1st YEAR PHYSICAL CHEMISTRY PRACTICAL COURSE, 2007-08

1 INTRODUCTION

In the PTCL you will meet a wide range of techniques - from the simple to those using complex instrumentation - with which to study the properties of materials. During your time here, we want you to gain an understanding of experimental techniques and methods. It is not easy to work in a laboratory; making precise measurements requires practice, experience and judgement. In the practical course you will, we hope, acquire these skills, which are a vital preparation for later research.

2 WORKING HOURS

You will work in the laboratories during a 4-week period between 11 a.m. and 5 p.m. on Monday and Tuesday, or Thursday and Friday. As you cannot work beyond 5 p.m., plan in advance so you can stop when the laboratory closes, without disrupting your experiment. From 9 a.m. to 11 a.m., and all day Wednesday, the laboratories are closed, so you cannot do experiments, but you can use the computers.

3 STARTING AN EXPERIMENT

Prepare fully for an experiment by reading in advance the instructions and any appropriate background material. If the experiment lies in an area not yet covered in lectures, you may need to do some further reading, but proper preparation before starting an experiment is always time well spent. You will normally work on experiments as one of a pair.

When you come into the laboratory to start each experiment, go to the technician or a demonstrator and ask them to give you a "Date experiment started" sticker which must be stuck into your data book. You will need to match this with a sticker of the same number when the experiment is signed off.

You have two days to complete each of experiments X.1 (Solution Chemistry) and X.5 (Optics) and one day for experiments X.2 (Error Analysis), X.3 (Kinetics) and X.4 (Vacuum Techniques). If you are unable to complete an experiment through illness or other reasonable cause, see Dr Cartwright who will make arrangements for you to do the experiment at some other time.

4 NOTEBOOKS

Keep a record of experimental data in a hardback notebook. You must hand this in to Dr Cartwright by the end of 4th week, Trinity term, in your 3rd year. Write your name on the spine of the book and maintain at the front an index of experiments completed.

As you do an experiment, record measurements and results in the space provided on the worksheets, or in your data book if there is no appropriate place on the worksheet. When you must take many measurements (for example, during a titration), it is usually helpful to plot the data as the experiment proceeds, even if the instructions do not explicitly suggest this. It will then be easier to spot dubious data and errors made during the experiment.

Ask a demonstrator to countersign your results during the time you are in the laboratory, to indicate that (s)he is satisfied with the way you are working. (This *must* be done while you are working, not later. Demonstrators will not sign your data once the experiment is over, and it may not be possible to get an experiment signed off if the data are not countersigned.) When the experiment is complete, fill in a comments slip and post it in the box near the booking board, if you have any helpful comments to make.

5 REPORT FORMAT

Your first year experiments are written up partly on the worksheets that comprise this manual, and partly in your data book. The instructions will make clear where you are to do the write-up. All work should be written neatly in correct English; demonstrators may not accept sloppy or grammatically incorrect reports.

6 SIGNING OFF

Each first year experiment must be signed off as soon as you complete it; there should be no need to take the worksheet away for further embellishment before the demonstrator can sign it. Take your completed worksheet and data book to a demonstrator in the laboratory in which the experiment is located and ask to be signed off.

Once you have been signed off, make sure that a white signing-off slip is completed and signed, and add the appropriate stickers to the signing-off slip and the end of your report.

7 APPARATUS

Major apparatus for experiments is set out on the benches. Glassware may be obtained from the glassware cabinets; for other requirements, see the technicians.

Clean the apparatus and bench area when you finish an experiment. Rinse out glassware and leave on the draining board. If the apparatus is faulty in any way, report this to a technician, even if you caused the problem. If not reported, a problem may remain undetected until the next students attempt the experiment. Do not leave unlabelled liquids in beakers or flasks - these can present a hazard to the technicians.

8 WEB SITE

A web site is available for the practical course in physical chemistry at the following URL: http://ptcl.chem.ox.ac.uk/~hmc/tlab/tlabtl.html. This contains safety and other information which you may find of value.

9 EXAMINATION REQUIREMENTS

Each experiment in the PTCL practical course is allocated a number of points, reflecting its difficulty, length and educational value. In the first year, you must complete all five experiments (which are worth a total of 21 points).

By the end of 4th week in Trinity term of your 3rd year, you must have completed 3-year courses of practical work in two of the three laboratories (Physical, Organic and Inorganic) and a 2-year course in the third. If you pass a supplementary subject, you need do a 3-year course in only one area and two 2-year courses. There is at present no 2nd-year requirement in physical chemistry, but you are advised to ensure that at least some experiments are completed during your 2nd year.

10 QUESTIONS?

Help on any aspect of the practical course in the PTCL is always available from Dr. Hugh Cartwright, room 34-04, Hugh.Cartwright@chem.ox.ac.uk

SAFETY

You will need to work carefully in the PTCL, as in any laboratory. Special hazards associated with an experiment are explained in the instructions, but laboratories are dangerous places, so you must always be alert.

If in doubt - ASK

You have a legal responsibility to work as safely as possible. Your use of chemicals is governed by the COSHH regulations (Control Of Substances Hazardous to Health). Under these regulations, we assess the hazards of chemicals before they can be used, and ensure that precautions are taken so that they are used safely. Our experiments are drafted to take the regulations into account, and formal assessments for all potentially dangerous chemicals are available in the laboratory for you to consult. If any particularly unpleasant or dangerous chemical is used during an experiment, detailed precautions are given in the instructions.

However, remember that the prime responsibility for safety rests with you.

The guidelines that follow are important; PLEASE READ THEM BEFORE STARTING WORK !

1. Emergency

Before starting work for the first time, check the location of emergency facilities. There are two exits from each laboratory - make sure you know where they are. Each laboratory has an emergency shower - find it! Check that you know where fire extinguishers are kept in case you need to use one and where the break-glass fire alarms are in case you discover (or create!) a fire.

2. Eating, drinking & smoking

No eating or drinking is permitted in the laboratory; there is an area on the ground floor where hot drinks may be bought from a machine. All University buildings are non-smoking areas.

3. Preparation

Always read the instructions for an experiment in detail before coming to the laboratory. Prior preparation of this sort will contribute to safe working and it also helps you to work more efficiently. Ask a demonstrator or a technician if any aspect of the experiment is unclear. Don't feel that to ask a question shows a lack of preparation; you may have discovered an ambiguity or mistake in the instructions that has not been noticed before, so if in doubt, always ask.

4. Safety glasses

Wear safety glasses whenever you work with acids or bases, evacuated equipment or equipment under pressure. Eye damage is easy to sustain and an accident can lead to serious damage or permanent blindness. Always be aware of what is happening around you, and if a potentially hazardous operation is being carried out near you, wear eye protection even if your own experiment is without risk. You are advised to wear prescription lenses rather than contact lenses, since chemicals may become trapped behind the latter, keeping them in contact with the eye.

5. Laboratory coats and shoes

You are not required to wear a laboratory coat, but it is advisable to do so during any experiment in which toxic or corrosive chemicals are being used, to protect yourself and your clothes from chemical spills. However, do be aware of the danger that laboratory coat sleeves may catch on equipment if you have to reach across the bench. Lab coats should not be nylon-based; this burns, melts and sticks to the skin, leading to very serious burns. Open-toed shoes or sandals should not be worn except in the computing area.

6. Tidy working

Keep your working area tidy. A cluttered bench is a common contributory factor to accidents, so do not collect dirty glassware as the experiment proceeds. Instead, clean and reuse or leave at the sink any glassware that you no longer need.

7. Disposal of chemical waste

When you have finished using a chemical, do not keep any excess at the bench, but immediately dispose of it as appropriate; this may be down the sink, in a waste container, or back into the original container. Ask the technician how to get rid of chemicals if you are in any doubt. Do not leave solutions for the technician to deal with, especially in unlabelled containers. Do not add non-chlorinated solvents to the chlorinated waste containers, or vice versa. On no account wash toxic organic material or metal salts down the drains; if you do, you may be drinking it a few days later.

8. Disposable gloves

If you are using toxic or corrosive chemicals you may wish to use protective gloves. When it is essential that such gloves be worn this will be indicated in the text; if on other occasions you wish to wear gloves, ask the technician for a pair, and replace them as needed. Note that some chemicals, such as platinum salts, can cause sensitization. Gloves should be worn when handling these compounds (but platinum metal itself is not a sensitizer).

9. Spills

Always clean up a chemical spill without delay. If you spill a toxic, flammable or corrosive chemical, call a demonstrator or technician immediately.

10. Pipetting

Do not pipette by mouth, even solutions that you believe to be harmless. The solution may be less innocuous than you think, or there may be contamination on the pipette that you may get into your mouth.

11. Flammable materials

When working with flammable liquids, start by checking that the area immediately around where you work is free of flames, and hotplates. A volatile organic chemical such as diethyl ether can be ignited by contact with the surface of a hotplate - no naked flame is required.

12. High-pressure gases

If you have not used a high-pressure gas regulator before, ask for assistance the first time you need to use it. Large gas cylinders contain a great deal of stored energy and have the potential to cause serious accidents.

13. Tubing and glass

When sliding rubber tubing onto glass (for example, using a pipette with a flexible filler) use the minimum possible force and a gentle twisting motion. If you use excessive force and the glass breaks you will drive broken glass into your hand or wrist and cause serious (possibly life-threatening) cuts. In case of difficulty, ask a demonstrator or technician for help; special gloves can be provided if necessary. To remove rubber tubing it is often safest to cut the rubber tube off rather than trying to pull it off.

14. Fire alarms

If the fire alarm sounds evacuate the laboratory without delay. If time permits, make your experiment safe if this will take no more than a few seconds, but do not take time out to retrieve personal belongings (or take a trip to the toilet!) The assembly point for this building is outside the main entrance on the opposite side of the road, infront of the old Dyson Perrins building. Do not stand in the road itself, since access must be kept clear for emergency vehicles.

15. Spills onto skin or eyes, ingestion or inhalation of chemicals

If you spill a chemical on the skin, wash it off immediately with plenty of water. If a chemical gets into your eye, inform your fellow worker or another person in the laboratory and immediately irrigate the eye with water from one of the taps - a flexible rubber hose is attached to many taps for this purpose. If you get a chemical in your mouth, wash the mouth out with water immediately. In every case the demonstrator and technician (who is a trained first aider) must be informed immediately so that further action, if necessary, can be taken.

16. Special rules for pregnant women

The foetus, especially in the first trimester, is particularly vulnerable to damage from chemicals. If you are pregnant, or believe that you might be, you are encouraged to talk to Dr Cartwright so that he can assess whether any restrictions on the experiments you will do would be advisable. You may alternatively wish to consult your College chemistry tutor or the College nurse who will liaise with Dr Cartwright if necessary.

17. Pre-existing conditions

If you suffer from any condition which may lead to sudden illness, or if you must take, even for a short time, drugs which may affect your performance in the laboratory, obtain a medical record sheet from Dr. Cartwright. In case of emergency, it may help if this has been completed and stuck inside the back cover of your laboratory data book.

18. Accidents

Report any accident immediately, no matter how minor, to the demonstrator on duty. There is a First Aid room in the PTCL library and First Aid kits in the technicians' preparation rooms in each laboratory. The technicians are trained in emergency first aid.

If you have any suggestions or concerns about safety in the laboratory, please see Dr Cartwright.