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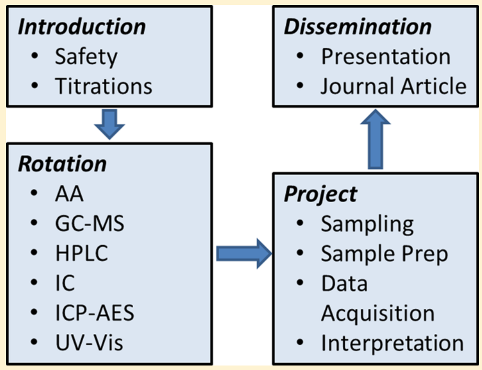
Merging Old and New: An Instrumentation-Based Introductory Analytical Laboratory

Mark B. Jensen*

Department of Chemistry, Concordia College, Moorhead, Minnesota 56562, United States

ABSTRACT: An instrumentation-based laboratory curriculum combining traditional unknown analyses with student-designed projects has been developed for an introductory analytical chemistry course. In the first half of the course, students develop laboratory skills and instrumental proficiency by rotating through six different instruments performing quantitative analyses of unknowns. In the second half of the course, students use these skills to design and perform a quantitative chemical analysis of a real-world sample of their choosing in which students direct each step in the analytical process: sampling, sample preparation, data acquisition, interpretation, reporting, and drawing conclusions. Unique features of the course include open lab periods, an online lab manual with embedded video demonstrations, and the publication of student projects in an online journal. Assessment results show that students report high levels of gain in relation to the desired outcomes of the overall project.



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The changing face of analytical laboratory courses has been the focus of much discussion and debate for more than two decades.[1−8] Courses that once emphasized titrimetric and gravimetric determinations of "unknowns" are being replaced by newer pedagogies such as role playing,[9] problem-based learning,[10−13] community-based projects,[14] and project-based labs.[5,15−17] These new approaches bring an important "real-world" dimension to the course that pushes students toward higher levels of engagement, interest, and ownership in the laboratory experience. Also, as instructors seek to modernize courses by replacing classical wet-lab analysis techniques with modern instrumental methods, the distinction between "quantitative analysis" and "instrumental analysis" becomes harder to discern.[6] While few will disagree that this evolution has brought much needed changes to analytical education, some instructors may now seek to find a balance between these old and new approaches. For instance, how can a modern analytical lab course embrace new pedagogies and focus on instrumentation, yet still retain the traditional emphasis on accuracy and precision with an exposure to a variety of laboratory techniques?

A 2007 strategic planning process in our department resulted in a number of new objectives related to our curriculum. Among these was the incorporation of more "real-world" experiences into our laboratory courses, with the goals of enhancing student learning in the laboratory, giving students a better understanding of the work scientists actually do, and generating more student excitement toward science in general and chemistry in particular. So with funding from an NSF Course, Curriculum, and Laboratory Improvement (CCLI) award, significant changes were made to both the analytical and general chemistry lab courses. The desired outcomes of the project were stated in the proposal as follows:

1. Student learning in chemistry will be stimulated through open-ended, inquiry-based laboratory experiences.
2. Students will better understand the process of science through personal involvement in this process.
3. Students will learn to critically evaluate scientific data and the conclusions drawn from that data.
4. Students will become proficient in the use of a variety of modern chemical instruments.
5. Student excitement about science in general, and chemistry in particular, will grow.

With these outcomes in mind, the analytical and general chemistry laboratories were restructured to incorporate multi-week, student-designed projects. The general chemistry projects are based on the fate of pharmaceuticals in natural water supplies, and the results of that work will be presented in a future publication.[18] Here we will focus on the newly designed introductory analytical course and an approach that seeks to integrate the best of old and new pedagogies by combining traditional unknown determinations with project-based labs and modern instrumentation.

COURSE STRUCTURE

The first analytical course in our curriculum (formerly Chem 232 − *Analytical Chemistry*) is required for all chemistry majors. It is offered each spring semester with typical enrollments of 15−30 students, mostly junior and senior chemistry majors.

The second course (formerly Chem 431 − *Instrumental Methods of Analysis*) is required only for our ACS majors, and is offered each fall with enrollments of 5−10 students. With the previously listed learning outcomes in mind, and understanding that most chemistry majors take only the first course, Chem 232 was redesigned with the goal of "modernizing" the analytical experience for all chemistry majors through an instrument-focused laboratory curriculum, and the incorporation of student-designed lab projects utilizing chemical instrumentation. In doing so, it no longer made sense to refer to the second course as "instrumental," so the course names were changed simply to *Analytical Chemistry I* (now Chem 330) and *Analytical Chemistry II* (still Chem 431). Much has been written about the changing nature of the first analytical ("quant") course and the incorporation of instrumentation over classical techniques.[2−4] In our case, Chem 330 retains an emphasis on statistics and aqueous equilibria, but now contains more complete introductions to atomic and molecular spectroscopy, as well as basic chromatographic methods. Chem 431 then focuses on signal-to-noise considerations, analog circuitry, LabVIEW programming,[19] and advanced instrumental applications in electrochemistry, optical spectroscopy, chromatography, mass spectrometry, and surface analysis.

In redesigning the Chem 330 laboratory to focus on instrumentation and incorporate student projects, several important features were introduced.

Rotation of Experiments

To give each student exposure to a wide range of modern instruments, the first half of the course is structured around a set of six experiments, each utilizing a different instrument: flame AA, ICP-AES, UV−vis absorption,[20] HPLC (reversed-phase),[20] ion chromatography,[21] and GC−MS with solid-phase microextraction.[22] Each of these experiments can be completed in 2−4 h, and students are free to work on whichever experiment(s) they choose in a particular week. The experiments consist of traditional unknown determinations using (in most cases) previously analyzed samples with known analyte concentrations, and students are evaluated on both the accuracy of their data and the correctness of their calculations.

Open Lab Periods

Historically, the Chem 232 lab would meet once a week for 4 h, and students would enroll in one of two sections offered. In moving to the new model for Chem 330, it is important that students have flexibility in their schedule to accommodate instrument availability and the demands of open-ended projects. We have therefore adopted a modified "open" schedule for the Chem 330 lab, similar to that suggested by Wenzel who argues that this format encourages independence and student ownership of projects, and eliminates any notion that science occurs in predefined time blocks.[5] In our case, 1 day of the week (currently Thursday) is defined as "lab day," when the lab is open and staffed by the instructor and/or teaching assistants from 8 am until 6 pm. Students are free to come and go as they please during that time, but they must sign up for instrument time in advance. Attendance is not required in any particular week (after the first week); however, firm deadlines are given for completion of the various course components. Given the variety of student schedules, students are allowed to work alone or with a partner on any particular experiment (and the project described below).

Online Lab Manual with Embedded Video

On any given lab day during the rotation phase of the course, up to six separate experiments can be occurring simultaneously. To facilitate student independence during this phase, an online lab manual was created and videos were embedded for each step in each experiment.[23] The online lab manual offers a number of other advantages as well, including the incorporation of digital photos, hyperlinks to commonly used procedures (e.g., pH meter calibration), the ability to perform instantaneous updates and corrections to any of the experiments as needed, and a tremendous reduction in the amount of paper used in the class.

Online Lab Prelab Quizzes

In many cases, students are using a particular instrument before it has been formally introduced in class. Before being allowed to use an instrument, a student must therefore first read selected pages from the course textbook and lab manual, then pass an online quiz (administered through our Moodle course management system) to ensure a base level of understanding of the instrument and its operating principles.

Student-Designed Project

The second half of the course is dedicated to student-designed projects in which the students are asked to develop and carry out the quantitative determination of one or more chemical species in a real world sample of their choosing, using one of the instruments from the first half of course. The project is summarized simply as "the determination of ______ in ______ using ______" where the students fill in the three blanks with their choice of chemical species, sample/matrix, and instrument, respectively. Students are expected to search the chemical literature (research journals, industry white papers, education journals, EPA methods) for a procedure, and then submit a short "proposal" in which they describe their project, provide the literature source, and produce a list of all chemicals and supplies required. Following approval, they are asked to examine the MSDS of each chemical and report any potential safety hazards. They then follow the literature procedure in performing their analysis, and are expected to report results with calculated uncertainties. The projects put emphasis on each step in the analytical process: sampling, sample preparation, analysis (data acquisition), interpretation, reporting, and drawing conclusions.[24] Students are encouraged to incorporate knowledge and experiences gained in other science courses (e.g., Ecology, Limnology, Biochemistry) into their projects to emphasize the multidisciplinary nature of chemical analysis, and to explore projects related to their intended career path. For example, students who had just taken Limnology determined phosphate, nitrate, and sulfate in river water with ion chromatography; two predentistry students chose to determine copper, zinc, and lead in human teeth using ICP-AES; a student now in a plant pathology graduate program used ICP-AES to measure potassium, calcium, magnesium, and phosphorus in two types of grasses; and several prepharmacy students have used various techniques to measure active ingredients in over-the-counter pharmaceuticals. To encourage students to take on more challenging projects, 20% of the project grade is based on the "ambitiousness" of the project.

Dissemination in an Online Journal

To draw particular emphasis to the reporting (dissemination) step of the analytical process, an online journal has been created (*Concordia College Journal of Analytical Chemistry*).[25] Each project is written by the student(s) in the style of a formal journal article, revised multiple times, then edited and formatted before online publication. The work of each class is published in a new volume, and these articles can be referenced and built upon in subsequent years.

■ LABORATORY SCHEDULE

The redesigned Chem 330 laboratory schedule is outlined in Figure 1. This model is similar to that reported by Hope and Johnson in which a series of traditional experiments is followed by projects based on the measurement of pollutants in urban air.[17] As mentioned above, week 1 is the only week in which students are required to attend lab at a particular time. During this time, the format of the lab is discussed, safety issues and procedures are reviewed, and students are introduced to the analytical laboratory by performing the only experiment of the semester involving titrations: a determination of KHP in a salt mixture through both volumetric and gravimetric titrations,[26] followed by a statistical comparison of the two methods using pooled class data.

Week 1: Introduction
- Course policies and procedures, safety
- First experiment: Statistical Comparison of Titration Techniques

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Weeks 2-7: Instrument Rotation
- Copper in Brass by Flame Atomic Absorption Spectroscopy
- Manganese and Nickel in Steel by Inductively-Coupled Plasma Atomic Emission Spectroscopy
- Benzoic Acid and Caffeine in Soft Drinks by Multicomponent UV Analysis
- Benzoic Acid and Caffeine in Soft Drinks by High Performance Liquid Chromatography
- Sulfur in Coal by Ion Chromatography
- Caffeine in Beverages using Solid-Phase Microextraction with Gas Chromatography-Mass Spectrometry

↓

Weeks 8-14: Student-Designed Projects
- Determination of ________ in ________ using ________
- Emphasize each step in the analytical process
- Use published procedure for analysis

↓

Week 15: Dissemination of Results
- Oral presentations in Analytical Symposium
- Publication of papers in the *Concordia College Journal of Analytical Chemistry* (online)

Figure 1. Laboratory schedule for Chem 330.

Weeks 2 through 7 are reserved for the rotation of the six instrument-based unknown determinations listed in Figure 1. Each experiment introduces students to an instrument they have most likely never used before (AA, ICP-AES, IC, HPLC), or requires them to use an instrument with which they have prior experience in a new way (GC−MS with SPME, UV−vis with multicomponent analysis). The variety of sample types used and the focus on accuracy and precision inherent to this more traditional approach work to develop students' lab skills, give them experience with a number of techniques, and build their confidence in the lab—all consistent with the desired outcomes associated with the introductory analytical lab for decades. These experiments also give the students the necessary background for selecting and carrying out their projects.

The student-designed projects in weeks 8−14 were described above. As opposed to the rotation of experiments that focuses almost exclusively on the sample preparation and analysis portions of the analytical process, these projects require students to collect the sample(s), interpret the data, draw conclusions, and report those conclusions. The intent, therefore, is for students to take greater ownership of their work, experience the ambiguities typically involved in data interpretation, and come away with a better understanding of the nature of real scientific work.

Project dissemination is the focus of the last week of the semester. Two days of regular class time are reserved for our "Analytical Symposium" in which the students share their projects with the rest of the class in short oral presentations, and the students submit the final drafts of their papers to be published in the online journal.

■ ASSESSMENT

The redesigned Chem 330 curriculum was first implemented in the spring of 2010 (16 students), with the same format being used again in 2011 (27 students), 2012 (18 students), and 2014 (29 students). (The author took a year-long sabbatical in 2013.) Assessment data related to this work was collected during the first three years using the Student Assessment of Learning Gains (SALG[27]) instrument administered both at the beginning and at the end of the course, and through a departmental survey of graduating seniors.

Results from the postcourse SALG survey are shown in Table 1. Each of the five learning outcomes is listed, along with questions related to that objective. Students were asked to rate the level of learning assistance each of the course components provided (for Outcome 1), or the level of perceived gain made in their own understanding of areas related to the course (for Outcomes 2−5). Students reported the highest gains (4.59) and the lowest standard deviation (0.73) in their understanding of the analytical process. As for the learning components related to Outcome 1, each averaged above 4 (much help), except the online prelab quizzes (3.41), with the online lab manual receiving the highest score (4.56). While students did report that the online videos provided much help (4.22), conversations with students showed that they felt the videos would be more effective if sound were included. So more recent videos have included sound with explanations of the performed procedure, and sound will be added to the older videos at some point in the future. When asked to comment on the resources available to them in the course, several students pointed to the effectiveness of the online manual and the embedded videos:

- "*The lab manual and especially the video demonstrations were invaluable to learning how to go about lab.*" (Spring 2012)
- "*The online lab manual was by far the most useful resource available to me as I completed this course.*" (Spring 2012)
- "*The online lab manual with videos was extremely useful in lab, and made it possible to run the labs without constant attention from a professor.*" (Spring 2011)
- "*The online lab manual was also helpful, especially the little videos.*" (Spring 2011)

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Table 1. Results of Chem 330 End-of-Semester SALG Survey over a Three-Year Period

Outcomes, SALG Survey Questions, and Statements for Student Response[a]	Weighted Average Scores[b]	Pooled SD Values[b]
OUTCOME 1: Student learning in chemistry will be stimulated through open-ended, inquiry-based laboratory experiences.		
HOW MUCH did each of the following aspects of the class HELP YOUR LEARNING?		
The online lab manual	4.56	0.80
The lab experiments	4.43	0.83
The student-designed lab project	4.34	0.87
The rotation of experiments	4.27	0.85
Video demonstrations in the online manual	4.22	0.98
Online prelab quizzes	3.41	1.05
OUTCOME 2 - Students will better understand the process of science through personal involvement in this process.		
As a result of your work in this class, what GAINS DID YOU MAKE in your UNDERSTANDING of each of the following?		
The analytical process	4.59	0.73
OUTCOME 3 - Students will learn to critically evaluate scientific data and the conclusions drawn from that data.		
As a result of your work in this class, what GAINS DID YOU MAKE in the following SKILLS?		
Critically evaluating scientific data and drawing conclusions from that data	4.04	0.83
Recognizing a sound scientific argument and the appropriate use of evidence	3.72	0.90
Critically reading articles about concepts discussed in class	3.58	0.97
OUTCOME 4 - Students will become proficient in the use of a variety of modern chemical instruments.		
As a result of your work in this class, what GAINS DID YOU MAKE in your UNDERSTANDING of each of the following?		
High-performance liquid chromatography (HPLC)	4.40	0.87
Atomic absorption spectrometry (AA)	4.23	0.88
Ion chromatography (IC)	4.20	0.83
Inductively coupled plasma atom emission spectrometry (ICP-AES)	4.13	0.91
Gas chromatography−mass spectrometry (GC-MS)	4.09	0.89
UV−vis absorption spectrometry	3.69	0.98
OUTCOME 5 - Student excitement about science in general, and chemistry in particular, will grow.		
As a result of your work in this class, what GAINS DID YOU MAKE in the following?		
Enthusiasm for chemistry	3.82	1.02
Enthusiasm for analytical chemistry	3.58	1.08
Interest in taking additional courses in analytical chemistry	3.13	1.13

[a]This is the scale for Outcome 1:1 = No Help; 2 = A Little Help; 3 = Moderate Help; 4 = Much Help; 5 = Great Help. For Outcomes 2−5, this is the scale: 1 = No Gains; 2 = A Little Gain; 3 = Moderate Gain; 4 = Good Gain; 5 = Great Gain. [b]*N* = 57.

- *"The videos on the lab manual were very useful."* (Spring 2010)

The lower score for the prelab quizzes drew attention to a problem with how the quizzes were administered. Students must receive 100% on a quiz to receive a passing grade, however they may resubmit their answers an unlimited number of times. The lower student response was found to at least partially result from an error that caused the correct answer to appear whenever a wrong answer was submitted. When this error was corrected, the scores for this question rose from 3.0 (2010) to 3.6 (2011). Still, the score is relatively low and this method of preparing students for performing a particular analysis will continue to be evaluated.

Two final points about Table 1 are noted: the lower score for "*interest in taking additional courses in analytical chemistry*" (3.13) stems in part from a large number of second-semester seniors in the class who do not have the option of taking *Analytical II*; and while students reported strong gains in their understanding of each instrument, it is consistent that they reported their lowest gains for GC−MS and UV−vis since these were instruments they had used in previous courses.

As described previously, the open-lab periods and the rotation of experiments are expected to increase students' sense of independence and confidence in the laboratory. While no question in the SALG survey directly addressed these aspects of the course, several written comments from students show its effectiveness in promoting independence and confidence:

- *"This lab course has allowed me to feel more comfortable working independently in the lab."* (Spring 2012)
- *"Probably most importantly, I have become a more confident chemist through the more individual lab setting."* (Spring 2012)
- *"The greater independence and rotation in lab helped me to grow and become more confident as a chemist."* (Spring 2012)
- *"Lab flexibility was awesome. I could get in the lab when I could, do what I needed to do and leave. I didn't feel the time pressure of past labs and that was really nice."* (Spring 2011)
- *"The open lab was a really good set-up. I also liked that we could choose which of the experiments we wanted to do that week. Being able to choose which procedure and when to go into lab was one of the reasons I enjoyed this lab so much."* (Spring 2011)
- *"The independence forced me to understand better."* (Spring 2011)
- *"The experiment rotation helped me become more comfortable with working in a chemistry lab setting thanks to the feeling of independence."* (Spring 2011)
- *"The freedom in lab led to a more responsible understanding of the procedures/better learning."* (Spring 2010)
- *"The laboratory activities were awesome. They really took the problem solving and basic lab skills to another level. I am much more confident in the lab now than I was before."* (Spring 2010)

In other selected comments, students point to ways in which their critical thinking skills were stimulated by the laboratory experience:

- *"The student-designed project was most helpful. Through the process, we learned to critically read and understand the article so we could later replicate the experiment. Also, when it didn't work out ideally, it was then our responsibility to try and determine what went wrong upon analysis of our data. Was it systematic or random error? To troubleshoot this, we not only had to fully understand the method of our article, but also the instrument method and conditions. It was a great learning experience."* (Spring 2012)
- *"The instrument lab has always scared me until I took this class. The sheer amount of money sitting in that room was really intimidating but now that I have a basic understanding of how each of the instruments works, I am much more comfortable in that room. I also now find myself*

Table 2. Results from Survey of Graduating Seniors Administered in 2009−2012

Statement Number (Related Outcome)	Statements for Student Responses	Mean Scores (SD)[a]			
		2009, $N = 0/17^b$	2010, $N = /17^b$	2011, $N = 11/12^b$	2012, $N = 17/18^b$
1 (OC 1)	The laboratory experiences in my chemistry courses have greatly enhanced my knowledge and understanding of chemistry.	4.0 (0.6)	4.3 (0.6)	4.3 (0.5)	4.5 (0.5)
2 (OC 1)	The laboratory experiences in my chemistry courses have greatly increased my own confidence in the laboratory.	4.3 (0.7)	4.4 (0.5)	4.5 (0.7)	4.5 (0.5)
3 (OC 2)	The laboratory experiences in my chemistry courses have greatly enhanced my understanding of the scientific method.	3.9 (0.7)	4.4 (0.5)	4.7 (0.5)	4.7 (0.5)
4 (OC 2)	The laboratory experiences in my chemistry courses have greatly enhanced my understanding of the type of work scientists actually do.	4.0 (0.6)	4.1 (0.7)	4.2 (0.6)	4.3 (0.7)
5 (OC 3)	The laboratory experiences in my chemistry courses have greatly enhanced my ability to critically evaluate scientific data and draw conclusions from that data.	4.1 (0.8)	4.2 (0.4)	4.6 (0.7)	4.6 (0.5)
6 (OC 4)	My chemistry courses have given me practical experience with a wide variety of modern instrumentation.	4.2 (0.6)	4.5 (0.5)	4.8 (0.4)	4.8 (0.4)
7 (OC 5)	The laboratory experiences in my chemistry courses have greatly enhanced my interest in chemistry.	3.8 (0.9)	4.5 (0.6)	4.4 (0.5)	4.6 (0.5)

[a]The scale was: 5 = strongly agree; 4 = agree; 3 = neutral; 2 = disagree; 1 = strongly disagree. [b]Number of graduating seniors having taken newly formatted course (Chem 330) out of total number of graduating senior chemistry majors. The remainder took previous course (Chem 232) or obtained equivalent credit at another institution.

thinking critically about which instrument would provide the best results when trying to determine a certain compound." (Spring 2011)

- "*I have gained many laboratory skills. Before when I was in the lab I didn't really know what was going on and was just blindly following the procedure. From this class I have been able to look at a procedure and experiment and realize what it is we're doing and why we are doing it.*" (Spring 2011)
- "*I have learned much about how to operate and think about instruments and the analytical process. Research has become simpler and less convoluted in my mind, and I feel more confident in my own skills as a chemist.*" (Spring 2011)
- "*I think my ability to critically look at scientific data and evaluate it appropriately improved with this class.*" (Spring 2010)

In 2009 a series of questions related to the objectives of this project was added to our survey of graduating seniors. These questions are given in Table 2, along with the outcome to which each is related, and the results for each year. Each question is a statement, and students were asked the degree to which they agree or disagree. In 2009 no graduating seniors (out of 17) had taken the introductory analytical class in its new format (Chem 330). This number rose to 29% (5/17) in 2010, followed by 92% (11/12) and 94% (17/18) in 2011 and 2012, respectively. It can be seen from Table 2 that in moving from 2009 to 2012, the level of student agreement with each statement trended upward, with most statements showing stronger levels of agreement in each successive year. The highest gains from 2009 to 2012 were seen in questions 7 and 3, which increased from 3.8 to 4.6 and 3.9 to 4.7, respectively. These questions related to the students' reported increased interest in chemistry (question 7) and their understanding of the scientific method (question 3). The highest overall agreement comes from question 6 in which students agreed that their chemistry courses had given them practical experiences with a broad range of instruments. On the other hand, the lowest level of agreement was found in question 4 related to the students' understanding of the type of work scientists do.

The graduating seniors were also asked to give what they considered to be the most beneficial laboratory experience they'd had at Concordia. Those who responded with one or more of the analytical classes (232, 330, and/or 431) jumped from 25% and 29% in 2009 and 2010, respectively, to 80% and 69% in 2011 and 2012.

Finally, adjusting the lecture content of the course to place a stronger emphasis on instrumentation appears to have had little effect on students' understanding of traditional first-semester analytical chemistry material, at least through a comparison of their performance on the ACS Analytical Chemistry exam taken as the final exam for the course. The average score (out of 50 possible) on the 2001 ACS Analytical Exam in the two years prior to implementation was 33.2 ± 6.5 ($n = 36$), whereas in the two years following implementation it was statistically unchanged at 33.8 ± 5.5 ($n = 43$).

AUTHOR INFORMATION

Corresponding Author

*E-mail: jensen@cord.edu.

Notes

The authors declare no competing financial interest.

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