Curriculum Decolonisation and Improving Sustainability in Undergraduate Chemistry Teaching Laboratories

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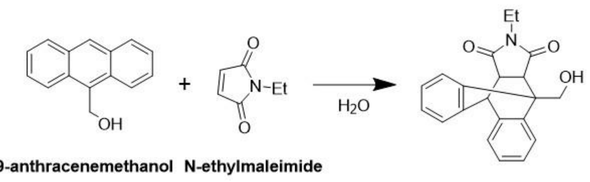
1. Introduction

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. In practice, this consisted of a number of key components:

- Developing a new synthesis practical using relatively environmentally friendly reagents and solvents, replacing a previous synthesis which involved refluxing in large volumes of chloroform (2-3).
- Assessing the viability of utilising 'microscale' columns to separate an acetyl ferrocene/ferrocene mixture (4).
- Attempting to understand the most important practical laboratory skills for graduates (5).

2. Initial attempts at a 'green' synthesis

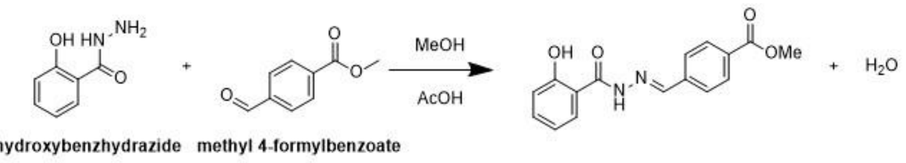
1. Diels-Alder reaction in water.



9-anthracenemethanol N-ethylmaleimide

- Unacceptable for safety reasons
- N-ethylmaleimide is a sensitiser
- Cannot be used safely with regards to an undergraduate laboratory.

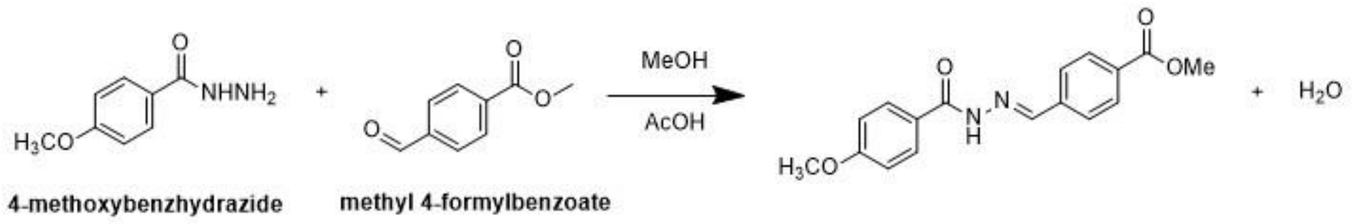
2. Condensation reaction between 2-hydroxybenzhydrazide and methyl 4-formylbenzoate.



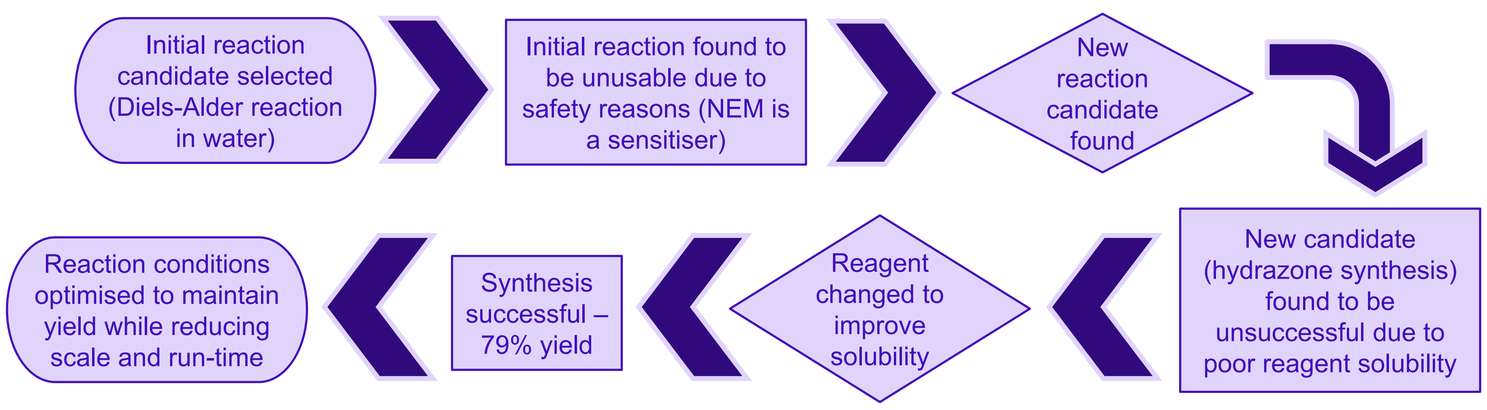
2-hydroxybenzhydrazide methyl 4-formylbenzoate

- Limited solubility of 2-hydroxybenzhydrazide
- Important as reagent needed to be dissolved in solvent (methanol) in order to react
- Provided a useful scaffold for the eventual successful synthesis candidate.

3. Synthesis of a hydrazone



4-methoxybenzhydrazide methyl 4-formylbenzoate



Key criteria	
Solubility	• 4-methoxybenzhydrazide was selected preferably over 2-hydroxybenzhydrazide due to superior solubility
Environmental impact	• Neither 4-methoxybenzhydrazide nor methyl 4-formylbenzoate are a known hazardous substance • Using GSK's solvent sustainability guide as an objective assessment of the sustainability of the solvents used, methanol (the solvent used in the greatest quantity), has a much smaller impact than chloroform used in the original synthesis
Industrial relevance	• Hydrazones are useful building blocks in organic synthesis • They have been widely studied due to their ease of preparation and diverse pharmacological potential

Reaction condition optimisation

		Reflux run-time / hours	
		3	1.5
Quantity of each starting reagent / mmol	10	79%	-
	5	70%	-
	3	79%	82%

Final steps

- ☑ Lab script developed
- ☑ COSHH form created
- ☑ Demonstrator notes produced
- ☑ Synthesis ready for incorporation for 25/26 academic year

Result

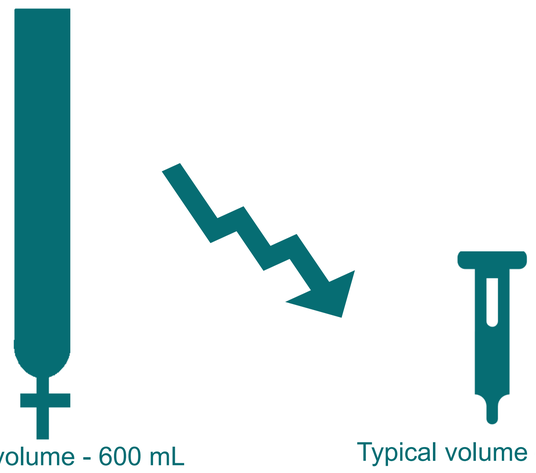
Consumption of chloroform in teaching labs reduced by over 1.5 L per year

4. 'Microscale' columns

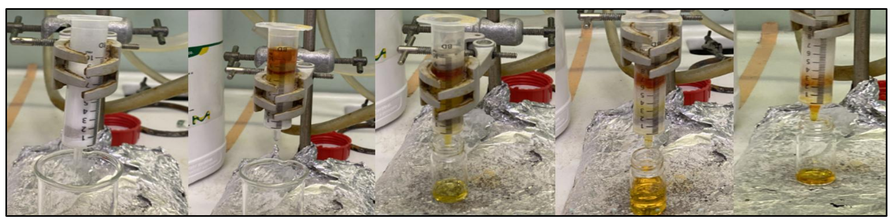
Traditional column chromatography involves large quantities of solvents and silica gel, leading to a large environmental impact

There have been efforts made to reduce this impact by transitioning to the use of 'microscale' columns, most frequently using a Pasteur pipette.

Successful attempts were made to use these 'microscale' columns for a current introductory experiment to column chromatography. The separation of a ferrocene/acetyl ferrocene mixture was successfully achieved using a disposable plastic syringe column.



Typical volume - 600 mL Typical volume - 10 mL



5. Engaging with industry

Attempts were made to engage with industry, primarily through a comprehensive Microsoft Form sent to senior personnel from leading pharmaceutical companies. This form aimed to address several topics and produced a few clear take-aways that will allow the curricula to be evaluated to ensure alignment with the responses :

1. The most important laboratory skills for graduates

Reflux, liquid-liquid extraction, HPLC, mass spectrometry, proficiency analysing NMR data and proficiency with software packages were among the most widely agreed important skills for graduates.

2. The most important 'green' solvents in industry

Ethanol and water were considered the polar protic 'green' solvents of greatest importance in industry.

Acetonitrile, ethyl acetate and acetone were considered the polar aprotic 'green' solvents of greatest importance in industry.

Toluene was considered the non-polar 'green' solvent of greatest importance in industry.

3. The most frequently conducted chemical reactions in industry

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There was little consensus on the most frequently conducted chemical reactions in industry among responses.

6. References and Acknowledgements

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