Learn Chemistry

Faster Greener Chemistry? Catalyst Synthesis and Evaluation

Resource Overview

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Introduction - About context/problem based learning

Context/Problem Based Learning (C/PBL) is a teaching methodology that aims to increase student engagement with a subject by designing courses based on real-life applications of the principles, techniques and experiments students encounter in their undergraduate courses. These real world contexts are presented in the form of problem scenarios which are ill-defined and have a number of satisfactory solutions. Learners work collaboratively to solve problems and acquire new knowledge and then present the outcomes or product. This approach provides the opportunity to develop valuable transferable skills such as communication, team working and problem solving. Students are encouraged to take control of their learning and real world examples are used as an effective means to promote real learning. Academic staff adopt the role of facilitator or guide during this process. For further information, the following review on context and problem based learning can be consulted; T.L. Overton, Context and Problem-Based Learning, *New Directions*, Issue 3, Oct. 2007, pages 7-12.
(see http://www.heacademy.ac.uk/assets/ps/documents/new_directions/new_directions/newdir3_link.pdf)

About this learning resource

This case study focuses on the synthesis, characterisation and evaluation of a range of up to nine manganese(III) salen complexes that are employed as catalysts in the oxidation of alkenes to epoxides. Students adopt the role of chemists employed in a campus company that specialises in chemical catalysts, Chem Cat Ltd. Their company has been contracted to carry out some consultancy work for a large pharmaceutical multinational, HugePharma Ltd. Students work in teams and report to their laboratory manager in Chem Cat Ltd. Their brief is outlined in a letter to their Managing Director from the Chemical Development Manager in HugePharma Ltd. The pharmaceutical company have specifically requested that green chemistry (also known as sustainable chemistry) principles be implemented to their full potential in the epoxidation process, as they need to maintain their Integrated Pollution Prevention and Control (IPPC) licence.

The students are required to work as part of a team to:

1. Prepare and characterise one of a range of Mn-salen complexes using a two step synthesis.
2. Evaluate the performance of the catalyst in a reference reaction (epoxidation of stilbene).
3. Assess the relative costs and the environmental impact of this process and of alternative procedures with reference to suitable metrics.

To do this, they need to devise and perform several laboratory experiments to obtain the results required, research relevant information and make a recommendation to HugePharma Ltd.

The context is based on information from the literature, including journal articles and reviews, patents, textbooks, some articles in Chemistry World and Chemical & Engineering News, and environmental protection agency websites. Salts of Mn(III), particularly those that contain salen type ligands (salen is bis(salicylaldehyde)ethylene diamine), have been found to show catalytic activities of significant interest. The main application of these properties to date has been in the asymmetric epoxidation of alkenes (e.g. Jacobsen's catalyst) and oxidation of a variety of organic substrates. As a result, Mn-salens have been employed at commercial scale to oxidise a wide range of organic compounds. The process often uses sodium hypochlorite (bleach) as the oxidant. It is an environmentally friendly reagent as it breaks down into sodium chloride, water and oxygen. It is also inexpensive and has an efficient atom economy. Modifications are being investigated to reduce the environmental impact further as several of the catalysts can be recovered and recycled, and reactions in the absence of an organic solvent and in supercritical carbon dioxide have been performed. Some useful references that provide more detail are listed in the literature review in the student briefing pack at the end of this guide. This case study resource aligns with the RSC Chemistry for Tomorrow's World priority areas of "Water and Air" and "Raw Materials and Feedstocks".

Who is the learning resource designed for?

The resource is designed for undergraduate students in the last two years of their degree. However, it could be modified to be suitable for students at an earlier stage by providing some additional supporting information on green chemistry principles and environmental legislation and adapting the assessment criteria and learning outcomes to suit the performance expected of learners at that stage.

There are also opportunities for further investigations at more advanced levels such as purification of the epoxide product by column chromatography, determination of the enantiomeric excess in the epoxidation product if chiral diamines were used to prepare the ligand, and variation of the metal (to Co or Fe). References are provided in relation to these other options in the literature review, but they are not dealt with in this guide.

Tutors are encouraged to use this guide as a flexible framework to produce a C/PBL student activity that is appropriate to their circumstances and meets their requirements. It is anticipated that the student contact hours available, prior knowledge of the students and the specific skills, knowledge and understanding to be developed will vary across cohorts and institutions. The tutor and student guides are provided as Microsoft® Word documents so that they can be modified to suit a particular situation and to allow the appropriate learning outcomes to be addressed. With this in mind, the developers have indicated which aspects of the delivery they consider to be core and which ones could be omitted or modified in the section that follows on 'Flexibility within this resource'.

How is the learning resource delivered?

The resource is designed to be delivered to students as a laboratory based course, with 8 three-hour sessions incorporated, four delivered in a laboratory setting, and four not, labelled as workshops. When combined with independent study and writing-up time, it is intended that this resource will require a total of 50 learning hours. (a total of 24 contact hours plus 26 hours of self study). As such it represents approximately 2.5 European Transfer Credit System (ECTS) or 5 UK credits of work or 1.5 US credits.

Some of the workshop sessions require access to computers for all students (Session 1 if some training on using wikis is provided and Session 6 when students are asked to find information on vendors and costing for raw materials). In each case, a one hour session in a computer laboratory would suffice and the remainder of the session can be spent in a classroom. A digital projector is required for Sessions 1 and 8. If possible, access to some computers during all sessions would be useful to allow students to input information into their wiki directly but this is not a requirement.

In addition to the group wikis that the students will produce, it is strongly recommended that a Virtual Learning Environment (or alternatively a 'central' wiki to which all of the students are invited) is used to support this case study. This will allow the tutor to host the Student Guide, supplementary material (presentations, journal articles) and any other information that is deemed appropriate, on a central resource that all of the students can access. It also provides a communication tool that may be useful in implementing the resource (providing general feedback, reminders of tasks to be completed and deadlines etc).

Navigating this tutor guide

A schedule for delivery which provides an overview of the resource is presented in Table 2. It contains a breakdown of what each lab and workshop session will entail, including aims and teaching and learning activities. Further details for each session are included in the relevant sections throughout this Tutor Guide. A simplified version of Table 2 can also be found in the accompanying Student Guide (also Table 2). Table 3 is a schedule of the weekly tasks to be completed by students which are aligned with the suggested tutor feedback / prompts / actions required. The Appendices to this guide contain the student briefing pack (Appendix 1) which is provided to students in the Student Guide for this resource. Appendix 2 deals with using wikis to facilitate and assess group collaboration and Appendix 3 summarises materials, apparatus and instrumentation requirements. The remaining Appendices 4-8 provide detailed tutor notes for Sessions 2-6 respectively and elements of these can serve as a sample answer when assessing student work.

This Tutor Guide in its entirety presents quite a daunting prospect because of its length. However, the Appendices account for almost two thirds of the content so it is recommended that the main body of the guide be printed separately for ease of use (It would also be useful to add the final page which lists the references). Appendices 1-4 would need to have been consulted before beginning this C/PBL case study and, thereafter, the relevant Appendix can be read before the previous session (e.g. read the tutor notes for Session 3 before Session 2). It is recommended that, for each session, the tutor brings along for reference the main body of the Tutor Guide, a copy of the Student Guide and the Appendices relevant to that session and to the next one.

Assessment

This case study resource has three main elements: planning laboratory experiments, conducting experiments and analysis of results and reporting of the data obtained. Therefore, assessment focuses on (i) the planning of the group project, most conveniently monitored using a wiki (see later in this section) (ii) student lab books on experiments conducted and (iii) the reporting, analysis and recommendations arising out of the data obtained. More details on assessment are provided at appropriate points in this guide and the assessment components and a guideline weighting are provided in Table 1 below. These are also presented in the Student Guide (Table 1) and introductory presentation, and can be adjusted if considered necessary.

Table 1: Assessment components

Activity	Group / individual	% mark allocation (guideline)
Contribution to group (based on effort and effective collaboration - evidenced by participation in lab and workshop sessions, summaries of meetings and contribution to group wiki)	Individual	10
Peer assessment by other group members* (based on frequency and quality of contributions, both online and face-to-face)	Individual	5
Lab notebook and individual work submitted on a weekly basis	Individual	30
Reflective piece (criteria are presentation, relevant content and structure, coherence and accuracy)	Individual	10
Final wiki report (Criteria are relevant content, accuracy, structure, clarity, literature references)	Group	30
Presentation - feedback from tutor and peers and assessment by tutor (criteria are visual impact, effectiveness of oral communication, relevant content, accuracy, structure, clarity, references to the literature and understanding).	Group	15

* Peer assessment is an optional but recommended assessment component and the weighting allocated (5%) is low to encourage its inclusion. The weighting can be increased if the tutor wishes to do so.
One option to consider for peer assessment of each member's contribution to the group is peer review software such as CATME (Comprehensive Assessment of Team Member Effectiveness), available to download for free at https://engineering.purdue.edu/CATME. This software was developed with support from the National Science Foundation. E-mail contacts for group members are uploaded by the tutor and a range of statements on team member contributions can be selected to be used.

Flexibility within this resource

The work that learners undertake for this activity focuses on the development of teamwork, communication and problem-solving skills as well as the synthesis and characterisation of organic and organometallic compounds. The context provided requires that the preparation and use of these compounds on a larger scale be evaluated on the basis of their "greenness" (the extent to which hazardous substances are generated or used). Therefore, a tutor can opt to focus more on the organic chemistry (ligands and alkene oxidations), the inorganic chemistry (catalyst complexes, oxidation reagents) or the green / industrial chemistry aspects involved. Alternatively, a collaborative approach could be employed between academics with organic, inorganic and green / industrial chemistry backgrounds. The green chemistry metrics employed in this activity are at an introductory level and, if students have prior knowledge in this area, it would be appropriate to extend what is required of them (further details are provided in the notes provided for Session 6). Consultation with the lecturing staff concerned would be recommended. In addition, a recent review on effective practices in teaching green chemistry by Andraos and Dicks is very informative on this topic.[1] They emphasise that there is no one correct "green" answer but instead there are a range of alternatives that need to be evaluated using criteria so that a decision can be made. This approach of considering multiple solutions and making decisions is quite challenging for most students but develops important key skills.
Some modifications that tutors may consider are:

- To combine Sessions 4 and 5 so that some students in the group carry out the standard epoxidation reaction and others use the alternative conditions during the same laboratory session.
- Not to carry out the alternative epoxidation reaction and use data from the literature instead.
- To ask students to prepare either a report or presentation on their findings and recommendations but not both.

It is important that learners are provided with sufficient time to perform the tasks assigned and to gain understanding. If the time available is less than that outlined here, some components of the case study will need to be removed and / or the associated tasks reduced.

Table 2: Schedule Showing the Aims and Teaching and Learning Activities for the Workshops (WS) and Laboratory (Lab) Sessions

Session (3 hrs)	Title	Aims	Activities (during and between sessions)
1 (WS 1)	Introduction to the project and planning for first synthetic step	Tutor aims: • To introduce the case-study and outline the learning outcomes, learning activities and assessment components and criteria. • To introduce the principles of green chemistry and environmental reporting requirements for commercial producers of fine chemicals. • To provide information on the literature references to be consulted to plan the first synthetic step in the production of a Mn-salen catalyst for alkene epoxidations. Each group of 2 to 3 students is assigned a different catalyst to prepare. • To obtain student e-mail addresses so that a wiki can be set up for each group (if not possible to do so before the first session).	• Introduction presentation and assignment into groups. • Discussion on green chemistry, environmental legislation (Annual Environmental Reports and Integrated Pollution Prevention and Control licences) and the role of catalysts and desirable properties they should have. • Discussion on selecting a suitable scale for the first step and preparation of a procedure. • Demonstration of a sample wiki if possible (adding and editing pages, uploading files, adding comments and checking page history).
Independent group and individual work in preparation before each lab session should take 1 – 3 hrs. Some guidance prompts are provided.			
2 (Lab 1)	First synthetic step (reaction of diamine and salicylaldehyde to form the ligand intermediate)	Learner aims: • To perform a suitable experimental procedure to prepare the ligand intermediate in the synthesis of the Mn-salen catalyst. • To characterise the intermediate prepared.	• Writing of procedure in advance. • Preparation of ligand intermediate. • Prediction of expected IR and NMR spectra for the salicylaldehyde starting material used and the intermediate prepared. • Begin group literature research on alternative procedures/oxidants. • Maintain independent lab book and group wiki.
3 (Lab 2)	Second synthetic step (preparation of catalyst)	Learner aims: • To prepare a suitable experimental procedure to be used to prepare the Mn-salen catalyst. • To characterise the Mn-salen catalyst prepared and determine the overall yield obtained. • To review information obtained on alternative epoxidation conditions using Mn-salens and recommend an experiment to be attempted.	• Writing of procedure in advance. • Preparation of catalyst. • Prediction of expected IR spectrum for the catalyst prepared. • Interpretation of IR and NMR spectra of the salicylaldehyde used and the intermediate prepared. • Preparation of a 1 page summary on literature research on alternative epoxidation conditions (recommend one experiment to be attempted) • Maintain independent lab book and group wiki.
4 (Lab 3)	Evaluation of catalyst performance in a reference reaction (epoxidation of *trans-*	Learner aims: • To prepare a suitable experimental procedure to be used to perform the epoxidation of stilbene oxide. • To perform the epoxidation of an alkene (*trans*-stilbene) using	• Writing of procedure in advance. • Epoxidation reaction using sodium hypochlorite in duplicate / triplicate. • Prediction of expected IR and NMR spectra for the

	stilbene)	sodium hypochlorite as the oxidant in duplicate or triplicate. • To characterise the epoxide product and determine the yield and conversion obtained. • To evaluate the alternative oxidation procedures found and recommend related future work.	alkene substrate used and the epoxide prepared. • Evaluation of alternative oxidation procedures found based on criteria provided and recommend which should be attempted in the future. • Interpretation of IR spectrum obtained of catalyst. • Maintain independent lab book and group wiki.
5 (Lab 4)	Evaluation of catalyst performance using alternative conditions	Learner aims: • To perform the epoxidation of an alkene using alternative conditions in duplicate or triplicate. • To characterise the epoxide product and determine the yield and conversion obtained. • To compare the effectiveness of all catalysts prepared in the reference reaction.	• Writing of procedure in advance. • Epoxidation reaction using alternative conditions • Complete catalyst performance evaluations. • Analyse data from all groups to determine the most effective catalyst in the reference reaction. • Maintain independent lab book and group wiki.
6 (WS 2)	Costing and assessment of environmental impact for oxidation procedures performed	Learner aims: • To cost the raw materials for two oxidation processes. • To compare the environmental impacts of two processes with reference to the appropriate metrics. • To plan for the scope of future development work on each process. • To compare the results obtained in the two epoxidation reactions.	• Use ChemSpider to find vendors and costings for raw materials and solvents for the synthesis of the catalyst and the epoxidation reactions. • Use guidelines on metrics and recommended reading sources to evaluate the environmental impact of each process as well as the potential for further development. • Analyse data from all groups to determine the most effective epoxidation conditions.
7 (WS 3)	Clinic for formative feedback on draft reports (on group wiki)	Tutor aims: • To provide learners with formative feedback on areas of the report they need to work on and on which aspects have been addressed satisfactorily. • To answer any student queries on the assignment and activities, and discuss any issues raised.	‘Clinic’ where each group: • has submitted a 1 page ‘work in progress’ summary in advance. • discusses any problems or queries. • receives feedback on their summary and on their wiki draft report as it is at that point.
Optional Additional Laboratories: purification of the epoxide product by column chromatography, determination of enantiomeric excess in the epoxidation product by chiral GC if chiral diamines were used to prepare the ligand, variation of the metal (to Co or Fe)			
8 (WS 4)	Oral presentations	Tutor aim: • To provide feedback to the learners on their presentation skills and the content of their presentation. Student aim: • To learn from each other’s presentations and to ask each other questions.	• Presentation to peers summarising work undertaken and recommendations made followed by questions from the tutor, guest tutor (if present) and peers. • General oral feedback from tutor (optional written feedback to each group from peers and tutors). • Completion of wiki report (with feedback from presentation incorporated), individual reflective piece, and, if required, peer assessment of other group members.

A word about wikis

There are many advantages to using a wiki when collaboration on a group project is required and these are dealt with in more detail in Appendix 2. To summarise, it provides an effective and flexible means for learners to work as a team on a report, presentation or web page while generating an archive of all information used and of all previous versions of the final pages. The main benefit to a tutor is that the quality and quantity of contributions made by each student can be tracked relatively easily and that the process as well as the product can be assessed. Wikis are regularly used in organisations to allow groups to collaborate on projects and documents and to share knowledge and the ability to use one is a valuable transferable skill. For example, a wiki has been established to develop policy in the area of green chemistry in California (http://cagreenchem.wikidot.com/start).

Although it is recommended that a wiki be used as a component of this C/PBL activity, an alternative can be adopted if preferred. Some type of online interaction among a group such as a discussion board or online group is very useful and, if this has been set up by the tutor, there is the advantage that they will be able to monitor progress being made. If this option is not used, the weekly group meeting summaries that are required from each group can be used to monitor progress and to check that all members are making a contribution. To provide a facility similar to the wiki for organisation of the work being undertaken, it would be useful for groups to use a ringbinder with sections that correspond to the main parts of the report to which useful documents and draft work can be added. Under these circumstances, it is recommended that this draft work and supporting information is submitted as an appendix to the final report.

Resource learning outcomes

On completion of this C/PBL resource, the learner should be able to do the following, within the context provided:

1. Use a procedure from scientific literature to write a laboratory procedure, including a list of materials and equipment required, for the preparation of organic and organometallic compounds on a suitable scale.
2. Prepare a short chemical risk assessment for the experimental work to be undertaken.
3. Plan time in the laboratory effectively in order to complete the synthesis and evaluation of the catalyst assigned.
4. Keep an accurate and current record of experimental details and data in a laboratory notebook.
5. Interpret experimental data and predict and assign spectra to confirm the identity and purity of the products.
6. Use appropriate databases to find relevant information on raw material costing for the process and on recent developments to improve the environmental impact it has (e.g. alternative solvents and oxidants).
7. Evaluate the efficiency, relative costs and the environmental impact of the two oxidation procedures used for the epoxidation of *trans*-stilbene.
8. Identify aspects of each reaction performed that adhere to the principles of Green Chemistry and those that do not.
9. Use the results obtained for all catalysts evaluated to predict how catalyst structure and efficiency are related.
10. Produce a professional report, including an executive summary and an assessment of the scope for each step to be improved, that is supported by the relevant experimental data and a laboratory notebook as well as references to the literature.

11. Prepare an oral presentation on the findings from the study to present to the company that commissioned the project.
12. Prepare a short individual reflective statement on the group process, transferable skills developed, and the extent to which the stated learning outcomes were met.

Transferable skills development

Students will be asked to reflect on the development of the skills listed below at the end of the project. It is recommended that they record notes as they go along on the areas they are finding challenging as well as progress that they feel they are making to make this task easier. The specific ways in which it is intended that key skills will be developed during this C/PBL case study are described below:

- Team work: learners work in groups to complete the task assigned, use a wiki to facilitate collaboration and meet between sessions to review progress.
- Organisation and planning: learners prepare procedures on a suitable scale and plan their time in the laboratory effectively.
- Communication skills: Learners present (oral presentation) and report (wiki and final report) on the scientific work performed in keeping with the context.
- Drawing conclusions and recommendations from data: learners justify decisions, assumptions and conclusions made with reference to results from other groups and supporting literature in order to produce a logical and clearly reasoned scientific report.
- Numeracy: learners apply the relevant green chemistry metrics to their experimental results.
- Professional role and responsibilities: learners adopt the role of a professional chemist and are required to consider the environmental impact and costing of the processes they have been working on.
- Problem solving: learners work in groups to address the brief presented in the context scenario.
- Information technology skills: learners use a wiki to collaborate and develop their ability to use word-processing, spreadsheet, presentation, chemical drawing and library database software.
- Metacognition: learners reflect on the process involved in working on the brief given, the extent to which the stated learning outcomes were met and to which their transferable skills were developed.

Implementation: Class organisation

This resource is designed so that students work in small groups (typically three students) to complete the brief provided together. A recommended maximum number of students for one tutor is 15. If a second tutor is available, the maximum cohort size would be about 30. The assessment components (Table 1) require that each student submits a laboratory book as well as a short reflective summary for individual assessment at the end of the C/PBL activity. The assessed group work covers the presentation and report and the group wiki which provides a record of how the group collaborated and their rate of progress. The report can be generated directly from the wiki either by converting wiki pages to PDF files or by printing the pages (depending on the software used). Alternatively, you may want to request that the text can be cut and pasted into a Microsoft® Word document once all collaboration on the wiki is complete. This option allows a word count to be performed easily as well as additional formatting such as page numbers. Alternatively, you may opt to read the report as generated on the wiki and not require that a paper copy be submitted. This decision is left to the discretion of the tutor as it will depend on what format you prefer to correct and whether you feel it would be useful for learners to produce a written report in a conventional format.

Weekly feedback

Advice on monitoring and correcting wikis is provided in this guide, but, to summarise, the tutor should log into each group's wiki approximately once per week if possible and provide brief feedback on the progress reported (in the group meeting summary). Feedback is also provided on any components of the group report that are submitted in a given week (e.g. experimental procedure, cost analysis) and the draft group reports are reviewed before Workshop 7. The remaining correction time is spent on the presentation and the completed reports. In this manner, students receive feedback at various stages throughout the process. Students' email addresses should be collected at the outset of the module and used to set up each group wiki. At the latest, each student should be invited to join his or her group wiki just after Session 1. Suggested assessment criteria are provided in Table 1 and further information on assessing individual contributions to wikis is presented in Appendix 2.

A schedule of weekly work to be submitted by students and the suggested feedback provided is shown in Table 3, but it will be at a tutor's discretion to decide the extent of the feedback that is reasonable for them to provide. The assignment of weekly tasks as individual or group activities can also be adjusted as the tutor decides is appropriate. The weekly work is designed so that students are preparing elements of their final report as they go along. Other considerations for the tutor are whether all submitted work should be typed or not and whether weekly work should be handed in as a printed copy and/or added as a file to the wiki. One factor that can be variable is how quickly students can obtain NMR spectra of their compounds. It has been assumed that they may not have them until the lab session that follows the one in which the compound was made. If the turnaround time is shorter, the schedule of work in Table 3 (and the corresponding Table 3 in the student guide) can be modified.

Student workload

In general, there is a piece of individual work each week as well as group work. In this way, students get to learn about compiling safety information on chemicals and prediction of NMR and IR spectra by themselves initially but, in later weeks, this is assigned as a group activity. To allow students to work efficiently, it is suggested that they be allowed to paste any relevant material already prepared in an electronic format such as reaction schemes and safety information into their laboratory notebooks. It is recommended that students be asked to submit any previously corrected work that was returned to them as a hardcopy with their final report (for group assignments) or with their laboratory notebook (for individual assignments) so that the extent to which feedback was incorporated can be determined. The ideal turnaround time would be that material is submitted 2 to 3 days after the weekly session and it is returned at the session the following week but this may not always be feasible. The day of the week on which work is due can be added to Table 3 in the space provided.

Student cohorts vary as do their workloads and you may opt to reduce some of the weekly requirements and overall assessment components depending on the length of time that you estimate it will take them to complete the tasks assigned (see Flexibility Within this Resource section also). For example, prediction of the main features of NMR and IR spectra of the compounds involved is a very useful learning exercise but it could be removed and students could just be asked to characterise their products using the spectra recorded.

Whenever possible, the tutor should take a back seat and direct the students to the Student Guide and recommended resources to obtain relevant information. This encourages learners to read and engage fully with the materials provided. The distribution of the workload associated with the tasks should be assigned by the students in their groups.

Groups

The authors recommend a group size of 3 students, but this will depend on the class size, ability and prior learning. It is recommended that these groups be assigned before the start of the first session and it is useful to prepare a table listing the group members and the diamine and salicylaldehyde starting materials that they have been assigned to use to add to the Student Guide or bring to the first session. It is recommended that the tutor tries to ensure there is a range of abilities and skills in each group and, if possible, that students get an opportunity to work with people they may not know very well. One other important consideration is that each member of a group should have similar class contact timetables so that it is easy for them to arrange to meet. You may like to suggest that each group choose a name for their team as this has been found to work as a good ice-breaker and develops a sense of group identity. Advise the students that there are 3 roles that should be assigned to group members each week on a rotating basis: Chair, Reporter and Editor. The Chair will prepare the agenda for meetings, will lead/run the group meeting/discussions, listen with an open mind to all group members, and ensure that everyone in the group has the opportunity to contribute. The Reporter should prepare a summary of the action items arising from discussions and meetings and should post these on the group wiki by the day of the week that you specify (usually 2 days before the next session). The Editor will review the wiki content to ensure a consistent style, coherency and an overall structure and will also liaise with authors when changes to content are required. Each student should adopt each role at least once during the project and the group should nominate these roles during each session (note than an Editor is not required until Session 2). It is recommended that you remind the students to nominate these roles at the start of each new Session.

It is useful to ask the students to stop what they are doing about 15 minutes before the end of each session and to identify all tasks that need to be completed before the next session, to review the description of the following session and to arrange a date and time to meet outside of the class contact time. It may also be helpful to ask a spokesperson from each group to very briefly summarise the progress of their group to the class.

It is not easy to ensure that equal time is spent with each group due to the nature of the C/PBL approach and tutors must do their best to ensure this is the case. If involved in a particularly interesting discussion with one group that merits more time, it may be a good idea to open up this discussion up to the entire class and in that way include all students. If a group has a problem that requires a significant proportion of your attention (e.g. the group is not functioning as a team, the students do not understand the tasks), it may be wise to meet with that group outside of normal contact time.

The most significant problem we have encountered when implementing this type of activity has been the difficulties that can arise when students are asked to work as a group. This is a necessary evil as some conflict is inevitable when groups begin to try to work together. If some tensions become apparent in a group over a number of weeks, it is likely to be as a result of one member not contributing, a personality clash or one person working on their own and not collaboratively. The methods of assessment used (wiki contribution, requirement to minute weekly group meetings, peer assessment) ensure that those not contributing or collaborating effectively will lose marks and the class can be reminded of this. If the problem persists, it may be necessary to speak to the particular group. It is also useful to remind students that the context is representative of the challenges that exist in the workplace when participating in a team and is a valuable learning experience.

A good overview for students on effective group work is presented in Chapter 3 of "Study and Communication Skills for the Chemical Sciences"; Overton, T., Johnson, S., Scott, J.; Oxford University Press (2011).

Table 3: Work to be completed after each weekly session & associated suggested feedback

Stage	For submission after the session by the following (add day of week) at (add time)	Suggested feedback
Week 1 (Introduction) Date for completion of tasks:	**Group** • Modified experimental procedure for step 1 (catalyst ligand preparation).* • Summary of group meeting posted on group wiki. **Individual** • Short version of chemical safety assessment.* • Maintain group wiki and independent lab book. * The procedure and safety assessments should be typed and structures should be drawn using chemical drawing software.	Any required changes. Acknowledgement. Grade and give brief comment to individual. General feedback either at next session or posted online before it. Brief comment on wiki, sign and date lab book at next session.
Week 2 (Ligand preparation) Date for completion of tasks:	**Group** • Modified experimental procedure for step 2 (catalyst preparation). • Short version of chemical safety assessment. • Summary of group meeting posted on group wiki. • Begin research on alternative epoxidation conditions for Mn-salen catalysts. **Individual** • Predicted IR and ^{1}H NMR spectra for salicylaldehyde starting material and ligand intermediate. • Maintain group wiki and independent lab book.	Any required changes. Any required changes. Acknowledgement. Acknowledgement. Grade and give brief comment to individual. Give general feedback at next session or online before it. Brief comment on wiki, sign and date lab book at next session.
Week 3 (Catalyst preparation) Date for completion of tasks:	**Group** • Modified experimental procedure for epoxidation of stilbene using standard conditions. • Summary of group meeting posted on group wiki. • One page summary on research carried out (on alternative conditions for epoxidation of alkenes with Mn-salen catalysts and recommended experiment to try). • Short version of chemical safety assessment. • Predicted IR spectrum for catalyst with suggestion as to why NMR analysis of catalyst is not useful. **Individual** • Interpretation of IR and ^{1}H NMR spectra obtained of ligand intermediate and of salicylaldehyde starting material. • Maintain group wiki and independent lab book.	Any required changes. Acknowledgement. Grade and give brief comment to group. Give general feedback at next session or online before it. Any required changes. Grade and give brief comment to group. General feedback either at next session or posted online before it. Grade and give brief comment to individual. Give general feedback at next session or online before it.. Brief comment on wiki, sign and date lab book at next session.
Week 4 (Catalyst evaluation 1) Date for completion of tasks:	**Group** • Modified experimental procedure for epoxidation of stilbene with catalyst using alternative conditions. • Short version of chemical safety assessment. • Predicted ^{1}H NMR spectra for stilbene and stilbene oxide.	Need to have identified to students which alternative procedure is to be used (e.g. H_2O_2 as oxidant). Any required changes. Any required changes. Grade and give brief comment to group. General feedback either at

	• Evaluate alternative oxidation procedures found based on criteria provided (yield, catalyst loading, solvent, type of oxidant, complexity of the procedure) and recommend which should be attempted in the future. • Summary of group meeting posted on group wiki. **Individual** • Interpretation of IR spectrum obtained of catalyst. • Maintain group wiki and independent lab book.	next session or posted online before it. Grade and give brief comment to individual. General feedback either at next session or posted online before it. Acknowledgement. Grade and give brief comment to individual. General feedback either at next session or posted online before it. Brief comment on wiki, sign and date lab book at next session.
Week 5 (Catalyst evaluation 2, alternative conditions) Date for completion of tasks:	**Group** • Compilation and sharing of data on group results (yields and conversions) for reference epoxidation reaction (using sodium hypochlorite). • Determination of most effective catalyst based on this data for the reference reaction and prediction of how catalyst structure & efficiency are related. • Summary of group meeting posted on group wiki. **Individual** • Interpretation of ^{1}H NMR spectra obtained of crude product from stilbene epoxidation using sodium hypochlorite. • Maintain group wiki and independent lab book.	Any required changes. Any required changes. Acknowledgement. Grade and give brief comment to individual. General feedback either at next session or posted online before it. Brief comment on wiki, sign and date lab book at next session.
Week 6 (Costing and green metrics workshop) Date for completion of tasks:	**Group** • Analysis of ^{1}H NMR spectra obtained of crude products from alternative stilbene epoxidation to determine percentage conversion. • Compilation and sharing of data on group results for alternative epoxidation reaction. • Comparison of results for the reference and alternative oxidation reactions. • Costing of raw materials and solvents for synthesis (including work up) of the catalysts prepared, and for their epoxidation reactions. Identify any resulting issues (most and least expensive materials). • Evaluation of the environmental impact of each process with reference to appropriate metrics and provide recommendations for further development. • Draft group report on wiki ready for preliminary review. • Work in progress summary on draft group report posted on group wiki (provide link from Table of Contents). • Summary of group meeting posted on group wiki. **Individual** • Maintain group wiki and independent lab book.	Check conversion determined and state any required changes. Any required changes. Any required changes. Any required changes. Any required changes (it should be possible to give feedback during the workshop session) Review before next session and prepare short summary on aspects dealt with well and those that need more work. Check to see if anyone has not been contributing at all. Acknowledgement. Brief comment on wiki.

Week 7 (Clinic for formative feedback) Date for completion of tasks:	**Group** • Incorporation of feedback from clinic workshop into the group's wiki report. • Consideration of the scope for each synthesis step to be improved and recommendations for future work. • Practise group presentation to ensure it is coherent, structured, accurate and meets the time requirements (the wiki itself can be used as a visual aid or, alternatively, it may be preferred that PowerPoint slides be prepared). • Summary of group meeting posted on group wiki. • Maintain group wiki.	Any required changes. Provide oral and brief written feedback after presentation at next session. Acknowledgement. Brief comment on wiki.
Week 8 (Oral presentations) Date for completion of tasks:	**Group** • Incorporation of feedback from presentation into the group's wiki report. • Final editing and completion of group's wiki report. • Submission of wiki report **Note for tutor:** inform students on final report format required (PDFs, Microsoft® Word or wiki), if it should be printed and whether to submit it for checking by plagiarism detection software. **Individual** • Reflective piece to be submitted. • Lab book to be submitted. • Peer assessment of other students in the group (optional).	Provide spoken and written feedback during session - use criteria in Table 1. Grade using criteria in Table 1 (see Appendix 2 also) and give optional general feedback or group specific feedback added to wikis. Grade and add some comments. Grade and add some comments. (Using CATME software would automate this process.)

Why do it? The philosophy and rationale for this case study

In designing this case study, we wanted to put learners in a position where there is an existing framework (a proposed synthetic procedure that addresses some green chemistry principles) but to provide an opportunity to investigate potential improvements. As this is a constantly developing field, flexibility to incorporate alternative procedures that become available for using Mn-salen catalysts to epoxidise alkenes has been built in. The intention is to allow students to develop an appreciation of what scientific research involves and to gain some experience of this approach in a group project. The expectation is that they will have a better understanding of what is expected of them when they go on to undertake an individual final year research project. In the authors' experience, this has been the case as students have been observed to be able to work effectively as soon as they begin their final year projects, particularly in relation to planning their work, finding relevant information and evaluating results obtained in order to decide what should be done next.

Additional anticipated benefits are (i) that learners will appreciate the holistic nature of their subject as the case study interlinks the applications of organic, inorganic, industrial/process chemistry and green/sustainable chemistry, (ii) that students develop a sense of identity as a future professional chemist and (iii) that an opportunity to be involved in and reflect on working as part of a team is provided.