Learn Chemistry

First Year Undergraduate Chemistry Laboratory Course Manual 2011-2012

Core Chemistry 1A and 1B: Discovery Block 3

Developed by Dr Jacquie Robson, RSC School Teacher Fellow 2010-2011 at Durham University

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Core Chemistry 1A and 1B

First Year Chemistry Laboratory Course Manual 2011-2012

DISCOVERY BLOCK 3

Name ……

Core Chemistry 1A session:
Day/Time: …… Group name: ……..

Core Chemistry 1B session:
Day/Time: …… Group name: ……

Contents

Safety in the first year laboratory (CG 021)

The Health and Safety at Work Act was introduced in 1974. Since then many regulations have been made under the act, for example, The Control of Substances Hazardous to Health (COSHH). The University has a statutory obligation to comply with these requirements and you, as a student, have a duty to abide by the regulations. The following notes are to guide you in good laboratory practice and to familiarise yourself with the safety aspects of your laboratory work.

Emergency Telephone Numbers

Internal telephones:	FIRE, POLICE, AMBULANCE	9999
	UNIVERSITY EMERGENCY NUMBER	43333

1. Staff with special responsibilities for safety:

Chairman of the Board of Studies:	Professor J S O Evans
Chemistry Safety Officer:	Dr J A G Williams
Undergraduate teaching laboratories:	Dr E Wrede (Physical Chemistry)
	Dr J A G Williams (Inorganic Chemistry)
	Dr E Khosravi (Organic Chemistry)

2. No work is to be carried out unless a member of staff is present.

3. All persons in laboratories (whether or not they are actually doing practical work) must wear safety spectacles and laboratory coats. Academic staff supervising undergraduates enforce this rule. In all laboratories, hair should be secured so that it does not hang below the neck. It is important to wear suitable clothing, and your footwear must incorporate flat heels, slip-resistant soles and uppers fully enclosing the foot.

4. Foods, drinks, cigarettes, cosmetics and mobile phones must not be taken into or used in areas where chemical substances are used or kept.

5. Bags and coats should be placed in the lockers provided outside the laboratory and not left in corridors or on benches.

6. All accidents and dangerous occurrences must be reported immediately to a member of staff or a demonstrator. The first aid box is located in the foyer area and a list of qualified first aiders is on the front. The accident book is kept in room CG 058 and the member of staff in charge of the laboratory must fill out a report for all incidents. An emergency shower is located in the foyer area and there are four eyewash stations beside the sinks. There is a chemical spillage treatment kit in CG195.

7. The fire action signs in the laboratory indicate the nearest fire alarm and the emergency exit. There are two carbon dioxide fire extinguishers on either side of the central pedestal and another in the instrument room. There is also a foam spray fire extinguisher on either side of the central pedestal and one at each fire exit. A general fire practice is held twice yearly to check the smooth operation of the procedure so you should ensure that you know where to go in an emergency.

8. Pipetting by mouth is not allowed. Use a bulb or automatic pipette.

9. Do not inhale vapours or make skin contact with any substances. Use gloves where necessary always remembering that they are semi-permeable.

10. Experiments must be conducted on clean working surfaces; any spillage should be cleaned immediately. A high standard of tidiness should be maintained at all times. Contaminated surfaces and equipment must be cleaned as soon as it is practicable after use. The equipment should then be put away. Do not clutter bench-space with unused equipment and bottles of chemicals.

11. Waste should be disposed of in the appropriate containers: solvents should be placed in either the C, H, N, O-containing waste solvent bottles (Category C Waste), or halogen, sulphur-containing waste solvent bottles (Category D Waste). Heavy metal waste should be placed in the appropriate bottle. Broken glassware should be washed and placed in the designated glass bin. Solid waste should be dried, placed in a polythene bag and placed in a solid waste bin. A sharps bin is located in CG195. Consult a demonstrator if you are unsure about the correct disposal procedure.

12. The COSHH assessment of any chemical you use or make will be given in the laboratory script. There are further safety warnings at the appropriate parts of the text. Staff and student demonstrators reinforce these. If you are in any doubt, consult a demonstrator.

13. No unauthorised experiments are to be carried out.

14. It is important to ensure that hands are washed and all protective clothing removed **before** leaving the laboratory.

Introduction

Chemistry is an experimental science and, as well as attending lectures, both the University and the Royal Society of Chemistry, who accredit your degree, require you to complete a designated number of hours of laboratory work. During the first year, 18 weeks of practical work must be completed. The first year practical course is split into four sections:

1. Induction (Week 1)
2. Skills (Weeks 2-7)
3. Discovery (Weeks 8-16)
4. Projects (Weeks 18-19).

During Blocks 3, 4 and 5, you will complete the Discovery section. This contains activities designed to extend and build upon the key skills you have developed and practised in the Skills section. Some experiments will lead on from some in the Skills section, some will be linked to lecture courses you have studies and others will introduce new chemistry and ideas. Ideas developed in previous sections is now assumed knowledge, so you may need to refresh your memory by reading back through older laboratory manuals and your lab notebook as part of your pre-lab preparations.

1.1 The pre-lab exercises

As in the previous section, before every laboratory session one or more pre-lab exercises must be completed. These may involve reading, watching video clips, answering questions, completing assignments or using interactive software to rehearse techniques. Many of the files and resources for these exercises will be accessed via DUO, the university Virtual Learning Environment, which you should now be familiar with using regularly.

Pre-lab exercises will often contain summative aspects (i.e. the marks will count towards the overall marks for the Laboratory Course), and they must be completed in the week before you attempt the laboratory activity. All pre-lab work must be finished an hour before the relevant laboratory session so that completion can be checked. For example, a student attending the Thursday laboratory session, which begins at 9.00am, must have completed the pre-lab exercises by 8.00am that same day. Anyone arriving at a laboratory session without having completed the pre-lab exercises will be sent away to complete them before being allowed to begin work in the laboratory. Failure to complete the pre-lab exercises on time will incur a marks penalty. Your time in the laboratory will become very pressured if you are sent away to complete the pre-lab exercises. Good time management is the key to success in most areas of university life, but particularly in your laboratory work!

If there are any problems with access to DUO or LabSkills using personal computers, there are open-access machines available for use in the library and at other points around the science site. There may also be provision in college. Ask for help if problems arise when accessing the pre-lab exercises. Failure to access the exercises will not be accepted as a reason for incomplete pre-lab

work unless the laboratory course leader (Dr J. M. Robson) is informed in advance of the deadline so alternative arrangements can be made.

1.2 LabSkills

Many pre-lab exercises will again involve you using LabSkills. This is an electronic, interactive laboratory textbook for you to use to gain confidence in assembling and using apparatus before you begin work in the laboratory. Interactive exercises are designed to allow you to practice key techniques and learn more about apparatus and safety as you progress through the course. During the Discovery section of your laboratory course, the pre-lab exercises will be less prescriptive in their use of LabSkills but you should continue to use it as part of your pre-lab preparations to ensure you have refreshed your memory of the key techniques before your laboratory session. LabSkills also contains useful glossaries and worked examples of calculations that you will find useful. It will be accessible in the laboratory for additional assistance if you need it.

1.3 The laboratory sessions

One laboratory session per week will be assigned to Core Chemistry 1A, and a second session per week for Core Chemistry 1B. Experiments that will count towards Core Chemistry 1A will contain a suffix of 'A' in the title (e.g. Experiment 9A) and will be carried out by everybody. Experiment titles containing a suffix of 'B' (e.g. Experiment 9B) will count towards Core Chemistry 1B and will be carried out only by those studying Core Chemistry 1B.

In the first term (Michaelmas), University weeks begin on a Thursday and end on a Wednesday. Those students only studying Core Chemistry 1A will be assigned one laboratory session per week and will carry out all of the 'A' experiments. Those students also studying Core Chemistry 1B will be assigned two sessions per week. The first session of the week is assigned to be the Core Chemistry 1A session, and the second session of the week is the Core Chemistry 1B session. For example, if the two allocated laboratory sessions are on Thursday morning and Monday afternoon, the Core Chemistry 1A session will be Thursday morning, when an experiment with the suffix 'A' will be performed, and the Core Chemistry 1B session will be Monday afternoon, when the 'B experiment for that week will be completed.

Laboratory sessions will be allocated during one or two of the following times:

Thursday	9.00am - 12.00pm
Friday	9.00am - 12.00pm
Friday	2.00pm - 5.00pm
Monday	9.00am - 12.00pm
Monday	2.00pm - 5.00pm
Tuesday	2.00pm - 5.00pm
Wednesday	10.00am - 1.00pm

You may only attend the laboratory at your allocated time.

A risk assessment is provided in this manual for the chemicals used in each experiment. This must be read carefully before attending the laboratory, and the advice followed throughout each laboratory session. All experimental work must be completed in that laboratory session and your lab notebook and work space signed off before you leave.

1.4 Set Allocation

Students in each laboratory session are allocated to one of three named sets of no more than 20 students. Sets are named after chemical elements and students are assigned to sets in no particular order. Lists showing members of each set are available on DUO and details should be written onto the front of the laboratory manual. During the second term (Epiphany), weeks begin on a Monday. This will necessitate some set changes within groups to ensure that students complete their 'A' experiment in their first session and their 'B' experiment in the second session. New set lists will become available before the start of the second term so students can check the experiment rota and identify the experiments they need to prepare for.

Each set will tackle a different activity each week, in a three week cycle, until everyone has completed each activity. The three experiments in the laboratory will then change and each set will again work through each experiment according to the rota.

Set names are as follows:

	Set 1	**Set 2**	**Set 3**
Thurs am	actinium	bismuth	cobalt
Friday am	dysprosium	erbium	fluorine
Friday pm	gadolinium	hafnium	yttrium
Monday am	potassium	lithium	molybdenum
Monday pm	niobium	phosphorus	osmium
Tuesday am	rhodium	strontium	tantalum
Wed am	uranium	vanadium	tungsten

Students will be allocated one set for their Core Chemistry 1A session and a second set for their Core Chemistry 1B session. Set lists will appear on DUO before the start of Week 2. Sets will perform experiments according to the following rota:

Week	**Set 1**	**Set 2**	**Set 3**
8	Experiment 8	Experiment 9	Experiment 10
9	Experiment 9	Experiment 10	Experiment 8
10	Experiment 10	Experiment 8	Experiment 9

For example, in week 9, everyone in Set 2 will carry out Experiment 10. Those studying only Core Chemistry 1A will only complete Experiment 10A. Those students also studying Core Chemistry 1B

will complete Experiment 10B during their second session of the week. You should note you're your allocated set on the front of your laboratory manual.

1.5 Assessment

Pre-lab exercises, mostly carried out via DUO, will contain assessed components. These exercises will differ between experiments, but assessed aspects will be highlighted. Completion of these exercises is compulsory and there will be a marks penalty for non-completion. The pre-laboratory exercise marks will make up 10% of the total marks for the practical course.

During each laboratory session, work and progress will be assessed. Completion of the lab notebook and performance in the practical tasks will be given marks. Occasionally there will be a small amount of post-laboratory work that will need to be completed to finish each experiment. Marks will be awarded during laboratory sessions throughout the year and will make up 10% of the total marks for the practical course.

In the Discovery section, a number of additional assessed components will be introduced in Experiments 10, 12 and 15 for Core Chemistry 1A and Experiments 10, 11 and 15 for Core Chemistry 1B.

1.6 Assessment summary (bold indicates activities to be completed during Skills)

	Core Chemistry 1A	**Core Chemistry 1B**
Pre-lab exercises (whole year)	**10 %**	**10 %**
Laboratory session marks (whole year)	**10 %**	**10 %**
SKILLS Experiment 5B: Determination of the enthalpy of vaporisation of ethanol	-	5%
DISCOVERY Experiment 10A	**20 %**	**-**
DISCOVERY Experiment 10B	**-**	**15 %**
DISCOVERY Experiment 11B	-	20 %
DISCOVERY Experiment 12A	20 %	-
DISCOVERY Experiment 15A	20 %	-
DISCOVERY Experiment 15B	-	20 %
PROJECT	20 %	20 % *
	100 %	**100 %**

*50 % of the project mark for students completing both Core Chemistry 1A and Core Chemistry 1B will be allocated towards the Core Chemistry 1A total marks, and 50 % will be allocated towards the Core Chemistry 1B total marks.

1.7 Supervising staff and postgraduate demonstrators

DISCOVERY Block 3 – week 8 to week 10

(Thursday 24th November to Wednesday 14th December 2011)

Senior Demonstrators (staff)	Junior Demonstrators (postgraduate students)
Dr Jacquie Robson*	Paul Brooks
Dr Ivana Evans	Philip Brown
Dr Corinna Hess	David Cole
Dr Hazel Sparkes	Lucy Clarke
Dr Pippa Coffer	Fabian Kempe
	Santi Marques-Gonzalez
	Alex Payne-Dwyer
	James Radcliffe
	Joe Ridout

*laboratory course leader - email: j.m.robson@durham.ac.uk

DISCOVERY
BLOCK 3
EXPERIMENT 8A

A REDOX TITRATION TO DETERMINE THE ETHANOL CONTENT OF WINE

8A. A redox titration to determine the ethanol content of wine

Potassium dichromate(VI), $K_2Cr_2O_7$, is a common oxidising agent used in redox reactions. It is commonly used in redox titrations. In this experiment, a solution of potassium dichromate(VI) will be standardised using a solution of ammonium iron(II) sulphate, $(NH_4)_2Fe(SO_4)_2.6H_2O$, as a primary standard. A known volume of this potassium dichromate solution (a large excess) will then be reacted with the ethanol in wine to produce ethanoic (acetic) acid:

$$2Cr_2O_7^{2-} + 16H^+ + 3CH_3CH_2OH \rightarrow 4Cr^{3+} + 11H_2O + 3CH_3COOH$$

The remaining potassium dichromate will be titrated with the ammonium iron(II) sulfate standard solution. The results will be used to calculate the percentage of ethanol present in the wine.

8A.1 Aims

- To design and perform a titration experiment to standardise (determine the concentration) of a solution of potassium dichromate(VII)
- To use the solution of potassium dichromate to determine the percentage of ethanol in a sample of wine
- To gain experience in the use of redox titrations and stoichiometric calculations

8A.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

A demonstrator will check that you have completed this task before you will be allowed to join the rest of your group to begin the laboratory activity.

1. Read the instructions in the lab manual thoroughly and use other resources (e.g. www.chemguide.co.uk or text books) to learn about redox titrations. Use LabSkills or http://chem-ilp.net to look up any unfamiliar words or phrases and to revise any relevant practical or calculation techniques. Focus particularly on the use of potassium dichromate(VI) in redox titrations, and the use of sodium diphenylamine-4-sulphonate as an indicator.

2. Prepare the lab notebook for the experiment. Then, beginning with the relevant half-equations, construct a balanced redox equation for the reaction between potassium dichromate(VI) and ammonium iron(II) sulphate and write it into the lab notebook. Omit the spectator ions from the final equation. Write down the reason why a solution of potassium dichromate(VI) must be acidified (usually using dilute sulphuric acid) before it can be used as an oxidising agent.

3. Using the brief instructions given in Part 2 of the Laboratory Activity section below and, referring to experimental instructions used for previous titrations (Experiments 3A and 3B), design a titration experiment to standardise (determine the concentration of) the potassium dichromate(VI) solution. This will include calculating the appropriate mass of ammonium iron(II) sulphate to be used in preparing the standard solution, and full instructions for the preparation of the solution and carrying out the titration.

The detailed instructions for this experiment should be written into the lab notebook.

You are provided with:

- solid ammonium iron(II) sulphate, $(NH_4)_2Fe(SO_4)_2.6H_2O$
- potassium dichromate(VI) solution, $K_2Cr_2O_7$, of unknown concentration (assume the concentration is between 0.018 and 0.023 mol dm^{-3})
- sulfuric acid (2 mol dm^{-3})
- aqueous phosphoric acid (8.6 mol dm^{-3})
- sodium diphenylamine-4-sulphonate indicator
- 200 cm^3 volumetric flask and stopper
- 250 cm^3 volumetric flask and stopper
- top pan balances
- analytical balances
- beakers
- glass stirring rods
- hotplate stirrers
- 25 cm^3 burette
- 20 cm^3 pipette
- 25 cm^3 pipette
- pipette filler
- deionised water
- white tile

The usual contents of the lab lockers will also be available for the experiment.

4. Read carefully through all the instructions and prepare carefully for carrying out the required calculations after the experiment.

8A.3 Risk assessment

CHEMICAL RISKS	RISKS	SAFETY
AMMONIUM IRON(II) SULPHATE	Irritating to eyes, respiratory system and skin.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Dispose of in heavy metal waste.
POTASSIUM DICHROMATE(VI) SOLUTION	May cause cancer. May cause heritable genetic damage. May impair fertility. May cause harm to the unborn child. Harmful if swallowed. Toxic by inhalation. May cause sensitization by inhalation and skin contact. Harmful: danger of serious damage to health by prolonged exposure through inhalation. Very Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.	Avoid exposure. Do not breathe gas/fumes/vapour/ spray. Wear suitable protective clothing and gloves. In case of accident or if you feel unwell, seek medical advice immediately (show label where possible). Avoid release to the environment. Dispose of in heavy metal waste.
2M SULFURIC ACID	At this concentration, treat as irritant.	Wear eye protection.
8.6M PHOSPHORIC ACID	Corrosive. Causes burns.	Wear gloves. In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. In case of accident or if you feel unwell, seek medical advice immediately (show label where possible).
SODIUM DIPHENYLAMINE-4-SULPHONATE INDICATOR	Irritating to eyes, respiratory system and skin.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

8A.4 Laboratory activity

This activity will be carried out in pairs. Choose a work area with a free locker and a nearby fume cupboard. Write your partner's name, the locker number and the fume cupboard number at the top of the lab notebook page for this experiment. **Inform the demonstrator of the locker and fume cupboard number.** This experiment has a number of steps so be sure to work efficiently with your partner to get the ethanol in the wine sample reacted with the oxidising agent at the same time as standardising the potassium dichromate(VI) solution. The will only be a limited amount of reagent present for the final titration so use it very sparingly.

Part 1: Oxidation of ethanol in wine using acidified potassium dichromate(VI)

The ethanol in wine will react with oxidising agents to form ethanoic acid. When performed quantitatively, the reaction can be used to determine the ethanol content of wine.

1. In a fume cupboard, turn on a stirrer hotplate (with a heating block) to warm up.
2. Using a pipette, transfer 1 cm^3 of wine to a 250 cm^3 B19 round bottomed flask.

3. Pipette in 100 cm^3 of potassium dichromate(VI) solution.
4. Using a pipette, add 25 cm^3 sulfuric acid (2 mol dm^{-3}).
5. Add a stirrer bar.
6. Set up B19 reflux apparatus. **Ask the demonstrator to check your apparatus**. Reflux the mixture for up to an hour. Note any colour changes.
7. Keeping the condenser attached, take the flask off the heat and leave to cool to room temperature. This can be assisted after a few minutes of cooling in air by using first a cold water bath and then by adding ice to the cold water bath.

Part 2: Standardisation of the potassium dichromate solution using a solution containing Fe^{2+}

This part should be carried out while waiting for the wine sample to reflux. A detailed set of instructions for this part of the experiment, including appropriate masses and volumes of reagents and sizes of required apparatus, should have been written into the lab notebook as part of the pre-lab exercises. The experiment can be carried out on the open bench.

1. Prepare a solution of ammonium iron(II) sulphate with a concentration of close to 0.1 mol dm^{-3} (record the exact concentration of the solution prepared). This solution must contain at least 50 cm^3 of sulfuric acid solution (2 mol dm^{-3}).
2. Place an aliquot of the ammonium iron(II) sulfate solution into a conical flask. Add approximately 20 cm^3 of sulfuric acid (2 mol dm^{-3}), approximately 4 cm^3 of phosphoric acid (8.6 mol dm^{-3}) and 3 drops of sodium diphenylamine-4-sulphonate indicator. Titrate with the potassium dichromate(VI) solution.
3. Use your results to calculate the concentration of the potassium dichromate(VI) solution to an appropriate number of decimal places. **Show your results to a demonstrator.**

Part 3: Determination of the percentage of ethanol in the wine sample

1. When the flask containing the ethanol sample, the sulfuric acid and the potassium dichromate(VI) solution is at room temperature, remove it from the fume cupboard and rinse a clean burette with it (using as little as possible). Fill the burette and record the initial titre. Use the solution sparingly.
2. Pipette a 10 cm^3 aliquot of the standard ammonium iron(II) sulfate solution into a 250 cm^3 conical flask. Add approximately 10 cm^3 of sulfuric acid solution (2 mol dm^{-3}), approximately 2 cm^3 of phosphoric acid (8.6 mol dm^{-3}) and three drops of sodium diphenylamine-4-sulphonate indicator.
3. Take an initial reading from the burette to an appropriate degree of precision. Add the cooled potassium dichromate(VI) mixture to the flask from the burette until the indicator changes colour and note the final titre value. Discard the chromium-containing solution in the heavy metal waste. Record all observations.
4. If the titre value is not sensible, make adjustments to either the pipette size, the burette size or the concentration of the solutions used to obtain sensible results. Ask the demonstrator to assist if required.

5. Repeat the titration until two consecutive concordant results are obtained (or until the sample has run out). Careful use of the refluxed mixture should allow at least five titrations to be performed.
6. Use the titration results to determine the amount of potassium dichromate(VI) left in the 250 cm^3 round bottomed flask after the reaction with ethanol, and thus calculate how much ethanol was present in the wine sample (assuming all of the ethanol was distilled). Thus calculate the % ABV (% alcohol by volume) of the original wine sample. This is a calculation requiring a number of steps. Comment on the value and compare it to the stated % ABV of the wine. Suggest reasons for any discrepancies. Use error propagation to determine the uncertainty in your final answer. **Show your results to a demonstrator**.

Wash and tidy up all the equipment, returning it from where it came from. Ammonium iron(II) sulfate solution and all solutions containing potassium dichromate(VI) should be carefully disposed of in the heavy metal waste. Tidy both the fume cupboard and the bench space. **Have your work space checked off by a demonstrator before leaving the laboratory**.

DISCOVERY
BLOCK 3
EXPERIMENT 8B

A REDOX TITRATION TO DETERMINE THE VITAMIN C CONTENT OF FOODS AND SUPPLEMENTS

8B. A redox titration to determine the vitamin C concentration of foods and supplements

In this experiment, the vitamin C content of different substances is determined using iodate, IO_3^-, as an oxidising agent, in the presence of iodide, I^-, in a redox titration.

Vitamin C, also known as L-ascorbic acid, is an essential nutrient for humans. It cannot be synthesised by the human body and must be consumed. Fruits and vegetables are the primary source of dietary vitamin C. Vitamin C deficiency can lead to the disease scurvy in humans. It is an antioxidant (prevents oxidation) and is often used as a food additive.

Ascorbic acid can be oxidised to dehydroascorbic acid using oxidising agents such as iodine. In this experiment, the iodine is produced by reaction between iodate and iodide and is immediately reduced to iodide by ascorbic acid. Once all the ascorbic acid has been oxidised, the free iodine combines with the starch indicator to form the blue-black starch-iodine complex.

8B.1 Aims

- To perform iodate titrations to determine the vitamin C content of a food supplement.
- To determine the vitamin C content of packaged fruit juice or fresh fruit.

8B.2 Pre-lab exercises

These exercises must be completed in the week before the experiment, and completed at least one hour before the timetabled start time of your laboratory session. You will not be allowed to enter the laboratory until the exercises are completed.

1. Read through the instructions for the whole experiment. Look up any unfamiliar words or phrases (e.g. using LabSkills or http://chem-ilp.co.uk or other resources) and practice any necessary techniques using LabSkills. Prepare the lab notebook for the experiment.

2. When acting as an oxidising agent, iodate (IO_3^-) is converted into iodine (I_2). Write out the half-equation for this reaction. In the presence of iodate, iodide (I^-) is oxidised to iodine. Give the half-equation for this reaction. Combine the oxidation and reduction half-equations to show the overall redox reaction between iodate and iodide.

3. Look up the structure of L-ascorbic acid and L-dehydroascorbic acid. Write the redox half-equation for the conversion of ascorbic acid to dehydroascorbic acid in the lab notebook, showing clearly the structures of the organic compounds.

4. The iodine produced when iodate and iodide react together is reduced by ascorbic acid. The iodine is reduced to form iodide and the ascorbic acid is oxidised. Write down the overall reaction between the iodine and ascorbic acid.

5. Prepare carefully for carrying out the required calculations at the end of the experiment.

6. Bring to the session a sample of fruit or vegetable to be tested for vitamin C content. Approximately 100 g will be required, and the sample food needs to be suitably soft to be ground in a pestle and mortar. Select fruit or vegetables that reportedly have high vitamin C contents are tomatoes, berry fruits or capsicum peppers (bell peppers). An alternative to a fresh fruit or vegetable sample is a sample of commercial fruit juice.

8B.3 Risk assessment

CHEMICAL RISKS	RISKS	SAFETY
2 M HYDROCHLORIC ACID	At this concentration, treat as irritant.	Wear eye protection.
0.002 M POTASSIUM IODATE SOLUTION (KIO_3)	Contact with combustible material may cause fire. Irritating to eyes, respiratory system and skin.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
0.6 M POTASSIUM IODIDE SOLUTION (KI)	Irritating to eyes, respiratory system and skin.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. In case of accident or if you feel unwell, seek medical advice immediately (show label where possible).
STARCH INDICATOR	Minimal.	Minimise exposure.

8B.4 Laboratory activity

This activity will be carried out individually. Choose a work area with a free locker, and write the locker number at the top of the lab notebook page for this experiment. **Show a demonstrator the pre-lab exercises and inform them of the locker number**.

Part 1: Determination of the vitamin C content of a food supplement tablet

1. Record the mass of one soluble vitamin C tablet on an appropriate balance.
2. Prepare a standard solution by dissolving the soluble vitamin C tablet in deionised water and making up to 250 cm^3.
3. Place 20 cm^3 of the standard solution into a 250 cm^3 conical flask. Add 150 cm^3 of deionised water.
4. Add 5 cm^3 of potassium iodide solution (0.6 mol dm^{-3}).
5. Add 5 cm^3 of hydrochloric acid (2 mol dm^{-3}).
6. Add 5 cm^3 of starch indicator.
7. Titrate the sample with 0.002 mol dm^{-3} potassium iodate solution.

8. Repeat until results are concordant.
9. Calculate the concentration of ascorbic acid present in the standard solution and the mass of vitamin C present in the tablet (in mg). Use propagation of errors to determine the uncertainty in your answer and quote it to an appropriate degree of precision.
10. Comment on how this value compares to the reported vitamin C content shown on the container.

Show your results to a demonstrator before continuing on to Part 2.

Part 2: Determination of vitamin C content in fruit, vegetables or fruit juice.

If a solid food sample has been brought to the lab:

1. Cut 100 g of fruit or vegetable into small pieces and grind in a pestle and mortar.
2. Add a few 10 cm^3 portions of deionised water to the sample whilst grinding and decant the liquid into a 100 cm^3 volumetric flask.
3. Filter the ground fruit and pulp through a thin filter paper and wash through with a few more 10 cm^3 portions of water. Collect the filtrate in a 100 cm^3 volumetric flask.
4. Make the solution up to 100 cm^3 using deionised water.

If a sample of juice has been brought into the lab:

1. Filter the juice sample if pulp or seeds are present.
2. Make 50 cm^3 of juice (measured using a pipette) up to 100 cm^3 in a volumetric flask using deionised water.

Follow the method detailed in Part 1 to attempt to titrate 20 cm^3 aliquots of the solution against the potassium iodate solution.

If problems are encountered when performing the titration, such as the titre values being too high or low, dilute the sample or alter the volumes used in the conical flask accordingly or use the juice undiluted.

Once useable results have been obtained, calculate the vitamin C concentration in your sample (in milligrams per 100g of food or milligrams per 100 cm^3 for fruit juice). **Show a demonstrator your results.**

Tidy your workspace and wash out all apparatus. **Sign out with a demonstrator**.

DISCOVERY
BLOCK 3
EXPERIMENT 9A

BOILING POINT ELEVATION

9A. Boiling point elevation

The depression of the freezing point of water upon addition of salt is a phenomenon that is familiar to us all. Equally important in chemistry is the elevation of boiling points upon addition of solutes. Both of these are known as *colligative* properties, which describe how solvents behave upon addition of solutes. Using thermodynamic concepts, it is possible to show that, at low concentrations of solute, the boiling point of the solvent is raised by an amount:

$$\Delta T = K_b b \quad (1)$$

where b is the molality of the solute, and K_b is the known as the boiling point constant. As with all predictions in physical chemistry, this remarkably simple expression, which will be covered in Core Chemistry 2, relies on a number of assumptions.

- The vapour, which is in equilibrium with the liquid, behaves ideally.
- The enthalpy of vaporisation of the solvent is independent of temperature over the range of temperatures concerned; i.e., from the boiling point of the pure solvent to the measured elevated boiling point.
- The liquid can be considered an *ideal solution*. Such a solution is one in which the interaction between a solute and a solvent molecule is the same as (or similar to) interaction between a solvent and another solvent molecule and a solute and another solute molecule.

9A.1 Pre-lab exercises

These exercises must be completed at least one hour before the start time of the laboratory session. Students not completing the pre-lab exercises task will be turned away from the laboratory until the exercises are completed.

1. Read through all instructions. Look up any unfamiliar terms or phrases using http://chem-ilp.net or another resource.

2. Prepare the lab notebook for the experiment in the usual way, then look up and write down the meaning of the term molality. Look up a value for the boiling point constant of water and note it including the reference (where the value was found). Prepare a results table for the experiment.

3. Prepare for the experiment by considering how the boiling point constant will be determined using the data to be collected in the experiment.

9A.2 Aims

- To quantify the magnitude of boiling point elevation upon addition of solutes.
- To determine the *boiling point constant* for water.

9A.3 Risk assessment

CHEMICAL RISKS	RISKS	SAFETY
SODIUM CHLORIDE	Irritating to the eyes.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

9A.4 Laboratory activity

This activity will be carried out in pairs. Choose a work area with a free locker, and write the locker number at the top of the lab notebook page for this experiment. **Show a demonstrator the pre-lab exercises and inform them of the locker number**.

1. Use a 100 cm^3 two-necked, round-bottomed flask with a reflux condenser in one neck and a temperature probe in a thermometer adapter in the other.
2. Measure out 75cm^3 of high purity water. This gives a sufficient depth for the end of the probe to be a couple of centimetres below the surface of the water.
3. Add a spatula of anti-bumping granules to the water.
4. If the NaCl provided is in large crystals, crush them up with a pestle and mortar.
5. Heat the water to boiling point using a stirrer hotplate and a heating block or using a heating mantle.
6. Record the highest stable temperature.
7. Once a reading has been taken, take the flask off the heat to minimise water loss through the condenser.
8. Take the reflux condenser off, when the water has cooled from the boil, and add a known mass (0.2-0.25g) of crushed NaCl to the water.
9. Put the apparatus back on the heat. Record the temperature once the solution is boiling.
10. Repeat steps 7-9 until about 10-12 additions of NaCl have been completed.

Use your results and equation (1) to determine the boiling point constant for water.[1] Use Excel to calculate the standard errors on your answer. Compare your value of the boiling point constant with a value from the literature. Ensure all working and any graphs produced are secured into the lab notebook. **Show your results to the demonstrator.**

[1] Note that it may be necessary to disregard some of the data points from the higher concentrations if there is a noticeable deviation from linearity. This is due to a breakdown of the third assumption discussed above. Despite the apparent simplicity, this is also a difficult experiment to obtain consistent results from, so do not be concerned if there is scatter in the data.

DISCOVERY
BLOCK 3
EXPERIMENT 9B

ELECTROCHEMISTRY

9B. Electrochemistry

In an electrochemical cell, chemical and electrical energy is interchanged. In a galvanic cell a spontaneous reaction takes place that generates electrical energy. Examples of galvanic cells include commercial batteries, fuel cells and many bio-electrochemical processes. An electrolytic cell runs in the reverse direction: it involves a non-spontaneous reaction which is made to occur by driving electrical energy through it. Examples of the use of electrolytic cells include the commercial manufacture of sodium via the electrolysis of molten sodium chloride (the Downs process) and many other manufacturing processes.

9B.1 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Refresh A-level knowledge of electrochemistry (e.g. by using the Redox Equilibria chapter in www.chemguide.co.uk or by using other appropriate resources). Read Chapter 18: Electrochemistry (p636-660) of Housecroft and Constable's *Chemistry* (4th Edition) or Chapters 10.2-10.6 in Atkins and de Paula's *Physical Chemistry*.

2. Prepare the lab notebook for the lab session.

9B.2 Aims

- To gain experience in the elementary principles of electrochemical reactions through a series of experiments with Daniell's cells.

9B.3 Risk assessment

CHEMICAL RISKS	RISKS	SAFETY
1 M COPPER SULFATE SOLUTION	Harmful if swallowed. Irritating to eyes and skin. Very Toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment .	Avoid release to the environment. Dispose of in heavy metal waste.
1 M ZINC SULFATE SOLUTION	Harmful if swallowed. Risk of serious damage to eyes. Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Avoid release to the environment. Dispose of in heavy metal waste.
0.5 M TIN(II) CHLORIDE SOLUTION (CONTAINING CONCENTRATED HYDROCHLORIC ACID)	Harmful if swallowed. Causes burns. Irritating to the respiratory system.	**Wear gloves.** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Wear suitable protective clothing and eye/face protection. In case of accident or if you feel unwell, seek medical advice immediately (show label where possible)
2 M SODIUM CHLORIDE SOLUTION	Irritating to the eyes.	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
METAL ELECTRODES	Sharp edges.	Avoid contact with skin and eyes.

9B.4 Laboratory activity

This experiment is to be carried out in pairs. You will work through a number of examples, construct some different types of galvanic cell, record measurements and investigate and discuss the cells as you work through. Find a partner and a lab locker. **Inform a demonstrator of your locker number**.

Read each procedure carefully and work through the exercises making appropriate notes and observations in the lab notebook.

Part 1: Background and observation of electron transfer

Electrochemical reactions involve the transfer of electrons. In some ways, these processes are similar to the transfer of protons in acid-base reactions, and some electrochemical cells involve proton transfer as one of the steps in the mechanism. The *standard hydrogen electrode* is one such electrochemical cell. There are, however, many important differences between reactions involving the transfer of electrons and proton transfer. This experiment will be focusing on electron transfer reactions. You should be familiar from your previous studies with writing half-equations to show electron loss and gain (oxidation and reduction processes) and combining them into balanced redox equations. Metals tend to undergo *oxidation* reactions and lose electrons. A general half-equation for the oxidation of a metal, M, is:

$$M_{(s)} \rightarrow M^{n+}_{(aq)} + ne^-$$

Conversely metal ions (M^{n+}) can be *reduced*:

$$M^{n+}_{(aq)} + ne^- \rightarrow M_{(s)}$$

The combination of the two processes gives an equilibrium reaction

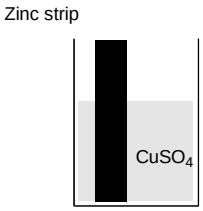
$$M^{n+}_{(aq)} + ne^- \rightleftharpoons M_{(s)}$$

and the potential difference between the metal and the solution of its ions is known as the *electrode potential* (E) of the metal.

You can observe the transfer of electrons between a metal and metal ions in solution by carrying out the following redox reaction

$$Zn_{(s)} + Cu^{2+}_{(aq)} \rightarrow Zn^{2+}_{(aq)} + Cu_{(s)}$$

Dip a piece of zinc in a beaker of 1 M $CuSO_4$ as shown below



Watch carefully and write down any observations in the lab notebook. Do not leave the zinc immersed in the copper solution for too long.

The arrangement of a metal electrode dipped into a solution of its own ions is referred to as a *half-cell*. Two (or more) half-cells can be combined to give a *full cell*. Since each metal has its own electrode potential, connecting two half-cells together results in an overall potential difference and electric current will flow. For reasons of convention, *the half-equation for the process occurring in a half-cell is always quoted as a reduction process*.

In order to make comparisons between half-cells constructed from different materials it is necessary to define a reference point. By convention the chosen reference half-cell is the *standard hydrogen electrode* (SHE). The half-equation for the SHE is:

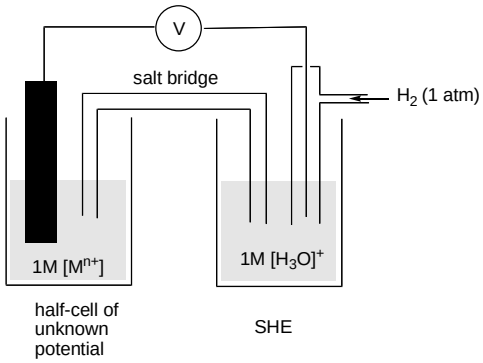
$$2H^+_{(aq)} + 2e^- \rightleftharpoons H_{2\,(g)}$$

You should note that in this cell it is possible for either H_2 to be oxidised or H^+ ions to be reduced. The reaction takes place on a platinum electrode, and while the platinum can help catalyse the reaction, it plays no role in the overall redox process.

Since

$$E^0_{cell} = [E^0_{reduction\ process}] - [E^0_{oxidation\ process}]$$

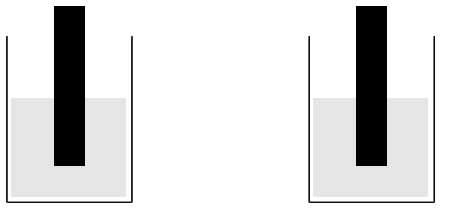
by taking the potential of the SHE as being 0.00V, we can use a cell like the one below to measure the *standard electrode potential* (E^0) of any other half-cell



Because the SHE is rather difficult to operate in a teaching laboratory, in this practical we will use a $Cu|Cu^{2+}$ half-cell as a reference point, and note that the standard electrode potential of this half-cell is +0.34 V. This will be explored in more detail as the practical develops.

Part 2: The Daniell cell

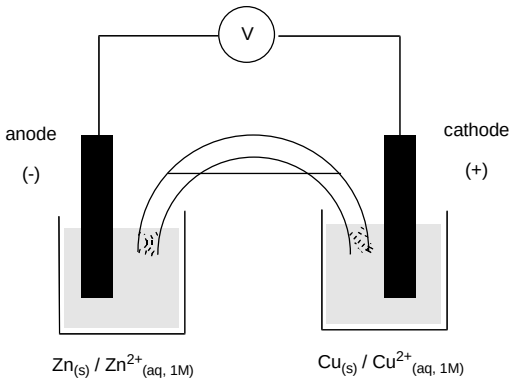
The Daniell cell, named after its inventor, John Daniell, can be made by constructing a copper half-cell and a zinc half-cell and connecting them. Clean a piece of copper and a piece of zinc with the fine emery paper provided. Label these metal electrodes with a dry board marker. Construct the two half-cells by placing the metal strips into a 100 cm^3 beaker containing approximately 50 cm^3 of a solution of the appropriate salt (1 M $CuSO_4$ solution and 1 M $ZnSO_4$ solution). Label the beakers clearly. The half-cells can be drawn out, or represented using cell notation (both shown below):



To connect the cells a *salt bridge* is used. To make the salt bridge, tightly plug one end of a plastic tube with cotton wool, and fill the tube with the solution of 2M NaCl provided. Plug the free end of

the tube with cotton wool and gently form the tube into a U-shape with a rubber band. It is important that solution is not flowing from the tube. The salt bridge acts as a barrier between the two half-cells, but at the same time permits charge-carrying ions to flow between them.

Connect the $Cu_{(s)}|Cu^{2+}_{(aq)}$ and $Zn_{(s)}|Zn^{2+}_{(aq)}$ half-cells as shown, using the crocodile clips and leads provided with the voltmeter to make the connections to the electrodes. Be careful – it is important to connect the voltmeter in the manner specified with the red (positive) and black (negative) leads connected as shown:



In an electrochemical cell, the oxidation reaction occurs at the *anode*, while the reduction reaction occurs at the *cathode*. Given that the diagram of the full cell is labelled correctly, draw it out into the lab notebook and write the cathode reaction half-equation and the anode reaction half-equation underneath the appropriate half-cell.

Instead of drawing a diagram like the one above each time a cell is to be described, it is more convenient to use short-hand notation known as a *cell diagram*. It is important to note that in a cell diagram the reduction process is ALWAYS drawn on the right hand side.

$$Zn_{(s)}|Zn^{2+}_{(aq,\ 1\ M)}||Cu^{2+}_{(aq,\ 1M)}\ Cu_{(s)}$$

The solid bar | represents a boundary between two phases (e.g. liquid and solid). The double bar || indicates the salt bridge.

Copy the cell diagram above into the lab notebook, and record the overall cell potential displayed on the voltmeter.

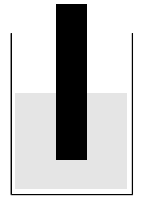
The standard cell potential can be defined in terms of the standard electrode potentials (those potentials compared to the standard hydrogen electrode, SHE) of the two half-cells by:

$$E^0_{cell} = [E^0_{reduction}] - [E^0_{oxidation}]$$

Given that the standard potential of the $Cu|Cu^{2+}$ cell is + 0.34 V, calculate $E^{o}_{Zn^{2+}|Zn}$ and record the value in the lab notebook, showing the calculation clearly. **Show a demonstrator your lab notebook.**

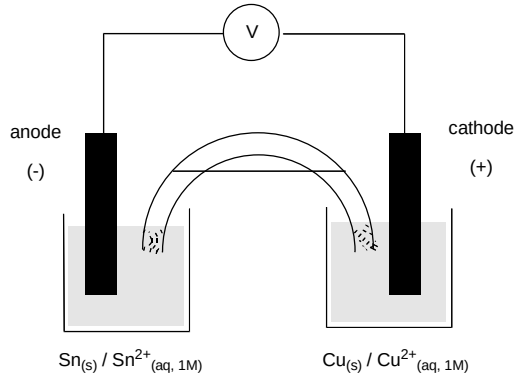
Part 3: Calculation of E^0 for other fuel cells

Carefully disconnect the cell and remove the salt bridge, giving each end a gentle rinse with distilled water. Clean a piece of tin with the fine emery paper provided and construct a half-cell using 0.5 M $SnCl_2$ solution. Label it clearly. [**Care! The $SnCl_2$ is made using concentrated hydrochloric acid. Wear gloves. Avoid contact with skin.]**



$Sn_{(s)} / Sn^{2+}_{(aq, 1M)}$

Construct the following electrochemical cell:



Describe this cell in the lab notebook in terms of a cell diagram. Record the cell potential. Calculate the standard potential of the tin half-cell. **Show your results to the demonstrator.**

The half-cells we have been discussing are all written as reduction reactions, and the potentials (E^0) calculated can be more accurately described as *standard reduction potentials*.

Consider the two half-cells which have not yet been examined in combination as a full cell:

$$Zn^{2+} + 2e^- \rightleftharpoons Zn_{(s)}$$
$$Sn^{2+} + 2e^- \rightleftharpoons Sn_{(s)}$$

Before constructing the cell write the two cell diagrams which describe the two possible cells that can be constructed from the zinc and tin half-cells into the lab notebook. Give the half-equations for the reaction at the anode and the cathode for each cell (stating which is oxidation and which is reduction), and calculate the overall cell potential in each case.

By convention we always write the cell so that the *spontaneous* reaction is read from left to right. A spontaneous cell in this form will have a positive value of E^0_{cell}.

Assemble the cell. The salt bridge may need additional 2M NaCl adding to it. Record the observed value of E^0_{cell}. Comment on how the calculated value of E^0_{cell} compares with the experimentally observed value. Indicate which of the two cell diagrams describes the spontaneous reaction. **Show your results to a demonstrator.**

Part 4: Gibbs energy changes for cell reactions

The thermodynamic driving force of the reaction (or Gibbs energy change, ΔG^0) can be readily calculated from E^0_{cell} as:

$$\Delta G^0 = -z\,F\,E^0_{cell}$$

where *z* refers to the number of moles of electrons transferred per mole of reaction and *F* is the Faraday constant = 96 485 C mol^{-1}.

Write the cell diagram for each of the three cells you have constructed and calculate ΔG^0 for each one (giving units). If the cell diagrams are written correctly, such that the spontaneous reaction is read from left to right, the E^0_{cell} should have *positive* values in each case. **Show your results to the demonstrator.**

Part 5: A concentration cell and the Nernst Equation

Build the following cell, paying close attention to the concentration of solutions. The 0.100 M solution of $CuSO_4$ will need to be prepared by dilution of the 1 M stock solution. Measure and record the cell voltage for this whole cell in the lab notebook.

$$Cu_{(s)} \mid Cu^{2+}_{(aq,\ 0.1\ M)} \mid\mid Cu^{2+}_{(aq,\ 1M)} \mid Cu_{(s)}$$

The potential difference in the full cell arises because of the dependence of each half-cell on the *concentration* of the reagents. The relationship between concentration and potential is given by the *Nernst Equation*:

$$E_{halfcell} = E^0 - \left\{ \frac{RT}{zF} \times \left(\ln \frac{[reducedform]}{[oxidisedform]} \right) \right\}$$

where:

$E_{halfcell}$ is [$E_{Cu/Cu2+}$ 0.1M] (i.e. the effective half-cell potential at the concentration employed)

E^0 is the standard cell potential (i.e. at concentration = 1 M)

R is the molar gas constant (8.314 J K^{-1} mol^{-1})

T is the temperature (decide which units are most appropriate)

z is the number of electrons transferred per mole of reaction

F is the Faraday constant (96 485 C mol^{-1})

In the case of a metal ion | metal half-cell, the concentration (or more accurately, the *activity*) of the metal (i.e. the reduced form) is taken to be 1.

Calculate the potential of the $Cu_{(s)}$ | $Cu^{2+}_{(aq,\ 0.1\ M)}$ half-cell and then the cell potential of the full cell. Compare this with the experimental value and comment on any significant difference in the values. **Show your result to the demonstrator**.

Empty your salt bridge, and rinse the tube thoroughly with deionised water. Place all metal ion solutions in the appropriate waste containers, and rinse beakers with deionised water. Clean and dry the metal strips and return them. Remove the zinc strip from the beaker, and clean it in a solution of dilute hydrochloric acid. Wash and tidy away any other glassware.

Using prior knowledge (and knowledge gained during this experiment), write down why the acidic solution effectively removes the deposit of copper metal from the zinc electrode.

Show a demonstrator your answer and have the work area signed off before leaving the laboratory.

DISCOVERY
BLOCK 3
EXPERIMENT 10A

PREPARATION OF AND COMPLEXOMETRIC TITRATION ANALYSIS OF A 1,2-DIAMINOETHANE COMPLEX OF NICKEL

10A. Preparation and complexometric titration analysis of a 1,2-diaminoethane complex of nickel

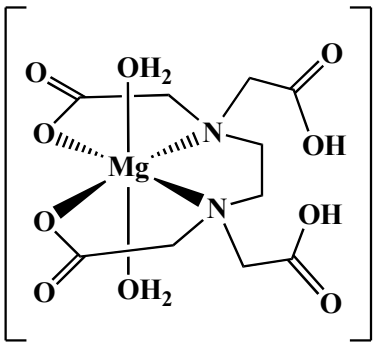
This experiment follows on from the preparation of the nickel complexes in Experiment 5A in Block 2. Good working practices for the use of nickel compounds were developed during this previous experiment and are expected to be followed at all times during these exercises.

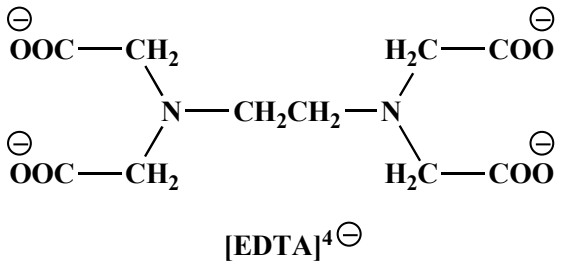
For a ligand to be useful as a titrant it must form strong complexes with metal ions and the equilibrium between the metal, ligand and complex must be highly in favour of the formation of the complex, so that the reaction is quantitative within the limits of error of the experimental procedure used. In other words, the equilibrium must lie to the right and K must be very large:

$$[M]^{2+} + L \overset{K}{\rightleftharpoons} [ML]^{2+}$$

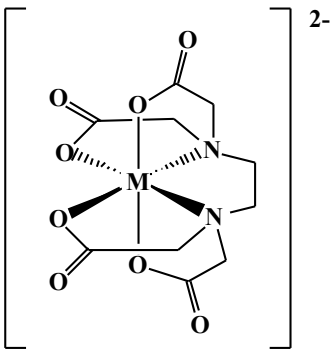
K = equilibrium constant (also known as the stability constant in this context)

Monodentate ligands are not usually suitable. A group of aminopolycarboxylic acid polydentate (or multidentate) ligands have been found to be ideal. Polydentate ligands are also known as chelating ligands and are designed to occupy up to 6 coordination positions depending on the requirements of the metal. The most common ligand titrant is ethylene diaminetetraacetic acid (EDTA). This





molecule can lose two protons to form $EDTA^{2-}$, which occupies four coordination sites around magnesium in solution, or $EDTA^{4-}$ (ethylenediaminetetraacetate), which occupies six sites around many first row transition metals, i.e.



Complexes containing polydendate ligands featuring five- or six-membered rings that include the metal centre are very stable. Thus the most useful ligand titrants are those that form many five- or six-membered rings, and in doing so fully saturate the coordination requirements of the metal ion. $EDTA^{4-}$ is one such ligand. It forms a 1:1 complex with many metal ions almost instantly. Note that EDTA forms bonds to metal centres not only through the nitrogen atoms but also through the oxygen atoms of the acetate (ethanoate) groups.

The stability constant, K, for such a process is:

$$[M]^{2+} + [EDTA]^{4-} \underset{}{\overset{K}{\rightleftharpoons}} [M(EDTA)]^{2-} \qquad \text{where } K = \frac{[M(EDTA)^{2-}]}{[M^{2+}][EDTA^{4-}]}$$

In general, any ligand that forms a 1:1 complex with a stability constant (K) greater than 10^7 may be used in direct complexometric titrations. If the value of K is less, the equilibrium would lie too far to the left and one would not expect to be able to carry out direct complexometric titration with a standard solution of the complexing agent.

In this experiment, you will prepare a complex of nickel and use complexometric titration with EDTA to determine the nickel content of the complex and thus the formula.

10A.1 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read carefully through all the information in the laboratory manual for this experiment, including Appendix 1, and look up any terminology that is unfamiliar.
2. Prepare the lab notebook. Note down the meaning of the term 'complexometric titration' when used in volumetric analysis. Draw the structure of 1,2-diaminoethane in full and state what type of ligand it is (e.g. monodentate, bidentate, tridentate etc.).[2]
3. Prepare a risk assessment document for Part 1 of the experiment. Write it onto on the left hand side of the lab notebook. Use previous risk assessment tables that have been provided in the laboratory manual as a template. The table should list all the chemicals used in Part 1 of the experiment, the R and S (Risk and Safety) numbers associated with that compound and the R and S phrases written out in full. Use the MSDS (Material Safety Data Sheet) documents to identify the appropriate R and S numbers. Cross reference these numbers with the lists of R and S phrases provided in DUO and copy them out in full into the table. Note that the R and S numbers are not normally given in your laboratory manual, but they should be included in your own risk assessment.

[2] It may be useful to refer to G. Schwarzenbach, 'Complexometric Titrations', Methuen, 1960, p. 79-82 or A. I. Vogel, 'Quantitative Inorganic Analysis', 3rd Ed., Longmans, 1962, p. 435-6.

10A.2 Aims

- To prepare a 1,2-diaminoethane complex of nickel
- To implement good laboratory practice for handling toxic and carcinogenic chemicals
- To construct a risk assessment for an experiment
- To standardise a solution using EDTA in a complexometric titration
- To analyse a nickel complex of 1,2-diaminoethane using a complexometric titration

10A.3 Risk assessment

Part 1

The risk assessment for Part 1 of the experiment is to be prepared and written into the lab notebook as part of the pre-lab exercises.

Part 2

CHEMICAL	RISKS	SAFETY
MAGNESIUM SULPHATE HEPTAHYDRATE	Minimal.	Do not breathe dust. Avoid contact with skin and eyes.
ETHYLENE DIAMINE TETRAACETIC ACID (EDTA) SOLUTION	Irritating to the eyes. Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. Avoid release to the environment. Refer to special instructions safety data sheet
BUFFER SOLUTION (pH = 10)	Corrosive. Irritant.	Do not breathe gas/fumes/vapor/spray. Avoid contact with skin and eyes.
ERIOCHROME BLACK T INDICATOR SOLUTION	Irritating to eyes, skin and respiratory system.	In case of contact rinse immediately with plenty of water. Wear gloves when handling.

Part 3

CHEMICAL	RISKS	SAFETY
CONCENTRATED HYDROCHLORIC ACID	Causes burns, irritating to eyes, skin and respiratory system.	In case of contact rinse immediately with plenty of water. Wear suitable protective clothing and gloves when handling.
MUREXIDE INDICATOR	Treat as toxic.	Harmful if swallowed. Avoid contact with skin and eyes.
CONCENTRATED AMMONIA	Flammable. Toxic by inhalation. Causes burns. Very toxic to aquatic organisms. Can cause irreparable damage, particularly to eyes.	Use only in fume cupboard. Wear suitable protective clothing, gloves and eye/face protection. Keep away from sources of ignition.

10A.4 Laboratory activity

Work in pairs for this experiment. Choose a work area with a free locker and select a fume cupboard. Write the number of these at the top of the lab notebook page for this experiment. Work in a fume cupboard throughout. **Inform the demonstrator of the locker and fume cupboard number**.

Part 1: Preparation of a 1,2-Diaminoethane-Nickel Complex

Work in a fume cupboard throughout.

1. Dissolve $NiCl_2.6H_2O$ (3.0 g) in deionised water (about 4 cm^3).
2. Add 70% 1,2-diaminoethane (ethylenediamine) (3.5 cm^3).
3. If necessary (i.e. if any insoluble material remains), filter the solution to remove any iron oxide impurities.
4. With gentle stirring slowly evaporate the purple solution to a volume of approximately 4 cm^3.
5. Add one further drop of 1,2-diaminoethane, then allow the solution to cool slowly to room temperature.
6. Cool to ice temperature and await the formation of purple-coloured crystals.
7. Collect the solid product by vacuum filtration, wash twice with acetone and dry in the air.
8. Record the mass of the product.

If the yield is low, further amounts of the product may be obtained from the mother liquor by dropwise addition of a small volume of ethanol followed by cooling in ice. Repeat this process of addition of alcohol and cooling until product formation starts again, then leave at ice temperature as long as possible.

Part 2: Standardisation of EDTA solution using Mg^{2+}

1. Prepare a standard solution of magnesium sulphate using accurately about 0.25 g $MgSO_4.7H_2O$ in a 200 cm^3 volumetric flask.
2. Add a 20 cm^3 aliquot of the standard solution to a 250 cm^3 conical flask. Add 2 cm^3 of buffer solution (pH = 10), approximately 40 cm^3 deionised water and 2-3 drops of Eriochome Black T indicator solution.
3. Titrate with the EDTA solution (which is approximately 0.005 M).
4. Repeat until concordant results are obtained.
5. Calculate the concentration of the EDTA solution, reporting it to an appropriate number of decimal places. **Show your results to the demonstrator before proceeding**.

Note 1: Since complex formation does not occur instantaneously, the titration must be carried out slowly near to the end point.

Note 2: If there are problems determining the end-point, ensure the apparatus is in the fume cupboard and, during the titration, add 1-2 drops of concentrated ammonia solution.

Note 3: For comparison, it is advisable to have solutions at hand that represent the two colours of the indicator. These may be prepared as follows: To two boiling tubes add an equal volume of the magnesium solution (approximately 2 cm^3), the same volume of water, a few drops of buffer solution (pH = 10) and a drop of the indicator. Then to one of the tubes add EDTA solution slowly until the indicator changes colour.

Part 3: Determination of nickel content of the 1,2-diaminoethane complex

Work in a fume cupboard.

1. Weigh out accurately about 0.31 g of dried 1,2-diaminoethane complex (prepared in Part 1) into a 100 cm^{-3} conical flask.
2. Carefully add concentrated hydrochloric acid (10 cm^3)
3. Boil for 10 minutes. Allow the solution to cool.
4. Carefully dilute with deionised water (approximately 100 cm^3).
5. Transfer to a 200 cm^3 volumetric flask and transfer all washings. Make up to the mark with deionised water.
6. Pipette a 20 cm^3 aliquot of nickel solution into a 250 cm^3 conical flask. Add approximately 100 cm^3 of deionised water and a small quantity of solid Murexide indicator sufficient to give a peach-yellow colour. Add concentrated ammonia dropwise, testing the pH after additions, until the pH is 7 and the colour of the solution becomes lemon-yellow. [Hint: at the appropriate pH, the colour changes with one drop.
7. Titrate with the EDTA solution. After approximately 15 cm^3 of EDTA has been added, make the solution back to pH $\geq$ 10 by dropwise addition of concentrated ammonia. Continue to add EDTA until the colour changes to magenta (red-purple), adding further drops of concentrated ammonia during the titration if the colour returns to peach-yellow. Just before the end point, add additional concentrated ammonia (approximately 5-10 drops).
8. Repeat the titration until concordant results are obtained.

10A.5 Post-lab assignment

The results of this experiment should be typed up into a report and submitted electronically via DUO within one week of the end of the laboratory session. Reports should be prepared individually and one report should be submitted per student. Joint submissions are not accepted.

The report should show your name, your partner's name and the title. It should be written in continuous prose and use the passive voice. It should be written as though the reader has no access to the laboratory manual and so should be a standalone piece of work. Begin with a short 'Introduction' section, including a paragraph summarising the experiment and some background about complexometric titration and the indicators used. Do not copy text from the laboratory manual or any other sources (except your lab notebook!). The 'Results and Discussion' section should summarise the results for each part of the experiment (include weighings recorded to an

appropriate number of decimal places, results tables with appropriate significant figures and all units and appropriate calculations with working, all presented in an appropriate format. Observations should also be included with the results).

Under each set of results, the appropriate calculations should be performed and any questions answered (detailed below).

Part 1 Results and Discussion

As well as including results and observations with any appropriate comments, you should answer the following questions:

1. The density of ethylenediamine is 0.899 g/mL and the Mr of Ni is 58.69 g mol^{-1}. Determine the molar ratio of nickel to ethylenediamine in the final product.
2. Suggest a likely formula for the unknown nickel-ethylene diamine complex.
3. Indicate any isomers that could be formed:

Part 2 Results and Discussion

1. Calculate the concentration of the magnesium sulfate solution and include the working.
2. Determine the concentration of the EDTA solution, including working.

Part 3 Results and Discussion

1. Report the mass of your nickel complex used in the analysis.
2. Calculate the % Ni in the complex (showing all working) and hence the relative molecular mass and suggest its formula from your knowledge of possible ligands and counter-ions present.
3. Compare the % Ni found with the theoretical % Ni in likely complexes. The complex is of the general form $M_xNiX_y.nH_2O$, or $NiL_mX_2nH_2O$, where M = cation, L = neutral ligand, X = anion or anionic ligand (e.g. halide).
4. Suggest the structure for the complex and indicate clearly which species are ligands bound to the nickel centre, which species are counter ions, and if there is any solvent of crystallization present.
5. Calculate a % yield for the nickel ethylenediamine complex based on the formula determined and the mass of nickel chloride used in its preparation.

The report should also include an error analysis of the results, and should state any references used. Reports should be concise. Marks will be penalised for over-long reports. Do not copy out the laboratory manual. Draw any chemical structures using e.g. ChemDoodle (available via the NPCS computers).

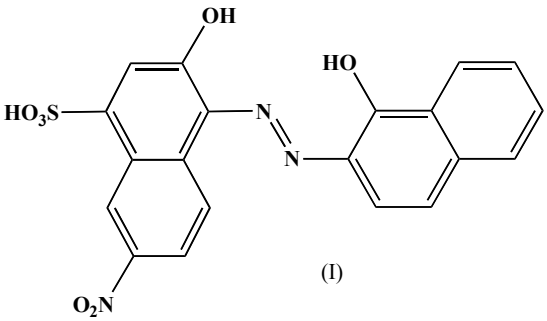
Reports must be submitted electronically via DUO within one week of the end of the laboratory session. Late submission will score zero marks. This report is worth 20% of the Core Chemistry 1A practical marks for the first year and should demonstrate knowledge gained from a wide variety of 'SKILLS' experiments during Block 1 and 2.

Appendix 1: Indicators for complexometric titrations

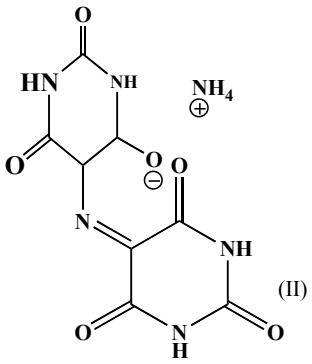
The indicator used in complexometric titrations is itself a ligand (a complexing agent) that forms a distinctly coloured complex with the metal ion being titrated. Such direct action indicators should possess the following properties:

1. The free indicator must possess a different colour from the metal/indicator complex.
2. The metal/indicator complex must be formed under the same pH conditions as the metal/EDTA complex.
3. The indicator must be very sensitive towards the meal ions so that only a small amount of it is necessary for titration.
4. The metal/indicator compound must be considerably less stable than the metal/EDTA complex, but not so weak as to dissociate appreciably in the vicinity of the end-point when the concentration of free metal ions becomes very small. Non-adherence to either condition will lead to diffuse end-points with high results in the first instance and low in the second.
5. The reaction between the indicator and metal ions must be rapidly reversible.

Two indicators used in complexometric titrations are shown below:



(I)



(II)

Eriochrome Black T, structure I, is the indicator used in the titration with the standard Mg^{2+} solution. Possible coordination sites of the indicator are the two imino nitrogens, the two hydroxyl groups and the sulphonic acid group.

Murexide, structure II, forms a pure yellow complex with nickel(II). The nickel-indicator complex may also be used as an acid-base indicator to adjust the pH, and is also used in the experiment for this purpose.

DISCOVERY
BLOCK 3
EXPERIMENT 10B

THE IONIC MODEL

10B. The Ionic Model

This experiment is a 'dry' practical. Details will be provided on the day you complete the experiment. Check the rota carefully to ensure you are aware when you will be performing this experiment. It may not be at the time you would normally expect to attend.

The instructions for this experiment and details of the write-up, which is summative, are given on the sheets you will receive during the session. Attend the lab promptly when you are scheduled to complete this experiment and meet the demonstrator outside of the laboratory. Bring only your safety spectacles; do not attend wearing your lab coat.

All work will be completed within the laboratory session (but not necessarily in the laboratory).

10B.1 Aims

- To reiterate the concepts and ideas learned during the Ionic Model lecture course.

10B.2 Pre-lab exercises

These exercises must be completed at least one hour before the timetabled start time of the laboratory session. Students not completing the pre-laboratory task will be turned away from the laboratory until the exercises are completed.

1. Read and revise your lecture notes from the 'Ionic Model' lecture course given by Dr I. Evans.
2. Read the relevant sections of chapter 5 of 'Inorganic Chemistry' by Housecroft and Sharpe, Second Edition, Prentice Hall, 2005.

You are not required to prepare anything in the lab notebook for this experiment. You may, if you wish, prepare some notes on the relevant topic in your lab notebook before the session, but this is not compulsory.

10B.3 Risk assessment

This experiment has minimal risk.

10B.4 Laboratory activity

Arrive promptly outside the laboratory to meet the demonstrator. Bring safety spectacles but do not bring lab coats. All additional instructions will be provided.

APPENDIX A: Assessment guide for laboratory reports

Reports will be assessed against the following criteria, which are not necessarily equally weighted.

	Structure	**Presentation**	**Technical Content**	**Results and Discussion**
First Class	Excellent, very clear, logical subdivision.	Well written in good English, cogent arguments presented. Conclusions concur with results obtained, results are clearly summarised.	Appropriate theoretical background included. Proper use made of theory expressions, etc.	Critical assessment of the results. Quality of sample based on data (spectra, errors). Graphs neatly plotted and correctly interpreted. Extended interpretation based on analysis of theory section.
Upper Second	Well organised easy to follow and a sense of direction throughout.	Clearly laid out, conclusions and summary evident and clearly written.	Good grasp of the necessary theory and its use.	Results analysed and assessed in sufficiently critical manner. Evidence of an appreciation of sources of error.
Lower Second	Satisfactory but some loss of way evident.	Straightforward to read, satisfactorily written, vagueness or hesitancy in conclusions and summary.	Only the basic theory behind the experiment is presented, no evidence of real understanding.	Satisfactory assessment of results and outcome of experiments. Critical evaluation not overly evident.
Third	No direction, no subdivision. Lack of clarity.	Somewhat disorganised and hard to read. Conclusion and summary incorrect or “off the mark”.	Gaps in understanding evident from what was presented.	Poor analysis of the results or sample quality, no attempt to assess sources of error or where things may have gone wrong.
Fail	No evidence of any organisation, absence of basic understanding, no coherence.	Difficult to read; slap dash presentation, absence of conclusion or summary.	No real presentation of background and its appreciation.	No assessment of the results, no discussion.