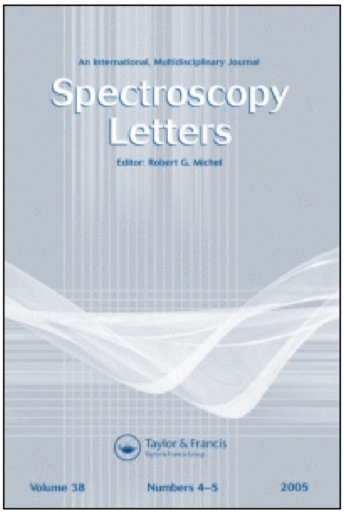

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Flame Atomic Absorption Spectroscopy in the Undergraduate Laboratory: Assessment of a NSF-CCLI Supported Project. What Did the Students Learn and What Did We Learn?

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Flame Atomic Absorption Spectroscopy in the Undergraduate Laboratory: Assessment of a NSF-CCLI Supported Project. What Did the Students Learn and What Did We Learn?

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Abstract: In traditional chemistry curricula, students may encounter a particular instrument only once in their undergraduate career. We have developed an approach that exposes students multiple times to selected instruments, with early encounters focusing on fundamental aspects of the techniques and later encounters building in complexity and sophistication. We recently finished the assessment phase of a curricular project centered on the incorporation of flame atomic absorption spectroscopy throughout the undergraduate chemistry curriculum. Our assessment indicates that this approach is successful and our initial goals were largely achieved.

Keywords: Assessment, flame atomic absorption, Trinity University

INTRODUCTION

The Departments of Chemistry and Geosciences at Trinity University were recipients of a National Science Foundation-Course, Curriculum, and

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Laboratory Improvement (NSF-CCLI) award to acquire a flame atomic absorption spectrometer (FAAS). The project was designed to expose chemistry students to the instrument multiple times throughout their undergraduate careers. An interdisciplinary feature was also included as activities were planned for students enrolled in introductory geosciences courses.

There are many literature examples of FAAS experiments in the undergraduate curriculum. Some of the more recent descriptions that focus on FAAS, as opposed to graphite furnace or inductively coupled plasma methods, describe multielement analysis of "real" samples for instrumental analysis courses[1] or sophomore-level quantitative experiments on synthetic samples[2] and cereals.[3] Still another describes the comparison of several techniques for the analysis of wisdom teeth.[4] These examples are typical of most literature experiment descriptions that are designed as stand-alone labs, usually targeting the sophomore, junior, or senior undergraduate audience.

The main premise of this proposal was that by providing chemistry students with multiple opportunities to use an instrument, they would develop a more sophisticated appreciation of the techniques and a more complete understanding than they would if they encountered the instrument only once in the chemistry course sequence. Experiments in lower division courses would stress the most important features of the technique but would also include other general concepts. Students who left the program after the introductory course would take with them the most important aspects of the technique. Students who continued would take upper division labs that build upon the previous experience. They would then be able to explore more complex topics. Expectations for students' input to and responsibility for experimental design would be higher in the upper division courses.

There were a number of secondary goals and foci to the original proposal. These included the desire to impact a large number of non–science major students. We hoped to increase interdisciplinary ties between the Departments of Chemistry and Geosciences. Because we wanted to use samples and analytes that would be of interest to students, samples that either the students collected themselves or had a biochemical link were typically chosen.

Concepts in the lower division courses (CHEM 1118: Introduction to Analytical Methods; and GEOS 1307: Exploring Earth) included multielement analysis, dilutions, quantification, comparisons of different methods of analysis, and interpretation of product labels. In the junior year course (CHEM 3334: Analytical Chemistry), concepts were expanded to include statistical analysis of data, sampling statistics and considerations, comparisons of different quantification methods, and cross-disciplinary applications. In the original proposal, the senior level instrumental analysis was included and would have exposed students to concepts of detection limits, atomic emission techniques, and experimental design. However, major curricular changes in the chemistry curriculum eliminated this course just prior to the beginning of this project.

The assessment plan involved tracking the impact of these repeated instrument encounters on the understanding chemistry students demonstrated about atomic absorption spectroscopy. Students were asked to fill out a brief, open ended questionnaire in each course using the atomic absorption instrument. After students completed the course sequence, their responses for all courses were sorted and compared. The answers for any particular student could then be evaluated in reference to their progression through the curriculum. The questionnaires were developed with the help of two outside experts. These experts participated in the evaluation of the student responses. The original plan called for chemistry majors to complete three questionnaires, but with the curricular change, only two questionnaires per student were collected. Nevertheless, some interesting observations can be drawn from the student responses.

Previous presentations[5,6] related to this project have focused on overviews, goals, and the philosophy of the approach. Another description is available on a Web site.[7] A project outline, experimental overview, and student report are available at the site. A more recent presentation[8] outlined the assessment results as well as those of another similar project. This report details the assessment results for the chemistry portion of the project.

MATERIALS AND METHODS

To accomplish our goals, we needed an instrument that could provide fast multielement analysis, user-friendly software, and the ability to handle many users and samples in a short period of time. Although a number of different instruments are available from different manufacturers, we chose a Varian SpectrAA 220 FS (Mulgrave, Victoria, Australia) with a Sample Introduction Pump System (SIPS). The fast sequential analysis feature facilitates analysis of several elements in any one sample. The autosampler feature means large numbers of students can use the instrument in one afternoon, and calibration curves can be automatically produced through instrument dilutions of one standard solution.

The Varian SpectrAA 220FS is controlled by Varian 4.10 PRO software. The manufacturer's methods book was used to determine the operational conditions for all elements.[9] In experiments where the concentrations of multiple elements were determined, a single calibration solution was prepared. The SIPS autosampler dilutes the standard to produce the calibration data. For all experiments, a five-point calibration plot and an air-acetylene fuel mixture were used. Typical concentrations of each element in standard solutions and instrument settings are given in Table 1.

Flame atomic absorption instruments can present multiple safety issues including explosive mixtures of fuels and accelerants, high temperatures, and UV radiation. Care should be taken when operating the instrument, and

Table 1. Instrument settings and standard concentrations for all analytes

	Cu	Fe	K	Mn	Zn
Wavelength (nm)	324.8	248.3	766.5	279.5	213.9
Slit width (nm)	0.5	0.2	1.0	0.2	1.0
Lamp current (mA)	10.0	5.0	5.0	10.0	5.0
Air flow (L/min)	13.5	13.5	13.53	13.5	13.5
Acetylene flow (L/min)	2.00	2.00	2.11	2.00	2.00
Burner height (mm)	13.5	13.5	13.5	13.5	13.5
Concentration (ppm)	5 or 10	20	6	5	2

instructors should be well versed in safety procedures and should abide by the manufacturer's operating procedures.

Compared with many other instrumental techniques, atomic absorption is easy to use and understand, at least at an introductory level. This means the technique can be used as a vehicle to introduce other topics. In the first-semester chemistry lab, students performed two experiments with the atomic absorption instrument. In the first lab, they determined the concentration of K, Cu, Mn, Fe, and Zn in vitamin tablets. Students dissolve a vitamin tablet in 25 mL of 6 M HCl with gentle heating. Solutions are then filtered and diluted to 100 mL with deionized water. This lab is based on well-known literature examples.[10,11] An overview of Beer's law and the instrumentation was provided. The lab stressed quantitative transfer, the use of calibration curves, and dilution methods. Students compared their results for the iron determination with those in a subsequent experiment involving the reduction of Fe(III) to Fe(II) with hydroquinone, followed by the complexation of Fe(II) with *o*-phenanthroline and quantification by UV-Vis spectrophotometry.

Later in the semester, students quantitated the amount of Fe(III) released by ferritin upon treatment with acid. Students mix 1.00 mL of a 0.50 mg/mL solution of ferritin with 2.00 mL of 2 M H_2SO_4. After 30 min the solution is centrifuged, decanted, and the supernatant is diluted to 25 mL and the amount of iron measured. This lab was adapted from a literature report.[12] Beer's law and the instrumentation features are reviewed in the lab handout. These results were again compared with those obtained in a subsequent UV-Vis experiment that monitors the kinetic release of iron.

Junior chemistry majors worked with atomic absorption in analytical chemistry (CHEM 3332). They collected soil samples from various sites on the Trinity campus and performed a metal extraction using EDTA[13] as well as an acid digestion with HCl.[14] Students then analyzed the samples for Cu, Mn, and Zn. They compared a calibration curve with the method of standard additions for the determination of Cu. They also determined percent recovery and compared their results with those from previous years. Standard reference materials are not used but could be easily added.

A questionnaire was developed with the advice of two outside experts. Approval to collect the assessment data was granted each year from the Trinity Institutional Review Board as noted on the form. Students were told that responses would not be graded nor would they be reviewed until the students had graduated. Completed questionnaires were collected from students in the first-semester CHEM 1118 and the junior-level CHEM 3332. Seven sections of CHEM 1118 were offered every fall semester in 2001, 2002, and 2003 with total enrollments of 128, 161, and 171 students, respectively. Some of these students subsequently enrolled in CHEM 3332 in fall 2003 and spring 2004 and 2005. Enrollments in these classes totaled 18, 19, and 16 students respectively. A total of 7, 10, and 14 students completed the questionnaire in both classes and only these 31 forms were evaluated. Answers for individual students were compiled on a single form and sent to the two outside reviewers. These materials were also evaluated within the chemistry department. Some students did not complete the forms for both classes, and some students did not take CHEM 1118 due to transfer or advanced placement credit. Responses from these students were not evaluated.

RESULTS

The questionnaire was designed by considering what aspects of atomic absorption are the most important that students should understand. This list includes the concepts that (1) atomic absorption measures the absorption of light by atoms, (2) the absorbed light occurs at a characteristic wavelength, and (3) the response follows Beer's law. The expectations for upper division students were higher. Concepts related to detection limits, instrument design, atomic emission, and sample preparation should be addressed and understood by upper division students. However, some of these concepts were not addressed when the instrumental analysis course was dropped from the chemistry curriculum. The questionnaire (Fig. 1) was designed to evaluate the depth and breadth of the students' understanding along these lines, without being too lengthy. Student responses are summarized in Table 2.

Table 2 attempts to classify answers in one of the following categories: good, attempted to answer, blank or "do not know," or vague or wrong. Some example answers are given as are the percent correct answers as tallied by one of the outside reviewers.

Not all of the students' responses on the questionnaires are easy to interpret, and the reviewers had slightly different interpretations to some responses. Answers to question no. 2 (what types of analytes can be quantitated) were typically along the lines of "ions" or "metals." Atomic absorption is, of course, used to measure the absorption of light by atoms. But the labs in CHEM 1118 are geared to determining the concentration of Fe(II) and Fe(III) in solution. So, an answer of "ions" or "cations" can be interpreted as an appropriate response for these students, where it might be interpreted as an

This questionnaire is being given to all students using the Varian SpectrAA 220 FS instrument in a laboratory course. Your answers on this form will be used to assess the effectiveness of incorporating this instrument throughout the chemistry curriculum. This is a required procedure as described in our grant proposal to the National Science Foundation. The NSF provided funds for 50% of the cost of this instrument.

Your answers will not be used to grade your performance in the laboratory course, but rather to grade the NSF project. Your instructor may require that you complete the form by some means but will not grade individual questions. Please answer each question to the best of your ability. If you do not know the answer to a question, simply state that you don't know. We are not interested in grading you but finding out and recording what you know about this instrument and technique. Use as much space as is necessary (use the back of this form or attach extra sheets) to provide a complete answer or the necessary explanations.

This study has been approved by the Trinity University Institutional Review Board. Your answers will be kept confidential. Your participation involves no risks or benefits to you.

Name:____________________ Class:______________ Date and Year:______________

1. Please describe your previous (if any) experience with atomic absorption (AA) and its related techniques.

2. What types of analytes can be quantified by AA or related methods?

3. Can AA be used to identify analytes? Explain.

4. Describe the relationship between concentration of analyte and absorbance in AA.

5. What are the approximate detection limits you might expect from flame AA?

6. In AA the samples and standards are often in an acidic solution. Why?

Figure 1. Student questionnaire.

incorrect answer for those in CHEM 3332. It is unlikely the students were referring to organic ions. Better wording of the question might have prevented this confusion. A few students responded very practically with statements to the effect "you can measure anything for which there is a lamp"! Ten (32%) CHEM 1118 students responded obviously incorrectly with answers such as "molecules" or with answers that were nonsensical such as "low concentrations" indicating a confusion of quantitative and qualitative concepts. Three (9.6%) CHEM 1118 students indicated they didn't know. All CHEM 3332 students at least attempted to answer the question. One reviewer counted five correct answers (16%) from CHEM 1118 students and 17 (55%) for CHEM 3332 students.

Table 2. Summarized results: Number of CHEM 1118/CHEM 3334 responses per question by category (total responses: 31/31)

	Types of analytes	Identification	Conc./ absorbance	Detection limits	Why acid solutions
Good answer	6/21	15/26	12/14	0/11	0/6
Examples	Elements/ metals, class analytes	No or wave-length specific	Beer's law, graph or equation	Number range or analyte specific	Digestion or base reac-tion/dissol-ution or salts or precipitation
Attempted answer	12/10	0/0	15/17	0/4	4/0
Examples	Ions, metal ions		Pro-portional, or linked	ppm	
Blank "do not know"	3/0	4/0	4/0	21/11	12/14
Vague or wrong	10/0 All/any that absorb	12/6	0/0	10/5	15/11
Reviewer: total correct	5/17	12/22	14/14	0/12	0/5

The responses to question no. 3 (can AA identify analytes) are more interesting. Most CHEM 1118 students and many CHEM 3332 students responded along the lines of "Yes, you can identify analytes by atomic absorption." Their explanations, however, made clear that they understood the strong link between the element of interest, the need to use a particular lamp, and the absorption of a specific wavelength. Because analytes provide unique signals, they concluded that identification is possible. Students generally did not recognize the practical limitations of using atomic absorption to identify species. Students in CHEM 1118 also confused the terms *concentration* and *identification* or did not distinguish between them sufficiently. CHEM 3332 students were more likely to answer "no" to this question (although many did answer "yes"), and they were more likely to recognize that it is important to know what you are looking for in order to choose the correct lamp. Four (13%) CHEM 1118 students did not answer the question. All CHEM 3332 students attempted an answer. One reviewer counted 12 (39%) correct answers for the CHEM 1118 students and 22 (71%) for the CHEM 3332 students.

Question no. 4 determines if the students understood Beer's law. Four (13%) CHEM 1118 students stated they did not know. All CHEM 3332 students attempted to answer the question and indicated at a minimum that there was some sort of proportional relationship, although they did not always state that this was a linear relationship. Twelve (39%) CHEM 1118 students and 14 (45%) CHEM 3332 students either named Beer's law, drew a graph representing the relationship, or provided an equation. One of the outside reviewers counted 14 (45%) correct responses for both CHEM 1118 and 3332 students.

Distinct differences in the responses were observed for the two student groups based on answers to question no. 5 (what are the detection limits). Twenty-one (68%) CHEM 1118 students attempted no answer or stated "I don't know," as did 11 (35%) CHEM 3332 students. All CHEM 1118 students who attempted an answer (10, 32%) did so incorrectly. Five (16%) CHEM 3332 students answered incorrectly or vaguely, for instance with "the dilution factor of the sample" or "concentration, pH." The other 14 (45%) CHEM 3332 students gave a reasonable answer or with an appropriate number range, indicating that at the very least, they understood the question. One outside reviewer counted no correct answers for CHEM 1118 students and 12 (39%) correct answers for CHEM 3332 students. As mentioned, the course that would have addressed the issue of detection limits was eliminated from the curriculum. Thus, students' knowledge of this topic comes not directly from the lab but from the related lecture and textbook material.

Distinct differences were also evident for the two student groups on question no. 6 (why are acids used) as no CHEM 1118 students could give answers that could be called good, compared with six (19%) in the CHEM 3332 group. Four of the CHEM 1118 students indicated dissolution or another answer that could be called partially correct. Many students in both groups were either unable to provide any answer or answered incorrectly or vaguely. Providing a good answer was clearly a stretch for students as the issue was not directly addressed in either class, and students needed to rely on knowledge largely from other classes. One outside reviewer counted no correct answers for CHEM 1118 students and five (17%) for CHEM 3332 students.

The outside reviewer who tallied answers to individual questions commented that the experiments were "sound," used real samples, and attempted to extract relevant information about the different systems. However, he concluded there was a general lack of awareness among the students on the basics of atomic absorption. The other outside reviewer calculated an overall ratio of correct to incorrect responses and found that the ratio increased for the 3332 course (1.64) as compared to the 1118 course (0.32). His overall conclusion was that the concepts were well taught, and the questions were carefully selected and covered the critical concepts.

DISCUSSION

Although staffing and curricular changes in both departments affected our ability to meet some of our initial goals (e.g., more interdisciplinary interactions between the chemistry and geosciences departments), several of our initial goals were met. Because the instrument was used in the first-semester chemistry and introductory geosciences courses, we affected a large number of students, many of whom eventually majored in non–science departments. We were also able to make use of samples that were of interest to the students. Some samples were collected directly by the students (e.g., water samples collected by GEOS 1307 students, soil samples across the Trinity campus by CHEM 3332 students), others were of broad societal interest (e.g., vitamin tablets, bottled water), and some were of interdisciplinary interest (e.g., ferritin analyzed by CHEM 1118 students).

It was evident from the assessment forms that students understood the most important aspects of atomic absorption after the first course. They understood Beer's law and the connections among analyte, lamp, and wavelength. These are arguably the most important concepts. Students also tended to understand the types of analytes that can be successfully investigated. We did not expect nor intend that introductory students would gain a more comprehensive understanding of atomic absorption. Other concepts such as detection limits and the use of acidic solutions were only or were at least better understood by the upper division students.

This study and the assessment approach could have been improved upon. Consultation with assessment experts may have improved the assessment aspect of the project. It also would have been useful to pilot test some questions. Rewording some questions would have made the interpretation of the student responses easier. However, this project officially ran from January 2001 through December 2003. The assessment portion of the program continued until the end of the spring 2006 semester. Pilot testing questions would further extend this time frame. Having a control set of responses would also have been useful. However, that would have entailed implementing the assessment form before the project was officially funded. That may not have been practical.

Maintaining a project and its subsequent assessment over this time frame is a daunting task. We routinely offer seven or more sections of our first-semester laboratory each year, and we frequently staff those sections with part-time instructors. Assessment forms were completed by students at a variety of points in the semester after they performed the lab. Standardization of the time at which students filled out the forms might ease the interpretation of their responses, but in practice such standardization may not be easy to implement.

Students were told their responses would not be graded. As a result, some students may not have been motivated to complete the questionnaire with much care. This may have artificially raised the number of "I don't know" responses to some questions. If students had understood their responses would be graded, they might have been more motivated to provide the best answer that they could.

Overall, we believe this project was a success. We affected a wide variety and large number of students. We successfully introduced first-year students to atomic absorption, and they developed an understanding of the most important concepts of the technique. We returned to AA methods in an upper division course where more sophisticated issues were addressed. Responses on the assessment questionnaire identified areas where student understanding is at a lower level than we expected. This is due in part to the lack of an instrumental analysis course, which would have more directly addressed the issue of detection limits, and in part to the confusion in lower division class over ion versus elemental analysis and quantitative versus qualitative information. Nevertheless, some issues such as introducing the term *detection limit* earlier in the curriculum, and spending more time explaining the use of acidic solutions, will be easily addressed in our current curriculum.

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