Department of Chemistry
Postgraduate Research Handout 2017/18

1. Welcome to the Department

1.1 Department of Chemistry

First of all, congratulations on your success in gaining a postgraduate research position and welcome to the Department of Chemistry at Bath. You are about to start on a very exciting phase of your life. If this your first time at the University Bath you'll find that it's very special place. If you're returning to Bath you will already know how special Bath is.

Postgraduate Studies is often much less structured than undergraduate studies. However, that doesn't mean you won't have lots to do: lots of work and lots of fun to be had!

There's also a lot to find out in the first few weeks - about the Department, the University and the City of Bath. We'll do our best to make sure that you settle in quickly so that you become familiar with the way that everything works at Bath, and in the Department of Chemistry. However, if you're not sure about something – please just ask.

The Department of Chemistry at Bath is a highly successful and growing department which carries out internationally recognised research in many areas of chemical sciences. Bath is the first choice for many people looking to pursue graduate studies in in Chemistry in the UK, and we have a large postgraduate community. Currently there are 42 full-time members of staff. We are a young department - the average age of academic staff is just 42. There are over 100 postgraduate students and this number is set to greatly expand in the coming years, so lots of people to meet.

Finally, I'm sure that you will have an enjoyable and rewarding time at Bath and if you do have any concerns either before you arrive or during your studies please do not hesitate to contact me by email: a.l.johnson@bath.ac.uk. Of course, once at Bath you can always pop by my office which is 1S 1.03b.

Dr. Andrew Johnson
Director of Postgraduate Studies

2. Department Structure

2.1. Who's Who in the Department

See http://www.bath.ac.uk/chemistry/contacts/ for a full list of Department staff. Some contacts most relevant to PG students are:

Head of Department	Professor Chris Frost
Deputy Head of Department	Professor Jonathan Williams
Director of Studies (PG Research)	Dr Andrew Johnson
Departmental Coordinators	Shula Dennard / Kate Remington

All staff are in 1 South unless indicated otherwise. The telephone numbers listed can be dialled from any internal University telephone. From outside the University, add (01225) 38 to the number (e.g. x 6504 becomes 01225 386504).

The directory of staff names and room numbers is located in the Foyer of the Chemistry Building, 1 South. If necessary, please contact Computing Services (using the "help" button at http://www.bath.ac.uk/bucs/help/contact/index.html) with your details if you need to be added to the appropriate list and email list.

2.2 Research Areas in the Department

Research in the department is divided into Physical, Inorganic, Organic and Computational Chemistry, with much of the research spanning two or more of these disciplines, facilitating multidisciplinary programmes of research. The Centre for Sustainable Chemical Technologies and the associated Centre for Doctoral Training link research between Chemistry and Chemical Engineering in many areas of green chemistry and renewable energy research, and also facilitates collaboration with other departments across the university.

Physical Chemistry
Solar cell research, electrochemistry, laser optical and microwave methods, LB films, biomimetic functional thin films, nanoscale & hierarchically structured materials and self-assembly, polymer-surfactant interactions, DNA electrochemistry, new polymer synthesis and sonochemistry, studies of protein-protein and enzyme binding, heterogeneous catalysis, materials for biofuel production.

Inorganic Chemistry
Catalysis, novel organometallic synthesis, hydrogen bond interactions, supramolecular coordination chemistry, bio-inorganic chemistry, X-ray crystallography and powder diffraction, synthesis of ALD and MOVCD precursors, transition metal chemistry, organometallic polymers, clusters and nanomaterials.

Organic Chemistry
Asymmetric synthesis, development of transition metals and enzymes as catalysts, asymmetric catalysis, biomimetic catalysis, bioorganic chemistry, antibody engineering, molecular evolution, enatioselective synthesis of aminoacids, supramolecular chemistry, molecular sensor design, natural product synthesis.

Computational Chemistry
Computer simulation of crystal growth, zeolite structures and minerals, studies of transition states of reactions, simulations of oxide materials for fuel cells and ceramic membranes and intercalation materials for lithium batteries.

Facilities and equipment
The department has a new research building with state-of-the-art laboratories and first class research facilities including:

- Atomic force and scanning tunnelling microscopes
- Surface plasmon resonance and surface plasmon enhanced fluorescence
- Mass spectroscopy suite
- Five NMR instruments (250, 300, 400 and 2 × 500 MHz)
- Extensive single crystal and powder diffraction facilities
- Small angle X-ray scattering instrumentation
- High power molecular modelling facilities
- Modern electrochemical instrumentation
- State-of-the-art laser optics

- Peptide synthesiser
- Fully equipped hazards-high pressure lab
- Raman, UV - Vis, FTIR, CD and fluorescence spectrometers

International and Industrial links
The Department has international links with countries including Germany, Sweden, the Netherlands, Switzerland, USA, Japan, China, Sri Lanka, France, Spain, Ireland, Malta, Singapore, Brazil, South Africa, Australia and New Zealand.

There are industrial links with companies including Glysure Ltd, National Physical Laboratory, Molecular Sensing plc, EpiChem, Unilever, Pilkington, Johnson Matthey, GlaxoSmithKline, Takeda, and Astra Zeneca.

3. Technical Information

3.1 Waste disposal

'Tradebe' handle the waste from Sigma and they have given instructions on how they would like the waste to be segregated. Please recycle all your empty SIGMA bottles in the bins outside 1 South. Please put a line through all the labels (glass & plastic) and remove the lids from the glass bottles. All lids can go in the plastic bin along with any plastic Sigma tubs. Any size of bottle can be taken but they should be washed out first.

Halogenated and Non Halogenated waste bottles: Please ensure that these bottles are not overfilled and the black lids not overtightened. *Only use "venting" lids for solvent waste bottles (identified by a special label)*, not normal lids. All full bottles must be placed in crates and not on the floor.

Each laboratory should regularly remove its hazardous chemical waste and take it down to the 1S waste holding area. Each bottle must be properly labelled (Aqueous or Non-Aqueous) and then stored according to chemical compatibility. Halogenated and Non-Halogenated solvents must be segregated. Waste labels can be obtained from the stores technicians. Vented caps MUST be used on all chemical waste bottles. Do **NOT** dispose of any chemical waste down the laboratory sinks or drains. Separate procedures exist for the disposal of unusual or highly hazardous waste – email bbcwaste@bath.ac.uk for further advice or with any queries.

3.2 Facilities

NMR Spectroscopy
The NMR facility in the Department of Chemistry at Bath currently consists of five instruments; one Agilent and four Bruker spectrometers. 1S 0.57 houses a Bruker AV300 and a Bruker AV400 spectrometer, located next door to our Bruker AV500 in 1S 0.03. The Bruker AV250 and the Agilent ProPulse 500 are in 1S 0.42. Basic training and safety guidance is provided to all researchers by Dr John Lowe or Dr Catherine Lyall before use of NMR instruments running in automation. Training will be arranged as part of your induction week.

The main purpose of the AV250 is to provide high throughput, rapid turnaround 1H spectra. It is fitted with a 60 position sample changer to allow continuous data acquisition throughout the day and night – 1H only during the day and 1H/13C during the night. Local users can keep track of samples submitted on the AV250, and print out spectra once complete, using a link from the 250MHz NMR webpage. A PDF of the processed spectrum is also emailed to the user immediately after acquisition.

The AV300 is also fitted with an automatic sample changer with capacity for 60 samples, allowing high-throughput data acquisition round the clock. Any researcher can use this machine, including final year undergraduate students during their research projects. It is normally used for the acquisition of standard 1D and 2D data. Like the 400 and 500, a fully autotunable probe allows data from a large range of NMR-active nuclei to be acquired. Local users can also keep track of samples and print out spectra online as for the 250, in addition to the emailed PDFs of spectra.

The AV400 is normally reserved for more specialised 'hands-on' applications, and a small group of students, postdocs and staff are trained to operate it. It is fitted with an autotune probe and, along with the 500, can acquire data from a wide range of nuclei (including 1H, 13C, 11B, 15N, 19F, 29Si, and 31P). In addition a separate probe allows the acquisition of 19F-1H correlations. Currently, it is often used for NMR studies of diffusion. A wide selection of modern gradient-based experiments is available, and spectra can be acquired at temperatures from −150 to +150 °C. Most research groups will have at least one person trained; anyone wanting training on the AV400 or the AV500 should contact Dr John Lowe in the first instance.

As with the AV400, the Bruker AV500 is used in manual mode by researchers who have opted to be trained in its operation. Complete with two autotune probes, it is capable of examining a broad range of nuclei (including 1H, 13C, 11B, 15N, 19F, 29Si, 31P and 103Rh). A full selection of modern gradient-based 1D and 2D experiments are available, and spectra can be acquired at temperatures from −150 to +150 °C.

In 2014, we have had our most recent spectrometer installed. The Agilent ProPulse 500 has a 96 position sample changer, for continuous data acquisition at a higher field strength. The autotune probe is capable of examining 19F spectra in automation, in addition to all other nuclei available on the AV300. When used in manual mode, samples can be acquired at temperatures between -80 and +100 °C.

An external NMR service is offered to customers from outside the University. Prices start at £30 per sample, please contact Dr John Lowe, j.lowe@bath.ac.uk, for further information.

Mass Spectrometry Service

The Department of Chemistry Mass Spectrometry facilities have recently been upgraded and consist of a new electrospray Time-of-Flight mass spectrometer (ESI-TOF), and a new electrospray quadrupole Time-of-Flight mass spectrometer (ESI-QTOF). These are used for a wide range of applications, including:

- Organic and inorganic synthesis product confirmation
- Impurity profiling and identification of unknowns
- Reaction monitoring

The **micrOTOF** (ESI-TOF) is used for routine accurate mass analysis, with high resolution acquisition. It is controlled by open access software, with a choice of generic methods for different mass ranges and polarities. Generally it is run using an autosampler to inject the sample into a solvent stream supplied by the LC pump, without any chromatographic separation. There is also the capability for liquid chromatography to precede the mass spectrometric analysis (LC-MS).

The **micrOTOF-Q** (ESI-QTOF) instrument is operated as a research instrument where users will have full interaction with the operating methods. As for the micrOTOF, this instrument is capable of acquiring high mass accuracy data, of publication quality. In addition, the inclusion of a 'Q' (quadrupole) allows for fragmentation experiments to be carried out, allowing for some structural elucidation to be done. In combination with the excellent NMR and X-ray facilities in the department, full structural identification and characterisation should be possible. A glove box is located next to the spectrometer making it possible to prepare samples in an inert environment and then to infuse them directly into the mass spectrometer via a break in the glove box, allowing for 'inert' mass spectrometric analysis to be conducted.

In order to use the facilities, it is important that you first receive some training. You can be trained to use the OpenAccess micrOTOF, or the research micrOTOFQ, or both. Contact Dr Anneke Lubben (a.t.lubben@bath.ac.uk) or visit the Mass Spectrometry Service web pages for more details or to sign up for training. Once you have been trained, you will be allocated a user name which will give you access to the service. Outside the normal working hours of 8.00am to 6.30pm weekdays, access is restricted using a card reader on the door to the foyer. In addition there is a keypad on the lab door to allow for access to the instruments themselves. This requires a 4 digit key-code, which is only given to you once you have undergone training.

4. Health and Safety

4.1 Safety policy

All safety matters are treated with the utmost importance within the Department of Chemistry, and a separate safety manual will be issued to all new researchers. To emphasise the importance of safety, some of the key points are repeated here.

The University has a Health and Safety Policy which is displayed throughout the campus. There is also a Safety, Health and Environment Unit (WH3.26) with staff who are able to advice on health and safety issues and who monitor the health and safety management of the University.

For further information, the Safety, Health and Environment website is:

http://www.bath.ac.uk/hr/stayingsafewell/hs-policy/index.html

or email r.bott@bath.ac.uk.

The University has a public liability insurance policy to cover any claims brought by students or members of the public against the University where the University has been negligent. This policy does not, however, cover students who come to harm because of their own fault, or cause damage of their own volition.

Risk Assessment and Operating Procedures
The Management of Health & Safety at Work Regulations, 1992, and other Regulations as well, require us to "suitably and sufficiently assess the risks to the health and safety of employees to which they are exposed whilst they are at work". This means that we must make "risk assessments" for every work activity carried out by, in particular, students and other researchers. This page indicates how the procedure may be put into effect in the Department of Chemistry.

Hazards and Risks
"Hazard" and "risk" are words which are synonymous in common use but in the technical jargon of Safety Management have different meanings: **the hazard** presented by a substance or activity is its potential to do harm (rock-climbing is a hazardous activity) and **risk** from a substance or activity is the likelihood that it will cause harm in the circumstances of actual use or that the hazard will be realised (rock-climbing may be of low risk if the proper equipment is used and the rules are followed).

The aim of making a "risk assessment" is to identify the hazards associated with an activity, to assess the seriousness of these hazards and to formulate systems of work, training or other methods (controls) to reduce the associated risks to a minimum or at least to an acceptable level.

This procedure has to be carried out by someone who is experienced and fully familiar with the activity *i.e.* a "competent person".

The Risk Assessment Procedure

We are required to

- identify hazards associated with activities or situations.
- somehow or other quantify the associated risk. (How likely is it that any hazard will be realised? How severe will the consequences be? How often does exposure to the hazard occur?)
- identify who is at risk.
- identify the control measures to be used to reduce the risk to a "reasonable" level.
- quantify the residual risk, and then
- record the assessment and implement the control measures.

Application to Chemistry

Straightforward as this procedure is, its translation to the variety of activities carried out in the Department of Chemistry is clearly not trivial, thus the following practice should be adopted.

How to Do it?

- ***Identify*** Which activity is to be assessed? Has it been done before or can it be broken down into tasks that have already been assessed (see the chemistry department safety wiki). If there is no existing Assessment, you will have to do it yourself.
- ***Carry out the procedures 1), 2) and 3) above, i.e. identify the hazards, quantify the risks and identify who is at risk.*** This is where you have to put in the work, reading around the subject and so on. The conclusions here may be subjective but you must be clear about the person or persons you are considering and their likely level of ability or competence. There is a possibility of using sliding scales of likelihood for the hazard being realised (unlikely, likely, very likely, certain) and for the severity of any injury (slight (Elastoplast), moderate (stitches), severe (hospitalisation), death), and frequency of exposure.
- ***Formulate control measures.*** These will include:
    - physical or engineering controls (*e.g.* sturdy, custom-built trolleys for moving gas cylinders, the provision of fixed racks for cylinders when they are in use) and this will allow you to spot any possible deficiencies in the physical provisions of laboratories;
    - protective equipment to be worn (*e.g*. safety glasses, lab. coats);
    - procedures to be followed (*e.g*. solvent bottles to be transported in proper carriers) and
    - any training that is required.

This process of categorisation is not simple. It calls on our own expertise and experience, our knowledge of the abilities of our students and our knowledge of how far any reasonable control measures to be used are going to be effective and it decides who gets to do what.

Write all this down: This is a legal requirement but, more than that, performing a Risk Assessment is not simply an end in itself. Rather it is a tool to help us to protect the health and well-being of our students and workers. As such, what is written down in the Risk Assessment document should be included as part of the training. The document should be fully available to, in fact required reading for, researchers to enable them to realise the hazards associated with the tasks they are to perform and to see what must be done or what they must do to protect themselves. The Risk Assessment document should be as full as possible and include within it a description of recommended operating procedures, if relevant, action to be taken in an emergency and any suitable references. So in fact this recording becomes part of the next stage.

Implement the control measures. *e.g*. provide the trolleys and racks and carry out the training, including reading the full text of the Risk Assessment.
Finally, if you think that the Assessment you have just made will be of use to others, make it available and have it included in the Department of Chemistry library of Risk Assessments on the Safety Wiki.

4.2 Safety Induction and Registration

As part of your induction week you **must** attend a Safety induction **BEFORE** starting any work in a COSHH regulated area. You will then be given a copy of the latest Departmental Safety Manual which you are required to read. You must complete **Form A** (page 15) and return it to the Health & Safety coordinator. A register of all attendees will be kept in the department.

Your induction will cover Fire and Chemical Safety. This will involve a presentation (including watching some videos) by the Departmental Health and Safety Officer.

You **MUST** also attend a session in the practical use of Fire Extinguishers, and will be expected to attend the next available training session arranged for research staff. Your certificate of attendance will be held on file and will cover a 3-year period (after which you will be required to attend for a refresher). To sign up for this session, send an e-mail to Mrs Sarah Elkins (s.l.elkins@bath.ac.uk).

If you will use gas cylinders in your work, you **MUST** also attend a session on Gas Cylinder Manual Handling. To sign up for this session send an email to Mr Russell Barlow (r.w.barlow@bath.ac.uk).

If your work involves use of lasers you **MUST** view the laser safety video (online) and complete the Laser Safety form. Contact Prof. Toby Jenkins (a.t.a.jenkins@bath.ac.uk) to obtain the form and directions for watching the video.

You should familiarise yourself with all the Safety noticeboards and where the Fire Extinguishers and First Aid kits are in your main work areas. You should know who is your postgraduate safety committee representative and your Laboratory Custodian.

If you are required to attend the University Medical Centre for Occupational Health Surveillance, please make sure you keep to your appointments. This can be normal for new research workers.

4.3 Safety Contacts

The Faculty of Science Health & Safety co-ordinator is Robyn Bott, who can be contacted at r.bott@bath.ac.uk. Alternatively, any queries about safety can be directed to your supervisor.

4.4 COSHH

COSHH Assessments must be completed **JOINTLY** by the Research Worker and the Research Supervisor BEFORE the procedure is carried out. The purpose of the assessment is to give careful consideration to the risks involved in using particular chemicals in a defined procedure such as a synthesis.

COSHH forms and much other useful safety information can be downloaded from the University Safety website:
http://www.bath.ac.uk/hr/stayingsafewell/index.html

5. Training Sessions

5.1 Mandatory Courses

Important: All new postgraduates MUST attend the Safety Induction on Monday 26th September 2016 (14:15 in room 1S 0.01) before starting any experimental work.

Fire Safety Training: will be held soon after the start of term (date TBC) and is **compulsory** for **all** new staff and postgraduate students. Sign up for one of the two sessions on the list during the Departmental Welcome or, if you miss this session, email Mrs Sarah Elkins (S.L.Elkins@bath.ac.uk) to reserve a place.

Courses which are compulsory for certain activities:

Laboratory Demonstrating: If you wish to take part in demonstrating in the undergraduate laboratories this year, you must attend this course, organised by Dr Andrew Johnson and Dr Fiona Dickinson. The date is to be confirmed – more details will be given in the Departmental Welcome. There are also equivalent courses run centrally, later in the year. These can be booked through the PGSkills website.

Solvent Purification System Training: If you will need to take dried pure solvents from this system, you must attend a training session before you use it. Attend the course run by Dr Randolf Köhn. The date and time for this training is to be confirmed – more details will be given in the Departmental Welcome. (no need to sign up, an attendance record will be taken during the course).

Gas Cylinder Manual Handling Training: This is **mandatory** if you will use gas cylinders during your PhD work, and you will not be allowed to move cylinders unless you attend this training. You will be contacted to sign up for this session near the start of term; if you miss this session, email Mr Russell Barlow (r.j.barlow@bath.ac.uk) to sign up for this course.