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# Conducting Reflective, Hands-On Research with Advanced Characterization Instruments: A High-Level Undergraduate Practical Exploring Solid-State Polymorphism

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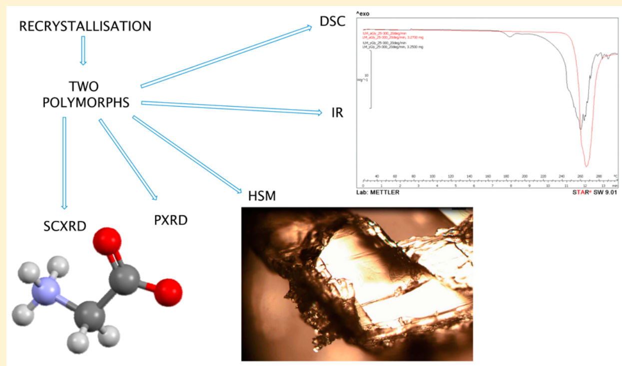
Supporting Information




**ABSTRACT:** An undergraduate practical exercise has been designed to provide hands-on, instrument-based experience of advanced characterization techniques. A research experience approach is taken, centered around the concept of solid-state polymorphism, which requires a detailed knowledge of molecular and crystal structure to be gained by advanced analytical techniques normally considered as the preserve of a research facility. Powder and single crystal diffraction techniques are primarily required and implemented via the unique approach of the students themselves using benchtop instruments dedicated to teaching, as opposed to more complex and difficult to access research instruments. Furthermore, the manual instructions for performing the practical are delivered via an adapted Electronic Laboratory Notebook system where, for each specific aspect of the practical, students note their intentions, actions, observations, and inferences. Assessors can access the notebooks and provide targeted online feedback for each individual section. Evaluation of the approach is based on interviews and surveys with the first cohort of 65 students that performed the practical.

**KEYWORDS:** *Upper-Division Undergraduate, Laboratory Instruction, Analytical Chemistry, Hands-On Learning/Manipulatives, Crystals/Crystallography, Hydrogen Bonding, Laboratory Computing/Interfacing, Thermal Analysis, X-ray Crystallography, Instrumental Methods*

## INTRODUCTION

Practical experience is crucial for a well-rounded education in chemistry. However, time,[1] resources,[2] and routes to deliver this are under extreme pressure,[3] leading to a decline in standards achieved. Hands-on learning and training is suffering as a result. In schools pupils are increasingly subject to demonstrations or detailed written instructions[4] whereby the "students can be successful even with little understanding of what they are doing"[5] and are unable to apply the tools and skills learned outside of the narrow teaching environment. In higher education, similar written instruction criticisms are valid, as students undergo formulaic practical exercises in very large teaching group sizes. Traditional methods of teaching practical skills, i.e., the "cook-book" approach, have not changed appreciably for decades. Students are often said to be "carrying out an exercise", rather than "doing an experiment", and exercises make limited intellectual demands.[6] Research indicates that students invest significant proportions of their time performing procedures without development of substantive understanding,[7] which does not provide sufficient skills training to prepare for future career work in industry or academia.[8−15]

It is not uncommon to have 60−80 students per cohort in a degree program, and practical training in large groups limits the quality of the experience and makes it difficult to provide training in advanced techniques. The primary limiting factors are logistics such as space constraints, timing, availability of educators, and access to advanced equipment. Hence there is a requirement for more imaginative delivery and timetabling of practicals, particularly in the latter stage of a degree program. The benefits of moving away from conventional teaching methods are well-known;[16−19] however, there is little evidence of addressing this. This paper presents an analytical practical exercise, akin to solid-state screening in the pharmaceutical industry, providing exposure to modern high-powered instrumentation and delivered to small groups of about 8 students at a time.

### The Challenge

For industry employability a strong analytical chemistry experience should be attained.[20] Increasing industry exposure has been achieved by two UK universities in different ways: (1) the University of Surrey with their "Analytical Club",[21] where a diverse range of companies (16 in this case) is involved in

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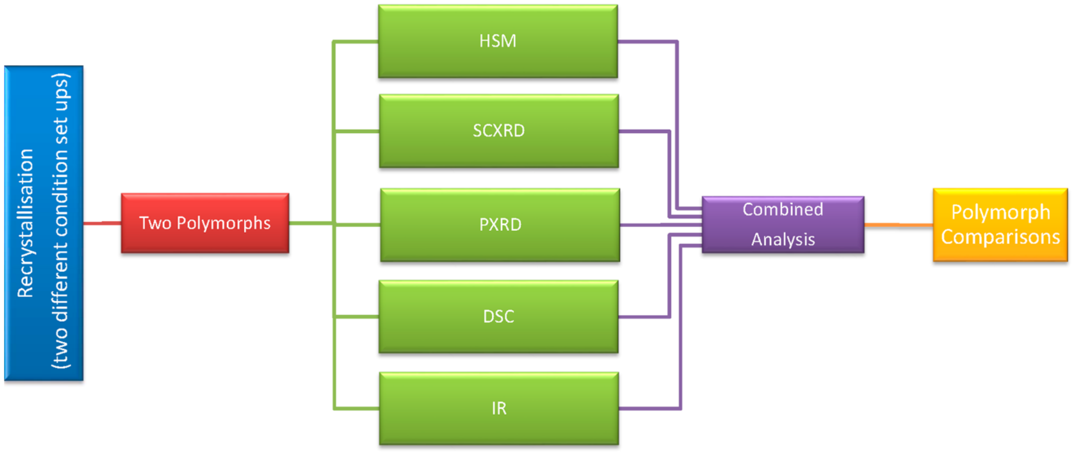


**Figure 1.** Schematic of the experiment and advanced practical techniques used in this exercise.

shaping degree schemes, tutoring, and providing real-world experience, and (2) the Glasgow Caledonian University's collaboration between students in physics and chemistry in a joint project.[22] A further shortcoming of undergraduate practicals is a lack of exposure to (industrial) standard operating procedures. For example, it is standard practice to use an electronic laboratory notebook (ELN) to document practical work and associated data. These standards are not met, and rarely even taught, as students use paper notebooks, and any data generated is dissociated from these.

The established model for practical training at the University of Southampton places first and second year undergraduates into teaching laboratories, following prescribed methods in large group sizes and gaining foundational experience in common analytical techniques, e.g., infrared (IR) and precollected nuclear magnetic resonance (NMR) spectra. However, access to advanced characterization techniques, specifically hands-on X-ray crystallography,[23−25] at the undergraduate level is restricted, mainly due large to group sizes and scarce resources. These techniques would only be (partly) experienced in the latter stages of a degree if an embedded research project were undertaken. There are examples of characterization data being made available to students, e.g., via a VLE (virtual learning environment),[26] as well as an alternative approach involving a virtual multifunctional X-ray laboratory,[27] which attempt to address this issue, but there is no substitute for actual practical experience. Final year research projects often provide an enriching environment; however, with increasing student numbers, quality is decreasing and the strain placed upon research groups is increasing. Additionally, the research project often gives exposure only to a narrow field of chemistry and set of techniques. While an "Analytical Club" seems a good approach, this is externally dependent, requiring continued collaboration with industries. Due to these factors, development of an in-house approach can be considered a more favorable option.

## Embracing the Challenge

The first design principle is to encourage student engagement, which has been a topic of debate for some considerable time.[28] The intention of this advanced practical is to break the monotony and formulaic approach of the current laboratory experience; while this serves well for basic skills training, it is less well suited for experienced students preparing for postdegree work. By being provided with an environment more akin to the research workplace and introducing a richer and more challenging variety to the practical experience, the student is not only more engaged but also prepared for employment. This is specifically achieved by

- increasing the level of independence required to complete not only individual tasks but the whole study;
- the use of multiple complementary analytical techniques, requiring piecing together data from disparate sources to draw conclusions on a larger scale than an individual technique;
- hands-on use of advanced characterization equipment, engendering greater participation and understanding;
- providing an alternative method to deliver practical instructions;
- alternative methods of recording and reporting experimental procedure, observations, and results and incorporating the assessment into this approach.

Solid-state polymorphism, a material existing in more than one solid form, is well suited to introducing these approaches for several reasons. Fundamentally, the concept has not been considered in depth prior to the experiment, although some foundational knowledge of structure and diffraction has been established from earlier years. Therefore, a greater level of self-led study and background reading is required. Second, the approach described herein develops understanding of the solid state through a crystallography-led practical incorporating a variety of techniques. To probe the phenomenon and its effect on physical properties it is necessary to draw on data from several techniques. Moreover, it is possible to produce polymorphs via different recrystallization techniques within the time frame of the practical, thus providing a beginning-to-end experience.

A similar research-led initiative, in that it is based on the topic of crystallography, was introduced by Wilson et al.,[29] who provided a supplementary course in a final year degree scheme. Drawing on samples generated by researchers in the department, students partake in the research process and explore aspects of chemistry that they have not previously had exposure to. The students gained an overview of the area of single crystal diffraction, had access to the diffraction laboratories, and gained some hands-on experience. However, it is important to note several differences with the scheme being presented here, primarily that the practical we describe is fully integrated into the undergraduate program with formal assessment. Moreover,


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our practical has adopted a 100% hands on approach to introducing a variety of techniques and encourages using multiple analyses to draw conclusions while enabling a full beginning-to-end experience with crystals grown by the students themselves.

## ■ THE EXPERIMENT

At the module level, five different practical exercises spanning different areas of chemistry are undertaken in a semester. A single practical takes two full days: 1 day a week over a fortnight. Accordingly this practical was delivered to 65 third year bachelors and fourth year masters students (in the ratio 40:25 respectively). To operate with an appropriate level of supervision a minimum of one PhD student laboratory demonstrator dedicated solely to this practical was required.

Glycine, a simple molecular organic system for which crystallization and polymorphic behavior are well documented, was chosen as the subject.[30] Crystallization is readily reproducible for two polymorphs ($\alpha$ and $\gamma$). Additionally it exhibits a phase transition between these two forms on heating: this is another new concept, which distinguishes students who fundamentally understand the practical.

A schematic of the practical is given in Figure 1 and consists of the following components: stock material (from Sigma-Aldrich) was used to generate two polymorphic forms via recrystallization under aqueous and strongly alkaline conditions. Once crystals had formed, structural analysis was performed via number of differing techniques including single crystal (SCXRD) and powder (PXRD) X-ray diffraction and infrared (IR) spectroscopy; the latter two techniques were also used on the stock material to allow structural determination and bulk purity. Thermal behaviors were also investigated for both polymorphs using hot stage microscopy (HSM) and differential scanning calorimetry (DSC). To complete the study, literature searching and interrogation of structural databases such as the CSD (Cambridge Structural Database)[31] were carried out to obtain background information and additional data to aid in the final comparisons. Using all data obtained, correlations and relationships between the polymorphs were assessed which involved

- polymorphs identified by cross referencing with published literature;
- "round-tripping", that is, calculating powder patterns from crystal structures and comparing to experimental PXRD data to assess if SCXRD is representative of the bulk material;
- analysis of intermolecular interactions in crystal structures, which could then be correlated with IR data;
- exo- or endothermic processes quantitatively identified by DSC;
- DSC results correlated to HSM observations, enabling identification of melting point and phase transitions;
- thermal behavior of the polymorphs related to crystal structure.

We present the practical as implemented in our institution; however, it is modular with scope for modification into more, shorter laboratory sessions (generate the crystals in one session and then characterize over several subsequent sessions). This permits one to cater for different size groups and fit into timetabling constraints requiring shorter timeslots. A modular approach also enables a curtailed experiment with lesser or limited analysis to be undertaken. The SCXRD data collection requires a few hours for each crystal and could be done in parallel with other techniques but is dependent on group size. Modifications due to equipment availability and access could use melting point apparatus or TGA in place of HSM; however, the phase transition may not be so easily identifiable.

The data collected provide a wealth of information and allowed numerous further interpretations and connections to be made; packing and arrangement of molecules within the unit cell could be visualized; comparisons between hydrogen-bonding networks in the different polymorphs could be made which provide an insight into stabilities and also aid the explanation of the thermal characteristics observed in the DSC and HSM experiments; polymorphic variations are also apparent in the IR spectra and could be related to hydrogen-bonding differences between crystal structures.

### Learning and Assessment Goals

The general learning objectives at the module level enable students to

- apply practical skills in a more open-ended context;
- apply core chemistry knowledge in addressing advanced problems;
- develop a range of key skills—experimentally and in presenting results;
- manage their own learning.

The learning objectives of this particular practical are for students to

- conduct elementary experiments on advanced characterization equipment;
- perform analysis of data from advanced characterization experiments and critically evaluate the results;
- understand the complementarity of different characterization techniques and how this provides insight beyond that of an individual measurement;
- recognize that specialized databases form part of the prior work that should be researched alongside the primary literature;
- be able to record experimental observations to the required standard using an alternative approach to the traditional paper notebook (i.e., an ELN).

In alignment with the learning objectives above, the high-level module assessment goals are that students

- communicate results, both verbally and in written form, of an open-ended investigation using appropriate scientific terminology;
- manage personal study time effectively;
- work effectively as a member of a team as required;
- work independently toward achieving well-defined objectives;
- prepare risk assessments;
- record practical work to a professional standard;
- collate and analyze data from a variety of sources.

More specifically, the goal of assessments for this practical are that students can

- appreciate the factors involved in accurately conducting characterization experiments;
- work up crystallographic data and critically assess the validity of the resulting crystal structure;
- compare and contrast results from complementary techniques to gain deeper insight into the phenomenon


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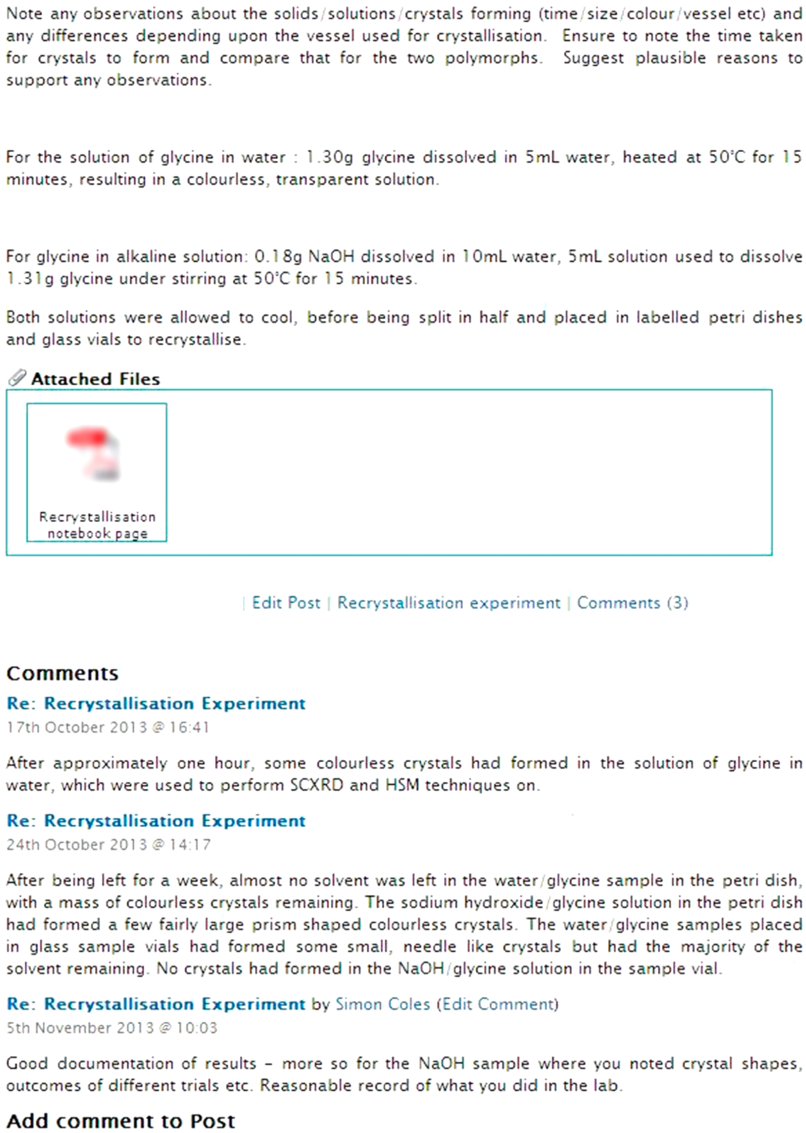

Note any observations about the solids/solutions/crystals forming (time/size/colour/vessel etc) and any differences depending upon the vessel used for crystallisation. Ensure to note the time taken for crystals to form and compare that for the two polymorphs. Suggest plausible reasons to support any observations.

For the solution of glycine in water : 1.30g glycine dissolved in 5mL water, heated at 50°C for 15 minutes, resulting in a colourless, transparent solution.

For glycine in alkaline solution: 0.18g NaOH dissolved in 10mL water, 5mL solution used to dissolve 1.31g glycine under stirring at 50°C for 15 minutes.

Both solutions were allowed to cool, before being split in half and placed in labelled petri dishes and glass vials to recrystallise.

**Attached Files**

Recrystallisation notebook page

| Edit Post | Recrystallisation experiment | Comments (3)

**Comments**

**Re: Recrystallisation Experiment**
17th October 2013 @ 16:41

After approximately one hour, some colourless crystals had formed in the solution of glycine in water, which were used to perform SCXRD and HSM techniques on.

**Re: Recrystallisation Experiment**
24th October 2013 @ 14:17

After being left for a week, almost no solvent was left in the water/glycine sample in the petri dish, with a mass of colourless crystals remaining. The sodium hydroxide/glycine solution in the petri dish had formed a few fairly large prism shaped colourless crystals. The water/glycine samples placed in glass sample vials had formed some small, needle like crystals but had the majority of the solvent remaining. No crystals had formed in the NaOH/glycine solution in the sample vial.

**Re: Recrystallisation Experiment** by Simon Coles (Edit Comment)
5th November 2013 @ 10:03

Good documentation of results – more so for the NaOH sample where you noted crystal shapes, outcomes of different trials etc. Reasonable record of what you did in the lab.

**Add comment to Post**

**Figure 2.** Screen shot of a student's notebook record containing the manual instructions, top, followed by any uploaded data or files, the student's comments, and finally an assessor's feedback.

of polymorphism through hydrogen bonding networks and relative stabilities seen through phase transitions;

- record experimental setups, observations, inferences, and conclusions in an ELN such that someone else can understand what has been done and can reproduce the experiment.

## ■ CHARACTERIZATION INSTRUMENTS

A range of equipment is required (one instrument for each of the techniques outlined in Figure 1), and it must be configured to be operable by students with no prior experience. Diffraction equipment used in a research context does not necessarily fit this criterion, and accordingly benchtop diffractometers (Rigaku MiniFlex 600 and a XtaLAB mini for powder and single crystal diffraction respectively) were used. Details of a collaboration with Rigaku can be found in a video testimonial[32] and the April 2014 edition of *The Bridge*.[33] The XtaLAB mini provides the same features and data collection experience as a larger research instrument, however a more compact design and simpler layout means that operation is much easier. The software is identical to that used on research equipment, however it can be configured in a semiautomated way so that advanced parameters are hidden.

The Miniflex PXRD instrument is similarly suitable for education. Other equipment used includes a Mettler Toledo 82HT Hot Stage for the HSM measurements. The DSC data (provided to the students) and IR spectra were collected on a Mettler Toledo DSC821$^{e}$ and a Thermo Scientific Nicolet iS5 FT-IR spectrometer, respectively.

## ■ PRACTICAL DELIVERY AND IMPLEMENTATION

The practical used a customized academic ELN, LabTrove,[34] to provide the experimental manual, split into sections for different parts of the experiment, see section 1 of the Supporting Information. To the best of our knowledge there are no significant reports in the literature of an ELN being employed (and tailored) specifically for delivery and assessment of educational exercises. However, a recent review[35] provides insights into the role of ELNs for record keeping.


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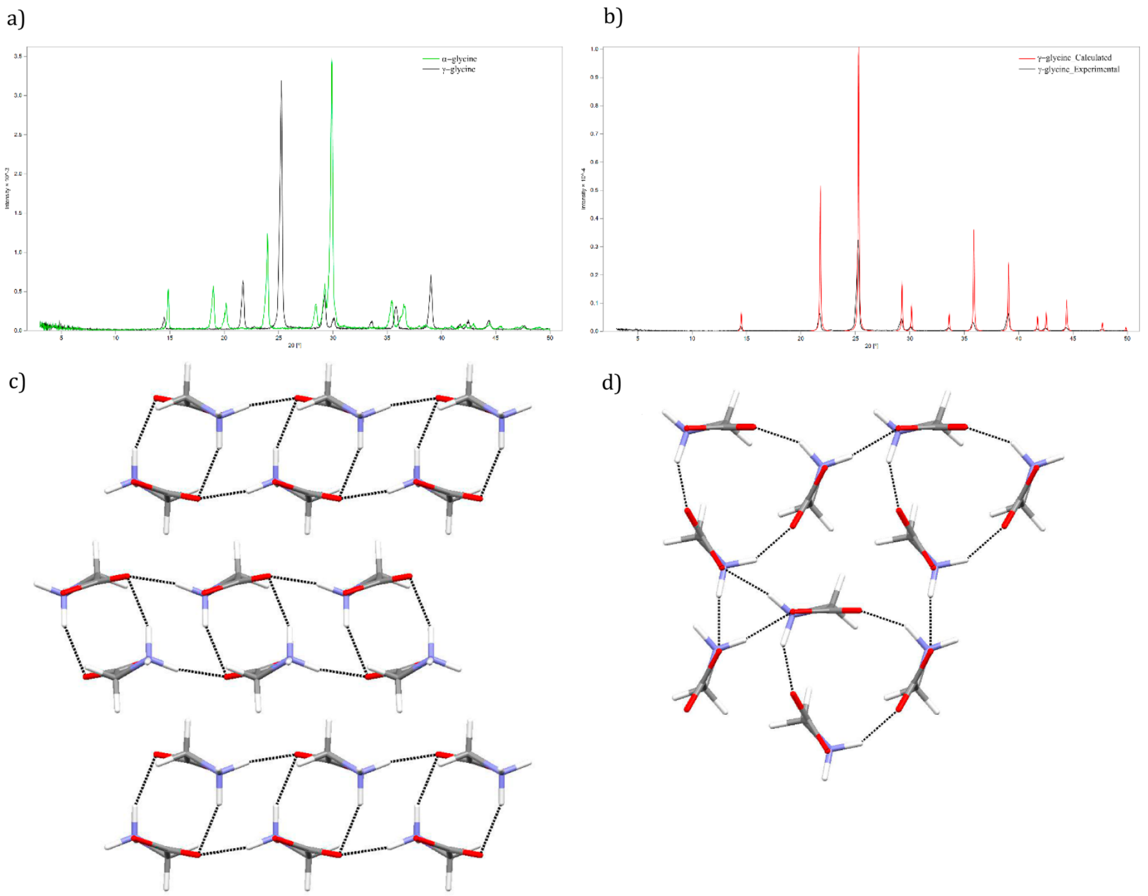

Figure 3. Examples of student data provided to rationalize observations: (a) PXRD comparison of polymorphs; (b) overlay of experimental XRPD with that simulated from SCXRD for gamma polymorph; (c) hydrogen bonding in alpha polymorph; (d) hydrogen bonding in gamma polymorph.

Students record their observations, inferences, conclusions, and data in a way that is linked to the different manual sections, and supervisors can view this content and leave feedback comments for each section during their assessment. The manuals for each component of the experiment, detailing practical requirements and instructions for specific equipment, are provided as a general template from a central notebook, available prior to the practical to allow students to familiarize themselves with the electronic system and experimental procedures. Students copy a template into their individual notebook, creating a new record to which details of process, observations, inferences, and data can be added. All the templates developed for this experiment have been packaged as a learning resource and are openly available via the University of Southampton EdShare repository at http://www.edshare.soton.ac.uk/13584/. The format of the ELN is nonprescriptive and provides several possible ways for a user to structure their work. Metadata (descriptive terms, or keywords) are used to link or group related records, e.g., the machine, technique, or software used, and are entirely user-defined, hence individual preferences can easily be met. This facilitiates navigation around the notebook and categorization of observations, while providing the individual some flexibility to organize their notes as they wish.

The key feature of this approach is that students can record all planning, observations, thoughts, and results together and these can readily be linked to the corresponding part of the manual. Furthermore, data from analytical instruments such as spectra, structure files, videos, images, and analysis documents can be uploaded and linked to the appropriate part of the experiment record (Figure 2). Students' observations are recorded as comments, to which others can also make comments if they have appropriate access privileges. In this case instructors can see all student records for assessment purposes, however students could only see their own notebook's contents.

## ■ ASSESSMENT

There were a range of assessment methods associated with the practical, including a critique of the Laboratory Notebook and COSHH (safety) assessment, "follow-up" questions (section 2 in the Supporting Information), a poster, a short presentation, and a short journal article. These were designed and delivered at the module level which encompasses a number of experiments and therefore align to the assessment aims defined for that level (vide supra). It is important that core chemistry knowledge is applied to address advanced problems—this is tested through the follow-up questions and by an understanding of the hazards and risks involved. Awareness of safety was tested through a COSHH (Control of Substances Hazardous to Health)[36−38] assessment form (see Supporting Information for blank form) prior to starting the practical. Students must identify and assess all chemicals and processes to determine the appropriate protocols and safety requirements necessary to undertake the experiment in a safe manner. Finally, students are assessed on their ability to manage their own learning by examining their ELN notes.



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J. Chem. Educ. 2016, 93, 131−140

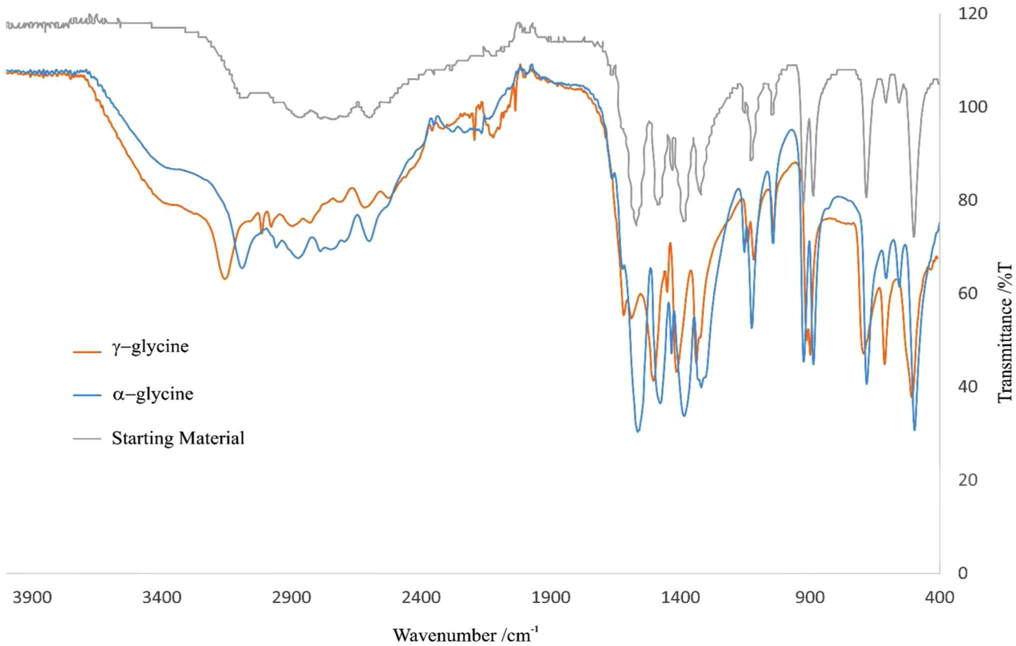


**Figure 4.** Comparative overlay of the infrared spectra of the glycine forms studied.

More specifically, at the level of the individual practical, the learning outcomes (vide supra) are matched to assessment in that students learn to

- Appreciate the factors involved in accurately conducting characterization experiments—primarily assessed by the extent of ELN notes.
- Work up crystallographic data and critically assess the validity of the resulting crystal structure—the data files and associated observations are available in the ELN while the ability to critically assess the results is assessed by observations in the ELN and the questions, paper writeup, and oral presentation.
- Record experimental setups, observations, inferences, and conclusions in an ELN such that someone can reproduce the experiment.
- Compare and contrast results from complementary techniques to gain deeper insight into phenomena such as hydrogen bonding and phase transitions; assessed by follow-up questions, paper writeup, and oral presentation.

The follow-up questions required bringing together data from all the complementary techniques to form conclusions. Examples (see Figure 3) include DSC and HSM data correlated to confirm melting points and recognize a phase transition; different melting points rationalized by analysis of packing and hydrogen bonding networks evaluated from crystal structures and IR; calculating a powder pattern from the crystal structure (Mercury software)[39] and comparing it to the experimentally determined PXRD pattern.

General observations on the level of understanding include the following:

- Relevant literature on glycine including PXRD patterns was readily found, and key peaks were identified and compared to experimental data. However, this led to PXRD being used to fingerprint rather than compare polymorphs.
- Many related IR bands to the functional groups present, but did not identify subtle differences due to intermolecular interactions. Figure 4 presents spectra where a student was able to identify from a good match that the starting material is the gamma polymorph, but failed to realize significant differences between the two forms around 1300−1600 and 2500−3150 $cm^{-1}$ reflecting changes in N−H stretching and bending due to its different hydrogen bonding modes.
- Comparison of HSM and DSC data generally led to the conclusion, with the help of the literature, to the presence of a phase transition.
- Different melting points were attributed to the different structures, however it was not readily recognized that the different hydrogen bonding networks are a major influencing factor (in fact a significant number failed to appreciate the existence of hydrogen bonds).

From this one can conclude that students can assess individual characterizations, but fail to appreciate the value of comparing, contrasting, and correlating techniques.

## ■ EVALUATION

The evaluation comprised two components, one a qualitative approach including observation of student behavior and conducting interviews. 1:1 interviews were conducted with 3 students by the PhD student demonstrator (L. Mapp). The responses to these interviews informed the design of the questionnaire (vide infra).

At the module level a general Student Experience Questionnaire is delivered via the BlackBoard VLE;[40] however, to fully and quantitatively evaluate certain aspects of this practical, a more in-depth method is required. The second element to evaluation was a specific questionnaire broadly probing student engagement, effectiveness of the hands-on approach, and effectiveness of the ELN system. The response rate was 45% (29 out of 65 students), and the full set of questions is in section 3 of the Supporting Information, while the full range of feedback comments is in section 4. A summary


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of the primary points probed is presented in Table 1—these points are generated either directly from one of the survey questions or by pooling the responses to >1 question that addressed the same issue (for the exact mapping of questions to categories in Table 1 see the Supporting Information).

**Table 1. Proportion of Respondents Recording a Positive Reaction to Specific Categories of Question**

| Question Category of Student Response | Positive Responses[a]/ % ($N$ = 29) |
|---|---|
| Was there concern about using this instrumentation prior to the experiment? | 30 |
| Was there a benefit from using the equipment? | 100 |
| Better understanding of theory as a result of practice? | 48 |
| Data interpretation facilitated as a result of practice? | 94 |
| (Positive) Attitude toward ELN before? | 35 |
| (Positive) Attitude toward ELN after? | 72 |
| ELN better than paper recording? | 53 |
| ELN helps order notes? | 57 |
| Sharing notes with assessor beneficial? | 100 |
| Feedback in ELN beneficial? | 66 |

[a]Questionnaire response rate was 45% (29 out of 65 students).

## Evaluation of the Hands-On Equipment Approach

The techniques employed were more complex than those used in previous aspects of the undergraduate course, and accordingly many found the prospect exciting and were positive; however, a significant number were apprehensive (worried about breaking expensive equipment).

A significant number considered their knowledge of the underlying theory to have improved. There is an overwhelming impression that the interpretation of results was facilitated as a result of actively collecting their own data and working it up to the result. For advanced instrumentation this is in contrast to the more commonly used passive learning process of being presented with precollected data or a demonstration.

Data analysis has two different challenges. First, obtaining a result can range from trivial to involved data workup requiring advanced skills. Second, one must draw on the results of several complementary techniques. Data workup was performed in the laboratory enabling peer-to-peer development and consulting instructors while the broader analysis was performed individually outside of the practical, which many found harder.

There are two areas where more attention should be focused. First, students should make a connection between the experiment and the related theory (this may be delivered in previous years). Second, students have not been able to fully compare and contrast data from complementary techniques and relate this to scientific concepts—e.g., linking hydrogen bonding to thermal behavior.

## Electronic Delivery, Recording, and Reporting

Table 1 shows an increase in positive attitude, from 35% to 72%, toward the use of an ELN after having used one. Student feedback attributes this to improvements in working in a digital environment (structure/ordering, legibility, different data types, etc.) and working in an online environment (reporting and feedback can be exchanged between student and assessor and accessed any time from anywhere).

Comparison of a traditional paper lab notebook and an ELN entry illustrates the benefits of a digital environment (Figure 5). It was envisaged that observations would be recorded directly in the ELN; however, in practice, due to logistics and not having a suitable method, a paper notebook was often completed while performing the experiment. The ELN records then became a combination of immediate note taking and informal postexperiment write-up—this had unforeseen advantages.

Recording observations at the time does not necessarily produce a record in the most suitable layout for reviewing or reporting. The ELN has the advantage that a comment can be added to any record at any time, ensuring that all aspects of one particular technique are collated and organized, regardless of when the information is added. Not only can students reflect and structure their thoughts from the laboratory, but also a digital format provides a legible and structured form, unlike most (hastily recorded) paper records. Moreover one can upload any format of data: data files collected during the experiment (raw or analyzed) as well as diagrams or other files, e.g., photographs or movies recorded on mobile devices. The ELN therefore became a mechanism to focus thoughts and rewrite rudimentary notes, leading to a more ordered, meaningful, and considered record. Submission of laboratory notebooks for assessment was favored as students felt it less formal and they could write exactly what they thought and would not be embarrassed by hastily recorded notes. Also, by using the structure of sections and a template, students made more comments than with a traditional notebook due to the template providing a prompt compelling an answer—thereby students think more about all aspects of the experiment.

There were, however, some less well received aspects to this system. A number preferred to write notes rather than typing, finding it faster, and sketching rather than using comments.

The online ELN may be viewed by an assessor. This improves previous approaches to the submission of laboratory notebook-based reports and delivery of feedback. From feedback (Table 1), students unanimously welcome this method of sharing laboratory notebooks with assessors and the majority find it beneficial as a route to receiving feedback. With the experiment split up into sections, it is possible to provide targeted feedback at a fine-grained level, i.e., for all sections of the experiment. This is in contrast to the usual level at which feedback is delivered, which is rather more general. This was thought to be advantageous as evidenced by the following comments: “easier to see where individual feedback was directed”; “got feedback on all aspects of the practical”. However, conversely, some students felt that this was too dispersed as it necessitated searching through the entire notebook to retrieve all feedback comments: “more detailed feedback although not all in one place”.

## Student Experience

The quality of the student experience was evaluated through analysis of the questionnaire responses. There were many points coming from this, indicating that students appreciated the approach. Progressing through from growing crystals, collecting a variety of data, and analyzing these to understand an entire phenomenon proved very constructive. Comments from students relating to the experience included the following: “It was interesting and valuable to see the process all the way through from the crystallisation to obtaining crystals, the analysis and finally solving the crystal structure.” “The crystallography practical was a great way for us as undergraduates to be able to perform something which we had never done in practice before.”


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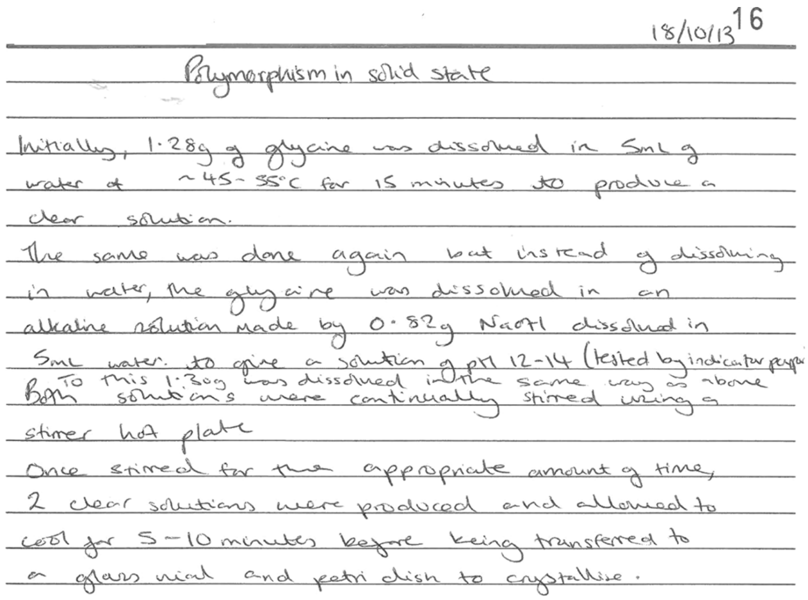
J. Chem. Educ. 2016, 93, 131−140

(a)



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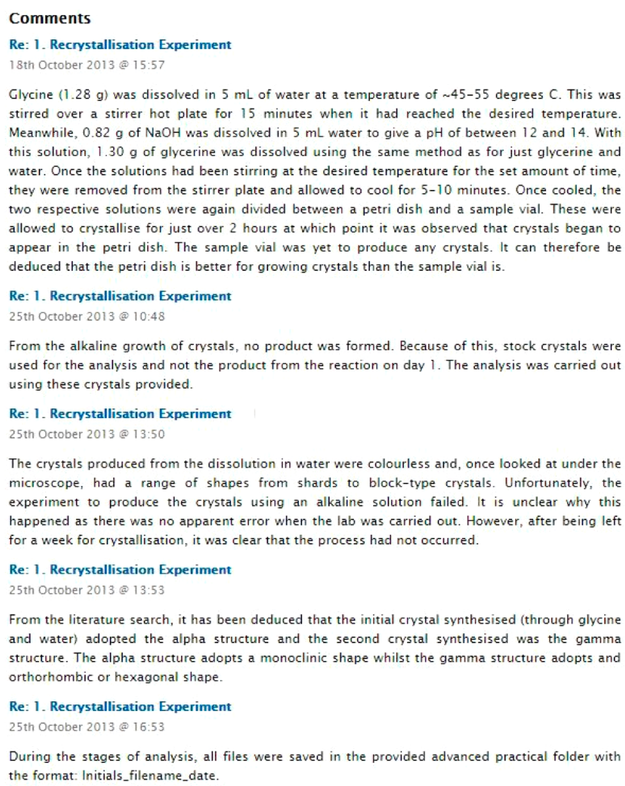
Polymorphism in solid state

Initially, 1·28g of glycine was dissolved in 5mL of water at ~45–55°C for 15 minutes to produce a clear solution.

The same was done again but instead of dissolving in water, the glycine was dissolved in an alkaline solution made by 0·82g NaOH dissolved in 5mL water. to give a solution of pH 12–14 (tested by indicator paper). To this 1·30g was dissolved in the same way as above. Both solution's were continually stirred using a stirrer hot plate

Once stirred for the appropriate amount of time, 2 clear solutions were produced and allowed to cool for 5–10 minutes before being transferred to a glass vial and petri dish to crystallise.

(b)



**Comments**

**Re: 1. Recrystallisation Experiment**

18th October 2013 @ 15:57

Glycine (1.28 g) was dissolved in 5 mL of water at a temperature of ~45–55 degrees C. This was stirred over a stirrer hot plate for 15 minutes when it had reached the desired temperature. Meanwhile, 0.82 g of NaOH was dissolved in 5 mL water to give a pH of between 12 and 14. With this solution, 1.30 g of glycerine was dissolved using the same method as for just glycerine and water. Once the solutions had been stirring at the desired temperature for the set amount of time, they were removed from the stirrer plate and allowed to cool for 5–10 minutes. Once cooled, the two respective solutions were again divided between a petri dish and a sample vial. These were allowed to crystallise for just over 2 hours at which point it was observed that crystals began to appear in the petri dish. The sample vial was yet to produce any crystals. It can therefore be deduced that the petri dish is better for growing crystals than the sample vial is.

**Re: 1. Recrystallisation Experiment**

25th October 2013 @ 10:48

From the alkaline growth of crystals, no product was formed. Because of this, stock crystals were used for the analysis and not the product from the reaction on day 1. The analysis was carried out using these crystals provided.

**Re: 1. Recrystallisation Experiment**

25th October 2013 @ 13:50

The crystals produced from the dissolution in water were colourless and, once looked at under the microscope, had a range of shapes from shards to block-type crystals. Unfortunately, the experiment to produce the crystals using an alkaline solution failed. It is unclear why this happened as there was no apparent error when the lab was carried out. However, after being left for a week for crystallisation, it was clear that the process had not occurred.

**Re: 1. Recrystallisation Experiment**

25th October 2013 @ 13:53

From the literature search, it has been deduced that the initial crystal synthesised (through glycine and water) adopted the alpha structure and the second crystal synthesised was the gamma structure. The alpha structure adopts a monoclinic shape whilst the gamma structure adopts and orthorhombic or hexagonal shape.

**Re: 1. Recrystallisation Experiment**

25th October 2013 @ 16:53

During the stages of analysis, all files were saved in the provided advanced practical folder with the format: Initials_filename_date.

**Figure 5.** A comparison of paper and electronic note taking for the same experiment.

Previously, students performed single reactions in a controlled environment with analysis being a single, simple analytical technique. The experiments were predescribed and planned to give a reliable outcome, therefore this practical provided a rich learning experience more akin to research. The following quotes illustrate this: "Feels like a two-week mini-project and comparable to the third year project [I] would otherwise be undertaking." "[Practical] was helpful for showing how characterisation can be used. Use of SCXRD is especially well received". Hands-on access to advanced equipment not normally available was warmly embraced, as evidenced by the following: "The hands-on approach to the experiment really allowed us to further our understanding of the technique as well as increasing our overall analytical ability in lab work." "New skill set, chance to experience another side of chemistry."

The format of the practical required students to manage their own time to complete tasks and negotiate within the group for instrument time: "Less regimented than years 1 and 2." "It was good to choose when and what you did, especially on the second day as had certain tasks to complete but could decide when and how." "Proximity of everything and working in the same room made checking access and when equipment was free very easy." Some work was completed in pairs, allowing peer and small group learning, which some found useful: "Can



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J. Chem. Educ. 2016, 93, 131−140

discuss between a pair if you don't fully understand something before asking another pair or a demonstrator." The semi-structured approach and pair work made students more comfortable in this new environment: "Made the work seem more manageable and less intense [than lone working]." This type of practical provided an excellent extension and reinforcement to previously learned theory—one student stated, "My understanding of how the theory is applied with the machines has improved." Using benchtop instruments introduces advanced techniques without the potentially overwhelming experience of research instruments. Accordingly this practical provides a transition from taught undergraduate to research-intensive project: "I now feel that I am more capable of operating this kind of equipment whereas before I wouldn't have been too confident."

Additional feedback evaluation statements are provided (section 4, Supporting Information).

## ■ FUTURE WORK

The design of the practical and the techniques involved are fit for purpose at this level, and the main developments should be made to the ELN system.

First, reaction regarding feedback delivery indicated that several students were unaware of its availability; it would be advantageous to implement alerting indicating that feedback is available and how to view it. Providing fine-grained feedback is time-consuming, so a "feedback library" of frequently used statements would be helpful to saving time and include more detail.

Second, the ELN was not implemented in the way the designer conceived it, therefore additional features could be included to support use in education. It was intended that note taking be made from the instrument-controlling computer, however then one must continually switch between programs. This is not very usable, and support for mobile devices is being investigated. This has the advantage of readily recording and transferring observations to the ELN in many different forms, e.g., images, videos, and notes—text, hand-written, or voice.

Finally, as repurposing of hastily recorded notes in order to generate a semiformal record, albeit unintentional, was very well received, we are investigating how this could be better supported.

## ■ CONCLUSIONS

A research-led practical with true hands-on time provides an excellent, and generally more rewarding, alternative experience than being embedded in an academic research group. Students are exposed to a wider range of advanced skills and techniques and, through a research techniques-led learning approach, experience a richer and more lasting learning experience. The approach taken fosters independence and engenders more control in conducting an experiment. This is akin to being involved in a research project and develops a range of skills beyond purely laboratory practical competency. Additionally, using an ELN for providing instructions, recording observations, and assessor interaction provides alternative approaches to laboratory practice, assessment, and feedback that enrich the learning and engagement of students, while also addressing industrial standards training.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.5b00071.

Instructions, assessment questions, questionnaire, and student feedback (PDF, DOCX)
COSHH assessment form (PDF)

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### Notes

The authors declare no competing financial interest.

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