

# **A Practical Guide to Implementing a Theme-based Modular Approach to Instrumental Analysis**

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## **Using this guide**

In 2007, Butler University made a decision to significantly modify the approach used in delivering the analytical chemistry curriculum. This change was made with the belief that students would gain the technical skills needed to best understand analytical chemistry while also developing professional and social skills. To that end, the lecture/lab combination common to most advanced analytical chemistry courses was redesigned so that lecture was now a separate course from the lab, and the lab was centered on different contextually themed modules.

This document provides guidance regarding the practical implementation of the theme-based modular approach to analytical chemistry based on personal experience. Different faculty have different approaches to implementation, but this instructor guide describes what has been most successful for the corresponding author. Also included are general details with regard to how the theme-based approach is incorporated into the Butler curriculum.

Where applicable, specific course materials and example work from the modules I have taught are available online to provide a deeper understanding of the type of experience that both faculty and students will have through the implementation of the theme-based approach. In addition, general department-wide documents related to grading and peer review have also been included, but are not necessarily specific to the theme-based modular approach. Certainly, not every possible question people may have about our methods are addressed herein and users of this guide are invited to contact the primary author if further clarification is needed.

For reference, a more complete and formal description of the modular approach is provided in *Analytical and Bioanalytical Chemistry*.

[Theme-based modular approach for delivering the undergraduate analytical chemistry curriculum.](#) O. Akinbo and M. Samide, *Analytical and Bioanalytical Chemistry*, 392 (2008) 1. DOI: 10.1007/s00216-008-2240-4

## **A theme-based modular approach**

The theme-based modular approach is focused on changing how students experience instrumental analysis. A more traditional approach might utilize a series of well crafted, faculty-tested laboratory experiments to involve individual students in the use of instrumental techniques. While this method provides breadth of coverage, it is often lacking in important skills such as experiment design, troubleshooting, team work, and communication.

Furthermore, students often lack ownership of the work and simply try to complete a procedure with the correct answer so that they can move to the next experiment. The theme-based approach removes the faculty-tested lab experiments and replaces it with a unifying theme under which students can explore and develop their own idea to test. This increases interest and ownership in the lab while providing students with specific tasks to accomplish using specific instrumentation. Students are also working in groups on one unified project (or a small series of projects).

A number of resources champion curriculum reform. The idea that knowledge can be directly transferred from the faculty to the student is proving false and students should instead be active participants in the learning process.<sup>1</sup> As a result of this call to curriculum reform, the NSF sponsored a New Traditions Project that resulted in a report which helped to identify mechanisms for implementing active learning. These elements include interactive lecture, team problem solving, open-ended laboratory experiments, a thematic approach, and formation of learning communities.<sup>2</sup> In addition, NSF-sponsored workshops on curricular developments in the analytical sciences produced a report that recommended context-based curriculum, small group learning, investigative laboratories, case studies, and development of oral and written communication skills.<sup>3</sup> The theme-based modular approach to the instrumental analysis laboratory was developed in response to these documents, making an attempt to put into practice those ideas that the chemical education research demonstrated were necessary to improve the learning environment for the students.

<sup>1</sup>J. N. Spencer, "New Directions in Teaching Chemistry: A Philosophical and Pedagogical Basis" *J. Chem. Educ.* 76 (1999) 566.

<sup>2</sup>C. R. Landis, M. A. Scharberg, S. Branz, J. N. Spencer, R. W. Ricci, S. A. Zumdahl, D. Shaw, "The New Traditions Consortium: Shifting from a Faculty-Centered Paradigm to a Student-Centered Paradigm," *J. Chem. Educ.*, 75 (1998) 741.

<sup>3</sup>T. Kuwana, "Curricular Developments in the Analytical Sciences: A Report from the Workshops (October 28-30, 1996, and March 13-15, 1997)," National Science Foundation Directorate of Education and Human Resources/Division of Undergraduate Education and Division of Chemistry, Arlington, VA, 1997, p 12.

### **Student Learning Objectives**

Taken from the original description of the theme-based approach, published in *Analytical and Bioanalytical Chemistry*, the following set of student learning outcomes applies to any theme selected for implementation of this model.

Once students have completed one or two differently-themed modules, students should have the:

1. Ability to identify scientific problems that are relevant to societal concerns.
2. Ability to conduct an appropriate literature search to gather project ideas, justify project significance and synthesize a procedure.
3. Ability to work within a team environment and provide peer review.
4. Ability to design, justify, and implement a solution for solving a scientific problem.
5. Ability to present scientific information in written and oral formats.
6. Ability to implement analytical methods under regulatory conditions.
7. Ability to apply chemical analysis to relevant societal issues through laboratory study and service learning

### **Logistics of using theme-based modules at Butler University**

At Butler University, the theme-based modular laboratory course has a course number of CH424. CH424 is the advanced analytical laboratory course and fits into the chemistry curriculum like other 400-level lab courses in physical chemistry, biochemistry, and inorganic chemistry. The advanced analytical lecture course is separate from the lab course, and students are not required to complete advanced analytical lecture prior to enrolling in the advanced analytical lab course. Rather, students may enroll in the CH424 course after completion of the first-semester introductory analytical chemistry course. Students receive 2 hours of credit for the 4 hour lab. Students are allowed by the University registrar to repeat CH424 for credit so long as each retake has a different theme.

Class size for all advanced labs at Butler is capped at 8 (for advanced lecture courses, the enrollment cap is higher), with an option of expanding to 10 if enrollments are high. We do this to foster an intimate environment where students can significantly contribute to the goals and outcomes of a project.

The chemistry department typically offers 1 to 3 sections of CH424 per year, depending on other teaching loads, sabbatical plans, and contributions to the honors program or the University core curriculum. Each section counts for 2 teaching hours toward a typical 9 hour semester load for tenured or tenure-track faculty. Therefore, the CH424 lab is often paired with one or two additional courses to make up a full semester teaching load.

The CH424 course is not an absolute requirement for the major, but is listed as an acceptable course for students to use to complete the major. It is also an option for students who are pursuing ACS certification, with the course providing 56 of the 400 necessary lab hours and counts as an analytical lab experience for ACS certification. If students are interested in advanced study in chemistry, it is recommended and encouraged that students take 2 different semesters of CH424, each section having a different theme. In this way, students gain broad exposure to multiple instrumental techniques while also gaining an in-depth understanding of each technique and how it is applied to a novel problem.

Because the course is not required for all students to complete, most students will complete only one section of the course. In a 5 year period, roughly 50% of our chemistry majors will complete one CH424. In that same time period, roughly 10% will complete two CH424.

### **Selection of the instructor for the theme-based analytical laboratory course**

While the theme-based analytical laboratory course is primarily taught and facilitated by the analytical chemistry professors, faculty specializing in other related chemical sub-disciplines can easily participate and serve as the instructor of record. In particular faculty specializing in biochemistry or molecular spectroscopy can contribute their expertise to create an interesting interdisciplinary theme which engages both faculty and student interest. Furthermore, team teaching within this modular structure encourages cross-discipline conversations and models how science is done in modern professional settings.

### **Choosing themes and projects**

The themes for this approach to advanced analytical chemistry lab can vary on the basis of faculty interest and expertise or student interest. The overall goal is to create a theme that is sufficiently broad to allow for interpretation and student development of ideas. While selecting a theme in which the faculty has expertise is helpful, it is not as important as selecting a theme on the basis of faculty interest. The theme-based approach can be used to begin exploration into a

new field of research or generate ideas for collaborative studies across disciplines. Selection of the theme should be done prior to the start of the class, and is often published along with the schedule of classes so that students can enroll in a course that aligns with their interests.

Selection of the project or projects within the theme is student-driven, but often is accompanied by some guidance from the instructor. In some cases, students are instructed to read and survey the research literature related to the theme and identify some knowledge gap that could be addressed through experimentation. A great way for students to organize their findings is to require a formal proposal. One example of a proposal guide used at Butler University is provided (Appendix A), but other variations have been employed. Often this planning process leads to rather grandiose plans where sample number and analysis time would limit the ability to collect meaningful data. The instructor should lead the students to an understanding of the types of data they will be collecting (using some sort of sample grid) so that the students can make a decision to trim the project down to a reasonable and manageable size. When students have very little exposure to a topic or the theme is too broad, the instructor can provide a bit more definition to the theme-based course using a contrived scenario or a list of topics from which the students can choose and explore. Examples of these types of guiding documents are provided (Appendixes B and C).

Another important factor in selecting both the theme and the projects within the theme is instrument availability. Themes should be selected that make the best use of departmental equipment, and students should be introduced to the instrument holdings prior to project selection. Often, students will find an interesting study from the literature that employs an instrumental technique unavailable at the institution. In this case, students could be asked to adapt the procedure to make use of current instrument holdings.

To encourage diversity of instrumentation, faculty should also encourage students to incorporate multiple instrumental techniques into their project(s). Examples of student-selected projects which incorporate multiple instruments are:

- Analysis of gunshot residue (GSR) by IC and ICPMS
- Biochemical analysis by fluorescence and electrochemistry
- Forensic analyses using FTIR, LCMS, and GCMS

In general, the theme and proposed projects often fit well together and, with faculty input, the resulting projects lead to interesting data from which the students can draw meaningful conclusions.

### **Example themes that have been used**

Described below are several themes and related student-selected projects recently implemented at Butler University. Each description includes the general structure of the course, the experiments performed by the students, the process by which the experiments were determined, and the outcomes from the student work.

**Bioanalytical Chemistry:** The bioanalytical-themed module was the first team-taught module at Butler. The two professors developed much structure for the course, including grading rubrics, writing style guidelines, and even lists of relevant literature references. This structure enabled two professors with different classroom teaching styles to adopt a uniform set of expectations for both the faculty and the students. In the end, this level of structure benefitted the students, who still had room to explore and develop their own ideas.

The lab work in the bioanalytical theme was implemented using 3 distinct projects, with each successive project providing students with more freedom and discovery. The first project involved a simple fluorescence experiment where students employed a Biotective Green reagent cocktail to quantify biotin in vitamin samples. Much of the student input was focused on adapting the reagent instructions in order to obtain meaningful data. The second project involved simultaneous implementation of two different methods for assaying vitamin C. Students were divided into two groups, and each group selected a different assay method from provided literature sources. Group 1 selected an electrochemical method, using a modified carbon paste electrode to determine ascorbic acid concentration. Group 2 adapted a chromatographic method to study the vitamin C. At the end of the experiment, the two groups shared data and were able to compare the two methods for accuracy and precision. In the final project, the class researched and developed a biosensor for analysis of glucose. This project was selected by the students with some guidance from the instructors. The student-built potentiometric sensor was compared to a commercially available blood glucose detector for accuracy. Unfortunately, the

success of the project was limited due to the cost of materials and time. However, students were able to explain their attempts toward achieving their goal.

**Forensic Chemistry:** The forensic chemistry module has been implemented three separate times at Butler, with each implementation following a different structure.

In the first implementation, students were allowed to select three different projects to study over the course of the semester. The students selected (1) the determination of ethanol in breath by FTIR, (2) analysis of arson debris for accelerants by GCMS, and (3) determination of an analyte in a complex matrix (urine) by ICP-OES. Students worked to validate each analytical method using standards and then attempted to create mock crime scene samples which could be analyzed. The most successful of these projects was the arson analysis. The students burned wood samples soaked in different accelerants and were able to use the GC pattern of peaks to match an unknown accelerant to a standard. The urine project was the most inconclusive, though the project did involve a community partner from a local test-strip manufacturing company. All three posters were presented to the department in poster format.

Learning from the past, the second implementation was more focused and involved a forensic chemist from another local university. With his expertise, we developed a set of evidence from the scene of a crime. The scenario is provided in Appendix B. The evidence included arson samples, actual pipe bomb fragments, contrived drug samples and powder, and ink from a note and several pens taken from potential suspects. With this suite of evidence, students were instructed to divide and identify methods published in the literature suitable for analysis of these types of samples. Once they identified methods, the groups reconvened and reported their findings. By the end of week two, students had selected several methods to study their samples: LCMS, GCMS, FTIR, and TLC. The group divided into teams and each team had primary responsibility for a particular project, but also had secondary responsibility to review and discuss another project. In this way, each student was exposed to multiple aspects of the project. Appendix E depicts sample results from this project.

The latest implementation of the forensic chemistry module provided



students with a list of potential topics to study, and the students were expected to research and select one project on which to focus their efforts. While the instructor made an attempt to guide the group toward analysis of inks and dyes, the group chose analysis of gunshot residue (GSR). The goal of their research was to link the GSR on a sample of cloth back to the GSR found in a bullet casing. Initial work focused on the validation of an ion chromatograph and an ICP-MS for various metals and ions common to GSR. However, validation also focused on less common ingredients in the hope that some “fingerprint” might be found specific to a particular ammunition manufacturer. Once validation was completed, students wrote a formal document describing their procedure and the results, focusing on statistical analysis of and confidence in their data. The last several weeks of the semester were then dedicated to the analysis of real samples. Students collected their own samples with the help of a local shooting range and performed analysis. Casing analysis was somewhat simple, but analysis of the cloth proved more difficult. The students realized a flaw in their collection method (and the fact that the range was busy and much GSR was present in the air) and so the attempt to quantitatively match casing to cloth proved inconclusive.

**Molecular Spectroscopy and Art:** In this module, students were provided with the theme of art and art conservation. Because of the nature of the topic, and the difficulty in collecting samples, specific analytes and questions were presented to the students. The questions were developed with the help of the staff scientist at the Indianapolis Museum of Art. Problem 1 introduced students to the issues surrounding the degradation of smalt pigments. Specifically, smalt “fades” over time from a brilliant blue color to a pink/brown. Studies show that the potassium leaches from the smalt (potash glass) and the coordination of the cobalt changes from tetrahedral to octahedral. This change in coordination leads to the color change. Students were asked to develop and implement a project that would contribute to the understanding of this process. Limited by time and equipment, students chose to examine two variables: rate of degradation as a function of composition and as a function of particle size. Using a small furnace, students formulated different cobalt glass samples and then ground and sifted them to create a uniform size. Analysis was accomplished by artificially fading

the samples through exposure to an aqueous environment at an elevated temperature. Within one week, samples were degraded. Problem 2 centered on brilliant but fugitive eosin-based pink pigments used by van Gogh and others in the late 1800s. Students chose to examine rate of decomposition of the pigment as a function of atmosphere ( $N_2$  vs. air), and determine the identity of any decomposition products (especially brominated products) in the gas phase. Fading occurred in a light chamber, and much of the fading occurred within the first few hours of exposure in the light chamber. Analysis of the materials was accomplished using the instrumentation available at the Indianapolis Museum of Art (SEM, reflectance spectrometer, optical microscope) and the instrumentation available at Butler (GCMS, FTIR). While preparation and fading occurred as expected, data from the spectroscopic analysis did not provide insight into the mechanism of fading for either of the two pigments. However, students were able to make two claims: (1) The smaller small particles acted as sacrificial particles, completely fading before the larger particles. Therefore, better control of the particle size might contribute to a better understanding of the decomposition system. (2) The amount of eosin pigment mixed into a paint binder is so small that any decomposition products are present in too small of an amount for conventional analysis. A larger paint sample or larger concentration of eosin pigment is needed for a quantitative study of the decomposition products.

Other themes specific to faculty interest have also been implemented at Butler University. Selection of a theme should be tailored to faculty and student interests.

### **Student use of time**

Students who enroll in a theme-based analytical laboratory course should expect to spend a considerable amount of time working on their project both during and outside of the regularly scheduled class time. The bulk of the chemical prep and development work will occur during the scheduled lab, along with group discussions and collaborative writing. Outside class, students often set up an analysis on an instrument or come in to evaluate data collected during an automated overnight analysis. In addition, reports and other presentations are typically completed outside of class time.

At Butler University, students who enroll in the theme-based labs are required to attend a 4-hour long laboratory period, where the bulk of the work is accomplished. However, situations often arise (instrument issues, solution preparation, illness, absence) that require students to work outside of the scheduled lab times. While this kind of class work cannot be mandated, students are encouraged to schedule times with the facilitating professor to come in to the lab and complete a task related to completion of the project. In fact, many students often spend an extra 2 hours (on average) working outside of the scheduled 4-hour lab time. In addition, much of the background research, report writing, peer review, and poster presentations occur outside of the scheduled laboratory time.

### **Controlling group dynamics**

When students graduate from their undergraduate institution and begin the transition to becoming professional chemists or graduate students, they will undoubtedly be required to work and collaborate with others. The theme-based modules offer students the opportunity to practice and hone their team-work skills.

In most cases, the group will easily identify several instruments which will contribute data necessary to addressing the project goal. Likewise, the group often finds a best method for utilizing their individual strengths for the benefit of the whole. However, students often gravitate to the one instrumental technique or job where they feel most skilled and they never gain exposure or experience on any other aspect of the project. In this respect, the instructor should encourage students to become involved in multiple aspects of the work. This can be done informally, with a gentle nudge or positive comment, or this can be planned, by requiring students to rotate to different aspects of the project. For example, in one implementation of the spectroscopy and art module, one student felt more comfortable only processing data, but not using the instrument to collect the data. In this case, the instructor guided the student to work with one other student to collect data. In this way, both students gained a better understanding of the project as a whole.

In the case where the instructor notices or learns that one or two students are not contributing at all to the work of the whole, the instructor may need to meet individually with the student(s) and grade accordingly, with the expectation that the rest of the group will still need to get the work completed.

### **Deliverables required from the students**

Several types of reports fit well into the theme-based approach to analytical lab. Throughout the semester, students are expected to complete short writing assignments that describe and outline the instrumental aspects of the work they are completing. These are graded and returned to the student so that they can incorporate the information into a final report. Once the experimental work and data analysis is completed, students work collaboratively on a manuscript-style report to summarize the significance, previous work, procedure, data, results, and conclusion. A formal poster and subsequent poster presentation summarizing their work for a larger audience is also required. Examples of the reports and posters are provided in Appendixes D and E. Oral reports are often incorporated in a “group-meeting” setting so that individual groups can report to the entire class on a smaller aspect of the overall project.

Often, students will not collect data that allows them to conclusively address their primary goal or research question. This does not mean that the experience failed or that they as a research team failed. Usually it is a question of time and resources and not a question of effort. In this case, students should be made aware of the successes they have had in their semester and should be encouraged to report on the process of the work and not simply on the outcomes. In this way, students still feel a sense of accomplishment and are able to take pride in their work. The learning process is as essential as any final results the students may obtain and through the attempts and failures involved with *doing* science, real learning takes place.

### **Grading**

Grading of all formal reports is accomplished through the use of writing guides and grading rubrics (Appendixes F through I) common to most of the upper-level lab courses offered at Butler. These rubrics were developed by a subcommittee of faculty who teach the upper-level laboratory courses. They are used to provide a level of uniformity in grading between themes and sections. In addition, a collaborative skills rubric is often incorporated to assess growth in professional skill development. In this way, students who make a more significant contribution to the success of the group can be rewarded in comparison to those who do not significantly contribute. Students are provided with the grading rubrics at the beginning of the semester and are expected to understand the rubrics before beginning work on any report. Mid-semester skills assessment has also been employed to encourage students with poor group skills to make changes in the latter part of the course.

### **The use of peer-review**

Peer review is desirable when implementing the theme-based approach. Using peer review, students can learn how to critically read and analyze draft manuscripts and make constructive comments. Peer review has also provided students with a different perspective on a project, which has led to a more unified understanding of the overall project. Since most students have not yet engaged in peer review, a guide is provided (Appendix J) to students which leads them through the process and give tips on what a successful review might look like. This document is used in many upper-level courses at Butler. Students evaluate and comment, but do not assign any kind of grade. Peer review is often a single blind process. The reviewer knows the author, but the author is not provided with the name of the reviewer. In a small class, however, the students usually figure it out.

At Butler University, peer review is only incorporated in the formal report writing process. However, peer review can be incorporated into multiple aspects of the project including formal written reports, poster preparation, group work skills, and oral presentations.

### **Concluding comments about the use of theme-based modules at Butler?**

Overall, the theme-based approach to the instrumental analysis laboratory has been very successful from the viewpoint of the student, the instructor(s), and the chemistry department. Students are exposed to different techniques and problems, while honing the skills necessary for success in the chemical community today. Faculty members in other areas of chemistry have participated in the theme-based approach and have also found the experience to be valuable for students and an invigorating way to teach. In addition, students make use of the skills they learned in the theme-based modules long after they graduate from Butler. The comments below serve as examples from former students who have benefitted from the theme-based approach (as a reminder the theme-based approach at Butler has the course number CH424).

“CH424 teaches the student to examine his own weaknesses and deficits in knowledge, educate himself on how to address these to the point that they are no longer weaknesses, and then start using this knowledge to solve problems.” CS, medical resident

“Method development, independent/critical thinking, and troubleshooting skills are all transferable skills which are applicable to any job; and every company relies on their

employees to create value through these means.” JG, industrial chemist

“The lab course required me to be more outspoken (something a shy person like myself steers away from!) which is definitely a skill and benefit that I used in my graduate school career as well as in my current lab job.” EL, MS chemist

“As a researcher today I have been able to complete projects very efficiently without lacking in significance, because I can now distinguish between what is feasible and important and what is not.” ES, graduate student