

Curriculum Decolonisation and Improving Sustainability in Undergraduate Chemistry Teaching Laboratories

Abstract

The discipline of chemistry has a crucial role in ensuring a sustainable global future, with 'green chemistry' now an essential component of scientific development across a diverse range of industries and in the production of a vast number of chemical products (1). These sustainable principles should also be effectively integrated from undergraduate level, including in undergraduate laboratories. In this research report and reflection, a newly adapted more environmentally friendly synthesis appropriate for undergraduate teaching laboratories is implemented, replacing a previous synthesis that involved the use of large amounts of both environmentally harmful and toxic solvents and materials. Attempts to improve the environmental sustainability of a current practical that introduces column chromatography are outlined, using 'microscale' columns to reduce the scale by a factor of 20 and thus minimising the unnecessary use of environmentally harmful silica powder. Environmental sustainability is not the only important element of a modern curriculum. The job market is highly competitive, so it is vital that laboratory courses are industrially relevant and that graduates aiming for careers within chemical sciences possess the practical laboratory skills deemed necessary and desired by employers. Attempts to consult with personnel from a number of pharmaceutical and manufacturing companies on the most important laboratory skills for chemistry graduates and the most frequently utilised 'green' solvents in industry are implemented.

Introduction and aims

The overall aims of the research project were to evaluate and improve the environmental sustainability and industrial relevance of the undergraduate chemistry labs at the University of Leeds. Modernising chemistry teaching is crucial for a number of reasons. Firstly, it is vital that undergraduate labs prioritise environmental sustainability, ensuring that the environmental impact and materials consumption of undergraduate programmes are minimised. Secondly, it is critical that efforts are made to reduce the use of toxic and harmful chemicals and materials in undergraduate labs where possible, as the safety and well-being of students should be at the forefront of any effective undergraduate programme. Finally, it is also essential that undergraduate courses are economically viable in terms of both cost (i.e., of materials) and income (i.e., enough applicants), ensuring financial stability in a time of ongoing higher education financial crisis. Since 2024, chemistry undergraduate programmes have closed at three institutions (Aston University, University of Bradford, University of Hull), causing job losses and restricting access to higher chemical education (2)(3)(4).

In practice, this consisted of a number of key and largely distinct elements, albeit connected through the same principles of 'green' chemistry and sustainability. The most significant portion of research was devoted to the development of a new practical using relatively environmentally friendly reagents and solvents. This was to replace a previous synthesis practical which involved refluxing in large volumes of chloroform. This is

an important change due to chloroform's negative environmental impacts (chronic toxicity to aquatic life, chromosomal damage in plants) and safety risks to humans (toxic, may cause cancer and fertility issues) (5)(6).

The second largest component of research was exploring the viability of alternative purification methods for use within the teaching laboratories. Specifically, utilising 'microscale columns' to separate a mixture of compounds. This was to assess whether it would be possible to use these 'syringe columns' in some instances, thus hugely reducing the consumption of both silica gel and reagents/solvents. Silica gel is a particularly hazardous material, as it can penetrate deeply into the lungs and can cause irreversible damage over time, contributing to the development of serious respiratory and autoimmune diseases (e.g., silicosis, lung cancer) (7).

The final significant section of research was attempting to understand the most important practical laboratory skills for graduates. This largely consisted of designing a survey for technical employees from major graduate employers within pharmaceuticals and manufacturing and analysing their responses.

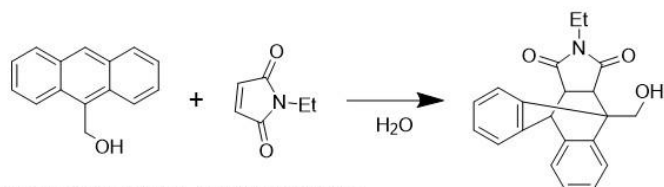
Designing a 'green' synthesis

When approaching the reaction design, it was important to not only be more environmentally sustainable but also to retain the learning outcomes from the original reaction. The principle learning outcomes of the original reaction (and the replacement) were to be industrially relevant, to be able to be completed within the duration of a typical undergraduate lab session (~6 hours) and to incorporate reflux conditions. Reflux conditions increase the rate of reaction as the reaction temperature becomes the boiling point of the solvent, allowing the production of vapours. The vapours are condensed by the condenser and prevented from escaping, being returned to the vessel via gravity. This accelerates the rate of reaction by conducting it at an increased yet controlled temperature without losing large quantities of the mixture, while also maintaining an ambient pressure. As a result, competency with reflux apparatus is an essential skill for all undergraduate chemistry students.

To improve the environmental sustainability, it was important to minimise the use of environmentally harmful halogenated solvents (e.g., dichloromethane, chloroform etc.). To be accessible for undergraduate students, it was also crucial that the final reaction could produce a significant yield of desired product (>70%) at a relatively small scale. The small scale is especially important as the synthesis would be carried out by ~100 undergraduates yearly, minimising both environmental and economic consumption, while the synthesis is also designed for students at the early stage of their course (who may lack the experience to confidently handle larger-scale reactions). Given that the main competency being tested by the previously used synthesis was reflux, the new synthesis also required incorporation of reflux conditions.

The initial approach was to pursue a Diels-Alder reaction in water. This reaction involved refluxing 9-anthracenemethanol and N-ethylmaleimide in water to produce a cyclohexene derivative formed by a 2,4 cycloaddition. Diels-Alder cycloaddition reactions are exceptionally industrially relevant, with their discovery being awarded the Nobel Prize in Chemistry in 1950 (8). This is because they are extremely useful laboratory

reactions because they are one of the few general methods of producing cyclic molecules (9, pp.430-431). They are pericyclic reactions, with two reactants joining together through a cyclic transition state (9, pp.430-431). Two carbon-carbon bonds form at the same time and in a single step (9, pp.430-431). This reaction could've been an excellent candidate given the very limited environmental and health and safety impact of water (in great contrast to those of chloroform) and the industrial relevance of Diels-Alder reactions generally.

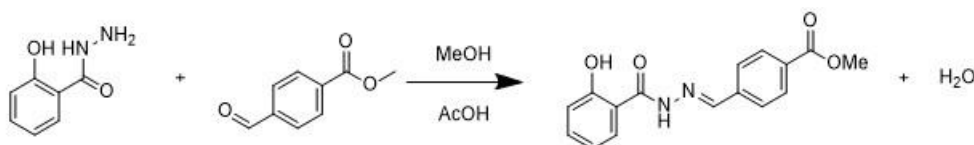


9-anthracenemethanol N-ethylmaleimide

Figure 1: Reaction scheme for the reaction between 9-anthracenemethanol and N-ethylmaleimide.

However, this approach was unfortunately unacceptable for safety reasons. N-ethyl maleimide is a sensitizer, so therefore cannot be used safely with regard to an undergraduate laboratory.

The second approach was to pursue a condensation reaction between 2-hydroxybenzhydrazide and methyl 4-formylbenzoate to produce a hydrazone. Hydrazones are useful building blocks in organic synthesis and have been widely studied due to their ease of preparation and diverse pharmaceutical potential (10, 11). This reaction involved refluxing in methanol with an acetic acid catalyst. After refluxing, the system was cooled in an ice-water bath, and the resulting precipitate was vacuum filtered and washed with diethyl ether.

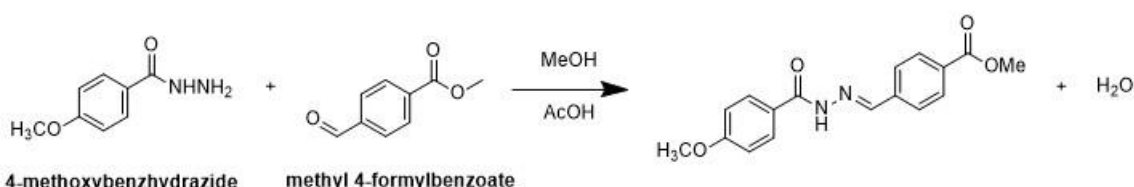


2-hydroxybenzhydrazide methyl 4-formylbenzoate

Figure 2: Reaction scheme for condensation reaction between 2-hydroxybenzhydrazide and methyl 4-formylbenzoate.

This reaction was not taken forward, as the solubility of 2-hydroxybenzhydrazide in methanol appeared to be very limited. This was a very important factor for this reaction as, in order to react, the reagent needs to be dissolved in the solvent. However, this reaction did provide a useful scaffold for the eventual successful synthesis candidate.

The final synthesis was a very similar condensation reaction. However, 2-hydroxybenzhydrazide was substituted for 4-methoxybenzhydrazide due to superior solubility.



4-methoxybenzhydrazide methyl 4-formylbenzoate

Figure 3: Reaction scheme for condensation reaction between 4-methoxybenzhydrazide and methyl 4-formylbenzoate.

Neither 4-methoxybenzhydrazide nor methyl 4-formylbenzoate are known hazardous substances. Using GSK's solvent sustainability guide as an objective assessment of the sustainability of the solvents used, it is clear that methanol (the solvent used in the greatest quantity), while still having some known issues, is exceptionally better than chloroform used in the original synthesis (12). In addition, considering only environmental impacts, methanol is classified as having no major known environmental impacts (12). While

it is true that both acetic acid and diethyl ether are classified as having some known issues and major known issues respectively (including major environmental issues in the case of diethyl ether), both of these solvents are used in very small volumes, so the use of these solvents in this case will have a negligible environmental impact (12). Similarly, in terms of economic sustainability, methanol, diethyl ether and acetic acid all have a similar price per litre compared to chloroform (~£20-30 per litre depending on supplier). In addition, methanol, diethyl ether and acetic acid are also all commonly used in large volumes within teaching labs, allowing for the possibility of bulk (cheaper) supply.

After this reaction was selected as the optimum candidate, a process then began to optimise the reaction conditions to both minimise scale and maximise yield of pure product. With the initial reaction, a starting quantity of 10 mmol of both starting reagents and a reflux time of 3 hours was used. These conditions produced an average yield of desired product of 79%, with analytical techniques used to ensure the purity of the product.

After reducing the scale and run-time to a starting quantity of 3 mmol of both starting reagents and a reflux time of 1.5 hours, the average yield produced remained at a similarly high level of 82%. Reducing the scale was preferential, as this reduced the consumption of materials and solvents (thus reducing the environmental impact), while reducing the reflux time was also favoured, as this would ensure the experiment would be able to be completed comfortably by all undergraduates within the duration of a typical synthetic undergraduate chemistry practical session.

As well as identifying a replacement reaction, all supplementary material for the procedure has been developed to ensure that it will be run this academic year, guaranteeing a transition to a more environmentally sustainable curriculum as soon as possible. Materials developed include a lab script, COSHH form and demonstrator notes. When constructing these documents, it was important to ensure that the lab script and demonstrator notes were in line with existing standards and that the COSHH form was compliant with safety regulations.

Microscale columns

After a chemical reaction has completed, the desired product will often contain impurities/be embedded in a mixture of products. As a result, purification techniques are required to isolate and purify the desired product. A very common method to do this (especially at an undergraduate level) is traditional column chromatography.

Traditional column chromatography involves large quantities of solvents and silica gel. Silica gel is an amorphous form of silicon dioxide, most commonly used in chemistry as a stationary phase within column chromatography. Silica gel is a material of concern, as it can penetrate deeply into the lungs and can cause severe damage over time, aiding the development of respiratory and autoimmune diseases, such as silicosis and lung cancer (7). There have been efforts made to transition, in some instances, to the use of so-called 'microscale' columns, using most frequently a Pasteur pipette. This can be beneficial as it reduces the environmental impact of the separation, reduces the economic cost of mistakes (especially important for often

inexperienced undergraduate students) and also reduces the risk of student overexposure to silica. Attempts were made to use these 'microscale' columns for a current introductory experiment to column chromatography at the University of Leeds. This experiment had involved the separation of a ferrocene/acetyl ferrocene mixture using traditional vertical glass columns. For context, a traditional column has a typical volume of ~600 mL, whereas a microscale column may have a typical volume of ~10 mL.

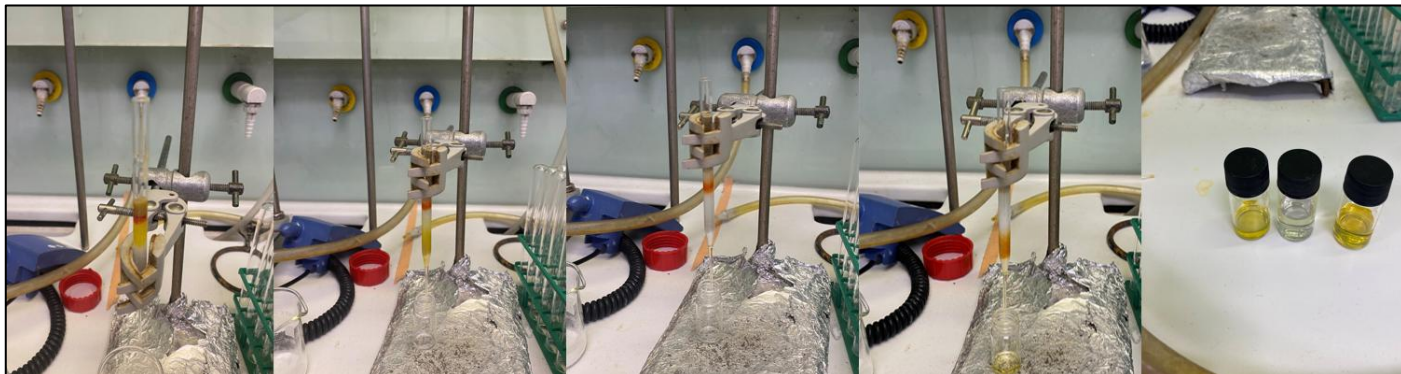


Figure 4: Pictures from the separation of the acetyl ferrocene/ferrocene mixture using a Pasteur pipette 'microscale' column.

There was a considerable amount of success in applying the use of Pasteur pipette columns, with a very noticeable and clear separation of the mixture being achieved despite minimising the scale significantly, from 1 g to 50 mg of material.

This same 'microscale' approach was then applied again using plastic disposable syringes as the column, achieving a similar level of success. Plastic disposable syringes are preferable to the use of glass Pasteur pipettes for first-year undergraduates, as there is a sizeable risk of breakage with glass Pasteur pipettes. This in turn creates a health and safety hazard. In addition, the environmental impact of using disposable plastic syringes is relatively minimal.

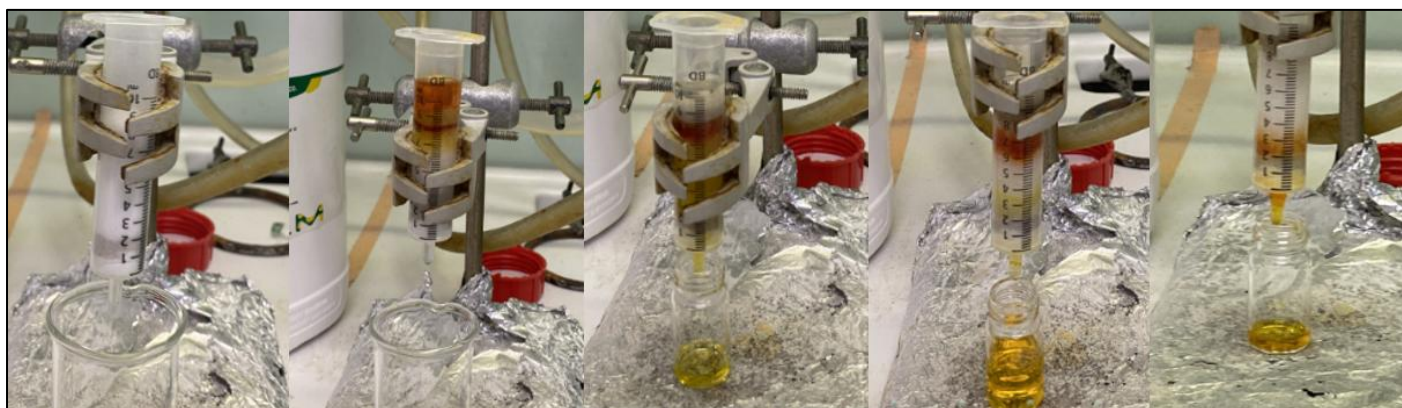


Figure 5: Pictures from the separation of the acetyl ferrocene/ferrocene mixture using a plastic disposable syringe 'microscale' column.

The success of the separation was confirmed qualitatively through NMR spectroscopy, and an instructional for the use of microscale columns in teaching labs for the separation of a ferrocene and acetyl ferrocene mixture was drafted. The instructional outlines a method of reducing the scale of the experiment. It is anticipated that this will be integrated in the 2026/27 academic year and reduce silica usage by several kilograms in each subsequent year.

Engaging with industry

A crucial component of the project was to look at improving the industrial relevance of undergraduate chemistry teaching laboratories. The chemistry landscape is constantly changing, so it is vital to make sure the course stays industrially relevant. This is important, as it ensures graduate employability is maintained and that the course is therefore economically viable. Engaging with senior personnel from a wide range of chemistry-relevant graduate destinations, including globally-leading pharmaceutical companies, comprised a significant portion of this element of research. This was mainly conducted through constructing a survey via MS Forms that aimed to address several key points. These key points comprised the use of 'green solvents' in industry, the most frequently executed chemical reactions in industry and the most important laboratory skills for graduates with regard to employability. While responses to the survey were somewhat limited, there were still enough responses to tentatively draw some meaningful conclusions.

The content of the survey questions was designed in order to address several key aspects that are important for industrial relevance. This included 'Likert'-style questions asking respondents to classify 'green' solvents in terms of the relative importance (low, intermediate, high) to their work. This is important, as understanding which solvents are more/less frequently used in industrial environments is crucial in ensuring undergraduate laboratory experiments can be designed with this framework in mind. On a similar topic, respondents were also asked to outline their most frequently executed classes of chemical reaction.

Another aspect included similar style questions but asked respondents to classify a range of common laboratory techniques in terms of the relative importance (low, intermediate, high) to their work. This was included because knowing which skills are most important in industry allows the design of authentically relevant undergraduate laboratory modules.

Finally, respondents were also asked to provide any comments about the practical experience of recent UK chemistry graduates, with a particular guiding question of "If you could add one thing to a teaching laboratory course what would it be?". This was included, as identifying any curricula gaps is crucial in ensuring the curriculum can be developed in a way to increase the long-term employability of graduates.

There were a few key highlights from the survey responses. Over 80% of correspondents considered water, acetonitrile and ethyl acetate as high-importance polar solvents for green chemistry. With regard to non-polar solvents, respondents considered toluene to be the 'green' solvent of most industrial importance.

When assessing laboratory techniques, reflux, liquid-liquid extraction and recrystallisation were considered to be the synthetic techniques of greatest importance by respondents (particularly useful due to the reflux component of the hydrazone synthesis). High-pressure liquid chromatography and mass spectrometry were considered the most important analytical techniques, while proficiency with software packages was also universally considered to be of importance by all respondents.

Predictably, there was little agreement on the most frequently executed chemical reactions in industry from respondents. A diverse and comprehensive coverage of a variety of different reactions is best placed to ensure students have industry-relevant practical experience.

The existing curriculum at the University of Leeds covers a wide range of techniques and industrially relevant reactions, particularly those highlighted by respondents as the most important. However, given the changing nature of both the pharmaceutical and manufacturing sectors (the two most significant employers of chemistry graduates), it would be beneficial to repeat this survey on a yearly basis and compare and contrast survey responses, ensuring that the curriculum remains appropriately agile to maintain industrial relevance. Given that the survey questions have already been constructed, it will be comparatively little effort to recirculate the survey periodically.

Conclusion

The overall goal of this research project was to evaluate and improve the environmental sustainability and industrial relevance of the undergraduate chemistry labs at the University of Leeds. In producing a new chemical synthesis using 'green' solvents for undergraduate labs that is already integrated into the curricula, progress was certainly made towards reducing the environmental impact of undergraduate labs. In replacing the previous synthesis, consumption of chloroform will now be reduced by over 1.5 L yearly. This progress was further reinforced through a successful exploration of the possible uses of 'microscale' columns, laying down a foundation for further changes in the future, with materials developed so that the 'microscale' column can be implemented for the 2026/27 academic year, reducing consumption of both reagent materials and silica. Finally, the survey responses are vital for the university to ensure that the course comprises all of the most industrially important techniques and skills. Ensuring employable graduates is as important as ever, especially in a changing professional world with the emergence of artificial intelligence. As a result, data such as this is extremely useful in working to improve and maintain the employability levels of students from the school, as well as ensuring the industrial relevance of labs overall.

Reflection

Conducting this research project has had a significant impact on my professional and personal development, allowing me to develop both leadership and technical skills. I have gained experience in independently producing professional COSHH assessments and undertaking key lab safety and compliance tasks. I have also been able to expand the range of synthetic and analytical techniques within my portfolio, including independently operating advanced spectroscopic instruments such as a 400 MHz NMR spectrometer. Effectively organising my time to ensure the completion of a number of tasks while meeting deadlines, including delivering a presentation about my findings to senior teaching lab staff, has no doubt had a profound impact on my ability as a leader. In terms of my future plans, I am currently seeking opportunities to undertake an industrial placement next year, primarily either within pharmaceuticals or manufacturing. Through completing this experience, I have been able to develop a number of vital transferable skills that I hope will accelerate my personal and professional development. Some examples include my ability to effectively problem-solve (e.g., adapting after the initial reaction candidate was unsuccessful), communicate within a professional environment (e.g., collaborating in-lab to ensure a safe working environment) and successfully manage projects. As a result, this project has significantly contributed to my long-term career development.

plans. In particular, experiencing laboratory research within a professional environment has informed my preference over my ideal roles for an industrial placement, especially compared to solely my undergraduate experience.

Footnotes

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