additional text is in italics. Questions and discussion points are in boxes within the text at appropriate places.

**Picture of general lab view**

The role of the medicinal chemists working in most of the synthetic chemistry labs is to convert “leads”, which are discovered in the early research phase, into “potential drugs” which can be developed into medicines to be taken by patients. A “lead” is a compound which has biological activity on an enzyme or receptor in a test tube or plastic multi-well plate. Medicinal chemists make related compounds that work at lower doses and that also work in whole mammals for a reasonable length of time with no harmful side effects. Chemists work in project teams focussed on a particular disease.

*The role of a medicinal chemist is primarily to synthesise new compounds. The structure of these new compounds is decided by analysing biological results from previous compounds and correlating them with their structures. It is a relatively rational approach but involves a considerable intellectual challenge. A good medicinal chemist needs theoretical and practical expertise in organic chemistry plus a good appreciation of the biological aspects of the project they are working on. To be able to design compounds that will have good absorption and duration in the body requires a thorough understanding of the physical chemistry involved and how metabolic processes work in the body.*

**Picture of fume cupboard**

All practical chemistry is carried out in fume cupboards. No chemistry is done in the open lab. This is a basic health and safety requirement to protect all chemists in the lab from hazardous chemicals and from solvent vapours. Most of the solvents that are used are flammable and therefore naked flames are banned. There are no Bunsen burners in our labs. This is a surprise to many people who see the Bunsen burner as an icon of chemistry. Electricity is used to heat chemical reactions using either hotplates for small reactions or mantles for larger flasks.

**Picture of chemist in lab coat and safety glasses**

When a chemist works at a fume cupboard a strong flow of air is sucked through it. A warning sounds if the front is opened too far for safety. The protection of chemist from the hazards of doing practical chemistry is very important. Safety spectacles and lab coats are always worn in the lab. Chemists who wear normal glasses have personalised safety specs made up for them. It is very unlikely for any chemical to get splashed into their eyes, however, for added safety, plastic bottles of dilute salt solution are available for washing contaminated eyes.

**Picture of computer model of ligand and enzyme**

It is very satisfying to make new chemicals that will work at very low concentrations at biological receptors or enzymes. It is an even greater challenge to design something which will be absorbed from the stomach or intestines after taking a tablet by mouth. The new compound normally needs to be long acting and not rapidly broken down by our livers. It also has to be safe and have no harmful side effects. Some of this design is done by simply drawing chemical structures on paper; some, like this compound blocking a flu virus enzyme, by using computer modelling.

**Picture of office area.**

Chemicals and lab coats, which may be contaminated with chemicals, are not allowed in the office area. The office is adjacent to the lab and is where the chemists work at computers to check the chemical literature and hazard data. They also discuss work with colleagues and write reports. There may be chemists from more than one project area with desks in here. There is a clear view into the lab for safety reasons so that any potential accident can be spotted. Heath and Safety are taken very seriously. There are training courses and the use of very hazardous materials needs authorisation by a senior chemist.

## Virtual tour

*The laboratory for this tour is partly split down the middle by a bank of fume cupboards. To be able to see all aspects of the lab, the panoramic photography was done in both sections – called Lab A and Lab B for convenience.*

**Synthetic chemistry Lab A**

**Fume cupboard A. Click another fume cupboard for more information.**

All practical chemistry is carried out in a fume cupboard. When a chemist is not working there, the front sash is closed to prevent the air conditioned lab air being lost to the outside environment. When the sash is open the airflow is 0.5 meters per second. The area of a fully open sash is about 1 m2 so about 0.5 m3 of air is drawn through per second. This stops the chemist from breathing in hazardous vapours from solvents and chemicals in use.

*With all the fume cupboards open, the whole of the lab air can be changed in about one minute.*

**Fume cupboard B. Click another fume cupboard for more information.**

All chemical reactions are carried out in fume cupboards – mostly in glass flasks or test tubes. A flask is being heated using a hotplate with an aluminium block on top of it. Blocks have special hollows in them to fit different flask sizes. Another flask is being cooled using a plastic bowl with ice in it. Nitrogen is being passed into both flasks via the blue tubes to prevent the chemicals reacting with oxygen or water vapour in normal air.

*Most reactions are stirred using magnetic stirrers (the blue or orange units in the fume cupboards). The chemist puts a bar magnet coated in Teflon® into the reaction and this is turned round and round by a second magnet attached to a motor in the base of the magnetic stirrer unit.*

*Reactions are regularly carried out under nitrogen because our supply is very dry and oxygen free. Commonly used reagents that are sensitive to water and or oxygen are: acid chlorides and anhydrides; transition metal catalysts (especially palladium complexes); organometallic reagents (Grignards, organolithium compounds). Students might suggest sodium and potassium.*

**Class discussion point:**

The nitrogen gas is obtained by evaporation from liquid nitrogen. Why would it be dry and free from oxygen?

Name some elements or chemicals which react with water and/or air.

**Fume cupboard C. Click another fume cupboard for more information.**

All practical chemistry is carried out in fume cupboards. Taps down each side supply services. One is for nitrogen. Two are connected to vacuum pumps that serve the whole building – traditional filter pumps using high pressure water are not used. Chilled water is pumped via a “flow” tap from a central tank, through a condenser and back through a “return” tap. This closed loop system saves water and vastly reduces the volume sent to waste.

**Waste bin and storage box for dry ice (solid carbon dioxide).**

All chemically contaminated materials such as filter papers; disposable gloves; plastic weighing boats; paper tissues and empty plastic bottles that contained chemicals are put in the red waste bin. This waste is incinerated on site to destroy all chemical contamination. The blue/green box stores dry ice at -78°C and is used to cool chemical reactions to slow down their rate. Generally, a 10° fall in temperature halves the reaction rate.

**Waste bin**

The waste bin (red) is used for all materials that might be chemically contaminated. These include filter papers; empty plastic bottle that chemicals were supplied in; disposable gloves; plastic weighing boats and paper tissues. All of this waste is incinerated on site to safely destroy all chemical contamination.

**Class discussion point:**

Why does incineration destroy hazardous chemicals?

What are the products of incinerating all of this waste material?

*The incinerator on site is run at a high temperature so that everything is burnt and so that no dioxins are formed. The exhaust products are water vapour, carbon dioxide and nitrogen (from nitrogen containing compounds). Students may be concerned about carbon dioxide emissions, but they are relatively low on a research site. Incineration is much more environmentally friendly than sending such materials to landfill. Chlorine in waste (e.g. PVC) ends up as hydrogen chloride which is absorbed by a sodium hydroxide “scrubber”. All solid particles in the exhaust are removed using electrostatic plates. All residual inert solid ash is sent for landfill.*

**Gloves**

Whenever a chemist is working with chemicals they wear disposable plastic gloves. They have good resistance to most chemicals. Once a sequence of operation is complete the gloves are removed, put in the waste bin and hands are washed. This approach to safe working is so important that a typical chemist would go through many pairs of gloves a day. Thick rubber gloves would be worn when very hazardous chemicals are in use.

**Vented cabinet and rotary evaporator. Click the cabinet for a view inside.**

The cabinet has a gentle air flow through it so that traces of solvent vapours do not get into the open lab. The rotary evaporator inside is used to get products out of solutions that are obtained from reactions. Solvent can be boiled off quickly at 30-60°C. By doing the evaporation under a partial vacuum the boiling point of the solvent is lowered. The process is speeded up by rotating the flask to produce a high surface area of solvent over the glass.

*The grey and blue unit to the left of the rotary evaporator is the pump which is connected to it. The pump has a solvent trap (grey cylinder – far left) attached to it on the exhaust side of the pump. The trap is therefore at atmospheric pressure and is cooled with solid carbon dioxide at –78º C so that all solvent vapours are condensed before they reach the atmosphere. This local system gives the chemist complete control of environmental protection and safety.*

**Class discussion point:**

What temperature does water boil at on the top of Mount Everest where the air pressure is significantly lower than at sea level?

*For interest*

|  |  |  |
| --- | --- | --- |
| ***Place*** | ***Altitude*** | ***Water boils at:*** |
| *London* | *sea level* | *100 °C* |
| *Denver, Colorado* | *1,625 m (1 mile)* | *95 °C* |
| *Quito, Ecuador* | *2,880 m* | *90 °C* |
| *Lhasa, Tibet* | *3,720 m* | *87 °C* |
| *Mt Everest (top)* | *8,920 m* | *71 °C* |
| *Dead Sea* | *- 400 m* | *101 °C* |

**Hand wash and safety station**

Chemists wear safety spectacles all the time in the lab – there are no exceptions. It is very unlikely for any chemical to get splashed into their eyes. However, for added safety, plastic bottles of dilute salt solution are available in the green box for washing contaminated eyes. There is also a bottle on the right hand pillar of each fume cupboard. Since chemists change gloves regularly the sink is important so they can wash their hands.

**Ultraviolet lamp housed in a closed box for safety**

Chromatography is an important technique for checking the progress of reactions. It is often encountered for separating coloured inks by running them up filter paper using water. It is easy to see that black ink spreads out into pink, blue, etc. and, being coloured, the eye can see the spots. In organic chemistry the components of the reaction are usually colourless (or white) so chromatography runs have to be put under UV light to visualise them.

The chromatography plates are usually coated with a layer of fine silica which includes a trace amount of a UV fluorescer. Most organic compounds absorb UV so they show up on the plates as dark spots.

**High performance liquid chromatography (HPLC).**

This stack of equipment is for HPLC. It is similar to paper chromatography that is encountered for separating coloured inks by running them up filter paper using water. In HPLC the mixture of chemicals from a reaction is injected onto one end of a steel tube packed with silica (SiO2) and separated by pushing solvent through the tube at high pressure. This is a very sensitive method for analysing chemical reactions and checking the purity of products.

**Radio / CD player**

This is a key piece of equipment and adds to the pleasant atmosphere of the lab. The fume cupboards and background equipment noise is fairly quiet so everything from Wimbledon commentary to grunge rock can be heard in labs. It all depends what the chemists fancy on the day.

**Lab note book**

It is really exciting for a chemist to make things for the very first time in their experiments. As the work is carried out, the chemists note down the quantities of reactants used, how the reaction was done and the analytical results on the products so that their work can be repeated. A full risk assessment is written before the experiment starts. The notebook is a legal record of what was invented when. Every page must be signed and dated.

Notebook pages are dated at the start of an experiment and signed and dated by the chemist concerned at the end of each experiment. They are then countersigned about a week later by a colleague from another team to ensure that all data is present. This provides a third “legal date stamp” for the experiment. New and inexperienced staff will have their notebook routinely checked by their supervisor to make sure that good risk assessments have being carried out and that all data is being recorded.

**Door to service corridor. Click to go through for a tour of safety signs.**

All the supplies of solvents and chemicals are delivered by stewards via a central service corridor in the building. Stewards also take away waste for incineration. Chemists use this route to move their chemical samples about too. The main pipes for nitrogen, vacuum and water services which go to all of the fume cupboards run down this corridor and are easily accessed by service engineers. Click on the door to visit the corridor and see safety signs.

Details of what can be seen on the photo montage tour are at the end of this document.

**Office area.**

Next to every laboratory is an office area where chemists have their desks and carry out on-line research on reactions and safety data etc. The project team will also use it for informal discussions. There is a clear view from the office desks into the lab which ensures that there is always safety cover even if there is only one chemist in the lab and another in the office. Chemicals and lab coats, which may be contaminated with chemicals, are not allowed in the office area.

**Vented weighing booth – back view; click to see the other side**

The booth contains a top pan balance and a narrow “curtain” of air flows across the front of the booth. This stops chemists inhaling dust and vapours. Chemicals must be weighed before they are used in reactions so that the reacting masses, derived from the reaction equation, are correct. The reaction product is also weighed to work out the chemical yield. Most reactions are done on a very small scale (0.01 gram) because that’s all that’s needed.

In the early research phase the medicinal chemists are looking for “needles in haystacks” i.e. the small number of compounds which have reasonable biological properties as opposed to the weak or inactive ones. Modern biological testing techniques are so sensitive that about 30 tests can be done on 3mg of stock material. That’s normally all a chemist needs to make.

To synthesise paracetamol a chemist reacts 4-aminophenol with ethanoic anhydride in a 1:1 ratio. Under the usual reaction conditions the amino group (-NH2) is more reactive than the phenol (-OH) and so reacts first. However if an excess of ethanoic anhydride is used, the phenolic group of the product might react to give a by-product as an impurity.

1. Using the molecular formulae shown, calculate the relative molecular masses.

2. If you weighed out 1g of 4-aminophenol, how much ethanoic anhydride do you need for a 1:1 reaction?

3. What is the maximum yield (in grams) of product that could be obtained?

4. If the actual yield of product is 0.8g, what is the percentage yield?

5. Assuming a perfect 1:1 reaction, what is the other product of the reaction and how much would be produced?



*The relative molecular masses are: 109, 102, 151*

*A mass of 0.936g of ethanoic anhydride is required.*

*The theoretical yield is 1.385 g*

*0.8g represents a 58% yield.*

*The other product is ethanoic acid; molecular mass = 60;*

*0.55g would be produced.*

**Automated chemistry robot – click for a closer view**

A robot is housed inside this vented cabinet. Automation is used to carry out repetitive tasks such as weighing sample bottles and adding accurate volumes of solutions to them. This means that the chemists can carry out many similar reactions at once without having to do the boring, but crucial, bits. Other equipment such as automated chromatography can be used to get the products pure from all of these reactions.

**Synthetic chemistry Lab B**

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*The catalyst is usually a derivative of palladium.*

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**Microwave oven**

No – not for snacks! Traditionally chemical reactions are heated using thermal energy to make them go faster. Generally a 10 degree rise in temperature doubles the reaction rate. The use of microwave energy is a very important technique in modern chemistry because it activates individual molecules in a reaction mixture much more than thermal energy. Reactions therefore go much faster due to the increased vibrational and kinetic energy of the molecules.

*Conversely, a 10°C fall in temperature halves the rate of a chemical reaction. That is why we store samples in fridges at 4°C. In a fridge would take 4 times longer to break down than if it were stored at a room temperature of 24°C. Students will appreciate that milk keeps for 4 days in a fridge and only one day on a window ledge at 24°C. The general rule about temperatures and rates is derived by putting typical values into the Arrhenius equation.*

*For a good review of microwave chemistry see Education in Chemistry, September 2002, vol 39(5), centre pages of InfoChem (pupil supplement).*

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**Vacuum oven**

Once a reaction is complete the chemist will usually have made the product as a powder or as crystals. The solid must be dried before it is sent to look for biological activity on isolated cells or on an enzyme. Residual solvent might interfere with the tests and give false results. The vacuum oven is very good at drying solids. The solid is gently heated and a vacuum is applied. Solvents boil at a lower temperature when the pressure is reduced.

When compounds made on a larger scale (typically 5g to 100g) they are dried in the vacuum oven. These compounds are used as intermediates in long reaction schemes and need to be free from solvents, otherwise the mass used in the next step may be weighed incorrectly, or may interfere in the next step.

**Vented weighing booth – back view; click to see the other side**

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Text for the photo montage tour of the corridor and teacher notes.

Students could be given the task of spotting all safety signs and safety features; identifying their meaning; explaining their positioning and commenting on their use where appropriate. A worksheet is provided.

Picture 1

This is the central corridor in the building which was designed for safety. All chemicals, solvents, waste and deliveries are taken through this corridor. On each side are labs, each with a “clean” office on the outer side of the building. This design ensures that “clean” people do not come into contact with chemicals unless they are wearing lab coats etc. The main pipes servicing the fume cupboards with nitrogen, vacuum and water can be seen running down this corridor.

Picture 2

This is the door to the lab opposite the one you have been visiting. There are 2 blue safety signs visible. What messages do they give? Next to the door is a holder for safety spectacles. There are also notices to advise visitors or night security staff of any atypical hazards in the lab.

Picture 3

This is the end of the corridor and the exit to the stair well. What does the green sign indicate? To the left of the door are three fire extinguishers.

Picture 4

There are three sorts of fire extinguishers located in the corridor. Two are labelled “Powder” and “Foam”. The label for the contents of the third one is not visible – what do you think it contains? (note the wide exit tube). Why is there a need for different types of extinguishers?

Picture 5

Just outside the door of the lab you have been visiting is the safety shower. What do you think it is for? There is no drain hole – why do you think that is? What happens to the water?

Picture 6

There are safety showers in all of the corridors. They have never been used in emergencies but are tested regularly.

Picture 7

The building was designed for safety. The central corridor has a number of fire proof doors along it to divide the big building into isolated zones so that the spread of fire is minimised.

Picture 8

Return to the door of the lab you have been visiting. Next to the door is a holder for safety spectacles.

Picture 9

Go back into the lab. Don’t forget to put your safety specs on!

**Task: Take a tour of a corridor outside one of the labs in a pharmaceutical company’s chemistry building.**

*In the Safety Signs tour there is a much larger photo montage tour of corridors and labs covering many safety features. There is also an extensive student worksheet and teacher support material.*

1. Locate as many safety warning signs and information signs as you can.

Fill in the table below for each sign you come across on your tour.

|  |  |
| --- | --- |
| **Description of the sign and/or wording** | **Comments about the meaning and where it is positioned** |
| **Eye protection must be worn** | On the lab door as a directive before entering lab |
| **Fire door keep shut** | The signs are hard to see but there is a very small blue circle on all doors - on doors to prevent fire spreading |
| Green fire exit **“running man”** sign | at main exits to indicate the escape route |
| Red **Fire extinguisher** location sign | close to the exit door so that there is always a safe escape route |
| **Emergency shower** information sign | it is in the corridor outside the lab so that any chemicals that are washed off do not contaminate the lab |
|  |  |

1. There are three sorts of fire extinguishers located in the corridor.
   1. The label for the contents of one is not visible – what do you think it contains? (note the wide exit tube).

**Answer**: carbon dioxide

* 1. Fill in the table below for each fire extinguisher

|  |  |  |
| --- | --- | --- |
| Extinguisher type | What type of fires is it good for? | What are the limitations? |
| Foam | Foam is for most materials on fire such a paper. | It is water based and can not be used for electrical fires or on anything that reacts with water – e.g. potassium. The foam cools the fire and restricts contact with air. This type of extinguisher is pressurised with either air or nitrogen to create the foam. |
| Dry powder | Dry powder is good for all types of fires. | It covers the fire with dry solid cooling it and preventing oxygen getting to it. It makes a mess but that is preferable to the result of a fire. It is the best all round type of extinguisher – especially in chemical laboratories |
| Carbon dioxide | Carbon dioxide stops oxygen getting to the flame. It can be used on most fires, being especially good for electrical and solvent fires but some chemicals will react with it. | It comes out of the extinguisher horn very fast and there can be a risk of spreading small burning objects around the area. |

1. You will see a safety shower.
   1. What do you think it is for?

**Answer**: It is for drenching a chemist with water in case of severe splashes from dangerous chemicals. It has never been used. A lab coat and any contaminated clothing would be thrown off before activating the shower.

* 1. There is no drain hole. Why not? What happens to the water?

**Answer**: The building is designed for safety. There are no holes through the floors which are solid slabs and are fire and water proof. Fire can spread from one floor to another through drain holes. A water flood can spread to the floor below through drains. It is therefore much safer to eliminate the drain hole and just mop up the water from the shower. The spread of water can be contained using absorbent in “sausages” round the area (similar to nappy liners).

1. There are holders for safety spectacles in the corridor but they are not easy to spot.
   1. Where are they located?

**Answer**: In plastic racks by the side of each lab door – usually under the white notice board

* 1. Why are they there?

**Answer**: So that visitors to the lab can pick up a pair before they go in.