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Much has been said about effective learning in chemistry and, in particular, how the laboratory component plays a key role in developing students' understanding of the topic. While an emphasis in developing students' laboratory skills or on the demonstration of a concept has merit, the controlled nature of the laboratory experience can limit the time given to the student to plan the experiment and to interpret the results *(1)*. Domin's chemical analogy of the laboratory manual articulates this view perfectly (*1,* p 110):

> Just as a catalyst speeds up a chemical reaction by providing an alternative lower energy pathway, the laboratory manual reduces the amount of time necessary to complete a laboratory activity by providing an instructional pathway that does not require the utilization of higher-order thinking skills. The laboratory manual has become an instrument that maximizes laboratory efficiency at the expense of fostering higher-order cognition.

Problem-based learning has been used very successfully in other disciplines. Aspects of this approach are being used to teach analytical chemistry in the laboratory *(2–5)* or as a text-based or Internet-based case study *(6–8)*.

This paper reports on a related approach used to teach analytical chemistry in which both constructivism and problem-based learning feature strongly. The approach extended across both the theory and laboratory components of the curriculum unit, which was mainly focused on chromatography. In addition the use of a portfolio as the assessment tool is discussed.

Pedagogical Aims

The following ideas informed development of the modules designed to provide an alternative learning environment:

- Laboratory work provides scientists with a unique and invaluable learning tool *(1)*
- What a student *does* matters most in the student's learning process *(9, 10)*
- Assessment drives the learning process *(11)*
- Learning objectives and assessment must be aligned if effective student learning is to occur *(9)*

Also, the unit under study was a final-semester unit with students hoping to gain employment on completion. Therefore, independent and lifelong learning skills developed by students undertaking some self-directed and independent learning, working collaboratively with their peers, reading the literature, designing, planning, and carrying out some experiments, and managing their time seemed particularly appropriate. In an effort to provide an effective learning environment and promote independent learning and to fully and transparently give the students the responsibility for their learning, the traditional lecture and laboratory manual were abolished. To further support this approach the assessment had to be radically changed. Students were asked to submit a portfolio that provided evidence of their learning.

Overall Framework

A curriculum unit in the previous semester had introduced the students to instrumentation-based chromatography and, in particular, high performance liquid chromatography (HPLC), gas chromatography (GC) and capillary electrophoresis (CE). To support their learning students completed several laboratory activities, including: quantitative determination of caffeine in soft drinks and preservatives in food, both by HPLC; determination of ethanol in wine and separation of fuel gases, both by GC; and separation of cations and preservatives by CE. In all cases, the analytes were ideally suited to the technique. For example, caffeine is a neutral organic compound that is soluble in aqueous solutions and absorbs strongly in the UV region. Therefore, it is an ideal analyte to study using reversed-phase HPLC with UV detection.

The curriculum unit under discussion in this paper is divided into two modules, one of which was designed to extend students' understanding of chromatography beyond that of the previous semester; the other module focused on environmental chemistry. This paper reports on the first module, which ran for seven weeks. In this chromatography module, we placed a strong emphasis on how to deal with difficult samples (those that were not readily amenable to RP–HPLC or GC), other separation mechanisms, and detection methods.

Amino acids were chosen as the target analytes as they are nonvolatile, which makes them unsuitable for direct analysis by GC and many amino acids lack a strong chromophore making them unsuitable for HPLC–UV–vis and CE–UV–vis detection (when sensitive detection is required). The reading material provided to students (see the online supplement for details) in the first instance avoided methods that were outside the scope of the laboratory (e.g., mass spectroscopy detection coupled to HPLC) and included methods that were available (e.g., evaporative light scattering detection, ELSD).

Using a Portfolio for Student Assessment

The use of a portfolio was a real change in assessment for most of the students who were accustomed to a final exam (usually closed-book). While students were keen to work in groups in the laboratory they unanimously supported individual portfolio submissions. To provide students with some structure for their portfolios the content of the portfolio was outlined with students asked to submit written materials in the following five categories:

1. A summary of the outcomes and learning from the first workshop
2. A laboratory report
3. Evidence of self-directed learning (minimum of three examples)
4. Examples of shared learning resources (students posted a minimum of two items on a course management system Web site)
5. A reflective log that details a before and after analysis of the student's learning experience

It was imperative that the students were confident about and understood the assessment approach used, so they were supplied with a handout (included in the online supplement), which explained the learning approach and use of portfolio assessment and what evidence of self-directed learning and shared learning resources might look like. The lecturer also provided students with examples (based on a different issue or problem) to further illustrate and demystify the individual assessment items (see the online supplement). Students were familiar with writing scientific laboratory reports as in the previous semester the lecturer and learning advisor together had actively worked with the students on writing both laboratory reports and essays. As a university-level employee, the learning advisor's key role is to support students in their learning, which includes helping students gain and develop writing skills, interpret assignments, and develop critical learning skills. Traditionally, this help has been largely one-on-one although more recently the approach has been to work with lecturers and their students.

What Did Students Do?

At the workshop in week 1, having thoroughly discussed this alternative approach to learning, the students worked in groups reviewing the reading material provided by the lecturer. After a class discussion that highlighted the main issues regarding amino acid analysis, the students further defined the issue they were going to explore for the next six weeks as "detection issues in the analysis of amino acids by HPLC and GC". The class broke into groups of 2–3. Each group selected an area to focus their laboratory work on (e.g, one group investigated amino acid analysis by GC, another group selected HPLC with ELSD). For week 2, the students had to write a summary detailing what they had learned during the first workshop. Also for week 2 students had to use the journal articles provided, as well as collect other relevant articles, to plan the experiments they would do.

In week 2, the students came back with rough procedures, chemical lists, and lots of questions. During this session, students had to read through material safety data sheets, and to determine the cost and availability of chemicals that had to be ordered. All of these informed the students as to what experiment they would run in week 3. For example, one group was keen to try methyl chloroformate, a derivatizing agent but then learned that the substance was explosive, only available in the United States, and would take at least six weeks to arrive by ship. For obvious reasons the students selected another derivatizing reagent. At the end of this session, students had refined their procedures and left to prepare (in their groups) a chemical and instrumentation requirements sheet that was to be handed in to the technician three days prior to the laboratory session.

The students decided on the composition of the amino acid mixture. The mixture they chose was taurine, lysine, serine, asparagine, glutamine, proline, and arginine. For example, students chose taurine because it is in sports drinks and proline because it is an example of an amino acid that is not easily derivatized for HPLC analysis.

Over the next five weeks (in a 5-h laboratory period each week) the students planned and carried out experiments to better understand the issues regarding amino acid analysis and, in particular, their detection. To support the work in the laboratory and to develop understanding of the topic chosen, students also completed some independent learning.

Student Example

To better illustrate the process, the path taken by two students is briefly documented here.

Two students decided to study amino acids in the laboratory using an ELSD coupled with HPLC. They were familiar with HPLC but not ELSD. They elected to separate the amino acids using a method described in the literature that involved using an ion-pairing reagent, trifluoroacetic acid, and a reversed-phase column.[1] Initial efforts failed to resolve the mixture. The students, after some discussion, tried a number of strategies to resolve the mixture, which included changing the column (in case it was a faulty one), varying the organic:water mobile phase composition, and increasing the ion-pairing reagent concentration but with no success. After some discussion in the laboratory, the students concluded that the ion-pairing reagent must not be suitable and went off to search the literature. The deflated students came back buoyant. A closer look at the literature revealed that TFA was not useful in retaining and resolving the very polar amino acids present in the student mixture *(12)*. Perfluorinated carboxylic acids with longer *n*-alkyl chains are required to resolve the more polar amino acids *(12)*. Pentadecafluorooctanioc acid (PDFOA) was particularly effective. They had successfully problem solved and along the way they had gained valuable skills and understanding. In particular, because the students were trying different strategies using the same technique and samples, the repetition was invaluable in reinforcing skills and in developing their problem solving skills, which is not possible in one 3-h laboratory. A related approach of incorporating reiterative laboratory sessions at the end of a unit has also had similar benefits *(13, 14)*.

Another "learning moment" that both challenged and informed these two students' learning was when they were experimentally determining the linear range and limits of detection (LOD) for their ELS detector, with the aim of comparing it to experimentally generated GC and HPLC–UV–vis data collected by their peers in the other groups. Initial attempts seemed problematic, as the anticipated linear relationship between concentration and detector response was not realized. Again, the students resolved this apparent error by reviewing the literature, which demonstrated that polynomial relationships and not linear relationships are typical at low concentrations for ELSD *(15)*. Here the repetition also consolidated their learning. They really began to appreciate what LOD, linear range, and other terms actually mean.

The laboratory work was central to the students' learning and in many cases provided the impetus for their independent learning. For example, some of the self-directed learning items these two students submitted in their individual portfolios included: a summary (table and paragraphs of text) of the different separation conditions and detector conditions reported in the literature for the separation of amino acids using HPLC–ELSD; an account of how an ELS detector works; an account of an alternative highly sensitive technique for amino acid analysis using liquid chromatography with mass spectroscopy detection; and a detailed account of ion-pair chromatography. As part of their portfolios they also prepared some shared learning resources and they had to justify the value of the resource that was posted on the course management site. For example, by week 3, one of these students posted a table with the formula, molar mass, and structure of the common amino acids. Some weeks later one of the individuals in this group provided a table that detailed the LOD obtained by their method so that their peers in the other groups could refer to it in their reports for comparison purposes.

What Was the Role of the Lecturer?

Behind the scenes, the activity was tightly structured. The lecturer needed to choose a topic that would provide a number of challenges for the students and expose students to the relevant content. Amino acid analysis was chosen but carbohydrate analysis would also have worked well. In another situation with access to a different suite of equipment and different unit content the focus might be very different. In addition to reviewing the literature to select appropriate reading for the first workshop, the lecturer also tried to predict the type of experiments that the students might want to run and purchased the necessary resources (e.g., derivatizing and ion-pairing reagents). In addition to the more technical role of a demonstrator who is responsible for maintaining a safe working environment by vetting students' laboratory experiments and promoting safe and appropriate handling of chemicals and equipment, a key role of the demonstrator (in this case it was the lecturer) was to encourage discussion, ask relevant questions, gently guide the students to new ideas and methods when they had exhausted their repertoire, and to celebrate with the students when they "got it". In questioning the students, the lecturer, depending on the context, sought to determine the students' understanding, to clarify some content, to challenge the students' understanding, or help them adopt new ideas. In many cases the questioning opened up some group discussion that in turn led to the lecturer's providing some explanation of concepts at a time when students were open to it rather than in a lecture where there is no context. Certainly a limitation of this type of learning is the need for an experienced demonstrator or lecturer, who is comfortable in facilitating student learning rather than "controlling" it.

Student Outcomes

The key student learning outcomes were met in completing the portfolio (Table 1). The students gained excellent hands-on skills on the instrument and several of the students commented on this in the evaluation sheet they completed after finishing the module. It was also quite evident to the lecturer that the students were competent in operating the instruments and in basic troubleshooting. They had clearly extended their knowledge and understanding (the knowledge and skills gained were unique to each student). The self-directed learning items produced by the students were of extremely high quality. It was evident that the students understood what they were writing about. They used their own words and descriptions rather than trying to cut and paste from a variety of sources. In addition, the introduction and discussion sections of the laboratory report reflected a deep understanding of the topic. They clearly demonstrated effective written communication skills. The focus on writing skills in the previous semester really did benefit the students; they were practicing the skill rather than struggling with such things as what makes a good paragraph.

The transferable skills that students developed were very clearly demonstrated during the class activities and to some extent in the portfolio entries. Problem solving, higher-order thinking and communication skills were absolutely necessary to successfully complete this module. The students developed and practiced these skills alongside developing their laboratory skills.

Table 1. Distribution of Student Learning Outcomes by Assessment Strategy

	Student Outcomes To Demonstrate					
Assessment Items	Laboratory Skills	Description of Analytical Techniques	Effective Communication Skills	Problem Solving Skills	Critical Thinking Skills	Knowledge of Occupational Health and Safety
Summative						
Laboratory Report	X	X	X	X	X	X
Summary of Workshop			X			
Self-Directed Learning	X	X	X			
Shared Learning Resources	X	X	X			
Reflective Log			X			
Formative						
Laboratory Activities	X	X		X	X	X
Workshops in Weeks 1 and 2			X	X	X	X

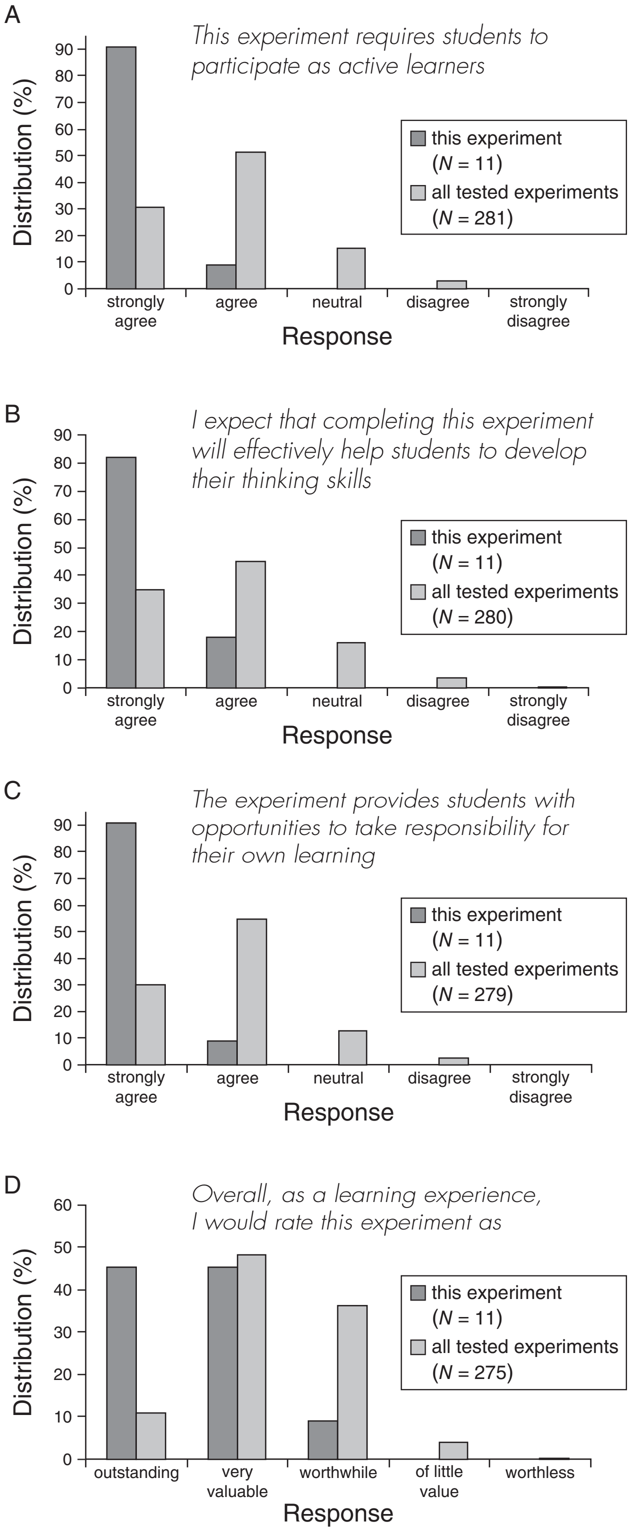

Figure 1. Selected evaluation data for this activity from the ACELL workshop, compared with aggregate data relating to all experiments tested. Acknowledgement: ACELL project *(17)*.

In the end, students gained so much more than these specified outcomes. Because they were largely responsible, they better understood and appreciated the need for independent learning and how it fosters lifelong learning.

While primarily for the students to reflect on their learning, the reflective log was very informative for the lecturer and learning advisor. It is clear from the reflective log that students recognized the importance of transferable skills, especially as they were about to enter the workforce. Time management, questioning, and thinking skills were mentioned by most students as being important and a sense of responsibility for one's own results also featured in students reflection.

The multiple "conversations in chemistry" were an unintended outcome of the learning process. This verbal articulation of chemistry is invaluable and is often lost in the traditional laboratory where the focus is on trying to get through the experiment and the thinking comes after, often in isolation.

External Evaluation and Feedback

The authors presented this activity at the Australian Chemistry Enhanced Laboratory Learning (ACELL) Workshop held in Sydney *(16)*. Chemists from 27 Australian and New Zealand universities submitted examples of best practices in student laboratory learning. One important aim of the ACELL project is to make available pedagogically sound undergraduate experiments, along with everything necessary for them to be easily adopted by other institutions—quality control is maintained by incorporating an element of peer review into the process. The aim of the workshop was to peer review the submitted experiments and provide the attending academics with ideas and experiments that they might adopt in their own institutions. Another function of the workshop was to demonstrate that each experiment is transportable to another institution; for this reason, each experiment was tested by up to 13 people (a mix of academics and undergraduate students from the participating institutions) who then completed some evaluation forms (which were administered by the ACELL team).[2] This feedback was collated and was later distributed to the authors *(17)*.

This activity was tested by 12 workshop delegates; their feedback was very positive. As can be seen in Figure 1, the experiments tested at the ACELL workshop were positively received by delegates, and even in such a group of valuable experiments, this activity stood out. Over 80% of those who tested this activity "strongly agreed" that it would help students develop their thinking skills, require them to participate as active learners, and provide students with opportunities to take responsibility for their own learning (Figure 1). This supports the view that this activity is successful in achieving its aims. The participants also "strongly agreed" that the activity would effectively help students to develop their conceptual and theoretical knowledge (80%) and their scientific and practical skills (>60%). The activity was rated as an "outstanding" learning experience by almost half of those who tested it (Figure 1D). The verbal feedback was also very positive, particularly, from the student participants. Some valuable feedback from this forum will be used to inform the next phase of this action research project.

Conclusion

Finally, this approach is certainly more difficult to adopt if less experienced teaching staff were employed in the laboratory or if the class size exceeded 20 or 40 (two groups). However, no matter what the class size we should be able to provide a more investigative, problem-oriented approach to our laboratories. We should be able to provide our students with greater opportunities to enhance their learning skills, and demonstrate to us what they know rather that what they do not know.

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Notes

1. Originally Application Note 0040E, Alltech Associates Inc., 1999. Now a part of W. R. Grace and Co., some descriptions of Alltech's products are available online. See, for example, the applications document, p 437, Peptides on 238MS C18 Column with Various Modifers, at *http://www.discoverysciences.com/uploadedFiles/Site_for_Catalog_2008/applications/Applications.pdf* (accessed Sep 2008).
2. Collection of these data was authorized by the Human Research Ethics Committee of the University of Sydney, approval number 12-2005/2/8807, and supervised by the ACELL directors and associate director.

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Supplement

Description of portfolio assessments and grading rubric; Details of teaching and learning strategy; Reading list for students

Guidelines for writing laboratory reports; Criteria for a scientific laboratory report