Novel approaches to individual hands-on crystallography learning for undergraduate students

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Development

Launched in 2013, the glycine polymorphism advanced practical[1] provided third year undergraduates with the ability to collect and analyse their own single crystal diffraction data. With the allocated time expanding from 2 to 3 weeks, the practical was reviewed. It was decided to reframe the exercise in order to provide students with an improved research experience, changing from a formulaic to a problem-led practical, whilst maintaining the excellent introduction to diffraction techniques offered by the original.

The review identified the following aspects to be retained:

- Students growing their own crystals.
- Hands-on experience with multiple analytical techniques (IR,HSM, PXRD, SCXRD).
- Analytical data workup and analysis.
- Crystal structure solution and refinement.
- Understanding the complete picture by bringing together results from different techniques
- Assessment criteria and style (at the chemistry module level).

Expanding the practical allowed for new components to be implemented and developed:

- A research exercise providing student choice and open enquiry.
- Planning an approach to the problem.
- Time and resource management.
- Use of analytical data to inform next steps.
- Independent structure analysis.

Research Problem

Students are told a pharmaceutical company wants to modify the properties of a tablet containing glycine by using co-formers to create co-crystals (or salts). At the end of their research, students will need to make a single recommendation from the co-formers provided.

Eligible co-formers are a range of organic di-acids, or simple inorganic salts (based on alkali or alkaline earth metals).

Limited access to analytical instruments.

Criteria categories are:

- New pure, single product
- Melting point: 130-160 °C.
- Non-hydrate
- Block crystals

Plan Experiment

Prelab exercise to relate analytical technique to data required to evaluate the desired product properties - encourages students to think about the kind of information they gain from each technique

Students select two co-formers from a list of eleven to mix with glycine in a prescribed ratio - gives student control over their own work, justification of selection is marked as there is no "right" starting point

Co-former information included, structure, mass, melting point and solubility in water at 20°C - students have to apply chemical knowledge to justify their selection and approach to solving the problem

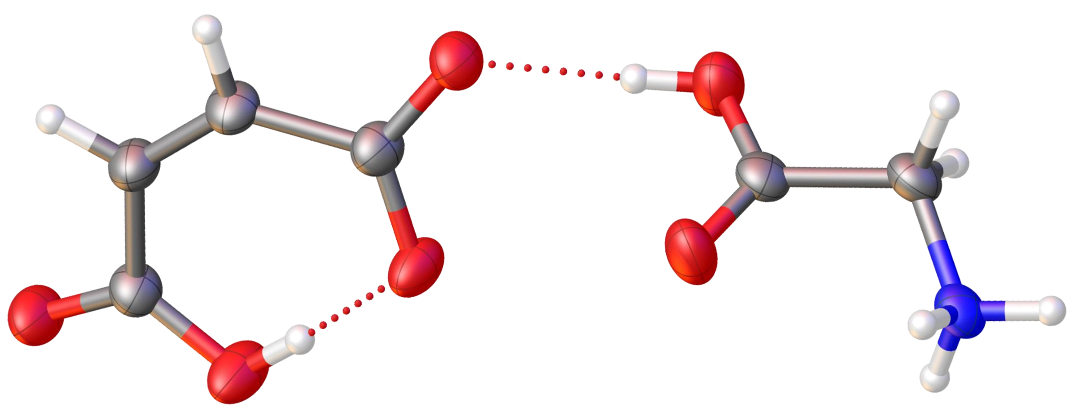
Plan out the order to run their analytical techniques to best reach a conclusion - encourages students to think about time and "cost "of each technique

Conduct Experiment

Mix components together, wait a week for the crystals to form - retains crystal growing element

Prelab videos show how the HSM, PXRD and SCXRD instruments operate - helps combat worry about using novel (and potentially expensive) equipment.

Students collect data on their own samples with only minimal intervention from demonstrators. Time on the instruments is limited and must be scheduled by negotiation with other students - students develop skills in independent working and time management. They also develop confidence using the instruments through repeated use.



Best Product:
Glycinium hydrogen maleate

Evaluate Results

Assess product against desired criteria - students gain insight not only into the results from individual techniques, but how a bigger picture can be seen from multiple techniques

After first products are analysed students can perform a second round of crystallisations - students use the first round of analytical data to inform their next choices.

Workup Data

An Olex2 workshop provides an introduction and familiarity with crystal structure analysis - this enables the students to independently solve their own structures

Collected raw data from instruments is processed into interpretable results - students develop the knowledge and understanding as to how to convert raw data into scientifically useful information.

Recommendation

After evaluating all available data on their products, students select the best possible match to the criteria and justify their selection - how they reach this recommendation is of more importance than the result itself, since it is unlikely they will obtain a sample that fulfils all the criteria.

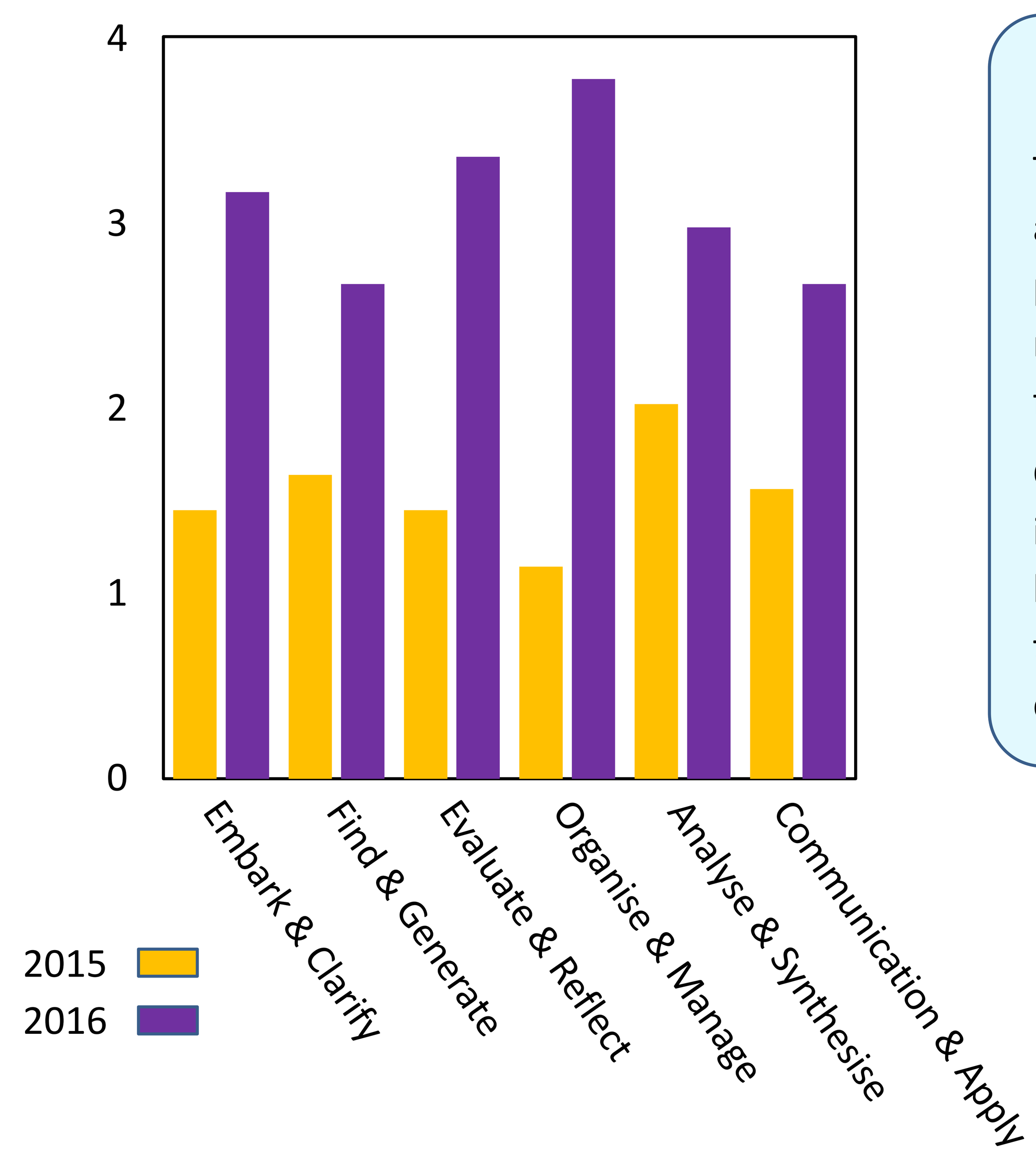
Student Feedback

A survey probed the following aspects:

- Enjoyment
- Engagement
- Skills development
- Resemblance to workplace or research experience

Notable findings include:

- 52% of students would like to see more practicals run like this (and a further 41% neither agreed nor disagreed)
- 93% of students felt the practical helped teach skills to become a better chemist
- 97% of students agreed that their understanding of the theory had improved (of which 41% strongly agreed).
- 80% of students felt this practical more closely replicated work done by professionals than previous practicals



Comparative RSD scores

Research Skill Development

The Research Skill Development (RSD) Framework [2] categorises six aspects of research on a scale from 1 to 5. 1 equates to highly structured, prescribed research, whilst 5 is unbounded research where the students make all the decisions (and corresponds to what would be expected for a final year PhD student).

Changing the practical has resulted in it evolving from a closed enquiry into a scaffolded research project. As such, students are able to develop higher level research skills than in previous 'cookbook' style practicals they have undertaken, whilst still providing a hands-on introduction to diffraction.

References

1. Coles, S.J. and Mapp, L.K., Conducting Reflective, Hands-On Research with Advanced Characterization Instruments: A High-Level Undergraduate Practical Exploring Solid-State Polymorphism, *J. Chem. Educ.*, 2016, 93, 131–140.
2. Willison, J., and O'Regan, K. Research Skill Development framework, 2013. (www.adelaide.edu.au/rsd/framework)

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