

Synthesis and Evaluation of Ammonia Borane: A Modular, Multifaceted Approach Introducing Experimental Design through Guided Inquiry

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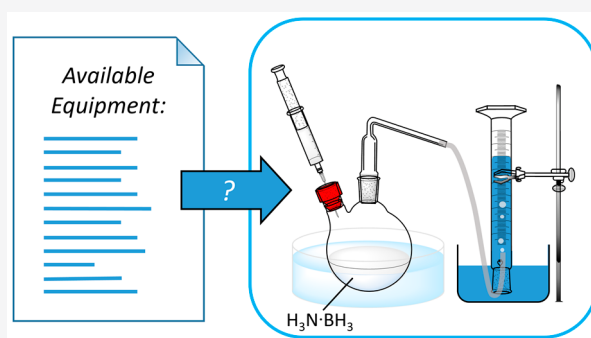
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Supporting Information

ABSTRACT: A practical focusing on the synthesis, isolation, and hydrolysis of ammonia borane (AB), $\text{H}_3\text{N}\cdot\text{BH}_3$, was developed for first-year undergraduate students. By requiring students to propose their own experimental setup to measure the amount of gas produced upon hydrolysis, experimental design skills were introduced and developed during the practical. As a result of the COVID-19 pandemic, remote and face-to-face versions of the practical were created to enable inclusivity in which experimental design skills were a key feature. Students identified and reported an appreciable increase in their experimental design skills. The multifaceted nature of the practical allows for flexibility in its implementation, dependent on students' prior knowledge, local logistical considerations, and the learning objectives of an institution.

KEYWORDS: First-Year Undergraduate/General, Inorganic Chemistry, Hands-On Learning/Manipulatives, Computer-Based Learning, Problem Solving/Decision Making, Computational Chemistry, Gases, IR Spectroscopy, Lewis Acids/Bases, Synthesis



Ammonia borane, AB ($\text{H}_3\text{N}\cdot\text{BH}_3$), first isolated in 1955,¹ is a well-known example of a Lewis acid–base adduct. Its high hydrogen density (19.5% by mass) led to interest in AB as a potential hydrogen storage material.^{2,3} The decomposition of AB to release H_2 can be promoted thermally, or catalyzed by a range of heterogeneous or homogeneous catalysts, including transition metal species (e.g., platinum,⁴ rhodium,⁴ palladium,⁵ and ruthenium⁵) and a range of Lewis and Brønsted acids.^{6–8} Selected bases have also been shown to promote H_2 release.⁹ Extensive reviews detail efforts to exploit AB as a future hydrogen source, with AB-hydrolysis systems tuned to maximize the amount of hydrogen generated and overcome practical issues such as portability.^{3,10} Aside from energy applications, AB has been used as a catalyst for the amidation of carboxylic acids,¹¹ a versatile reducing agent for numerous organic compounds,¹² a hydrogen source for the reduction of CO_2 to methanol,¹³ and a precursor to boron nitride.¹⁴ The demonstration of underlying chemical theory (Lewis acidity/basicity and the formation of adducts), as well as links to industrially and commercially relevant processes, led Arulsamy to develop a practical for upper-division chemistry students involving the efficient synthesis and hydrolysis of AB (published in this *Journal*).¹⁵

Experimental design skills are listed as a potential role of the undergraduate chemistry teaching laboratory,^{16,17} and the development of these skills benefits students' future laboratory-based study and careers.^{18,19} The incorporation of exper-

imental design into such levels of study has recently been reported in this *Journal* by Farley et al.²⁰ The use of these skills is exemplified in the integrated Masters chemistry degree programs (MChem, MSci) delivered in many higher education institutions in the United Kingdom, where a final-year research project forms a substantial part of the degree. Masters degree programs accredited by the Royal Society of Chemistry (RSC) are expected to provide students with, among other outcomes, “the ability to adapt and apply methodology to the solution of unfamiliar types of problems” and “the ability to plan and carry out experiments independently and assess the significance of outcomes”.²¹ With almost 50% of U.K. chemistry students graduating with an MChem degree in 2017, and over 50% of RSC-accredited courses at an integrated Masters level (MChem or MSci qualification types) in the U.K., the development of experimental design skills during the degree program is vital.^{22,23} However, many laboratory classes (especially those in the initial years of study where foundational principles are being taught) are *expository* or “cookbook”

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style, where students follow a procedure to arrive at a result.¹⁸ An alternative concept is that of level of inquiry, which is based on the levels of provision of concepts, solutions, or methods in a laboratory investigation.^{24,25} Experiments with low levels of enquiry provide all information and methods needed, while those with some attributes missing (thus requiring students to identify, recall, or construct concepts or answers, such as deciding on a particular data analysis method) comprise higher levels. Experimental design, requiring a student to create their own experiment (or aspects therein), is therefore a higher level of inquiry than those where a procedure is defined and provided. However, studies have shown that the majority of the laboratory experiments analyzed contained the lowest levels of inquiry. Those at higher levels of inquiry occurred more infrequently and, in some cases, were completely absent.^{24,26} This is consistent with Farley et al. (and references within),²⁰ who identified nonexpository practicals (i.e., with high levels of inquiry) as rarer, despite their recognized value in preparing students for more independent laboratory activities.

Although often simpler to implement due to a defined path through the practical content, practicals with low levels of inquiry have little or no scope for students to design or apply different approaches. This is also noted by Roberts, who states that "...most laboratory courses, in spite of their "hands-on" nature, are designed (consciously or unconsciously) to teach students how to follow directions. Rarely do students design or evaluate the experiment themselves".²⁷ Issues such as equipment availability, concurrent supervision of multiple approaches, and requirements to complement students' existing skills/knowledge may all act as deterrents to the wider implementation of nonexpository practicals/practicals with high levels of inquiry. These challenges may be more prevalent in the earlier years of students' university education, where practical and experimental design skills are likely to be less developed. The identification of the importance of experimental design skills is not restricted to staff; a survey of graduating students from UK chemistry degree programs identified experimental design skills as belonging to a "development deficit", whereby the skill is developed less than the relative usage of the skill postgraduation.²⁸ Therefore, the inclusion and development of experimental skills is a key topic for the investigation for future experimental chemistry courses.

While practicals with higher levels of inquiry are traditionally suggested for higher years of study, the inclusion of experimental design in introductory (first-year) undergraduate practicals has been reported previously.^{29,30}

Coincident with Arulsamy's report, we had also developed a practical involving the synthesis and hydrolysis of AB. This practical has been delivered to our first-year cohorts (ca. 180–220 each year) since 2018. Although mostly similar, there are some differences in our synthetic approach compared to that of Arulsamy (Table 1). In our work, oven-dried glassware and the use of a drying tube are sufficient, and the use of dried THF negated the need for filtration through Celite at the isolation stage.¹⁵ Additionally, students are instructed to devise an apparatus to measure the volume of hydrogen that evolved from samples of their prepared AB on hydrolysis and, hence, calculate the apparent purity of their product. This part of the practical offers a compromise between the introduction of a completely unscaffolded, nonexpository approach and one which is completely prescribed. Experiments involving AB have previously been explored at a high level of inquiry by Pagano et

Table 1. Comparison between This Practical and Previously Reported Work by Arulsamy¹⁵

Description	Arulsamy, 2018	This Work
Target level	Upper-division undergraduate	First-year undergraduate
Synthetic conditions	40 °C, 1 h THF Reagents ground before use	40 °C, 2 h Anhydrous THF Reagents used as supplied
Equipment used	Mineral oil bubbler Water bath	Drying tube Temperature-controlled heating block
Isolation	Filtration through Celite pad Rotary evaporation	Conventional filtration Rotary evaporation
AB-hydrolysis method	Addition of dilute H ₂ SO ₄ (aq)	Addition of RuCl ₃ ·xH ₂ O(aq)
Spectroscopic method/s	NMR (¹ H, ¹¹ B) IR	IR Prediction of IR spectra (DFT) Examination of crystal structure
Computational aspects		Synthetic method provided Hydrolysis method proposed by students
Experimental design aspects		Discussion of proposed hydrolysis method Lab report
Communication of results	Lab report	Mixture of nonexpository and expository
Practical style	Expository	

al., who developed an advanced course-based undergraduate research experience (CURE) project involving the optimization of catalytic hydrolysis of AB through ligand design.³¹ This work offers an alternative to such a high theoretical and inquiry level experiment, by introducing the concept of experimental design to inexperienced students. As part of our ethos of developing practicals that cross traditional subdisciplines in chemistry, for example, various activities have been incorporated. These include the interpretation of an IR spectrum assisted by computational DFT-modeling, the visualization of crystal structures using commercial software, and the inclusion of a calibration exercise. These introduce aspects of physical, computational, and analytical chemistry to what might be regarded as a largely inorganic exercise.

In response to the COVID-19 pandemic, we have also developed an alternative version of the practical which is completed entirely online, while still preserving the primary design objectives referred to above. We have previously investigated the role of remote teaching exercises as a vehicle for teaching experimental design during periods when laboratories are unavailable.³²

This report therefore complements the previously published work and presents a matrix of face-to-face and remote activities that can be tailored to the requirements of different institutions and their students. Our results and experiences from the past two years (prepandemic and in-pandemic, 2019/2020 and 2020/2021, respectively) are presented.

EXPERIMENT

Outline

All versions of the practical comprise three parts, completed in a single 6 h session which is delivered to first-year undergraduates. The components for each version of the practical are summarized in Table 2, and the relevant

Table 2. Summary of Components for 2018–2020 and 2020/2021 Practical Classes

Component	Part Name	2018–2020 Face-to-Face Version	2020/2021	
			Face-to-Face Version	Remote Version
Synthesis of AB	A	● ^a	●	○ ^b
Designing experimental setup for hydrolysis measurements	B	●	●	●
Computational analysis of AB and H ₃ P·BH ₃	B	●		
Analysis of crystal structure of AB	B	○	○	○
Hydrolysis of NaBH ₄ for calibration of experimental setup	B		●	○
Presentation of experimental setup	B			●
IR spectroscopic analysis of AB	C	●	●	○
Hydrolysis of AB	C	●	●	○

^a○ = component undertaken by student. ^b● = component present in provided form for students to examine and analyze (video, data, or raw measurement values).

adaptations discussed below. Detailed instructions for each component are provided in the Experimental Supporting Information. The skills developed in each component are presented in Table 3.

Part A: Synthesis and Characterization of AB

The reaction between NaBH₄ and (NH₄)₂SO₄ is conducted according to the method reported by Ramachandran and

Gagare.^{5,15} The importance of anhydrous synthetic conditions was emphasized to students, who were instructed to dry their flask thoroughly before use, and to use a CaCl₂ drying tube during the initial reflux period. Anhydrous THF can either be dispensed from a solvent purification system (SPS) or used as received (anhydrous grade) from commercial sources. In both cases, solvent was stored over molecular sieves and sealed to prevent the ingress of moisture. Brief exposure to air, in order to measure and add solvent, was acceptable and did not result in a noticeable loss of yield. However, exposure to water (through incomplete drying of flasks), or a reaction temperature significantly higher than stated (e.g., 60 °C or higher), resulted in decreased yields of isolable product and/or purity.^{5,10,33} In these cases, previously prepared samples were provided for analyses in part C. Yields typically ranged from 0.1 to 0.7 g, although greater masses (and, therefore, yields exceeding 100%) were occasionally obtained as a result of incomplete removal of THF.

Students recorded an IR spectrum of their isolated AB, which was discussed in their postlaboratory report. The IR spectrum of AB offers numerous bands of interest for students to examine, although at the most basic level, students can be simply directed to the region of interest for the B–N stretching vibrations (780–800 cm^{−1}).³⁴ The impact of isotopic substitution on the reduced mass, and the corresponding effect on vibrational frequency, serves as a good discussion point for students who are familiar with the theory behind IR spectroscopy.

Part B: Computational Analysis (2018–2020)

Part B is completed during the 2 h reflux period in part A. Originally, this part comprised a computational study, where students calculated the structures of AB and H₃P·BH₃ using Gaussian09 software.^{35,36} This built upon students' prior skills in performing calculations and demonstrated the application of theoretical analysis to support experimental findings. Geometry optimization and determination of bond vibration frequencies for AB and H₃P·BH₃ were calculated using DFT at the B3LYP level of theory using the 6-311G (+,2d,p) basis set, including prediction of the IR spectra (Figure 1). Students examined and compared the fundamental stretching vibrations of the B–P/N and B/N/P–H bonds, reconciling their relative frequencies

Table 3. Skills Developed for Each Component, with Prerequisite Skills Introduced through Previous Practicals^a

Component	Part Name	Skills Developed	Prerequisite Skills
Synthesis of AB	A	<i>Handling glassware/reagents</i> , use of anhydrous conditions, safe working practices	Handling glassware/reagents
Designing experimental setup for hydrolysis measurements	B	Experimental design	Awareness of accurate measurements
Computational analysis of AB and H ₃ P·BH ₃	B	Computational analysis, interpretation of results	Computational analysis
Analysis of crystal structure of AB	B	Computational analysis, interpretation of results	Computational analysis
Hydrolysis of NaBH ₄ for calibration of experimental setup	B	<i>Measurement</i> and observation, assessment of results and their reliability/reproducibility, <i>weighing small amounts</i> , <i>use of sharps</i>	Measurement and observation, handling of reagents, assessment of results and their reliability/reproducibility
Presentation of experimental setup	B	Presentation and communication skills	
IR spectroscopic analysis of AB	C	Interpretation of spectra	Recording and analysis of infrared spectra
Hydrolysis of AB	C	<i>Measurement</i> and observation, assessment of results and their reliability/reproducibility, <i>weighing small amounts</i> , <i>use of sharps</i>	Measurement and observation, handling of reagents, assessment of results and their reliability/reproducibility

^aSkills listed in *italics* denote skills developed in face-to-face versions of the practical.

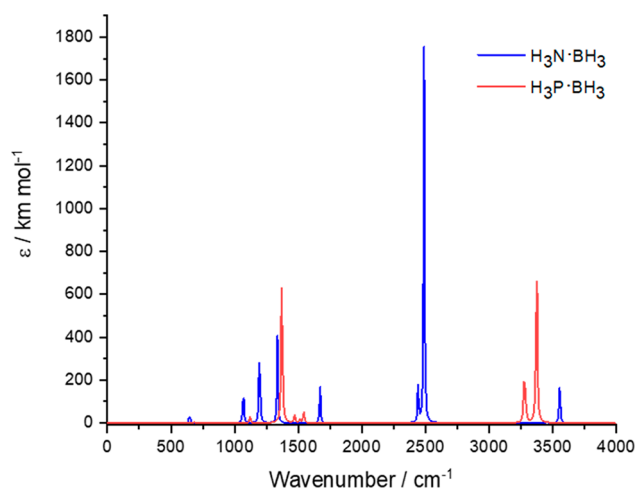


Figure 1. Overlay of predicted spectra of AB ($\text{H}_3\text{N}\cdot\text{BH}_3$, blue line) and $\text{H}_3\text{P}\cdot\text{BH}_3$ (red line).

with factors such as reduced mass and bond strength (related to force constant). These were then used to aid the assignment of their experimentally recorded AB spectrum. The differences between the predicted and experimental results were then used to encourage students to consider the limitations of such computational analyses (calculations are performed on molecules in the gas phase, and thus, significant intermolecular interactions are not considered). Additionally, students visualized the crystal structure of AB using software (Mercury, VESTA, and/or CrystalMaker),^{37–39} to identify and rationalize intermolecular attractions, namely, favorable $\text{B}-\text{H}\cdots\text{H}-\text{N}$ electrostatic interactions.

Part C: Hydrolysis of AB (2018–2021)

Once isolated, students investigated the purity of their AB product through hydrolysis or methanolysis, catalyzed by ruthenium(III) trichloride hydrate.⁵ In contrast to the expository nature of part A, this part of the practical is deductive, and the results are partially known (i.e., students calculate in advance the amount of gas that *should* be evolved, but the actual amount that will be released is unknown). This lies between *inquiry-based* (inductive approach and undetermined outcomes) and *problem-based* (deductive approach and determined outcomes) styles of laboratory instruction, as reviewed by Domin.⁴⁰ Students were provided with a list of available equipment and glassware, and some prompts to

consider in their proposal. Once a design had been proposed, students discussed this with staff, to refine and amend as appropriate, before calculating the masses of samples needed, volume of catalyst solution, and the volume of gas to be evolved. A maximum volume of 250 mL of H_2 could be evolved owing to the size of the measuring cylinder. This limited the scale of the hydrolysis reaction to ca. 100 mg of AB per measurement, although students often chose smaller scales (ca. 50 mg) to ensure the maximum capacity of the measuring cylinder was not exceeded, or the expected volume produced was easy to measure (100 mL rather than 121 mL, for example). Students were required to have their proposals and calculations checked by staff before commencing any investigations; this is especially critical owing to the potentially rapid evolution of extremely flammable H_2 in a highly exothermic reaction. This approval process also acted as a natural point to discuss experimental setups with students, and how their measurements could be used to determine the apparent purity. Common discussions related to a consideration of the ideality of the evolved H_2 gas, partial pressures (and how they can be accounted for), safe working practices, and the minimization of errors in experimental setups (for example, through leak testing, in addition to the consideration of errors associated with the hydrolysis reaction and measurements themselves). Some of these points tied in with lecture content, providing opportunities for students to recall, connect, and apply material in a different setting.

The experimental setups proposed by students varied slightly but often revolved around a central theme of water displacement from an inverted measuring cylinder. This method features in some secondary school practical curricula⁴¹ and was undoubtedly familiar to some students as a result. Other suggestions involved the use of a gas syringe, or the measurement of water displacement by the mass of water displaced into a secondary vessel. The majority of students devised their experiments individually, but those that required more support were guided toward an idea by a series of prompts from demonstrators or teaching staff. Students were neither penalized nor rewarded for proposing particular ideas, and discussions did not influence any marks. Students were ultimately guided toward the setup shown in Figure 2. This allowed for valid comparisons across the whole cohort and minimized logistical issues from managing numerous different setups during the session.

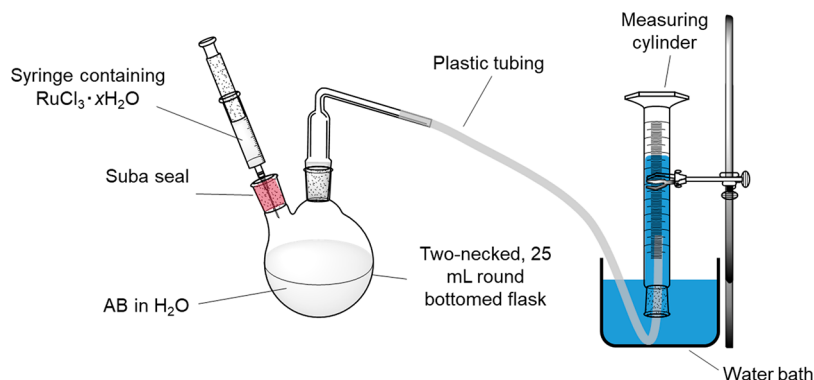


Figure 2. Typical setup for hydrolysis measurements to evolve H_2 from decomposition of AB. Water bath surrounding reaction vessel omitted from the diagram.

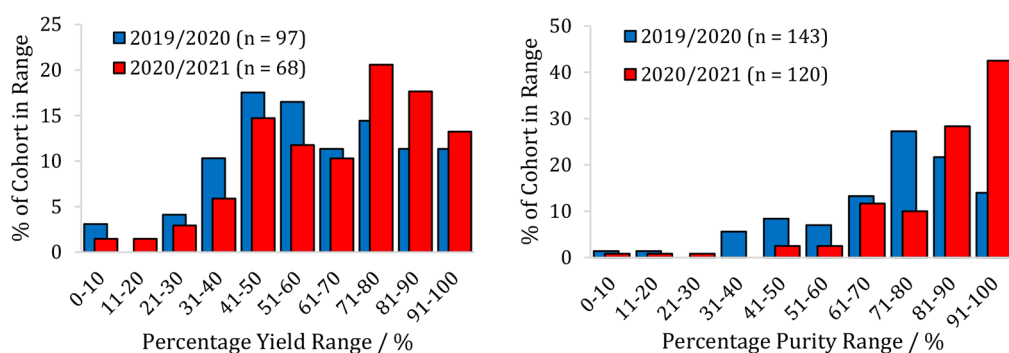


Figure 3. Percentage yields of AB obtained by students in the 2019/2020 (blue, $n = 97$) and 2020/2021 (red, $n = 68$) academic year (left). Calculated percentage purities of AB obtained by students in the 2019/2020 (blue, $n = 143$) and 2020/2021 (red, $n = 120$) academic year (right).

279 Postlaboratory Report

280 Following completion of the practical, students submitted a
281 report. In our case, students were asked to describe their
282 experimental procedures, analyze their IR spectrum, calculate
283 the yield, and determine the percentage purity of their AB. By
284 considering the crystal structure provided and analyzed in part
285 B, students measured bond lengths, identified the B–H...H–N
286 electrostatic interactions, and compared and contrasted the
287 results to a provided crystal structure of ethane.

288 ■ CHANGES AS A RESULT OF COVID-19

289 Part B: Trialing Devised Gas Collection Apparatus 290 (2020/2021)

291 Given that a majority of students' learning had migrated
292 online, the limited laboratory time students had was directed
293 toward the development of students' practical skills. Con-
294 sequently, the computational task in part B was substituted for
295 a lab-focused activity.⁴² In this new part B, students designed a
296 "control" experiment involving the ruthenium-catalyzed
297 hydrolysis of NaBH_4 of known purity,⁴³ in analogy to the
298 hydrolysis of AB catalyzed by ruthenium(III) chloride hydrate
299 described by Ramachandran and Gagare.⁵ The same setup was
300 then used for the hydrolysis of AB in part C, as described
301 above, and was the same as that devised in previous years
302 (Figure 2). The scale (no more than 250 mL of H_2 to be
303 evolved; ca. 75 mg) was also similar to that used in part C.
304 This inclusion had various benefits. Students could gain
305 familiarity with using the apparatus (particularly as it involved
306 the use of unfamiliar equipment such as rubber septa, flexible
307 sealing film, syringes, and needles) prior to evaluating the
308 purity of their AB samples. Students sometimes encountered
309 anomalous results, mainly arising from an improperly sealed
310 apparatus, and would need to develop problem-solving skills to
311 resolve these issues. Repeat measurements enabled students to
312 estimate experimental error. The use of a reagent of known
313 purity allowed the introduction of the concept of a "correction
314 factor" for their experimental setup, which could then be
315 calculated and applied to their measurements of AB performed
316 later. As many of these skills and themes had been introduced
317 previously, this task provided opportunities for contextual
318 reinforcement.

319 Remote Version (2020/2021)

320 As a result of the COVID-19 pandemic, significant adaptations
321 were made to enable delivery of this practical in the 2020/2021
322 academic year. For the ca. 10% of students unable to attend
323 practical classes in person, an online alternative was developed.

This lab involved a mixture of tasks and media types, to
facilitate active learning and prevent the exercise from
becoming entirely focused around one task type. The 6 h
synchronous, remote practical involved a live introductory talk,
a prefilmed video of the synthesis of AB, and provided
experimental data for AB. Staff assistance was provided
throughout the session, both on video and via the chat
function in Microsoft Teams. Students were required to engage
with the video and answer various questions related to
practices involved. They also had to devise and present their
experimental setup for determination of AB purity (see below).
The delivery of an online lab alongside the face-to-face version
meant that all students could complete the practical, regardless
of location or due to illness/self-isolation.

The video component was filmed in-house and provided an
audio–visual demonstration of the lab procedures, with
embedded captions. Importantly, the video was filmed in the
same laboratory setting and using the same apparatus as those
completing the practical face-to-face, so those students unable
to attend the lab could gain familiarity with the equipment and
environment that they would ultimately encounter in-person.
The impact of instructional laboratory videos on students'
familiarity, confidence, and understanding in the subsequent
laboratory classes has been reported previously.^{44–46} The
video was also made available to students who completed the
practical face-to-face and complemented a series of short
"techniques" videos also prepared in-house. In-lab questions,
indicated throughout, were included to stimulate discussion
between students, and between students and staff. These
covered both theoretical (e.g., mole calculations and balancing
equations) and practical topics (e.g., drying glassware and
repeating measurements).

Alongside the students in the face-to-face practical, those
completing the remote practical were tasked with designing an
experimental setup for the hydrolysis experiments. Both sets of
students were given the same equipment list. As students were
required to devise their own experimental procedures and
apparatus, there were no videos for these sections of the
practical. Remote students could discuss their suggestions with
staff via Microsoft Teams; as with the face-to-face lab, prompts
were given, with no negative consequences if students deviated
from them. Students were then asked to create and present a
short (5 min) summary of the practical using Microsoft
PowerPoint, including a discussion of their chosen analysis
method. Following the completion of the presentations, the
group and staff reconvened to discuss the students' devised
experimental setups and any assumptions, benefits, or
limitations of the proposals. Once the presentation was

complete, students analyzed a set of sample data as part of their postlab report.

RESULTS

Student results from the face-to-face practical in the 2019/2020 ($n = 143$) and 2020/2021 ($n = 120$) academic years were examined (Figure 3, Table 4). A small number of students (ca.

Table 4. Mean (\bar{x}) and Median (\tilde{x}) Yields and Calculated Purities of AB, with the Percentage of Students Reporting a Correction Factor for Their Purity Calculations^a

Academic Year	Yield / %			Purity / %			Correction Factor Reported	
	\bar{x}	\tilde{x}	n	\bar{x}	\tilde{x}	n	%	n
2019/2020	61	60	97	71	75	143	^b	^b
2020/2021	66	72	68	82	87	120	73	120

^aNB. The number of results (n) for each data set is reported adjacent to each value. ^bNo data obtained, as the calibration step (part B, 2021) was introduced after students conducted the earlier iteration of the practical.

10%) obtained product yields or purities of AB that apparently exceeded 100%. These values have been excluded from the numerical analyses presented. Median (\tilde{x}) values are also quoted, to account for the possibility of extremely high or low values skewing any averages presented from mean (\bar{x}) data.

For the 2020/2021 academic year, the average yield (66%, $n = 68$) is marginally higher than that of the previous year ($\bar{x} = 61\%$, $n = 97$) (Table 4). This may be attributed to the change to SPS-dispensed solvent, from commercially available solvent, for this academic year. A large increase in the median yield is observed between 2019/20 and 2020/21 ($\tilde{x} = 60\%$ and 72% , respectively). A large increase in the calculated purity of the product was also observed between the two years (Figure 3, right, $\bar{x} = 82\%$ vs 71% previously). Owing to the COVID-19 pandemic, students in the 2020/2021 academic year had less “hands-on” practical experience than their peers the previous year. The introduction of the hydrolysis of NaBH_4 prior to

measuring the purity of AB allowed students several “trial runs” with a control, which anecdotally appeared to increase the students’ familiarity and confidence when setting up and handling the glassware in part C (e.g., knowing where leaks commonly occur and how to fix them). This additional measurement part is likely to improve students’ skill levels in these techniques and may partially account for the observed increase in reported purities of AB (owing to more accurate measurements, rather than an increase in purity itself). However, the use of different solvent sources between the two years means that this cannot be stated for certain. Trial syntheses of AB showed that use of commercially available anhydrous THF ($<0.05\%$ H_2O at time of packing⁴⁷) resulted in a reduction in yield versus use of THF freshly dispensed from a SPS, but the isolated reaction products were of comparable purities. The implementation of a correction factor could also result in a pseudoincrease in purity, by consideration of systematic errors in measurement.

Students were not required to apply a correction factor to their results (although such measures were mentioned in the introductory talks and lab manual). However, pleasingly, over two-thirds (71%) of the 2020/2021 cohort implemented a form of correction from their hydrolysis setup trialed in part B, allowing for a more accurate determination of the percentage purity of their prepared AB. Students described their corrections by calculation of a deviation from the expected purity or volume of H_2 expected to be captured. This deviation was quoted either as a percentage or a single value. The deviation was then applied to the measured volume of H_2 evolved from the sample of AB. In some cases, students calculated the difference between the purity of the NaBH_4 sample and that which they had calculated from their hydrolysis measurements and added this deviation to their calculated AB purity. Some students also considered the stated purity of the NaBH_4 provided and adjusted their correction factors accordingly.

Logistical and Safety Considerations

Students typically experienced no major difficulties completing either the face-to-face or remote delivery form of the practical.

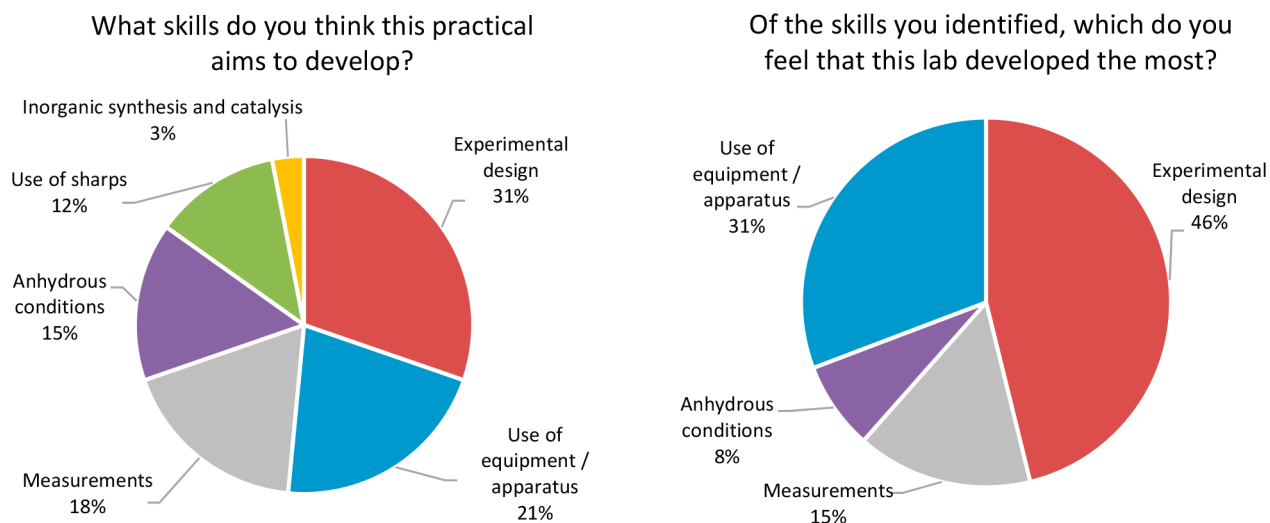


Figure 4. Skills that students identified the lab as aiming to develop (left), and those that students felt the lab developed the most (right). As students could identify any number of skills, the 100% value for each chart refers to the number of responses, rather than students. Skills not identified as being developed initially (Figure 4, left) were omitted from the data shown in Figure 4 (right).

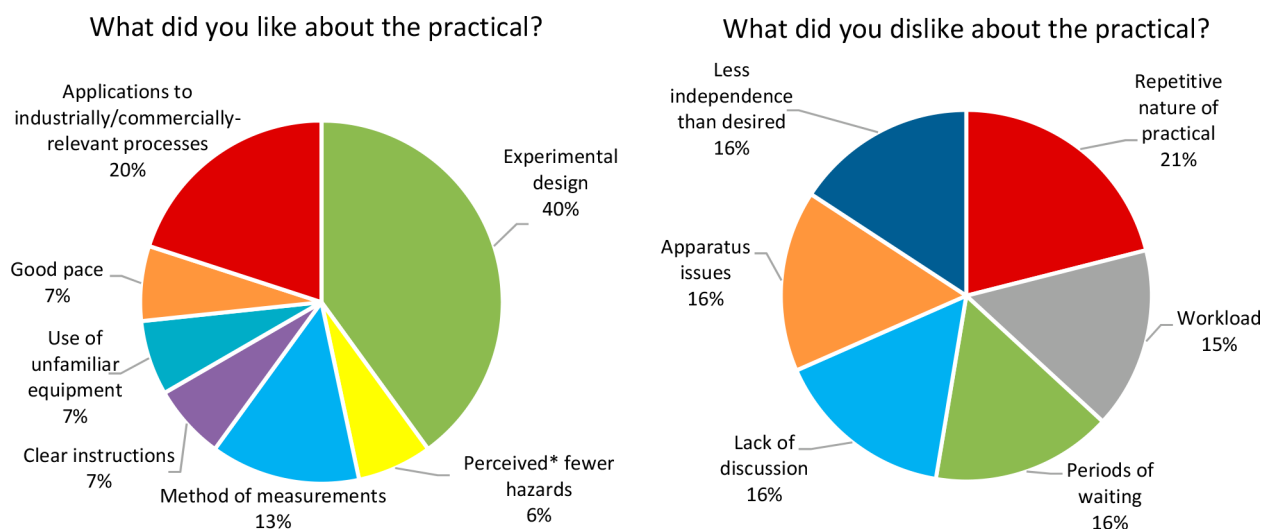


Figure 5. Aspects of the practical that students identified as liking (left) or disliking (right). Asterisk indicates that the perception of fewer hazards was implied by those that responded with this answer and was not stated in the practical or any documentation. As students could identify any number of aspects, the 100% value for each chart refers to the number of responses, rather than students.

Common mistakes included not using an adapter when attaching the condenser, forgetting to insert the temperature control probe from the hot plate (which can lead to significant decomposition of the product, even at 50 °C), and leaks occurring in the hydrolysis setup. The first two of these could be addressed by encouraging students to recall earlier practicals involving the glassware setup. Leaks in the hydrolysis setup can be reduced by using sealing film, and/or clips to secure the tubing adapter in place. Additionally, ensuring students added the catalyst solution in a controlled manner prevented a rapid internal pressure increase (which can result in lifting the adapter out of the vessel), reducing the potential for gas loss. From a safety perspective, the most hazardous aspects of the practical are the use of sharps and the generation of significant volumes of flammable hydrogen gas. NaBH_4 is flammable and contact of large quantities with water can result in the ignition of any flammable materials nearby owing to the exothermicity of the reaction. H_2 is flammable, forms explosive mixtures with oxygen, and can be generated rapidly in large quantities depending on the scale of the reaction. For these reasons, staff approval of masses and volumes to be used is an *essential* step in this practical. Samples of AB can undergo decomposition over time, resulting in pressure increases, and should be destroyed at the end of the practical by careful addition of small amounts at a time to a dilute aqueous acid solution (HCl or H_2SO_4) in a beaker.¹⁵ Further discussion of safety considerations is provided in the [Experimental Supporting Information](#).

OUTCOMES

Students were surveyed to examine their experiences of the practical. Although the survey was open to those that had completed either version of the practical this academic year, responses were only received from those who completed the face-to-face version. Of those who responded ($n = 17$), nearly two-thirds (65%) said that they enjoyed the practical. Students identified a range of skills that they felt the lab aimed to develop,⁴⁸ which aligned with those intended for the practical. Pleasingly, experimental design (31%) was the skill most identified, with almost half of these respondents saying that this was the skill that they developed the most from the

practical (Figure 4). All students stated that the lab had developed their identified skills, with 81% of students reporting a slight or significant improvement in their experimental design skills.

The skills identified by students (Figure 4, left) broadly align with those identified by staff, in particular, the use of experimental design to propose a hydrolysis setup for parts B and C. The use of anhydrous conditions, and sharps, also match the skills intended by staff to be developed. Staff identified reaction setup as a key skill, which appears to have been also identified by students more generally (“use of equipment/apparatus” in Figure 4, left). The identification of measurement skills as the previously identified skill developed the most was surprising, as this was previously considered by staff as a secondary (but useful, nonetheless) outcome of the practical.

The aspects of the practical which were liked had a wider range of responses (Figure 5, left). Of the responses, 40% mentioned experimental design as an aspect of the practical that was liked. Students also liked the applications of the practical to industrial or commercially relevant processes, presumably from the links to hydrogen storage and energy materials covered in the introductory text. This was a pleasing finding and provides further support for the inclusion of links to real-life applications.^{49–51} Several students expressed satisfaction with the opportunity to undertake experimental design, rather than being given prescribed instructions to follow; some even expressed a wish for even greater independence, to allow them to try their designed setup even if it would ultimately fail. Owing to time and pandemic-induced restrictions, this was not possible to implement this academic year but will be considered for future development. Students identified IR spectroscopy and calculations as the easiest parts of the practical. Given that our students had previously conducted a practical dedicated to an introduction to IR spectroscopy, and that the acquisition and analysis of IR spectra were features in several of the practical sessions in the same term, this demonstrated an effectiveness of our pedagogic strategy for developing skills.

Students identified recording accurate measurements and the underlying theory as the most challenging aspects of the

practical. The aspects of the practical that were disliked were more evenly distributed than those which were liked (Figure 5, right). Dislike of the repetitive nature of the practical (21%), waiting for processes to occur (16%), and issues with apparatus (16%) were also noted anecdotally in the sessions themselves. Pleasingly, none of the students identified the experimental design aspect of the lab as being something they disliked. A few students identified experimental design as being *challenging*, but some also pointed out that they did not regard this as a negative point and expected a challenge, owing to this being the first time they had encountered such a task in the lab. Of the students that stated they needed support (69%), an almost equal percentage (64%) sought help from staff, while approximately a third (36%) consulted colleagues who had completed the practical before. This reinforces the role of the laboratory as a site for peer-based learning,^{52–54} in addition to the support mechanisms formally provided by staff.

OUTLOOK

Going forward, it is intended that the practical will be delivered primarily in-person at this institution, following the significant, pandemic-induced drop in hands-on practical experience. The online components are to be preserved and are provided as a backup for those who are unable to attend the lab session. However, the flexibility afforded by this modular approach to the practical means that a mixture of both delivery methods is viable for teaching this practical postpandemic and will be adapted as required.

CONCLUSIONS

The synthesis and hydrolysis of ammonia borane has been utilized in a laboratory session which introduces students to the concept of experimental design. This experiment expands on that reported previously and consists of a set of components which can be added, removed, or adapted in a modular approach.¹⁵ The flexibility of pathways through the skills matrix associated with this practical allows other institutions to choose a set of learning objectives appropriate to their own needs, creating a multifaceted practical which can include a fully remote version that preserves the experimental design aspect. The face-to-face laboratory practical, which has run successfully for the past three years at our institution, can easily be adapted to varying levels of experience, theoretical understanding, and laboratory situations. The balance between fully expository and nonexpository styles allows inexperienced students the chance to improve experimental design skills in a guided environment, without requiring numerous apparatuses or supervision of multiple approaches at once. Furthermore, the location of the experimental design parts between inquiry and problem-based laboratory instruction styles enables tailoring of the instructions to either of these classes to be achieved readily, as desired by the instructor. The practical has also been adapted for both fully remote and face-to-face teaching in the time of COVID-19. An apparent increase in both yield and purity of product was observed alongside adaptations which emphasized gaining familiarization with the measurement apparatus. The development of experimental design skills has been recognized by students, with a corresponding improvement also identified.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.1c00849>.

Experimental details with safety and synthetic considerations (PDF)

Lab manuals for all experimental components, for both remote and face-to-face versions (PDF)

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Notes

The authors declare no competing financial interest.
Notes for instructors are available from the authors upon request.

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