Using Small Scale Techniques to Assess Laboratory Learning

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The laboratory part of a chemistry courses is considered essential by most chemistry instructors, yet few instructors attempt to assess the laboratory learning with anything other than written lab reports. At best lab reports may only provide feedback on students observational skills, communication skills, and their ability to follow directions, skills that students should have, but are hardly the essence of laboratory training. Those instructors that do attempt to assess students laboratory skills often do so with paper and pencil exams. An alternative, much favored by biologists, is a station to station exam. As students move from station to station in the laboratory there is some material to examine or a simple task to perform. This format has been used occasionally in chemistry with tasks that usually involve a simple skills such as reading a burette or making a simple measurement (Silberman et al., 1987). In introductory courses there have been very few attempts to implement laboratory practical examinations that assess the ability of students to devise and carry out an experiment.

Most chemistry instructors would agree that the ability to manipulate lab equipment and follow directions are important skills, but the ultimate goal of laboratory training is to teach students how to solve problems in the laboratory. The only systematic assessment of student's ability to devise and carry out an experiment occurs in graduate school, this in a discipline that prides itself on being an experimental science.

As suggested by Johnstone (1990), laboratory problems can be grouped into the four categories shown below.

Category	Problem	Method	Solution	Problem type
1	given	given	given	Instructor demonstration
2	given	given	devised	Typical lab problem
3	given	devised	devised	Laboratory assessment problem
4	devised	devised	devised	Laboratory research

The problems range from an instructor demonstration in which the problem, method and solution are worked out and shown to the student, to a research project in which all aspects of problem are devised by the student as a researcher.

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Typically most undergraduate lab problems fit category 2 as exemplified by a typical problem in a students laboratory manual. The problem is stated, the method has been worked out, and a student follows a detailed set of instructions, Laboratory assessment activities should aim for category 3, only the problem is presented, the student must devise the method.

In recent years interest in laboratory assessment has been on the increase and several individuals have attempted to devise laboratory assessment activities (Haddon, 1991; Haddon, 1992; Wood and Sleet, 1993; Silberman, 1996). Interest in laboratory exams has been encouraged by the inclusion of a laboratory practical component in the international Olympiad exam. However, many of the questions on these exams do not really assess all the skills that enable a student to design and carry out an experiment. Consider this question on a recent Olympiad exam. Determine the concentration of Mg^{+2} and Ca^{+2} in the "Bottled Water" sample. If a student were simply given this instruction and then given a laboratory with a large assortment of equipment and reagents, this question would certainly be a test of ability to design a laboratory experiment, but answering would require much more time and effort than one could reasonably expect from even the most advanced high school student. Instead, the question includes a detailed titration procedure, similar to the procedure found in a technicians handbook. A sample instruction:

> Add 40 mL of distilled water, 5 mL of pH 10 buffer solution and some calmagite indicator to 5.00 mL of the "Bottled Water" sample. Titrate this sample with EDTA solution to a clear blue endpoint.

While it is true that the student has to know what a titration is and how to do it, not much creative effort or thought is involved. The problem clearly fits in category 2. The question was graded on the accuracy of the students final determination of the percent concentration of Mg^{+2} and Ca^{+2} in the "Bottled Water" sample. The question as presented and graded is a test of the students technical skill in the laboratory. Questions that require students to follow a detailed set of directions to analyze or synthesize something simply do not test most of the skills and abilities related to problem solving.

Until recently there has been an almost complete absence of practical examinations in chemistry that attempt to assess student ability to design and carry out an investigation. This probably stems in part from the fact that such practical examinations are perceived to be time consuming to prepare, administer and grade. With the development of small scale laboratory techniques and experiments it becomes possible to develop practical exams that test students "laboratory thinking skills" and to use problems posed in the laboratory as assessment tools. Using small scale equipment and methods greatly reduces the time and expense associated with laboratory examinations. Consider the following laboratory practical exam question suitable for a high school students (Problem 1.)

Problem 1

Devise and carry out an experiment to test the hypothesis that the volume of any liquid, divided by its mass is a constant. You may use any of the designated materials at your desk.

The student is given the following materials, 2-3 plastic transfer pipets, a small container, paper, a pencil, access to a balance and a few liquid samples. The liquids can be provided in plastic transfer pipets. All the necessary materials can easily fit in a small container such as a small box, beaker or plastic bag.

The task seems to be a simple one, but there is no set of instructions and no procedure. Students must develop these by themselves. For many students this problem proves difficult. Students must decide: 1) how to reproducibly measure volume; 2) what liquid volumes to use; 3) what liquid(s) to use; 4) how many volume-weight determinations need to be measured; 5) how the data should be treated; 6) what constitutes proof of a hypothesis. They also must use a good experimental procedure. This includes such simple things as using the same pipette in the same way for each liquid sample or choosing a appropriate size container for weighing samples. Simple technical skills such as the ability to accurately weigh or pipet samples are also part of this problem. This problem is a far better test of a student's ability to think creatively in the laboratory than a follow the recipe problem. When this problem was given to a large number of high school students many had difficulty with it.

Consider a much more difficult problem that involves acid-base chemistry (Problem 2).

Problem 2

You will be given four (4) solutions in plastic thin-stem Beral type pipets labeled A, B C, and D. The solutions contain either HCl or NaOH in the concentrations listed below Each acid solution also contains phenolphthalein.

Possible acid solutions	Possible basic solutions
1.0 M HCl	1.0 M NaOH
0.5 M HCl	0.5 M NaOH
0.1 M HCl	0.1 M NaOH

The most concentrated acid in your group of four solutions has been carefully standardized. Use this solution to determine the precise concentration of the most dilute basic solution.

The chemicals and equipment needed are simple, 4 transfer pipets with the acid and base solutions and some kind of container to mix solutions in such as a wellplate or small flasks. Solving this problem requires considerable knowledge of acid base chemistry and a thorough understanding of how to plan a series of experiments to solve a problem. Students must: 1) develop a reasonable strategy for solving problem; 2) figure out how to identify acids and bases; 3) determine the most concentrated acid and the most dilute base; 4) collect and manipulate data; 5) perform repeated titrations; 6) draw correct conclusions from observations. All of this must be done with careful and accurate laboratory technique. This problem is far more difficult than asking students to perform a titration of an acid with a base. It presents a considerable challenge to most high school or college freshman chemistry students.

A useful feature of many smallscale laboratory problems is that they can easily be modified. In problem 2 the number, concentration, and kinds of acids and bases can be changed. This allows an instructor to give a seemingly different question to every student in the class. The problem can also be made easier or more difficult with a few simple changes. For example, the problem is much easier if a separate container of phenophthalein is provided or much more difficult if the concentration of the acids is much higher than the concentration of the bases.

Grading problems like these is more difficult than simply checking an numerical answer or the physical properties of a compound that has been prepared. The strategy is the one used to grade an essay exam question. Decide what a best correct answer would be and then assign a point value to various parts of this answer. A sample grading scheme for problem 1 above is shown below:

1. Determination of the liquid's volume and mass **2 pt.**
Possible methods and credit awarded:
a) The student determines volume by squeezing out some of the liquid into small graduates cylinder to measure the volume, but weighs the graduated cylinder empty and with the liquid to obtain mass. *1 pt.*
b) The student gets the weight of the liquid by weighting the Beral pipet before and after some of the liquid is squeezed out. *1 pt.*

2. Recognition that both volume and mass of the liquid must be measured several times. **2 pt.**
Rationale: Two data points will always give a straight line so do not yield any useful information. Three data points are the minimum number, but the line that results can be very sensitive to any one error in measurement. Four or five data sets will produce even more reliable results.

3. Plot of data on graph paper **4 pt.**
a) The student chooses consistent, reasonable intervals for both axes and labels them clearly. *2 pt.*
b) The student uses the data sets to draw a reasonable straight line. *2 pt.*
4. Interpretation of straight line on graph to mean that there is a constant ratio. 2 pt.

Extra credit could be awarded if the student
a) realizes that the ratio being determined is not density but the reciprocal intensive property.
b) uses the correct number of significant figures.
c) repeats measurements rather than relying on a single value.

Grading can be complicated because students often find more than one acceptable solution to a problem. A grader must be alert for alternate solutions that may be even better than the one the grader has devised. Creativity and innovation should be rewarded by giving extra credit for novel or creative solutions to the problem.

For example consider the following problem.

Problem 3

Devise and carry out an experiment to determine the pK_a of an unknown weak acid. Describe the method you developed to solve this problem.

Materials available

Chemicals	**Equipment**
Phenophthalein solution	Beral type transfer pipets
0.1 molar weak acid	24 well wellplate
0.1 molar NaOH	optional, pH meter
Universal indicator with color chart	

The problem tests student understanding of the relationships among pK_a, pH, and the titration of weak acids. Although it is common for students to manipulate the mathematics of these relationships in an algorithmic fashion, it is less common for students to determine these factors experimentally. Even if they have performed this experiment either in a macroscale or a microscale format, it still is a very discriminating experiment when the design options are not given. There are two equally good solutions.

Answer 1
1. Titrate 25 drops of the acid sample to a phenolphthalein endpoint with the base.
2. Add 25 more drops of the acid sample, assuring that $[HA] = [A^-]$; this means in turn that pH = pK_a.
3. Determine the approximate pH of the solution by adding universal indicator, by using universal indicator paper, or with a pH meter if this is available.

Alternate answer
1. Titrate a sample of the acid to the endpoint, counting the number of drops of base.
2. Then, titrate a second sample with exactly half of the previous volume of base. The same condition can be reached, that is, $[HA] = [A^-]$.
3. Determine the approximate pH of the solution by adding universal indicator, by using universal indicator paper, or with a pH meter if this is available

A sample grading scheme for problem 3 might look like the one shown below.

1. Measurement of a known volume of weak acid solution to titrate. **1 pt.**
2. Titration of a known volume of acid to an indicator endpoint. **1 pt.**
3. Determination of the pH when the acid in half-titrated. **2 pt.**
a) The student in no way addresses the pH at the half-titrationpoint. *0 pt.*
b) The student follows the likely approach procedure. *2 pt.*
c) The student uses an alternate procedure that is also reasonable. *2 pt.*
4. Determination of the pK_a **1 pt.**
a) The student is within 2 units of the true pK_a *0.5 pt.*
b) The student is within 1 unit of the true pK_a *1 pt.*

Extra credit could be awarded if the student
a) understands and discussed the uncertainty in the answer.
b) repeats measurements rather than relying on a single value.

Developing smallscale lab practical problems

When designing laboratory assessment problems it is important to begin by reviewing the laboratory curriculum to make sure that the questions developed test students' knowledge of the concepts actually taught. It would be unfair to ask students to design a experiment about pK_a if that material has not been taught. Before actually beginning to devise a problem it is a good idea to briefly write an assessment objective for the problem. For example the assessment objective for the problem 3 above was to test students depth of understanding of the relationships among pK_a, pH, and weak acid-base titrations. Another point to consider is the availability of resources. There is no point in making up a question that requires a pH meter if only one is available for an entire class. Assigning a time frame for an assessment problem is often difficult, because problem solving can be a slow thoughtful process. A problem that may seem very simple for an instructor may take a first year student a half an hour or more. Perhaps the best way to assess a problem's difficulty is to try it out with a volunteer.

A way to evaluate problems before they are used in class is to compare them with a list of lab skills. If lab skills are classified with the following scheme, good problems generally include some skills from each category.

1. Simple laboratory skills
   a. Weighing
   b. Measuring volume of solids, volumes of liquids
   c. Using heating equipment, burners and/or hot plates
   d. Using pipettes and burettes
   e. Following directions
2. More complex skills
   a. Preparing solutions (from solids)
   b. Preparing solutions from more concentrated solutions.
   c. Determining density of liquid, solid and gas
   d. Determining concentration by titration
   e. Identifying a compound using chemical test
   f. Measuring and recording physical properties
   g. Measuring and recording chemical properties
   h. Recording data
3. Complex skills involving higher order thinking
   a. Interpreting data
   b. Designing an experiment
   c. Testing a hypothesis
   d. Critiquing an experiment
   e. Data analysis

When designing problems, there are some common pitfalls. The problem may be trivial or one that virtually every student can answer from experience. An example of this is evident in problem 4 below, that was suggested by a participant at the beginning of a work shop on laboratory assessment.

Problem 4

Which will dissolve faster in water, granulated sugar or sugar cubes?

The obvious flaw is that every student already knows the answer. A less obvious flaw is that the determination is a trivial experiment. A student puts both in water and watches the process.

Another example of a poorly designed question.

Problem 5

Watch the chemical reaction that will be demonstrated for you. The proctor will add a piece of aluminum foil to an aqueous solution of Copper(II)chloride. Record observations of chemical or physical changes you see.

This is not a test of laboratory skills at all! At best it is a test of observation skills. An alternate approach could have been used that would make it in part a test of lab skills e.g. Give the students several salts and ask them find out which ions can remove the protective oxide layer from aluminum.

The final example is a question that appears to be laboratory based problem, but is primarily a test of math skills.

Problem 6

Determine the thickness of the zinc coating on the zinc coated washers provided.

The problem with this task is that the actual lab operations are simple; weight the washer, remove the zinc coating with HCl, dry the washer, and weight again. The difficult part is the calculations of the surface area of the washer. Although this a challenging problem for many high school students, it is challenging because of the calculations rather than the chemistry. Keep in mind that a well designed assessment problem tests content knowledge, but also skills related to experimental design, communication, observation and reasoning.

Laboratory assessment tasks allow a teacher to assess studen's ability to use laboratory thinking skills to solve a problem. They also demonstrate that it is possible to use practical laboratory problems to quickly gain information about a student's working knowledge of variety of chemical concepts and his/her ability to apply knowledge in practical situations. This combination of chemical knowledge and practical skills necessary to solve a problem is the essence of what we assume laboratory experiments really teach. Small scale assessment problems can provide us with a set of tools to test this assumption. ◩

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