

# Bristol Chemical Synthesis

*'A Doctoral Training Centre for a better future  
by education and training in the science of  
Chemical Synthesis'*



# People

- Prof. Kevin Booker Milburn (Director)
- Dr Eoghan McGarrigle (Course Manager)
- Mar Ruiz Molina (DTC Administrator)
- All Staff in the School
- Industry Partners (eg GSK, AstraZeneca, Pfizer, Novartis, Syngenta etc)

- 50 EPSRC funded 4-year PhD awards
- Intake of 10 students pa starting Oct.09
- Additional studentships from industry
- Unique training programme in first 6-months
- 3.5 year PhD research project in Chemical Synthesis
- Opportunity for industrial placement
- Unique 'Brainstorming' sessions for group problem solving
- Unique e-learning techniques experience

# Conventional PhD (DTA)

- 3 years + 3 months in duration
- Project and supervisor decided before the start of PhD
- Ideal for students who have clear view of the project they wish to study and the supervisor(s)



# 4-Year DTC Route

- 4-year in duration
- Project and supervisor *not* decided until 6-months *after* start of PhD
- First 6-months '*Postgraduate Advanced Chemical Techniques*' (PACT)
- Ideal for students who are interested in Chemical Synthesis and who would like to experience a broader range of synthesis techniques before embarking on a specific PhD project

# PACT

- Aim - to broaden and strengthen research technique before commencement of PhD project
- 6-months in duration
- Enable students to decide on specific PhD project
- Assessment by DTC Management - write-up, course test and 'Health Check' (oral examination)→ formal progression to PhD project

# PACT- Structure

- Postgraduate DLM enabled experiments
- Research Broadening Sabbaticals (RBS)
- Lecture Courses
- Brainstorming Sessions
- 'Director's Cut' Problem Sessions
- Journal Club

# DLM-aims

- Will enable mastery of important synthesis enabling/supporting techniques
- Real versions of experiments then made available for students to experience in research labs
- All DTC students will be provided with a quality laptop to carry out DLM and related activity

5. Bragg's Law simulator - Mozilla Firefox

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[http://synthesis.chm.bris.ac.uk/dlm/file.php/4/Experiment\\_and\\_theory/2. Experiment and Theory v4/page\\_07.](http://synthesis.chm.bris.ac.uk/dlm/file.php/4/Experiment_and_theory/2.Experiment_and_Theory_v4/page_07)
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5. Bragg's Law simulator

### Table of Contents

- A. 1. Experiment Video
- B. 2. Unit cells
  - 1. Lattice systems
  - 2. Bravais Lattices
- C. 3. Symmetry operations
- D. 4. Lattice planes
- E. 5. Bragg's Law simulator
- F. 6. Systematic absences

## 5. Bragg's Law simulator

Diffraction by crystals is explained by Bragg's law. A number of different planes are considered to run through the crystal lattice. Constructive interference caused by diffraction from adjacent planes occurs when the extra distance travelled of one wave with respect to another is equal to a whole number of wavelengths. This can be explored using the simulator below. By adjusting the angle of incidence and the crystal plane under investigation, the extra distance travelled can be manipulated until constructive interference occurs.

### Instructions

**Bragg's Law:**  $n\lambda = 2d \sin(\theta)$

**d spacing = 3.2 Å**

**$l = 1.78$**

Number of extra wavelengths travelled      Extra distance travelled

11.4°

**Info**

**X-ray:**

**Mo** **Cu**

Find:
Next Previous Highlight all Match case

Done

Crystal growth flowchart - Mozilla Firefox

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Crystal growth flowchart

# Crystal growth

## Table of Contents

- A. Crystal growth flowchart
  - 1. What does a suitable crystal look like?
- B. Recrystallisation
- C. Sublimation
- D. H-Tube methods
  - 1. H Tube method 2
- E. Layering
- F. Vapour Diffusion
- G. Annealing

## Crystal growth flowchart

The nature of your sample will determine what techniques for growing crystals are appropriate. This flowchart demonstrates the options available.

It may be worth setting up different techniques at the same time so that you don't wait significant amounts of time for one method to not produce any crystals.

Stable to heat?

```

graph TD
    Start([Stable to heat? Yes]) --> Q1{Does sample recrystallise from a hot saturated solution.}
    Q1 -- Yes --> A1[Test crystals]
    Q1 -- No --> Q2{Cool more slowly - crystals?}
    Q2 -- Yes --> A1
    Q2 -- No --> Q3{Try a different solvent}
    Q3 --> Q1
    Q3 --> Q4{Try sublimation - crystals?}
    Q3 --> Q5{Try a layering solution - crystals?}
    Q3 --> Q6{Vapour diffusion - crystals?}
    Q3 --> Q7{Anneal solution - crystals?}
    Q3 --> Q8{H Tube - crystals?}
    Q4 -- Yes --> A1
    Q4 -- No --> A2[Try growing crystals of a salt of your sample e.g. NH3+Br- or triflate.]
    Q5 -- Yes --> A1
    Q5 -- No --> A2
    Q6 -- Yes --> A1
    Q6 -- No --> A2
    Q7 -- Yes --> A1
    Q7 -- No --> A2
    Q8 -- Yes --> A1
    Q8 -- No --> A3[Leave sample in solution for extended period of time.]
    
```

Done

Annealing - Mozilla Firefox

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## Crystal growth

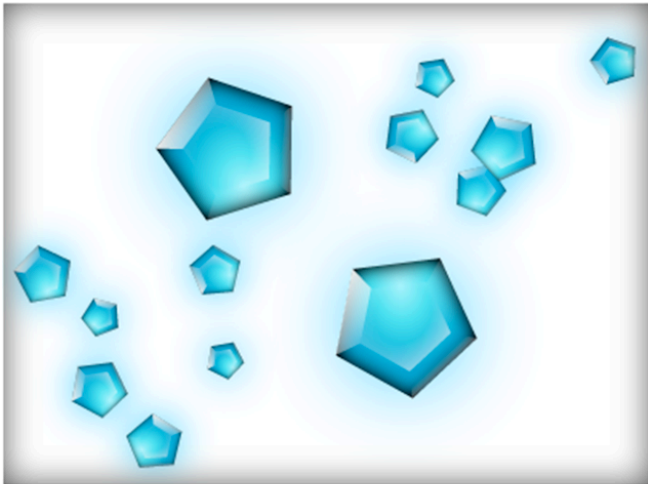
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- A. Crystal growth flowchart
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- G. **Annealing**

## Annealing

Annealing is not appropriate for thermally unstable samples.

Crystals can be grown using the process of annealing. There may be a spread of crystal sizes in solution. The small crystals may be dissolved by gently heating the solution leaving the larger crystals. By cooling slowly, the larger crystals act as nucleation sites and increase in size. By repeating the process, larger crystals of a suitable size can be grown.



**Stage 1**

Heat solution gently dissolve the smaller crystals.

Prolonged heating is to be avoided as this will dissolve all the sample

Heat

Done

Sublimation - Mozilla Firefox

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# Crystal growth

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## Sublimation

Sublimation is not suitable for thermally unstable samples.

Sublimation works by heating a sample under vacuum until it vapourises. The vapour is deposited on a cold finger and crystals can grow over time.

### Sublimation

Step 1

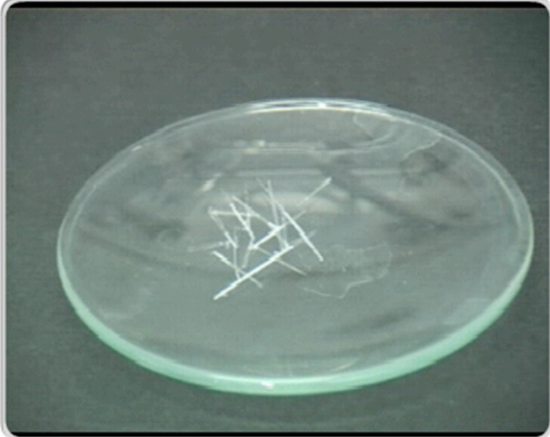
Step 2

Step 3

Step 4

Step 5

#### Step 5



00:09 / 00:12

Needle crystals have formed on the cold finger. Some have also grown so large that they have fallen off in the flask.

Done



# RBS-aims (3-days per week)

- 3 x 6-week residencies in a research lab
- Aims to broaden research technique beyond that of undergraduate experience
- Gain experience of three different research groups and supervision philosophies

# Brainstorming (2h)

- Each session will look at a particular PhD project on offer (from Research Portfolio)
- Enable students to consider all PhD projects on offer
- Exposure to full range of Chemical Synthesis research in School
- Enable students to work as a group to solve problems
- Enable students to come to an informed decision on what PhD project to start in month-7

# Directors Cut Problems

- On a Monday morning Director will place a PhD viva quality synthesis problem on the board in the Research Hub
- Students to solve this problem as a group by Friday
- Students present solution to Director on Friday
- Issues arising or gaps in groups knowledge will be dealt with by staff providing a short workshop on the issue (positive feedback loop)

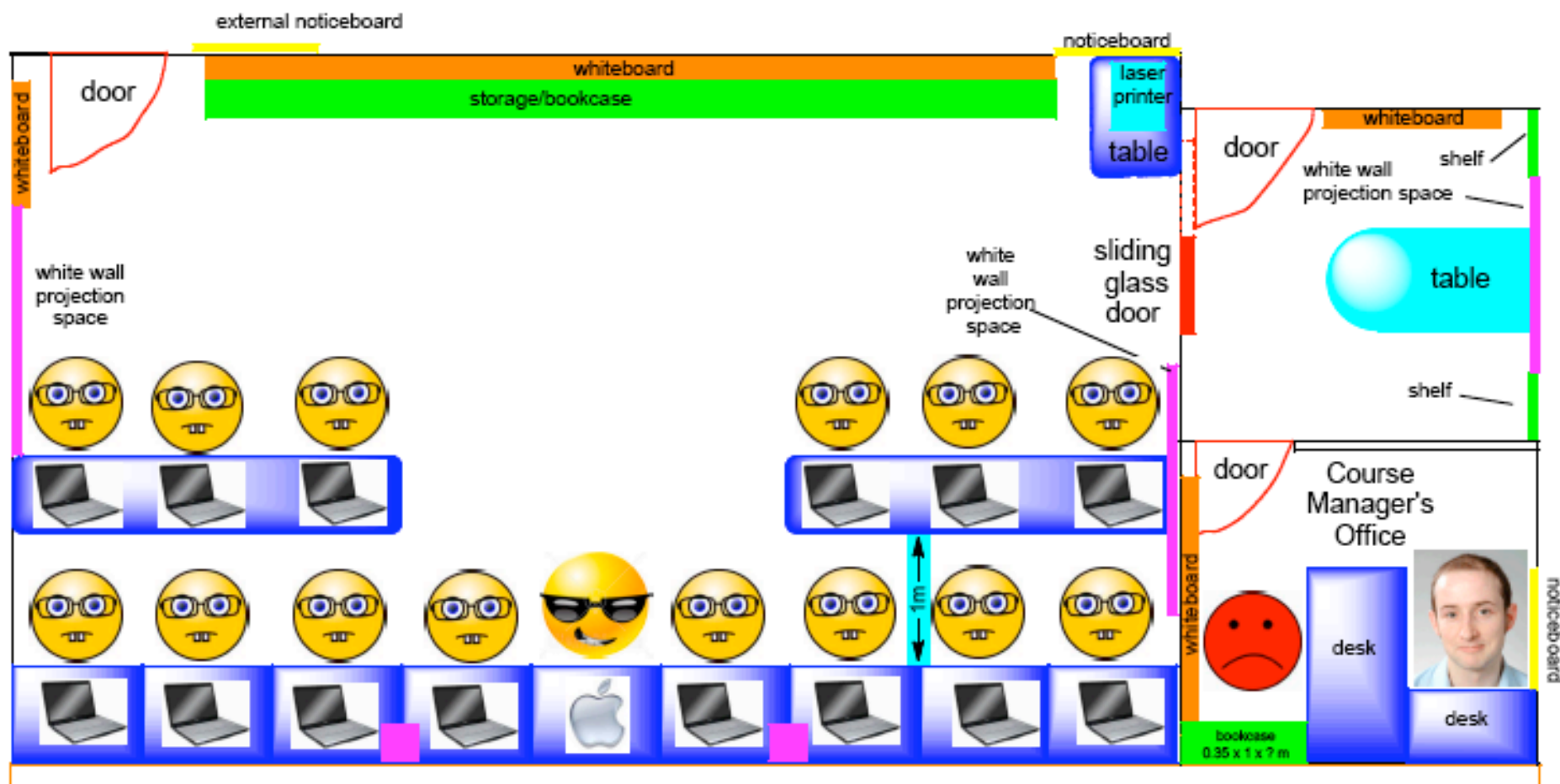
# Years 2-4

- Phd Project
- Industrial lectures and workshops
- IP & Patent Law course
- Industrial Interview Technique course
- Staff Problem sessions
- Cohort led Brainstorming sessions
- 1st Year Cohort Mentoring
- Industrial Placements
- Cohort Designed DLM experiments

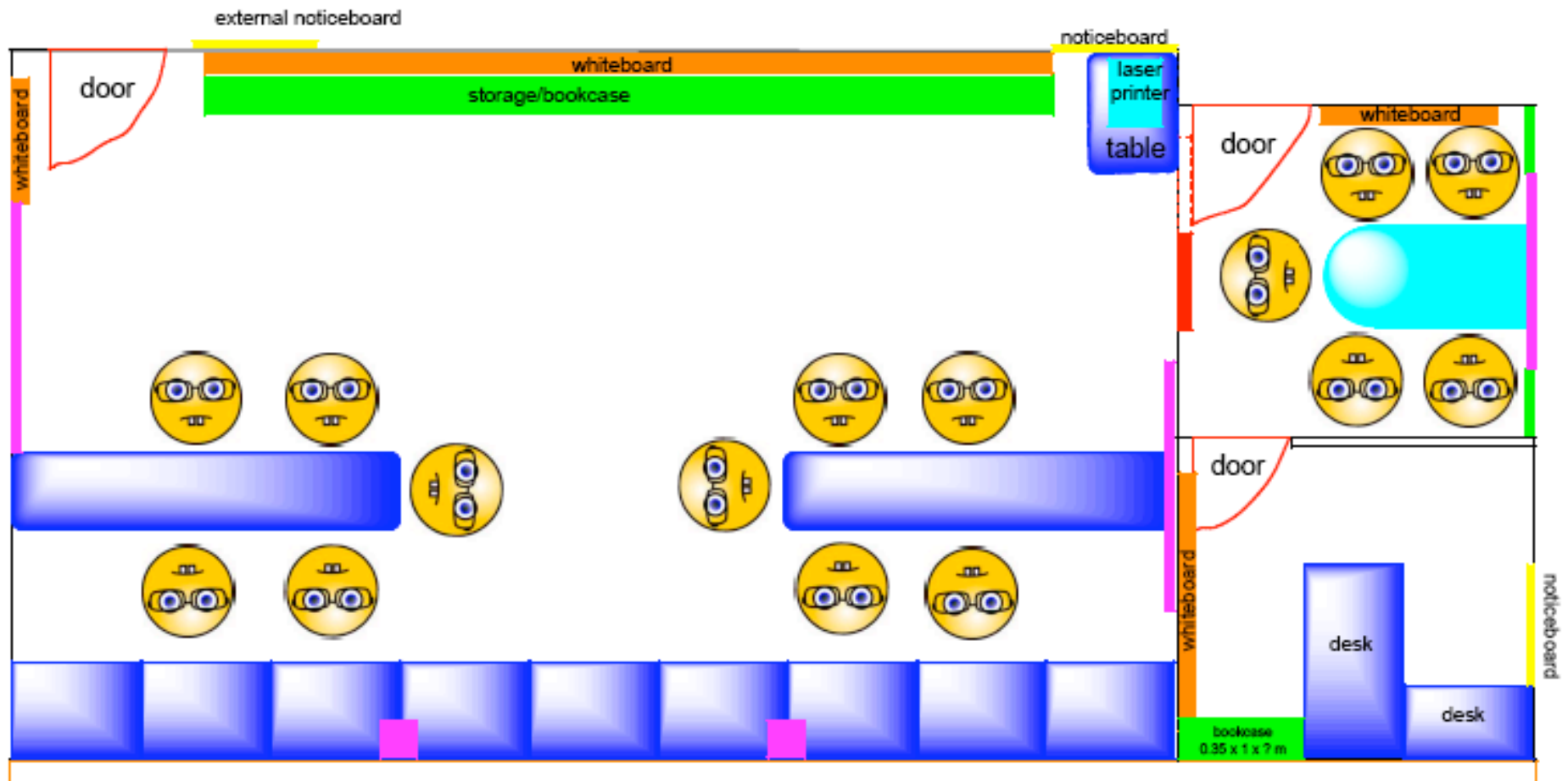
# Research Hub

- Purpose built space for all PACT activities in the first year
- Brainstorming wall
- Full AV equipment for presentations and lectures
- Printers, laptops, filing facilities
- Meeting room for all DLM activity in 1st and subsequent years

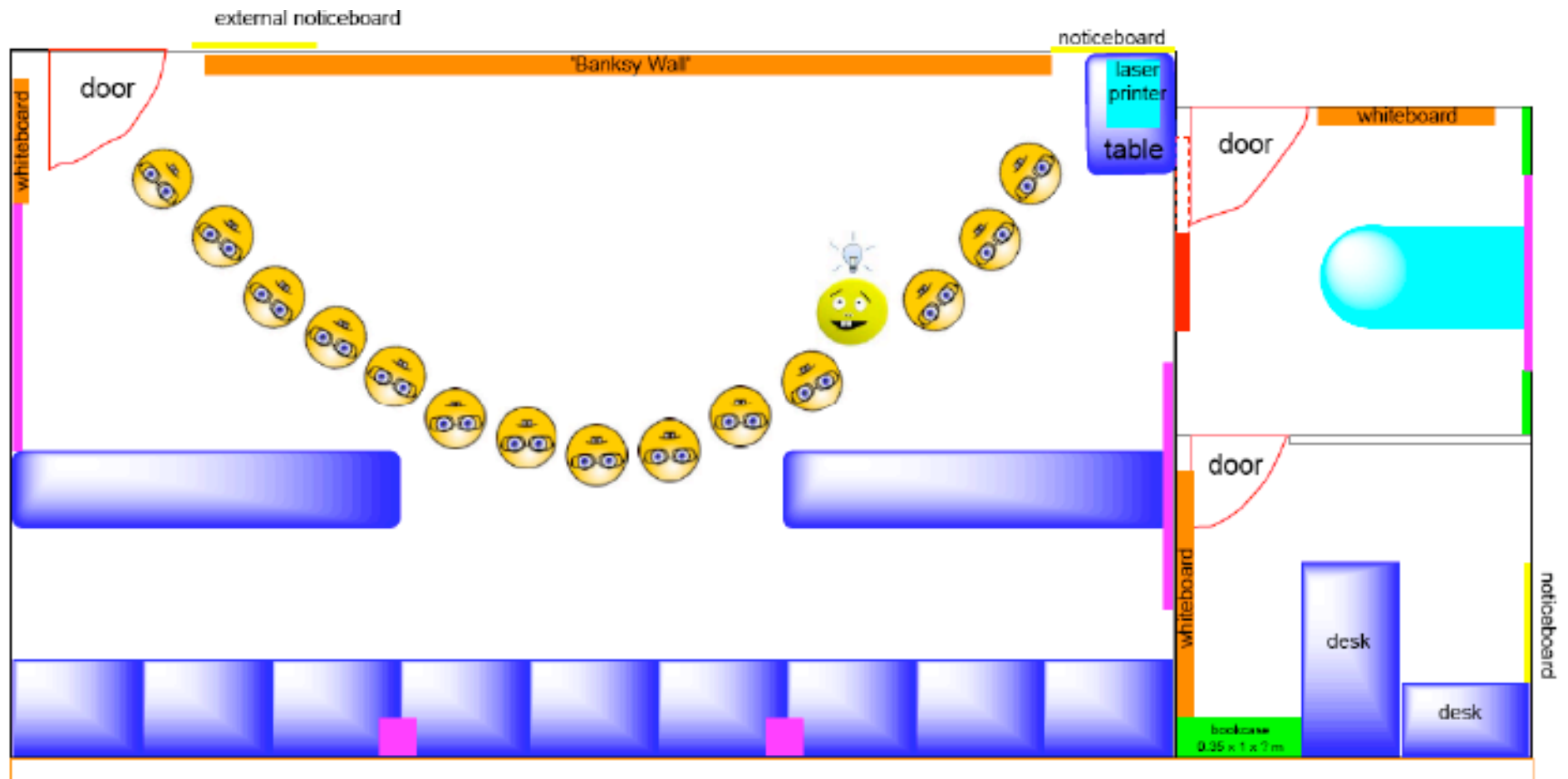
# The Bristol Chemical Synthesis DTC Research Hub



# The Research Hub - problem/sub-group discussion mode

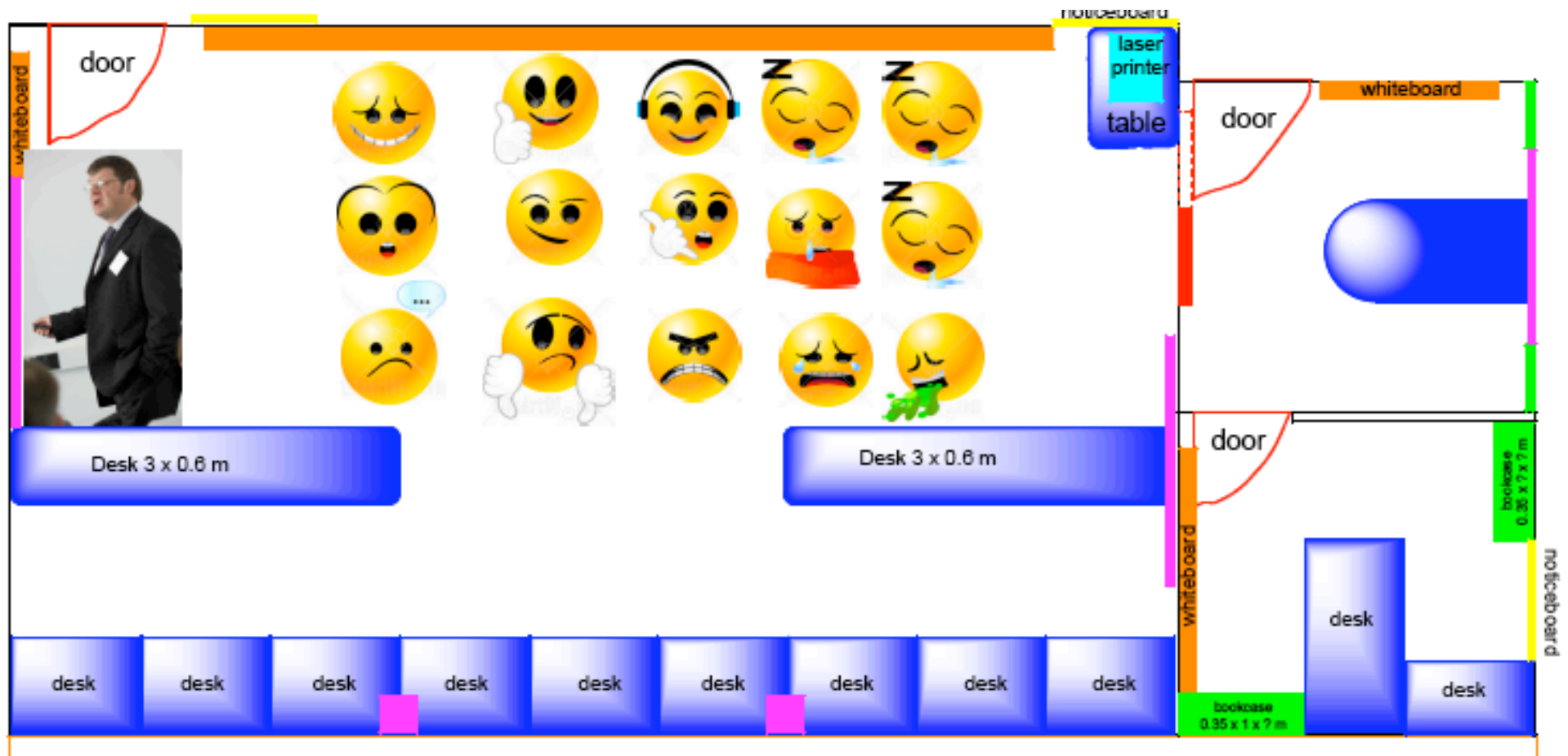


# The Research Hub - Brainstorm mode





# The Research Hub - Lecture mode



# First DTC Cohort





# Brainstorming in action!





## Breakout in action!



# Tour of Hub and Further Info

Please contact:

Dr Eoghan McGarrigle

[eoghan.mcgarrigle@bristol.ac.uk](mailto:eoghan.mcgarrigle@bristol.ac.uk)

# Application to study

Competition for DTC places will be fierce  
this year - apply asap!

See website for application procedure

<http://www.chm.bris.ac.uk/DTC/>