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Design and delivery of virtual inquiry-based organic chemistry experiments

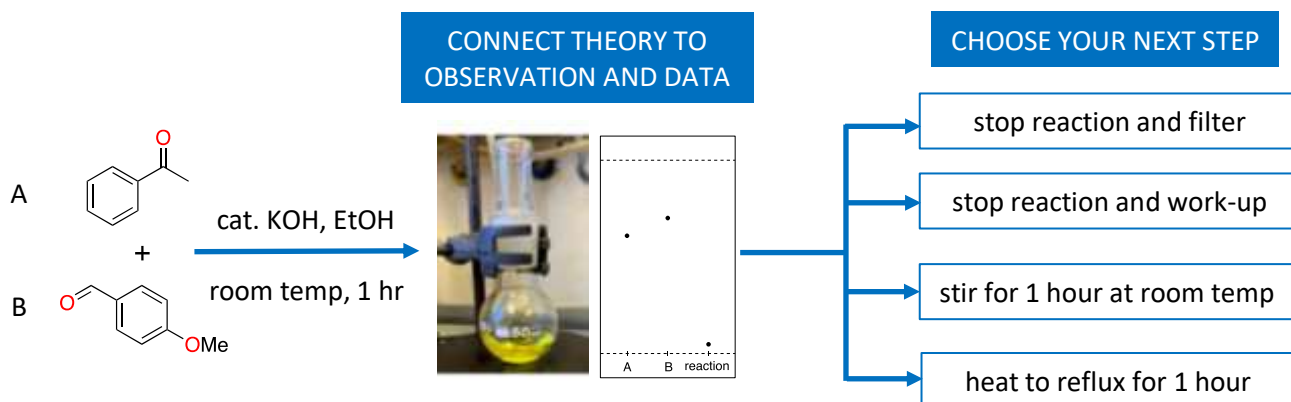
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5 ABSTRACT

Guided-inquiry experiments are an important tool for helping students develop scientific practices such as hypothesizing and problem solving. In organic chemistry, these types of experiments can help students learn how to connect the theory of the reaction to the observation and data to decide how the reaction is proceeding or if it needs adapting. Due to reduction of in person teaching during the COVID-19 pandemic, we developed a set of virtual inquiry-based organic chemistry experiments where students make the same decisions as they would do with a hands-on inquiry experiment. Thus, these simulations allow students to learn similar problem-solving skills. In this paper, we provide details of the simulations and the educational outcomes when they were used to replace hands-on inquiry experiments. We also include suggestions for its use post-pandemic.

15 GRAPHICAL ABSTRACT



KEYWORDS

Laboratory Instruction, Organic chemistry, Internet/Web-based learning, Inquiry-based/Discover Learning

INTRODUCTION

Guided-Inquiry experiments provide an important opportunity for students to develop core scientific skills. Unlike expository (also known as ‘cookbook’) experiments, guided-inquiry experiments are more open-ended and allow students to generate the procedure.¹ This means they can apply theory to hypothesize, problem solve and design experiments. In organic chemistry, guided-inquiry experiments allow students to solve ‘day-to-day’ problems that are encountered by practicing synthetic chemists.² Thus, they are an important tool in training students for graduate careers in the chemical sciences.

There are many examples reported in this *journal* of inquiry-based organic experiments where students can apply core organic theory, including the aldol reaction,^{3,4} nucleophilic addition to carbonyls,⁵ E2 elimination,⁶ electrophilic aromatic substitution,⁷ the Wittig reaction⁸ and metathesis.^{9,10} There are also examples where whole or partial organic laboratory courses have become inquiry-based with positive outcomes for the development of scientific skills.¹¹⁻¹⁴

At the University of Leeds, inquiry-based experiments form a core part of the level 2 synthetic chemistry laboratory course to help students transition from being able to perform expository experiments towards being able to perform research projects. With these experiments students must connect the theory of the experiment to what they can see in the flask (observation) and the TLC of the reaction (data) to hypothesize if the reaction is working, if it is complete and/or if by-products are forming. They also have to make these same connections between theory, observation and data to decide if they should modify the reaction conditions or stop the reaction to isolate the product. Students must additionally decide which purification method should be used.

Due to the COVID-19 pandemic, laboratory educators around the world have been prompted/forced to move courses online.¹⁵ The main challenge has been to replacement for face-to-face experiments with alternative activities. A variety of methods have been reported which include the use of videos or live online demonstrations,^{16,17} simulations of experiments,¹⁸⁻²¹ virtual reality experiments,^{22,23} chemistry kits for home use,²⁴⁻²⁶ kitchen experiments,^{27,28} shifting to literature-based assignments,²⁹⁻³² or a combination of these approaches.³³ The pivot to online teaching has presented difficulties to ensure the same learning outcomes are met at with face-to-face laboratories, however

there are also cases where online instruction provides benefits such as improving access to
50 underrepresented groups.³⁴ At the University of Leeds, we were required to reduce the amount of time
that students spent in the laboratory. This meant choosing which skills needed to be developed with
face-to-face laboratory teaching in lieu of other skills that be developed through online activities. Using
a simulation, where students could work through open-ended organic experiments, we believed we
could engage students in the same cognitive processes and develop the same skills as from our hands-
55 on inquiry experiments.

We also had a desire to use this simulation in our laboratory courses beyond the COVID-19
pandemic. Prior to the switch to online instruction, many students engaged with the guided inquiry
experiments in the way we desired, however some students found the experiments overwhelming from
the combination of having to think more deeply through their experiments while concomitantly
60 performing the laboratory manipulations within the time pressure of a laboratory class. As such, these
students often avoided connecting the theory to experiments to focus solely on getting the right
product. They put pressure on teaching assistants to analyze their reactions for them and tell them
what to do. We therefore reasoned that training students to perform open-ended experiments outside
of the laboratory would reduce their cognitive load and allow them to think more freely about the
65 theory of the experiment without the pressures of the laboratory.

In response to these perceived needs, we developed a set of virtual inquiry-based organic
experiments that we believed to simulate the way students would have to work through the equivalent
open-ended experiments in the laboratory. The experiments, delivery and outcomes of these virtual
experiments are also reported.

OVERVIEW OF THE VIRTUAL ORGANIC INVESTIGATIONS

The simulations were created as interactive webpages using Google Sites

<https://sites.google.com/view/organicinvestigation/home>.³⁵ Google sites has an easy-to-use interface
for creating and editing webpages which does not require knowledge of programming. The primary
75 reason for using Google sites was to ensure that the simulations would be easily accessible to
students, as the only requirement is a device with access to the internet, and that they would be

accessible to instructors and students from other institutions. There are recent examples of simulations being created through an institution's LMS system.¹⁹ This has the advantage of ensuring that students log into the LMS system to track their use of the simulation, however this means they are not accessible to those outside the host institution. By using an open, web-based simulation, it is not possible to track students in this manner. However, our interest was in recording students' application of theory to decide how to navigate through the online experiments. To do this, students submit Google Forms incorporated into the webpages of the simulation which are sent to the instructors. This allows the tracking of students' use of the simulations.

With each experiment, students are given a method for the start of the reaction, a picture of the flask, and TLC of the reaction. They are then presented with a series of choices. They can choose to (1) stop the reaction then isolate the components by filtration or work-up, (2) continue to stir the reaction further, or (3) heat the reaction (Figure 1). Based on the choice made, the reaction will proceed to a new webpage which reflects what would happen if students had made that same choice with the hands-on practical. For example, if students choose to filter a fully dissolved reaction mixture, then the following step will show no solid was collected and the ¹H NMR of the filtrate is mainly the reaction solvent.

These guided prompts force students to connect the mechanism of the reactions to what they observe in the reaction flask and the TLC data to determine if the desired product is forming, if the reaction is complete, if any side reactions are occurring, and if any by-products have formed. When students are choosing what steps to take next, they need to hypothesize what will happen as a result of their choice. If students are choosing to purify their reactions, they must choose the appropriate method by thinking about the structure-property relationships of the chemicals in the reaction flask.

Prior to moving onto the next step, students complete and submit a Google Form to explain what has happened in the reaction/purification step and a prediction of that will happen next. These forms prompt students to develop connections between the theory of the reaction with the observation of the reaction/sample and TLC/NMR data. Google Forms allows all the responses to be collated in a spreadsheet and sorted by students. This means the instructors can follow the student's pathway through the virtual experiment and see the explanations they have given for each stage. Students are

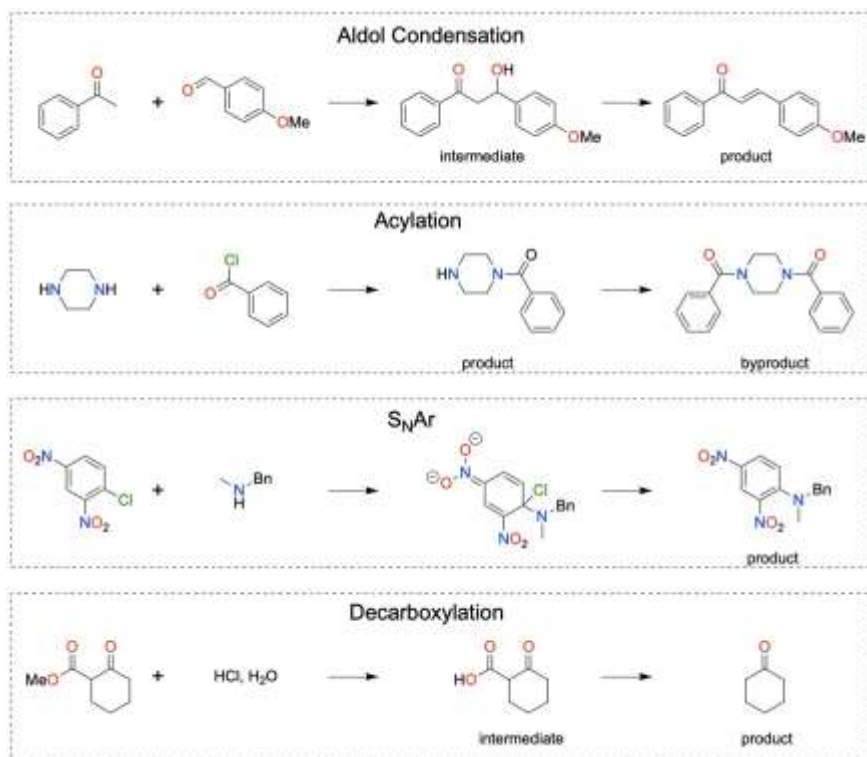
105 graded on achieving a pathway that leads to the correct product in high yield and for making the
correct connections between theory, observation, and data. The instructions on the simulations state
that they should only be completed by University of Leeds students, and that students from other
institutions should follow their instructor's guidance. For instructors from other institutions, we have
110 provided a template to create their own Google Form (supplementary information) for their own
students to use. This will ensure instructors receive their own students' submissions.

We chose four inquiry experiments that were based on core organic mechanisms which students
would have previously encountered before – the aldol condensation, acylation, S_NAr substitution and
decarboxylation (Figure 2).

The figure consists of two side-by-side screenshots from a virtual organic chemistry simulation. The left screenshot displays a chemical reaction scheme at the top, showing acetophenone reacting with anisaldehyde in the presence of catalytic potassium hydroxide (cat. KOH) in ethanol (EtOH) to form a chalcone derivative. Below the reaction is a 'Method' section with text: 'A solution of acetophenone (1 mmol) and anisaldehyde (1 mmol) in ethanol (10 mL) was treated with a catalytic amount (ca. 20 mg) of solid KOH and stirred at room temperature for 1 hour.' This is followed by an 'Observation' section stating 'The reaction forms a yellow solution.' and a photograph of a round-bottom flask containing a yellow liquid. Below that is a TLC analysis section with a 9:1 Petroleum ether:ethyl acetate solvent system. A chromatogram shows four spots labeled A, B, C, and D. A legend identifies A as acetophenone, B as anisaldehyde, C as the reaction mixture, and D as the co-spot. The right screenshot shows a digital interface titled 'Choose how the experiment should proceed'. It features a dropdown menu currently set to 'Change'. To the right are four blue buttons: 'Stop reaction and filter', 'Stop reaction and perform work up', 'Move to next step (temperature)', and 'Move to next step (time)'. Below these are three text input fields for student responses, each with a 'Your answer' label. A purple 'Submit' button is at the bottom left, and a circular icon with a pencil is at the bottom right.

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Figure 1: Screenshots of the virtual organic investigations.



120 Figure 2: The four virtual organic experiments.

With each experiment, the reaction does not proceed smoothly towards the product with the initial conditions, so some adaptation is required to improve the yield. For the aldol condensation, the aldol reaction occurs readily at room temperature, but the condensation (E1cB step) is slow. This can be determined from the observation, as the intermediate is polar and dissolves readily in ethanol, and the TLC, which shows the starting materials have formed a new polar compound. If the students choose to stop the reaction and purify the reaction, they will isolate the intermediate. If they stir the reaction, further the condensation product starts to form. This is evidenced by the non-polar product precipitating out of the reaction solvent and forming a non-polar spot on the TLC plate. Heating the reaction to reflux facilitates the E1cB reaction more quickly and so more precipitate forms. Choosing this pathway yields the highest amount of product.

The acylation reaction simulation was created to give students the practice of problem solving through reactions that yield undesired products. The desired product is the mono-acylated amide, but bis-acylation can occur as an undesired byproduct. Evidence that the acylation is working can be

135 determined through the formation of the hydrochloric salt, which precipitates out of solution. With the initial stir for 1 hour, the reaction is incomplete. If the reaction is stirred for longer at the same temperature the product is formed cleanly, however if the reaction is warmed then bis-acylation starts to occur. This can be seen in the TLC of the reaction.

For the S_NAr reaction, the reaction does not occur at room temperature and requires heating. 140 Stirring the reaction for longer is ineffective. When the reaction is heated, it forms a yellow solution. This indicates the formation of the fleeting anionic from the S_NAr intermediate and is as a sign that the reaction is proceeding.

For the decarboxylation reaction, the hydrolysis of the ester to the carboxylic acid intermediate occurs readily at room temperature but the loss of carbon dioxide does not and requires heating the 145 reaction. Understanding that the acid has formed can be determined by the polarity of the new spot in the TLC and from the lack of effervescence. When the reaction is heated the new TLC spot, indicating the final product has formed, runs much higher on the plate and effervescence can be seen in the reaction.

DELIVERY OF THE ACTIVITY

150 The virtual experiments were delivered as a two-day activity to second-year Chemistry and Medicinal Chemistry students as part of their practical module in the 2020/21 academic year. The lecture portion was delivered through Microsoft Teams due to COVID restrictions limiting face-to-face teaching.

A two-hour online workshop was held on the morning of the first day, where the learning outcomes 155 and overview of the task was delivered (see workshop resources). Students were informed of their assigned simulation and then worked in breakout rooms to deduce their reaction mechanism facilitated by the instructors. After this, they discussed further, how the products/intermediates/by-products could be visualized by TLC and through observations such as precipitation.

Following the workshop, students were able to start the virtual experiment. Two-hour online 160 support sessions were held on the afternoon of the first day, and on the morning and afternoon of the second day. If students had questions about their experiments, they could drop-in and ask the instructors. The first drop-in session was the most popular one for students attending and asking

questions. By the last session, very few students needed support from the instructors. In Microsoft Teams, channels were created for each simulation, so that students who had been working together to deduce the mechanisms could support each other. It is possible that peer-support was also occurring outside of these official channels. Submissions of the forms could be monitored over the course of the activity and it was found that all students were able to complete the task and submit their forms within the two days assigned.

EVALUATION OF STUDENT SUBMISSIONS

In total 79 students completed the activity. Table 1 breaks down the number of students who completed each simulation. Grades were based on whether students worked through a successful pathway to yield the correct product, by the quality of their argument and its support using what they predicted would happen next (see supporting information). Pleasingly, it was clear that students were making the connections between theory, data, and observation in the way we would have expected them to with a hands-on inquiry experiment. Examples of students making these connections is given in Table 1.

Most students followed a pathway that successfully yielded the desired product in good yield. The majority of students were also able to provide high quality descriptions of the reactions by connecting TLC data with the mechanisms of the reactions. For these reasons, the grades for students were relatively high. The main discrepancy between students was whether they could also connect the observations with the mechanism and intermediates of the reaction. Those that did so ended up with grades of around 80%, and those whom could not received grades of around 60% (these grades align with the 1st and 2:1 classifications used in the UK). As the average scores and standard deviation in Table 2 show, there was an approximately even split between the number of students achieving these scores. There was also very good consistency in the average grade and standard deviations between each of the simulations, showing they were equal in difficulty and no student was advantaged or disadvantaged based on which simulation they had been assigned.

Table 1: Examples of student's explanations

	Example
Connecting theory to data	The TLC plate shows that the starting materials have reacted to completion as the reaction mixture only has one visible spot, indicating a single product. Meaning there is no acetophenone or anisaldehyde left in the reaction mixture. This must be the intermediate as it has a must lower Rf value (0.05) than both the aldehyde (0.70) and the ketone (0.61), meaning it is much more polar. This increase in polarity is due to the OH group in the intermediate compound.
Connecting theory to observation	It is clear that this is the intermediate rather than the end product because it is in solution. The intermediate is more soluble in polar solvent (ethanol) than the final product as the OH present in the intermediate means it is a more polar compound, hence if this step had produced the final product then there would likely be a precipitate present rather than a solution.

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Table 2: Student grades

Simulation	% Mean	Std Dev
Aldol Condensation ^a	68	10
Acylation ^b	69	11
Decarboxylation ^c	73	12
S _N Ar ^d	70	10

^aN = 18; ^bN = 24; ^cN = 19; ^dN = 18.

STUDENT FEEDBACK

After the activity, students were asked to complete an anonymous survey to evaluate what they believed they had learnt from the experience. Twenty-nine students completed the survey, which represents a 37% response rate.

On a scale of 1-5 (1 being high, 5 being low), students were asked to rate their priorities when completing the simulation (Figure 3). Pleasingly, understanding the theory was a high priority for most respondents, as was giving the correct explanations and predictions. In contrast getting a high yield was rated as a low priority amongst students.

Students were asked to provide open-ended responses to what they felt they learnt from the activity. Almost all respondents commented that this activity helped them link theory to experiments.

"I have learned more about the planning of an open-ended experiment and what thought processes to use to enable me to adapt to the experiment if steps taken have not been successful"

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“I've learnt to think deeper about what I am actually carrying out in the practical rather than just reading from the book.”

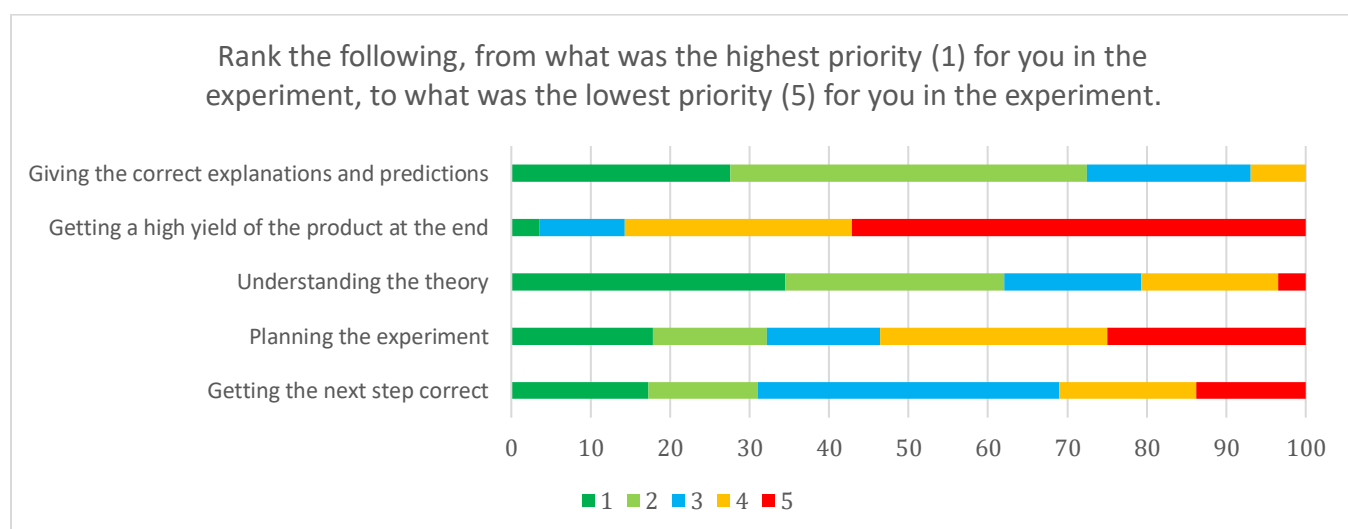
When asked if this simulation felt like performing an actual experiment in the laboratory there was an even split for “Yes” (51%) and “No” (49%) between respondents. Students could follow this up with a comment. Those who responded “Yes” indicated that it helped them understand how to perform open-ended experiments.

“Having to understand the theory behind it, whilst planning the next step really gave an insight into what it would be like to plan an experiment which works, and what results you would obtain (i.e. TLC, NMR)”

Amongst the comments from those that responded “No” were that the virtual experiments did not provided hands-on practice with laboratory equipment. Interestingly, many students also cited time pressures as a reason it did not feel like a real experiment, but that this lack of this pressure actually encouraged students to think more about theory than they would have in a hands-on experiment.

“It wasn't the same as doing it in the lab because there wasn't the same time pressure to make quick decisions...I liked that it didn't have the time pressure because it meant I actually got to think about what was happening and really understand what I was actually doing that I don't always get in labs.”

Overall, these comments supported our claim that the virtual experiments would allow students to think more about the theory of the experiment without the pressures of the hand-on aspects of the inquiry experiments.



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Figure 3: Students ranking of high and low priority factors when completing the simulation ($N = 29$).

SUGGESTIONS FOR FUTURE DELIVERY

From our analysis of student submissions and their responses to the survey, we believe this activity leads to students learning how to apply theory to perform open-ended organic experiments in the way they would do for hands-on inquiry-based experiments. As some students have suggested, they are more likely to connect theory to experiments through the simulation. In this sense, it has provided an adequate replacement for face-to-face laboratory experiments during the COVID-19 pandemic. However, as students also indicated, it does not replicate the full practical experience of a hands-on experiment. Therefore, when face-to-face laboratory classes can resume, this activity would be a useful exercise for students to complete before conducting inquiry-based experiments in the laboratory. We envision these virtual experiments working in a similar vein to simulations of lab techniques which are used prior to hand-on expository experiments.³⁶ Through these virtual inquiry experiments, students can learn how to apply theory to practice, then perform hands-on inquiry experiments to integrate this newly acquired skill with other practical skills. Interestingly, many of the students who completed the survey also made this suggestion.

"I think it can be used as a separate learning tool in order to get a deeper understanding of what is happening, without all the other stresses of being in the lab. I think it could be used as a very useful pre-lab tool for experiments."

CONCLUSION

In summary, we have developed a set of open-access, virtual inquiry-based organic experiments that help students learn how to apply theory to open-ended experiments. As such, this activity can help students develop some of the skills needed to transition from expository laboratories to research projects. These resources were used successfully with a cohort of second year Chemistry and Medicinal Chemistry students. Evaluations of student submissions demonstrated that students were making the connections between theory and the virtual experiments. Student feedback also supported the notion that they had to apply theory to work through the simulations. However, students did not feel the simulations replicated all the skills that they would gain from performing a hands-on

experiment. Therefore, these simulations could provide a platform for teaching students how to apply
255 theory to open-ended experiments before performing hands-on inquiry experiments and integrating
these skills with hands-on practical skills.

ASSOCIATED CONTENT

The Supporting Information is available at <https://pubs.acs.org>.

Workshop resources (PDF)

260 Experiment Workflows (PDF)

Supporting Information (DOCX)

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