

First Year Undergraduate Chemistry Laboratory Course Manual 2011-2012

Core Chemistry 1A and 1B: Induction

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Core Chemistry 1A and 1B
First Year Laboratory
Course Manual
2011-2012

INDUCTION ACTIVITIES
WEEK 1

Name

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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, cosmetics and cigarettes 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 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.

Induction activity 1A₁: Using a lab notebook

During practical sessions this year, a lab notebook will be used to record all experimental data. The notebook should be an A4 glue-bound hard-backed book and should have been purchased from Chemistry Stores before the start of the course. This exercise is a paper-based group task to teach how the lab notebook should be used.

The lab notebook itself will not be used in this first laboratory session. From the second laboratory session of the year, however, its use is compulsory. Marks will be awarded for continued good use of the notebook throughout the year.

Tasks

Split into groups of four. Each member of the group to take it in turns to introduce themselves, state their name, their college and explain a bit about themselves (e.g. where they are from). (5 minutes)

Look at a copy of Notebook Sample Entry 1. This is a bad example of notes made in a lab notebook. Read it through carefully then discuss with the other members of the group how the lab notebook entry could be improved. (15 minutes)

Within the group, decide upon a set of good practice guidelines for keeping a good lab notebook during a laboratory class and write them down on the A3 paper using the marker pen. (10 minutes)

Analyse a copy of Notebook Sample Entry 2 and see how many of your good practice guidelines it satisfies. Discuss in the group other improvements that could be made to the notebook entry, and use this discussion to amend the good practice rules appropriately. (10 minutes)

Show the amended and finished set of good practice guidelines to a demonstrator and ask for a copy of the document entitled 'Use of a lab notebook'. Read it carefully and check off how many of the good practice guidelines developed by the group feature in the document. Highlight any omitted guidelines and note them. (10 minutes).

Post-lab exercises

Securely stick a neat copy of the 'Use of a lab notebook' document into the back cover of the lab notebook. Copies will be available in the lab or they can be printed out from DUO. This document should be referred to throughout the laboratory course to ensure the lab notebook is completed appropriately.

Collect or print off from DUO a copy of the laboratory safety rules ('Safety in the First Year Laboratory CG 021') and ensure that they are stuck securely in the front cover of the lab notebook.

Write your personal details on the first page of your lab notebook following the instructions given in the 'Use of a lab notebook' document. Complete these tasks before your next laboratory session.

Use of a lab notebook

A lab notebook is a record of all the work carried out in the laboratory. In industry or in academic research, it is an important legal document that can be used to provide evidence about the discoverer or date of discovery of new chemicals or processes. In the undergraduate laboratory course it is important to develop the skill (or art!) of writing a good lab notebook. The records will be needed to generate lab reports at some points in the course, and the keeping of the lab notebook will be assessed regularly. The lab notebook should be purchased from Chemistry Stores before the start of the course. It needs to last the year so look after it and ensure that all additional sheets are glued/taped/stapled securely.

The requirements for keeping a good undergraduate lab notebook are summarised below.

How a good lab notebook is organised

The first page of the lab notebook should be used as a cover page and should include name, course, college, chemistry advisor and email address (so it can be returned in case of loss). The second page should be left blank to be used as a contents page. This page should be completed as the lab course progresses. Begin to write experimental data into the lab notebook from the third page onwards. Only write on the right hand side page. The left hand side page is to be used for a risk assessment of the chemicals used and for noting down other information e.g. adaptations to the original method. Make sure all pages are numbered and dated. All writing in the lab notebook should be in ink (blue or black). A ball point pen is better than a fountain pen as it is less likely to smudge if water is splashed on it.

Lab notebooks need to be looked after carefully. Do not soil them with chemicals as they may transfer hazardous substances out of the laboratory. In the first year laboratory, there are pull-out lab notebook work stations along each bench to ensure a chemical-free environment for completing the notebook. Space all work out well on the page so that the information is clear to anyone reading it.

What to include

The lab notebook is not a copy of the contents of your lab manual. It should expand upon the instructions given in the lab manual. The experiment title and reference (given in the lab manual) will allow cross referencing of the experiment notes with the information given in the lab manual. It is important to include the name(s) of any lab partners or group members and the date so that work can be monitored. If a second page is required, begin it with the experiment title and reference and the date, before continuing with the experiment notes.

The aim of the experiment should concisely explain the task for that lab session e.g. 'Aim: to synthesise $\text{Fe}(\text{acac})_3$ '. If it is a synthesis experiment (where a product is made), a (correct!) chemical equation for the reaction should be provided.

The experimental plan explains precisely what is to be done in that session. Occasionally, detailed experimental plans are provided in the lab manual and the lab notebook can state that these were followed directly. If the lab manual provides only an outline method, a more detailed method should be prepared in the lab notebook before entry to the lab session so that work can begin immediately. For experiments that require the development of a method before the lab session begins, editing may be needed during the session if changes are made. These changes should be clearly noted on the left hand side page alongside the method during the lab session. When writing a method, use clear language and simple direct statements in a

numbered list so that instructions can be followed easily in the laboratory. Do not use personal pronouns (such as 'I' or 'we'). The experimental plan section should also be used to note any special safety instructions given in the laboratory manual, or to write a risk assessment for the chemicals used in the experiment. A diagram should be used to illustrate novel or unfamiliar apparatus, and should show the cross-section of the equipment. Keep it simple. Label where appropriate. Do not use diagrams for common apparatus or procedures.

Observations, measurements and data should be recorded immediately and in full (with units, where relevant). Take the lab notebook to the balances to record masses. Do not use scraps of paper and then transfer the data to the lab notebook later. Never use Tipp-Ex. Never write over errors; simply cross them out neatly with a single line and write the correction alongside. Record all observations, measurements and data honestly. The lab notebook is a record of exactly what was observed and measured, not what is predicted to happen or be observed. When working in pairs or groups, ensure that an individual record of all the observations, measurements and data, is kept at the time of the experiment. If data are entered directly onto a spreadsheet, ensure that a copy is printed off and stuck into the lab notebook immediately afterwards. Do not copy data from someone else after the experiment. If data are to be shared with a partner or group, clearly flag the observations and data as belonging to someone else. Data should be recorded in a table, where possible, and the table should be written in vertical columns using headers and units at the top of each column. Individual cells in the table should only contain a number; units only appear in headers.

A graph or graphs may be necessary for the experiment. Graphs should be generated from a table of data and should usually be hand drawn in the first instance. Each graph should be drawn on graph paper, should have the experiment title and date written on it, and should be stuck in to the lab notebook as soon as possible. Axes should be labelled with the quantity and the unit. If possible, give error bars on each point. Always use a ruler to draw straight lines.

The discussion section needs to include clear presentation of any calculations with working that can be followed by an assessor reading the lab notebook. Use words to explain the meaning of different steps, and include units throughout. Comments should be made about how the results relate to any hypotheses or how they answer a question posed in the experimental aims. The conclusion should state the experimental findings and should include any error analysis and any notes about unusual findings or improvements that could be made if the experiment were to be performed again.

Lab notebook checklist

This list is not intended to be comprehensive, but it is a very good place to start!

Lab notebook entries for experiments should contain the following before they are handed to a demonstrator for marking at the end of each session:

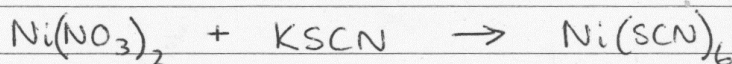
- Experiment title and reference number(s).
- The date in full.
- The names of any laboratory partners or group members.
- Aim of the experiment.
- Equations, where required
- Experimental plan/method and explanation of any experimental decisions.
- Diagram of equipment (if required).
- Observations and comments on the chemistry.
- Tables of data (where required) with column headings and units (no units in cells), either written directly into the lab notebook or printed off from Excel and securely attached.
- Graph of data (where required), either hand-drawn onto graph paper and stuck in or prepared in Excel, printed off and attached securely. Remember to include a title and label axes (including units). Graphs should be self-explanatory and not need notes from other pages of the lab notebook to be understood by an observer.
- Discussion of key ideas and answers to questions posed in the laboratory manual.
- Conclusion.

Use ink. Work clearly and legibly. Show working.

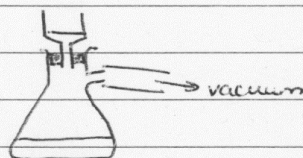
Notebook Sample – Entry 1

2/4/11

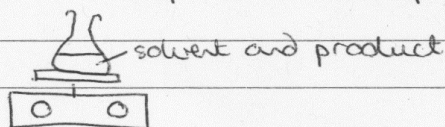
3A Making a nickel complex



I put the green nickel nitrate into 3 cm³ of hot ethanol. I mixed it with the solution of potassium ~~thiocyanate~~ thiocyanate I'd made earlier. After cooling, I filtered it using Buchner apparatus.



I then added 3.8 grams of KSCN in ethanol and water and used a hotplate to evaporate the solvent.



When 5 ml was left, I cooled it ~~and~~ until my crystals formed then I filtered it again.

Mass of nickel salt: 3.0

Green crystals gave green solution. Got crystals Yield OK.

Mass of product: 2.6 g % yield 23

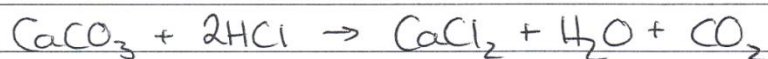
Blueish purple crystals (but Jane's were more blue than mine).

Notebook Sample – Entry 2

25th October

Experiment 4A : Determination of the rate of reaction between CaCO_3 and HCl .

Aims : • to determine the rate of reaction when CaCO_3 reacts with HCl :

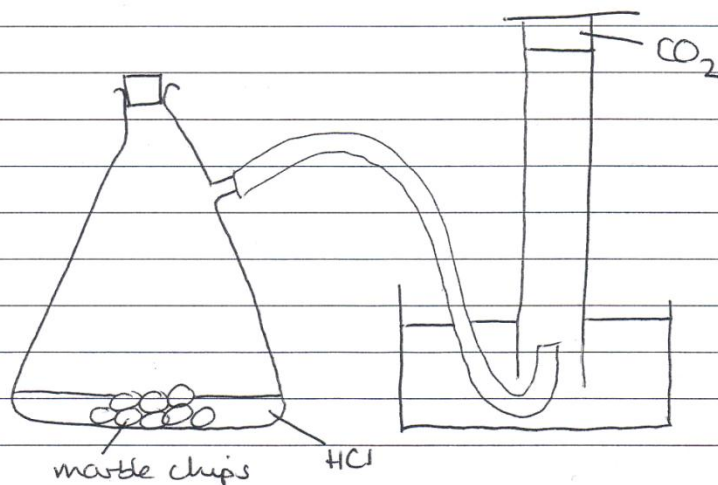


• to determine the effect of varying acid concentration on this rate

Plan : instructions followed on p16 of lab manual

Concentrations of acid used : 0.5 mol dm^{-3}
 1.0 mol dm^{-3}
 2.0 mol dm^{-3}

Apparatus :



Notebook Sample – Entry 2 (continued)

Results

0.5 acid:

1.0 acid

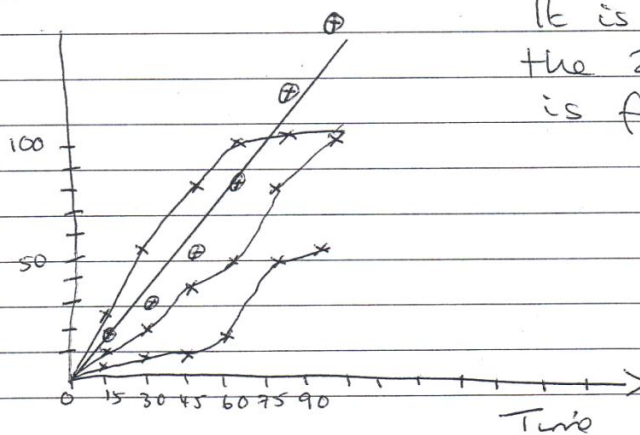
<u>Time</u>	<u>Volume</u>	<u>Time</u>	<u>Volume</u>
0	0	0	0
15	3 cm ³	15	10
30	9 cm ³	30	22.2
45	10 cm ³	45	36
60	15 cm ³	60	49
75	50 cm ³	75	79.0
90	55 cm ³	90	more than 100

2.0 acid

<u>Time</u>	<u>Volume</u>
0	0
15	25 ml
30	53 ml
45	80 ml
60	over 100 ml
75	over 100 ml
90	over 100 ml

Conclusion

Graph



It is obvious that the 2.0 acid is fastest.

Induction activity 1A₂: Determination of the density of brine

Introduction

The density, ρ , of a substance is the ratio of mass to volume ($\rho = m / V$). The density of a solution can vary with the concentration of the dissolved solute and with temperature. In this activity the mass and volume of brine (sodium chloride aqueous solution) will be measured, and the density of the solution will be calculated. This value will be compared to the values calculated by other members of the group. The accuracy and precision of the measurements will also be determined.

Aims

- To become familiar with working in the undergraduate laboratory, including familiarisation with the layout of the laboratory and some of the apparatus available
- To determine the density of a sample of brine (sodium chloride solution)
- To consider the accuracy and precision of the density determination

Equipment

50 cm³ beaker
250 cm³ beaker
10 cm³ measuring cylinder
50 cm³ measuring cylinder
10 cm³ pipette
25 cm³ pipette
top pan balance
analytical balance
thermometer
brine (sodium chloride solution, 2 mol dm⁻³)

Procedure

Work in pairs or threes. Divide the work up between members of the group to ensure that everyone uses each piece of apparatus and that work is completed efficiently. This is a task designed to take less than one hour and there is only 45 minutes allocated to measurements. All practical work and clearing up should be completed in this time, leaving 10-15 minutes to analyse the results. If time is running out, limit the number of trials.

1. Place ~100 cm³ of sodium chloride solution in a beaker. Measure the temperature of the solution and record it in the table.

Beaker and top pan balance

1. Place a clean dry 50 cm³ beaker on the top pan balance. Tare. ['Tare' means zero the display – usually by pressing the 'Tare' button].
2. Remove the beaker from the balance and pour a small amount of sodium chloride solution into it. Using the graduations on the beaker, read the volume and record it in the table.

- Place the beaker on the top pan balance and record the mass of the sodium chloride solution in the table.
- Repeat for trials 2 & 3.

Measuring cylinder and top pan balance

- Place a clean dry 50 cm³ beaker on the top pan balance. Tare.
- Pour a small amount of sodium chloride solution into a 10 cm³ measuring cylinder. Using the graduations on the cylinder, read the volume as precisely as possible and record in the table.
- Pour the sodium chloride solution into the beaker and place on the balance. Record the mass in the table.
- Repeat for trials 2 & 3.

Pipette and top pan balance

- Place a clean dry 50 cm³ beaker of the top pan balance. Tare.
- Using a volumetric pipette, add sodium chloride solution to the 50 cm³ beaker. Record the volume of solution added to the beaker.
- Place the beaker on the balance and record the mass in the table.
- Repeat for trials 2 & 3.

Analytical balance

Repeat each of the above sets of procedures using the analytical balance (in the balance room) in place of the top pan balance. Do not spill any liquid in the analytical balance room. These balances are very expensive and liquid transfers should be carried out outside of the balance room to minimise the risk of spillages.

Results

Temperature of water: _____ °C

Beaker and top pan balance

Trial	Volume / cm ³	Mass / g	Density / g cm ⁻³
1			
2			
3			
Average density			

Beaker and analytical balance

Trial	Volume / cm ³	Mass / g	Density / g cm ⁻³
1			
2			
3			
Average density			

Measuring cylinder and top pan balance

Trial	Volume / cm ³	Mass / g	Density / g cm ⁻³
1			
2			
3			
Average density			

Measuring cylinder and analytical balance

Trial	Volume / cm ³	Mass / g	Density / g cm ⁻³
1			
2			
3			
Average density			

Pipette and top pan balance

Trial	Volume / cm ³	Mass / g	Density / g cm ⁻³
1			
2			
3			
Average density			

Pipette and analytical balance

Trial	Volume / cm ³	Mass / g	Density / g cm ⁻³
1			
2			
3			
Average density			

Analysis

Complete the tables by calculating the density of the solution for each trial. Calculate and record the average density for each set of apparatus. Using the manufacturer's values for the precision of each piece of apparatus, calculate the range of the possible densities (smallest and largest) for each method. Considering these ranges, report one value for density for each method to an appropriate number of decimal places. [Note: more sophisticated treatments of errors will be used as the laboratory course progresses].

Apparatus used	Lowest density / g cm^{-3}	Highest density / g cm^{-3}	Density (reported to appropriate precision) / g cm^{-3}
Beaker and top pan balance			
Beaker and analytical balance			
Measuring cylinder and top pan balance			
Measuring cylinder and analytical balance			
Pipette and top pan balance			
Pipette and analytical balance			

The actual density of 2 mol dm^{-3} sodium chloride solution at 25°C is 1.0812 g cm^{-3}

Calculate the % error for each density average and record the data in the table below.

[Reminder: % error = $(\{\text{average calculated density} - \text{actual density}\} / \text{actual density}) \times 100 \%$]

	Top pan balance	Analytical balance
Beaker		
Measuring cylinder		
Pipette		

Questions

Discuss the following questions within the group and note down answers on these sheets. Once complete, report your findings to a demonstrator.

1. Which measurement of density was most accurate? Why do you think this was?

Induction activity 1B: Preparation of a transition metal complex

1B.1 Introduction

The study of transition metal complexes, which consist of one or more transition metal ions bonded (by coordinate bonds) to surrounding ligands, is often described as coordination chemistry and dates back to the turn of the 20th century. It is an area of research that is very much alive today. Coordination complexes are found in many aspects of our everyday lives: in nature, in components of industrial chemical processes (e.g. as catalysts) and in pigments. Examples include haemoglobin and the essential mammalian vitamin B₁₂. At the cores of these two metalloenzymes lie a transition metal ion (Lewis acid) surrounded by ligands (Lewis bases). It is the interplay between these two components and consequently of the metal complex as a whole (determined by the nature of the ligand, metal-ligand bond strength, ligand lability, geometry, and reactivity, etc.) that is the key to their function.

In this activity, a simple synthetic procedure will be followed to prepare a sample of Fe(acac)₃. Acetylacetonone (also known as pentane-2,4-dione) reacts with alkali to produce the acetylacetonate ion (pentane-2,4-dionate, acac⁻). Acetylacetonate is a very versatile ligand for coordination chemistry compounds.

1B.2 Aims

- To refresh and extend prior knowledge of coordination chemistry
- To become more familiar with working in the undergraduate laboratory
- To carry out a synthetic chemistry experiment (to prepare a transition metal complex)

1B.3 Pre-laboratory exercises

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

1. Ensure that all the post-lab exercises from the first laboratory session are completed. This includes setting up your lab notebook ready for use.
2. Refresh your knowledge of coordination chemistry by reading through suitable sections of relevant A-level textbooks, or by reference to www.chemguide.co.uk (visit 'Some essential complex ion chemistry' and 'Some essential transition metal chemistry' in the 'Inorganic Chemistry' section. Links available on DUO). It will be very helpful to read up on why M³⁺ ions are acidic in solution.
3. Read through the laboratory activity section in the laboratory manual and highlight unfamiliar words or apparatus.

4. Log in to DUO and, in the Core Chemistry 1B Laboratory Course main menu, open 'Foundation Chemistry LabSkills' and click to begin. Click on the 'Equipment Glossary' or the 'Reagent Glossary' at the top of the screen to find the meaning of any unfamiliar words contained in your laboratory manual.
5. From the Foundation Chemistry LabSkills main menu, select 'Filtration'. Select 'Technique' from the menu and read through the information, clicking along the progress bar at the bottom to move through. When complete, click on 'Gravity Filtration Video' and watch all stages, then click on 'Buchner filtration video' and watch through. Both of these methods of filtration should be familiar from prior studies, but, if not, take time to learn about the differences between them and their uses.
6. It is likely that you have used a Bunsen burner for heating in previous practical work. In the undergraduate laboratory, it is more likely that a hot plate will be used, accompanied by a magnetic stirrer bar (also known as a 'stirrer flea'). To prepare for using this apparatus, open the Chemistry Interactive Lab Primer, either via DUO or using a web browser (<http://chem-ilp.net>). Select the 'Lab Techniques' tab and select 'Heating' from the menu. Click on 'Hot Plates' and read all information about the use of stirrer hotplates and magnetic stirrer bars.
7. Complete the pre-lab quiz in DUO. Marks for this test will count a small amount towards your pre-lab mark for the Core Chemistry 1B course. Be sure to take the time to check over answers after submitting the quiz to receive your marks and some feedback.
8. Prepare the next page of the lab notebook ready for completing the experiment. Refer to the 'Use of a lab notebook' document (which should now be stuck into the back of the notebook). Work out or look up (e.g. using a textbook or <http://www.chemspider.com/>) the structure of acetylacetone (acacH) and acetylacetonate (acac⁻) and draw the structure of both into the lab notebook. If required, draw a diagram of the Buchner filtration apparatus to assist during the laboratory session.

1B.4 Risk assessment

CHEMICAL	RISKS	SAFETY
Ferric chloride hexahydrate	Harmful if swallowed. Irritating to skin. Risk of serious damage to eyes.	In case of contact with eyes, rinse immediately with plenty of water. Wear eye/face protection
Acetylacetone (pentane-2,4-dione)	Flammable, harmful if swallowed.	Do not breathe gas/fumes/vapour/spray. Avoid contact with skin and eyes.
Sodium hydroxide	Causes burns.	In case of contact rinse immediately with plenty of water. Wear gloves when handling
Dilute hydrochloric acid	At this concentration, treat as irritant.	Wear gloves.

1B.5 Laboratory activity

Preparation of Fe(acac)₃

Work in pairs but keep individual records in lab notebooks as the experiment proceeds. Choose a work area with a free locker and a fume cupboard to work in, and write these numbers at the top of the lab notebook page for this experiment. **Show a demonstrator the prepared lab notebook to confirm completion of the pre-lab exercises and inform them of the locker and fume cupboard number.**

- Using a top pan balance, measure out 1.0 g of iron(III) chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) into a conical flask (100 cm^3). Add a magnetic stirrer bar and add deionised water (15 cm^3).
- In a separate 100 cm^3 conical flask mix acetylacetone (acacH) (2 cm^3) and NaOH solution (6 cm^3 ; approximately 2 mol dm^{-3} , found on the laboratory benches).
- Carefully add the acetylacetone / NaOH solution prepared in step 2 to the stirred iron solution and heat to approximately $50 \text{ }^\circ\text{C}$ using a stirrer hotplate for about 5 minutes.
- Allow the solution to cool before collecting the precipitate by vacuum filtration (Buchner filtration).
- Wash the product with deionised water ($3 \times 5 \text{ cm}^3$) on the filter paper and air dry with suction still on.

6. Record observations in the lab notebook throughout. **Show a demonstrator that a product has been isolated.**
7. Once dry, weigh the sample on a top pan balance, and record the mass of product obtained. The acetylacetone and NaOH used in this reaction were in excess and thus the $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ determines the yield. The stoichiometry between $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{acac})_3$ is 1:1. Calculate the percentage yield for the experiment and record it, with working, in the lab notebook. [Hint: do not simply use the mass of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ to determine the % yield. First determine the number of moles of this starting material. Ask the demonstrator if you are stuck.]
8. Place the sample in a clean, labelled sample bag (not a weigh bottle!). Wash up all apparatus, thinking carefully about the disposal of any waste (ask if unsure), and return all equipment. **Hand your sample to a demonstrator for marking, and show them the lab notebook for marking.**

Investigating the formation of $\text{Fe}(\text{acac})_3$

To understand the chemistry that has just been performed, carry out the following exercises in pairs and discuss then record the results appropriately (and independently) in the lab notebook. Before beginning, decide upon an appropriate way to present the results for steps 2-6.

1. Place a small amount (about enough to cover the tip of a spatula) of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ into Test Tube 1 and add approximately 2 cm^3 of deionised water. Ensure the solid is dissolved by adding a bung and shaking. **Never cover the top of a test tube with a thumb or a finger to shake. Always use a bung.**
2. Add a drop of acetylacetone (from a Pasteur pipette) into Test Tube 2. Add approximately 2 cm^3 of deionised water and swirl. [Note: since this exercise does not involve precise analytical work, 2 cm^3 of deionised water does not need measuring out at all precisely using a pipette or measuring cylinder. Adding approximately 2 cm height of water to the test tube should suffice.]
3. Measure the pH of each solution by placing a drop of solution onto pH indicator paper and record the results in the lab notebook.
4. Add two drops (use a Pasteur pipette) of acetylacetone (acacH) from Test Tube 2 into Test Tube 1 (the iron(III) solution), and record the observations.
5. Slowly (dropwise) add approximately 1 cm^3 of 2 mol dm^{-3} NaOH solution to Test Tube 1, now containing the FeCl_3 and acacH, and record your observations.

6. Slowly (dropwise) add approximately 2 cm³ of dilute HCl solution to the same test tube, and record your observations.
7. Dispose carefully of any waste (ask if unsure), wash and return all equipment and ensure all power sockets and hotplates are switched off. **Show a demonstrator the clean and tidy workspace, the clean and tidy fume cupboard and the equipment locker to get signed off.**

1B.6 Written exercises

Study the results table for the test tube tests and consider the observations and the synthetic experiment that has been performed when deducing answers to the following questions. Answers should be discussed within the pair and written individually into the lab notebook.

1. Draw the structure of Fe(acac)₃.
2. Write an equilibrium equation to explain the pH that was recorded for the solution of acacH.
3. Explain the effect of the addition of NaOH on this equilibrium, and deduce and explain why NaOH was added to the acetylacetone solution before it was added to the iron(III) solution in the synthesis.
4. In a solution of FeCl₃·6H₂O, the ion [Fe(H₂O)]³⁺ is present. Using the formula of the hexaqua ion to begin, give an equilibrium equation, or a series of equilibrium equations, that explain the pH recorded for FeCl₃·6H₂O solution.
5. Using these equilibrium equations, deduce the chemistry that explains the observation in Step 4 and the changes observed during Step 5.
6. Deduce the chemistry that occurs in Step 6, above, and explain the observations made.

Show the demonstrator the completed exercises for marking before leaving the laboratory. If any equations or deductions are insufficient, pairs will be required to stay and re-work the answers.