Using worksheets and the Internet to improve student learning outcomes

Madeleine Bussemaker, Shannan Maisey and Duncan Wild
The University of Western Australia
bussem01@student.uwa.edu.au, maises03@student.uwa.edu.au, duncan.wild@uwa.edu.au

Undergraduate laboratory classes are being reviewed and in some cases scaled back because they are expensive to run compared to the learning outcomes for students. We believe that practical experience is essential and should remain an important part of undergraduate chemistry courses. However significant adaptations should be made to broaden the skills that students can take from these classes. We proposed to make changes which increased the value of laboratories as a tool for educating new scientists without increasing the workload for staff or students. The assessment tasks were altered to focus on learning outcomes and resources were improved and made available online and in hardcopy for students and teachers. Worksheets were introduced for each laboratory experiment and the number of full laboratory report assessments halved. The provision of worksheets placed an emphasis on the key chemical concepts and aided students in their understanding of scientific writing conventions. Students were provided with an explicit guide to writing laboratory reports and given feedback on their writing technique. The submission system was moved to the online student platform WebCT to increase flexibility and improve the quality and speed of feedback. These changes were met favorably by students, and the subsequent improvement in quality of student work was noted by the assessors. The study highlighted the importance of demonstrators as teaching staff and the need to provide them with adequate training and resources. While we acknowledge that further development is required we believe that by broadening the focus of assessment beyond chemical theory the needs of cross discipline students were met while still providing chemistry majors with a solid laboratory background.

Introduction

Laboratory classes are synonymous with undergraduate chemistry, however the intensity of resources required to run these classes compared to learning outcomes achieved by students has been bought into question (Reid and Shah, 2007). The inclusion of chemistry units in an increasing number of interdisciplinary degrees demands the integration of skills across a wider range of learning areas, at higher levels of undergraduate study. Nevertheless, the professional practice of chemistry requires laboratory skills which must be introduced to students as a fundamental aspect of the field, thus laboratory classes must remain an integral aspect of the chemical education experience. It is our opinion that the learning outcomes and efficacy of assessment of laboratory classes must be addressed in order to meet these new demands and justify their cost. Conventionally, laboratory courses have been developed to focus on the specifics of the experiment, rather than on the broader learning outcomes and value to the student (Meester and Maskill, 1995). This study highlights the value that a review of conventional laboratory courses can have on enriching these classes for a more diverse student base. In order to broaden the learning outcomes of the given laboratory course, current practices were revised in such a way that four core skill sets were addressed in greater detail (Reid and Shah, 2007). These core skills are universal for most sciences, and provide valuable foundations for the future professional development of students:

- **Practical skills**: Universal laboratory practices and techniques which are assessed formatively and developed throughout the laboratory course.
• *Chemical theory:* Laboratories need clear guidelines as to what key chemical theory is being evaluated. This needs to be an extension of theory covered in lectures, and student understanding must be challenged and assessed.

• *Scientific skills:* Students need to be encouraged to interpret and critically assess their results, and encouraged to draw meaningful conclusions in the context of their experiment.

• *Scientific writing skills:* Presentation and reporting of data and using scientific writing conventions.

Laboratory classes generally present a number of experiments deemed to be of theoretical relevance to the course material. Relevant practical skills are intrinsically included, as are some aspects of chemical theory. In order to meet the specified learning outcomes, the assessment tasks for laboratories must challenge students to develop all of these four skill areas.

Beyond first year, laboratory assessments are traditionally in a report format of 3-4 pages. Tuition in correct scientific writing before assessment is often overlooked because there is an in-class emphasis on the practical experiment, and as a result reports are often of low quality (Walczak and Jackson, 2007). Feedback from students at our institution has highlighted that the time taken to write laboratory reports is of high concern to students and should be taken into consideration when examining the efficacy of assessment of these courses. We propose that for the above criteria to be met, course assessment must be reviewed to more clearly meet student needs by:

• providing guidance before assessment
• having an effective feedback loop
• being relevant and interesting
• be engaging for students and thus more time efficient.

This study was performed within the context of a second year analytical and physical chemistry unit (Chem2220) in which students have been required to perform eleven laboratory experiments in six weeks (2 x 3 hr sessions per week). The total time spent on practical experiments represents more than half of the total contact hours of the unit. It was determined that the learning potential of these sessions could be improved by addressing the four core skill elements previously discussed. The laboratory course was reviewed, and the assessment and feedback components of the laboratory course were significantly overhauled to meet these criteria. This was achieved by improving the resources provided to students and laboratory demonstrators, changing the assessment tasks, as well as moving to an online submission system using the student platform WebCT.

**Methods**

**Changes to assessment**

Previously, students were required to submit two full reports per week. We implemented a change whereby students were encouraged to concentrate and focus on the presentation of scientific writing in one full report per week. An explicit guide to write a laboratory report was created and provided to students online and in their laboratory manual. This guide (Appendix 1) provided details about when and where select information and data should be used in a report. Discussions on appropriate writing techniques were initiated by demonstrators in class and provided in written feedback. There was an assumed second year level of literacy and referencing skills. The second lab of the week was assessed on the submission of a worksheet, which was provided to the student in a similar layout to a full written report. Tables and spacing were provided as examples of how to correctly present data and questions that encouraged students to engage in higher order conceptualisation of the experiment’s chemical theory were included. The final two assessment tasks of the course were created to allow a greater degree of
autonomy for students: in these assessments, students were required to develop their own experiments and to submit a full write-up in the style of a journal article (Appendix 2).

Development of resources

Student resources
Learning resources for the laboratory course were developed to enable students to complete the assessment tasks and encourage self-directed learning. These included a laboratory manual, online discussion forum, and other electronic resources including worksheets and a guide to scientific writing. The experiments were revised to ensure the objectives of the laboratory were clear, core concepts were highlighted, and the relevance of laboratory skills was identified. Pre-laboratory work (pre-labs) was included for each experiment, including a list of essential concepts, and for some laboratories a research proposal was required. Worksheets were developed for each experiment in the laboratory manual. The worksheets gave students adequate space to write experimental data and observations in class and students were encouraged to do so. Questions were developed to guide the students through the processing, evaluation and discussion of results; and discussion questions were developed to encourage a higher level of conceptual understanding and to bridge the gap between experiment and theory. Each section and question had marks allocated so that the students were aware of the weighting. An example of a worksheet, with marking guide and solutions is provided in Appendix 3.

Teaching resources
Postgraduate student demonstrators perform the majority of teaching in chemistry laboratories, generally with no formal training beyond a two-day introductory course. It is a requirement of second year teaching that demonstrators have a minimum one year of experience, with two to three years being typical. Weekly demonstrator meetings were implemented to train demonstrators before labs were conducted. This enabled experienced demonstrators to discuss issues that commonly appeared in previous years, and to explain laboratory equipment to new demonstrators. A demonstrator’s manual was introduced with the intention of creating a resource that provides guidance for student feedback and marking. The manual contained solutions for each lab, a writing template and a guide to providing online feedback. The solutions provided literature values pertinent to the laboratory and methods of calculations. For theoretical questions, model answers including main discussion points were provided, with a breakdown in marks if required. The writing template gave a reference standard which demonstrators could use to assess and provide feedback. The manual did not, however, contain an explicit guide to giving feedback; rather, demonstrators were allowed autonomy with the medium in which feedback was provided.

Online submission and feedback
An online system for laboratory submission and feedback was implemented. Each report was created as an electronic assignment sent to students’ WebCT accounts on the day of their laboratory. Each assignment included details of the assessment, a Word document of the worksheet for the particular laboratory, and a drop box that allowed students to upload required documents for submission. For full report laboratories, a copy of the writing guide provided on WebCT and in the student laboratory manual was also included. Marking was carried out by demonstrators in an electronic format, and once marked, reports with comments were returned to student drop boxes along with a grade. A variety of tools were used for feedback, including the track changes function in MS Word, comments with Foxit Reader for PDFs, and comments in the grade box on WebCT. Students were notified immediately through WebCT once feedback was available. Due dates and times were set for each experiment and student submissions were date stamped automatically, allowing demonstrators to keep track of late submissions.
Evaluation of alterations to the laboratory course and format

Surveys were carried out to gauge student perceptions of the changes made to the Chem2220 laboratory course. This was done with reference to the first semester unit Structure and Determination Chemistry, Chem2210 (not a prerequisite) which has a similar laboratory and assessment schedule. The survey was administered in class during the first week of laboratories. Students also completed an evaluation survey based on their experience of the new laboratory format, and this was administered in the penultimate lecture. The surveys included questions about worksheets, the online submission format, and feedback received from assessment. Demonstrators and the course coordinator were also asked to comment informally on the online submission system, assessment tasks and teaching resources provided. The development of student writing skills was assessed by comparing first and final week laboratory reports. Student reports from previous years were also used as a tool for comparison.

Results

Perception and expectations survey

The students were surveyed (49% response rate) about their initial perceptions of an online submission system, resulting in 45% positive responses, 34% negative, and 21% indifferent. Students liked the convenience of submitting online. However, the main concerns raised included technical issues with the submission of documents, and word processing. In a comparison of the two units, Chem2210 and Chem2220, student perceptions of demonstrator feedback both before and after the laboratory course were evaluated. The general perception of feedback in Chem2210 was that it had been useful but students wanted more feedback received closer to the submission date. When asked what type of feedback students would like to receive, a large number cited explicit points regarding how to improve their work.

Post laboratory course survey

Thirty seven (47%) responses to the post laboratory survey were collected.

Usefulness of worksheets

The worksheets were highly rated among students in aiding their understanding of the key aspects of the laboratory (89% useful or very useful rating). Eighty six percent of students found the worksheets useful for writing full reports (Figure 1). Common reasons cited were that the worksheets showed students what was expected of their work and provided guidance with questions and layout. Other reasons cited for the usefulness of the worksheets were that they helped the students with formatting, and that they were convenient and time-saving to use.

![Figure 1: Evaluation of the usefulness of worksheets in helping students to understand key aspects of the laboratory and writing a full laboratory report](image)
Confidence in writing a full report
At the completion of the laboratory course, 78% of students felt that their scientific report writing was adequate for higher level units. The main rationale cited for proficiency in report writing was the amount of practice; other reasons cited included that the skills required had been taught in detail this year, and that the student was comfortable with the task. Students who felt their skills were inadequate were not confident with specific aspects of the reports such as aims and conclusions, error evaluation, and literature searching. Other reasons for not feeling confident were due to shortcomings in demonstrator feedback such as a lack of or inconsistency in feedback.

Feedback
There was a positive response to feedback (Figure 1) and students were most appreciative of feedback regarding where marks were lost and where improvements could be made. Students also responded positively to demonstrators who returned their reports within a short period of time. However, 70% of respondents did not receive feedback within the desired seven days, leading to complaints that feedback was sparse or lacking. The online system did not significantly affect the students’ perceived access to feedback.

Online submission
After the completion of the laboratory course, more students preferred the online submission system to the previous hardcopy system (Table 1). It should be noted that the format of the post-laboratory survey questions was slightly different to the perceptions survey, with five options in the prior survey and a yes/no question in the post evaluation survey which specified that the reports must be word processed. Student responses indicated that the online submission system was convenient, saved on printing and paper, and allowed more comprehensive and legible feedback.

Table 1: Attitude towards online submission before and after the laboratory course

<table>
<thead>
<tr>
<th></th>
<th>Before (47 responses)</th>
<th>After (35 responses)</th>
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<tbody>
<tr>
<td>Positive response</td>
<td>45%</td>
<td>57%</td>
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<tr>
<td>Negative response</td>
<td>34%</td>
<td>26%</td>
</tr>
<tr>
<td>Neutral</td>
<td>21%</td>
<td>17%</td>
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</table>

Teacher perceptions
A comparison between full laboratory reports from the first and the last week of the experimental component of the course, and in relation to reports from previous years courses’ demonstrated an apparent improvement in writing technique. Over the six week laboratory course, the students’ ability to separate results from discussion, provide a clear aim and to draw meaningful and technically correct conclusions were developed. Final week reports from previous years did not demonstrate the same level of writing skills.

The following observations were made by demonstrators.

- Worksheets were used in class.
- There was an increased use of computers in the laboratory for entering data.
- There was notable increase in discussion between students and with demonstrators regarding data presentation, report format and writing style.
- Students embraced the more autonomous assessment tasks of the final two experiments.
- The discussion forums on WebCT were used extensively by students to assist in completing laboratory assessment tasks.

Overwhelmingly, students reported to demonstrators the intensity of the laboratory course as highly demanding on their time. Worksheets were not seen as significantly time saving for students, however some demonstrators reported that the worksheets were easier to mark and
provide feedback. Demonstrators reported a perceived increase in the quality of the feedback they gave, predominantly because of the convenience of typing.

The instruction manual was well received by demonstrators, with verbal feedback confirming that most used it on a regular basis. A valuable suggestion for improvement was the need for sectionalised advice for pre-laboratory class discussion, in class trouble shooting, and more explicit answer guides such as a sample of previous work. A suggestion to include common calculation errors and mistakes was raised as a means of reducing marking times. Reactions to the pre-laboratory meetings were positive and were regarded by demonstrators as useful tools for enhancing their demonstrating abilities.

**Discussion**

The revision of laboratory resources, and the subsequent overhaul of experiments was done with the aim of expanding the course to address all four core elements; practical techniques, chemical theory, scientific skills and scientific writing skills. Where each experiment developed practical skills, the method of assessment determined which skills were emphasised. Worksheets were provided for every experiment except for the last two laboratories. When worksheets were the sole assessment tool discussion questions highlighted the chemical theory involved in that experiment and tested this knowledge. The worksheets acted as training wheels to guide the students in their scientific report writing and when coupled with the writing guide, the emphasis shifted to scientific writing skills for full report assessments. The final two experiments increased autonomy and required students to combine the skills that had been previously been taught separately, shifting the emphasis to a higher level scientific skill set. This added degree of freedom was met with marked enthusiasm and we believe this is because of the relevance of the two laboratories as with students more willing to ‘own’ an experiment they designed themselves. Based on these outcomes we would recommend an earlier introduction of experiments of a similar format.

The purpose of this study was to improve the learning experience of a second year laboratory course through refinement of resources and assessments which included the introduction of worksheets. The use of worksheets in this context was novel and well-met by students, and overall their implementation considered successful. The worksheets were initially expected to be time saving to complete and grade however from the perspective of the students and some demonstrators they provided no time saving benefit. Nevertheless, students adopted the worksheets as a useful tool for completing the assessment tasks, and demonstrators noted that student scientific writing skills improved. Feedback from the post-evaluation surveys indicated that the worksheets aided students’ ability to independently present scientific data. The worksheets successfully highlighted the key chemical theories for each experiment, and ensured that pertinent questions were considered and answered. Consequently, students were assessed on their understanding of higher level concepts, as well as learning what techniques are necessary for a critical analysis of data.

The benefits of increased accessibility and flexibility of an online submission system was well received by students and demonstrators. The benefits of the online system as a feedback tool should be emphasised when compared to previously used systems. More detailed, legible feedback can be made available to the students with a higher efficiency for the marker. If made available online in a timely manner, feedback is readily accessible to the student in a significant improvement on the time taken for the collection and redistribution of hardcopy reports. Timeliness of the feedback has been an issue in previous courses, and the new online format has allowed the unit coordinator to monitor the turnaround time of assessments.

This study highlighted the importance of demonstrators as teaching staff. Laboratory classes typically represent more than half of the student contact hours, making demonstrators an important learning resource for students. If the outlined learning objectives are to be met,
demonstrators must be adequately trained and provided with the appropriate tools to provide good tuition and valuable feedback. The creation of the laboratory demonstrator manual was a positive step, and presents a valuable medium to encourage the transfer of knowledge across semesters, especially given that the turnover of teaching staff and demonstrators can be high. Its continued evolution has tremendous potential to strengthen the learning value of the course.

The delivery of feedback however was not consistent across the teaching staff and this is because there was no formal technique or guide provided. There was no policy on marking turnaround time, which was detrimental to the feedback loop and subsequently student development. This is clearly an area that requires further development to improve the course. In relation to this; the timeliness of feedback was hindered by the intense timetabling of the laboratory course, and could be improved by conducting the same course over a longer time period. Tailoring the laboratory assessment to build on learned skills is dependent on an effective feedback loop. We believe that a greater emphasis must be placed on high quality and timely feedback for the restructure to be truly effective.

**Recommendations**

Based on this report we have compiled a number of recommendations for higher level chemistry laboratories.

- Worksheets are a valuable learning tool which must be tailored to the learning outcomes.
- Demonstrators must be briefed on the pedagogical importance of feedback and given resources for providing timely and valuable feedback to students.
- Demonstrator manuals and weekly meeting are essential to ensure the transfer of knowledge between course coordinator and both past and previous lab demonstrators.
- Intense laboratory courses retard the feedback loop and increase student dissatisfaction; thus we would recommend weekly laboratories over a longer period of time.
- We would recommend the online submission system for laboratory courses.

**References**


Reid, N. and Shah, I., (2007), The role of laboratory work in university chemistry, Chemical Education Research and Practice 8 (2), 172-185

Appendix

1. Writing guide provided to students

EXPERIMENT SUBMISSION INSTRUCTIONS AND TEMPLATE

Worksheets
Worksheets have been created to help you complete your lab classes and to reduce the time taken writing up reports.
You should:
• Bring worksheets to lab classes.
• USE worksheets in lab classes.
• Use worksheets to complete the lab assignment.

You shouldn’t
• Just hand in a worksheet when a full write up is required (it won’t be accepted by your demonstrator).
• Fill out your worksheet without giving good thought to answering questions and presenting data with correct significant figures and errors.

Lab Assignments
Each lab will have an assessment task you will need to complete in order to gain marks (no marks for attendance alone). There are three types of assignments.

• Work sheet synopsis – these are short reports and the most common.
• Full lab write-ups – information on these below.
• Group journal article assignment – more information will be provided for these labs at a later date.

For full write ups there is a template of how you should submit it below, including descriptions of what goes in each section. Sectional questions should be answered with full sentences so that the reader does not have to refer to the answer sheet to understand the text. In the case of full write-ups the worksheet is intended as a guide. If you have any questions please don’t hesitate to ask your friendly demonstrator.

Title The title of your laboratory experiment.

Aims

In this section you need to summarise the aims of the experiment. Be sure to include the following aspects;

i) What are you setting out to measure?
ii) What experimental techniques will be employed to do so?
iii) What are the theoretical models that will be employed for data analysis?

Please note it is not just a direct copy from the lab manual, you need to use your own words to explain this and show you’ve done some background reading!

Methodology

Unless you have deviated, or have been asked to deviate, from the method stated in the lab manual it is sufficient to make reference to the lab manual for this section.
For the group journal assignment and the AAS Zinc experiment you are devising your own method thus must write a full explanation. It is essential that someone reading a method section can replicate your experiment exactly this means that you need to include details of the analytical reagents used (company, strength etc) as well as the specific techniques used (did you crush your tablet? How many times did you rinse your soil?). A really good way to learn what to include in a method section and the correct ‘tone’ of writing is to look at a Journal article. The *Journal of Analytical Chemistry* is a perfect guide for these experiments and available through the library website.

RESULTS

This section is where you include what your tests yielded. It is not a place to make excuse for an experiment that went wrong (i.e. I didn’t get a calibration curve because my solutions were not made properly is not a result you want to be reporting!)

It’s here that you include any tables of data, any plotted data, and any calculations employed. If you have undertaken repetitive calculations, it is sufficient to show the workings of one calculation. It is not advisable to simply show the results of the calculations without workings, as by doing this the marker has no idea what you have done, and hence is not in a position to determine partial marks.

Please understand the difference between results and discussion. Results are just that, your data without any interpretation or inference. It is a skill to differentiate between the two and something you should take note of! A good method for separating the two is to looks for words like; because, therefore, as a result of, hence, however etc as these generally indicate some sort of analysis of the result and should be included in your discussion.

***********VERY IMPORTANT***********

Numerical data needs to be quoted with estimates of the experimental error. It isn’t acceptable to make a statement of data without including the level of uncertainty because the reader needs to understand the reliability of making decisions based on the figures quoted, precision and accuracy are very important!

If at all unsure, then refer to the paper available on WebCT on “Treatment of Errors”

Failure to do so will result in the loss of valuable marks!

***********VERY IMPORTANT***********

DISCUSSION

This section is where you discuss your results, in the context of literature values and/or expectations as set out in the Aims section. You would also discuss here deviations from the literature values, and reasons for this (i.e. systematic errors.). It is an evaluation of the experiment...given your results what can you deduce? Does it agree with past experiments (i.e. from literature!)

If you are comparing multiple data sets, then do so here. For example, in experiment one where the t- and f-tests are used for statistical analysis, you should perform the analysis in this section. The raw data used in the analysis should however be presented in the Results section. You should draw upon the research question outlined in your aim and ensure that your discussion covers this.
Conclusion

This is the final section of the report and you must summarise the main results of the experiment, reiterating the comparisons with literature values. This needs to be stand alone so if one were to skim read your report and just read the aim and conclusion they will know exactly what was done, why, what was found and what did this mean.

While it’s difficult in these experiments to come to really meaningful conclusions we encourage you to put some effort into this section. Read a number of conclusions from Journal articles and see if you think you can determine what makes a good and what makes a bad conclusion.

Please be sure to include answers to all questions on the worksheets in your full lab write-ups, these will generally fit well into the presentation of your results and highlight key discussion points!

As a final comment, we often see work submitted that appears to have been written, and then not re-read. Please realise that producing a document requires that you write it, and re-read a number of times (i.e. proof reading). Bad prose sticks out like a sore thumb, and leaves a sour taste in the mouth of the report marker...

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2. Example of an experiment for a full write-up in the style of a journal article.

ZINC IN STREPSILS COLD LOZENGES

Pre-lab
According to the instructions below devise an experimental plan on how to quantitatively determine the zinc in your cold remedy product. Think about how to construct a calibration curve, the laboratory techniques you will have to use and the equipment required. Be sure to include calculations for your calibration curve construction.

Experimental description
The manufacturer of a certain cold remedy product claims each tablet contains 22 mg of zinc glutonate. You will use Atomic Absorption Spectrophotometry (AAS) to determine the quantity of zinc in a tablet and test the manufacturer’s claim.

The formula of zinc glutonate is Zn(C₆O₇H₁₁)₂ and the structural formula is shown below:
The ideal concentration for AAS analysis for zinc is about 5 ppm. Make up a solution of the tablet dissolved in water which will be about 5 ppm Zn if the manufacturer's claim is correct. Prepare four zinc standard solutions to bracket the expected value. Perform the AAS analysis in triplicate. Report the actual amount of zinc as zinc glutonate in the tablet and discuss the experimental uncertainties associated with your determination.

As you will be developing the method for this experiment please give full details in your report. It will be beneficial to read a few journal articles from a publication like the *Journal of Analytical Chemistry* to get an idea of what information you need to include and how.

**Marking key provided to students**

<table>
<thead>
<tr>
<th>Total (10)</th>
<th>Criteria 1</th>
<th>Criteria 2</th>
<th>Criteria 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aim (1)</td>
<td>Clear description of what is going to be measured. (0.5)</td>
<td>Clear description of the experimental techniques to be employed (0.5)</td>
<td></td>
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<tr>
<td>Methods (3)</td>
<td>Clear description of methods (1)</td>
<td>The methods are suitable for the aims (1)</td>
<td>The methods are suitable for a precise analytical study (1)</td>
</tr>
<tr>
<td>Results (3)</td>
<td>Relevant results are reported (1)</td>
<td>Presented in a logical manner (1)</td>
<td>Correct error analysis (1)</td>
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<tr>
<td>Discussion (2)</td>
<td>Adequate discussion of relevant results (1)</td>
<td>Discussion of experimental factors (1)</td>
<td></td>
</tr>
<tr>
<td>Conclusion (1)</td>
<td>Clear summation of results (1)</td>
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**Notes for demonstrators**

**Pre-lab board notes**

Aims – to perform an accurate analytical study, and to do a quality control experiment on the cold lozenges provided.

- Need to perform a full error analysis.
- Each measurement on the AAS should be done three times.
- They should come to the laboratory with an experimental plan – you will need to check this before they commence.

**Expectations**

Aim (1)

Method (3)

- A full error analysis should be conducted.
- Zinc calibration standards are made with a zinc salt rather than pure zinc as zinc is too reactive.
- The expected amount of zinc should sit in the middle of the calibration curve.
- Need a good explanation of methods, not just calculations.

Results (3)

- The standard deviation of triplicate measurements should be reported.
- Need to be presented in a logical way – tabulated, graphs labelled, *Linplot* used (*Excel* doesn’t compute errors in the unknown).
- Amount of zinc glutonate reported (not just zinc).
Discussion (2)

- Was their analysis close to the manufacturer’s values i.e. did their error range overlap the manufacturer’s?
- Sources of error?
- Any possible matrix effects?

Conclusion (1)

Notes from previous years:
You can let them make their own mistakes as it is not a time consuming exercise, although watch out they don’t get too off track. Here are some hints for the demonstrator;
- Zinc absorbencies stop being linear at about 10 ppm.
- The cold tablet provided may not match the details given in the lab book so they may need to dilute it more than once.
- Get the students to come to you and check their calibration dilutions before they proceed as this is a common source of mistakes.
- Emphasis is on writing a good lab report with a robust method – remind students they have to defend their results.
- Common mistakes – forcing the y-intercept through 0, the y-intercept is a measure of result accuracy – the smaller it is the more reliable the results
- To dissolve the zinc lozenge they might want to introduce solvent chemicals – remind them what solvents they use when they dissolve these lozenges in their mouths (hot water and stirring!)

3. Example of a worksheet including demonstrator notes and answers, given in blue italics.

STABILITY CONSTANT OF THE MONOTHIOCYANATOIRON (III) ION

1. Using the precise concentrations supplied with the standard solutions calculate the exact concentrations of Fe$^{3+}$, SCN$^-$ and H$^+$. Note: the numbers provided are the target concentrations you need to calculate what was actually used in your experiment!

<table>
<thead>
<tr>
<th>Solution</th>
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<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
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<tbody>
<tr>
<td>(a) [Fe$^{3+}$]/mol L$^{-1}$</td>
<td>$6 \times 10^{-4}$</td>
<td>$1 \times 10^{-3}$</td>
<td>$2 \times 10^{-3}$</td>
<td>$4 \times 10^{-3}$</td>
<td>$8 \times 10^{-3}$</td>
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<tr>
<td>(b) [SCN$^-$]/mol L$^{-1}$</td>
<td>$2 \times 10^{-4}$</td>
<td>$2 \times 10^{-4}$</td>
<td>$2 \times 10^{-4}$</td>
<td>$2 \times 10^{-4}$</td>
<td>$2 \times 10^{-4}$</td>
</tr>
<tr>
<td>(c) [H$^+$]/mol L$^{-1}$</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
<td>0.4</td>
</tr>
<tr>
<td>(d) $I$ (ionic strength)</td>
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Some students have a lot of problems with this question. They get confused about the target vs. actual concentration. You usually need to check their calculations in the lab (that they’ve used the actual concentration on bottle) and talk through with them which ion is the limiting reagent. Get them to check if they are using the right size volumetric flasks for their calculations because there’s often a mix of 25 ml and 20 ml volumetric flasks. Also, often there is confusion about using the graduated pipette…a pre-lab explanation of correct technique will help.
What are the associated errors for each concentration of \([\text{Fe}^{3+}]\)?

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<tr>
<th>Solution</th>
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<tbody>
<tr>
<td>([\text{Fe}^{3+}])/mol L(^{-1})</td>
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<td>V/ml</td>
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</table>

**Error of measurement:** pipetting and volumetric glassware errors are converted to a percentage, and the percentage is added together. Translated to absolute error, should be small.

2. Record the absorbance of solution 5 against a reference cell containing 0.4 mol L\(^{-1}\) perchloric acid at 10 nm intervals over the range 400-600 nm, plot the spectrum and determine the wavelength at which maximum absorption occurs. Once this wave length is known the rest of the measurements need only be taken for the specific wavelength. Record the temperature of the solution.

(3 marks = 1 mark data, 1 mark plot, 1 mark temp and max nm)

Make sure you highlight why we do this in the pre-lab: The idea is to optimise the absorbance thus we scan the wavelength until we find a maximum. Students should get an arch shaped plot (see Q9). It’s a good idea to walk around while they’re doing this and ask them to explain why it’s necessary to do this now but not for every concentration of \(\text{Fe}^{3+}\).

Temperature of solution _______________   Maximum wavelength____________

3. Measure the absorbance at the selected wavelength and record the temperature. (2 marks)

While students are doing this part talk to them about what they think the error is. Most will be able to tell you that there is a reading error from the dial, (1/2 smallest increment) some might mention that the machine will have its own error. This is correct and is 0.3% of the observed absorbance thus total error is the combination of the two.

Now is also a good time to talk to them about why they are using 5 solutions to make a calibration curve, what happens if one point is an outlier? How can they tell if their calibration curve is robust? Ask them if they can tell an outlier by eyeballing the data etc.

<table>
<thead>
<tr>
<th>Solution</th>
<th>Absorbance</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.085 ± 0.0052</td>
<td>21°C</td>
</tr>
<tr>
<td>2</td>
<td>0.120 ± 0.0054</td>
<td>21°C</td>
</tr>
<tr>
<td>3</td>
<td>0.210 ± 0.0056</td>
<td>22°C</td>
</tr>
<tr>
<td>4</td>
<td>0.335 ± 0.0060</td>
<td>21°C</td>
</tr>
<tr>
<td>5</td>
<td>0.470 ± 0.0064</td>
<td>20.5°C</td>
</tr>
</tbody>
</table>

4. Calculate the value of \(A\), \(A/c_f c_s\) and \((c_f + c_s) A/c_f c_s\) for each of the five solutions, include errors.

(5 marks = 3 marks calc, 2 mark errors)

<table>
<thead>
<tr>
<th>Solution</th>
<th>(A)</th>
<th>(c_f)</th>
<th>(c_s)</th>
<th>(A/c_f c_s)</th>
<th>((c_f + c_s)A/c_f c_s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.085 ± 0.0052</td>
<td>m(Q1)</td>
<td>Q1</td>
<td>7.15 x 10(^5) ± 6.6 x 10(^4)</td>
<td>571 ± 63.1</td>
</tr>
<tr>
<td>2</td>
<td>0.120 ± 0.0054</td>
<td>Q1</td>
<td>Q1</td>
<td>6.06 x 10(^5) ± 4.2 x 10(^4)</td>
<td>726 ± 60.3</td>
</tr>
<tr>
<td>3</td>
<td>0.210 ± 0.0056</td>
<td>Q1</td>
<td>Q1</td>
<td>5.30 x 10(^5) ± 2.71 x 10(^4)</td>
<td>1170 ± 75.8</td>
</tr>
<tr>
<td>4</td>
<td>0.335 ± 0.0060</td>
<td>Q1</td>
<td>Q1</td>
<td>4.23 x 10(^5) ± 2.4 x 10(^4)</td>
<td>1780 ± 153</td>
</tr>
<tr>
<td>5</td>
<td>0.470 ± 0.0064</td>
<td>Q1</td>
<td>Q1</td>
<td>2.97 x 10(^5) ± 1.2 x 10(^4)</td>
<td>2430 ± 39</td>
</tr>
</tbody>
</table>

5. Determine \(K_1\) from a plot of \(A/c_f c_s\) versus \((c_f + c_s) A/c_f c_s\) and also determine \(\varepsilon\) for the \(\text{Fe(SCN)}^{2+}\) at the wavelength of maximum absorbance. Include errors for both values. How does your value of \(K\) compare to the literature value?

(9 marks = 2 marks for plot, 2 marks for \(K\), 2 marks for \(\varepsilon\), 2 marks errors, 1 mark lit value)
- $K_1$ is the slope of the graph,
- $\eta$ is calculated from the intercept where $y$ intercept $= \eta K_1$.
- Errors should be combined as before then translated to a level of uncertainty using Linplot.
- The accepted literature values for $K$ at 25 degrees is 138 (J. Chem. Educ., 1963, 40 (2), p 71)

6. Explain why it is important that the ionic concentration of the solution remain constant in order to experimentally determine the stability constant and what effect it might have if changed. (2 marks)

Students should briefly explain that $K$ is reliant on the activity of the products and reactants. Therefore considering activity is directly related to the ionic strength of solution a change its value between solutions will interfere with results. It would be a source of error in the calculations if the ionic strength were to change between solutions (If the ionic strength were to change, the different solutions could not be used to find the stability constant as it would be different for each.)

7. Why must a value for temperature accompany a reported value for $K$? (2 marks)

Equilibrium constants (and thus stability constants) must be provided in the context of the temperature for which they are taken. This is because enthalpy, although not always written, is a product of each reaction.

The Van’t Hoff equation shows that all equilibrium constants change with temperature.

$$\frac{d \ln K}{dT} = \frac{\Delta H^o}{RT^2}$$

Thus to produce a result using multiple absorbance values and concentrations, the temperature must be constant.

8. What is meant by the term ‘stability constant’ how is it related to the equilibrium constant? (2 marks)

The stability constant, $K_{stab}$, is the equilibrium constant for the equilibrium that exists between a transition metal ion surrounded by water molecule ligands and the same transition metal ion surrounded by ligands of another kind in a ligand displacement reaction. The individual ligands are displaced stepwise, and an equilibrium expression could be written for each step, but it is more common to write an overall expression for the overall ligand displacement reaction. Like all equilibrium constants, stability constants vary with temperature (http://www.avogadro.co.uk/definitions/kstab.htm)

9. Why do you need to find the wavelength with maximum absorption to take your readings? How would your calibration curve be affected if you didn’t? (2 marks)

Maybe hint at this in class and try to get students to consider the shape of the curve they derive from question 2. If they consider the derivative for the rate of change is zero at the maximum then error is minimised. If they used a non-optimized wavelength slight variation either side would drastically affect the absorption value and thus the validity of their calibration curve. The maximum also gives the best signal to noise ratio. Students may also comment on the effect that potential non-linearity of absorption could have on results (although not a huge issue here because the range of concentrations is not large).