JOURNAL OF CHEMICAL EDUCATION

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# Incorporating Students' Self-Designed, Research-Based Analytical Chemistry Projects into the Instrumentation Curriculum

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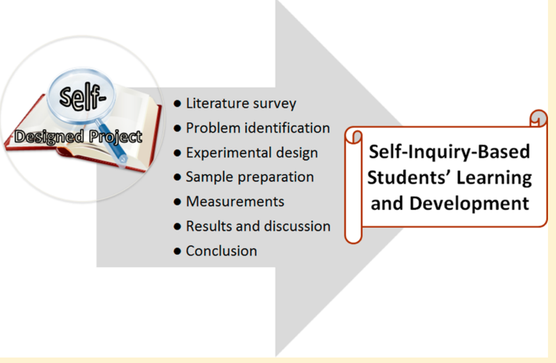
Supporting Information




**ABSTRACT:** In a typical chemistry instrumentation laboratory, students learn analytical techniques through a well-developed procedure. Such an approach, however, does not engage students in a creative endeavor. To foster the intrinsic motivation of students' desire to learn, improve their confidence in self-directed learning activities and enhance their problem-solving skills, students' self-designed, research-based analytical chemistry projects have been developed within the course of Advanced Chemical Methods for junior and senior undergraduates at SUNY College at Old Westbury. In addition to a series of manual-based laboratories held on a regular basis, students also do a literature search to formulate their own ideas that are tested using appropriate analytical instruments. By participating in self-designed projects, students independently go through the key components of analytical chemistry with greater engagement: from literature survey, problem identification, experimental design, sample preparation, measurements, results and discussion to conclusion, in a self-inquiry-based learning environment. The instructor's observation, assessment of written reports and students' self-evaluations indicate successful implementation of the projects and students' progress toward the goals mentioned above. This project provides students a research-based chemistry laboratory for self-inquiry-driven learning and independent thinking.

**KEYWORDS:** *Upper-Division Undergraduate, Analytical Chemistry, Curriculum, Inquiry-Based/Discovery Learning, Problem Solving/Decision Making, Instrumental Methods, Undergraduate Research*

## INTRODUCTION

The ultimate goal of chemistry higher education is to provide students with the tools to solve chemistry problems. According to American Chemical Society (ACS) guidelines for undergraduate professional education in chemistry, a chemistry program should provide excellent content and builds skills that students need to be effective professionals. Laboratory experience and development of professional skills, such as problem-solving skills, chemical literature skills, etc., are the target of chemistry education proposed by the ACS Committee on Professional Training (ACS-CPT).[1] This important attribute has been discussed and somewhat reflected in analytical chemistry curriculums.[2−4] Nonetheless, challenges still exist in preparing students to acquire necessary problem-solving skills in the laboratory.[4] In a typical undergraduate instrumentation laboratory, students often follow a chemistry "cookbook" to merely execute a well-defined instrumental analysis. They rarely have the opportunity to make their own choices and resolve problems.

Over the past decades, Deci and Ryan had developed Self-Determination Theory (SDT),[5,6] which states that intrinsically motivated learning can only occur when an individual feels free to make choices in a challenging activity, where this challenge can be met and conquered. Extensive research has been done to explore the relevance of SDT to many fields.[7,8] The intuitive appeal and strong evidentiary support for SDT have made it popular as a means of education. Incorporation of student-designed research projects in a chemistry curriculum for a seminar course was reported, providing a positive effect on students' intellectual and independent growth as scientists.[9] By participation in research-driven, inquiry-based microbiology laboratories, the ability of undergraduates to contribute to original research was strongly enhanced.[10] Student-designed experiments were also successfully adapted for upper level biochemistry laboratories, which improved students' confidence and ability in performing independent research.[11] Inquiry-based teaching has, in fact, been advocated since the 1960s.[12] Psychological research and theory suggest that by engaging students in problem solving processes, they can learn both content and thinking strategies.[13] Weaver and co-workers reported that more elements of inquiry would be determined by students rather than by the instructor in an open inquiry-based laboratory; moreover, a research-based pedagogy provided students with immense benefits over traditional laboratory experiences, and even over inquiry-based laboratory experiences.[14]

On the basis of those considerations, besides regular manual-based laboratory exercises for junior and senior undergraduates,

**Published:** January 17, 2015

student self-designed analytical projects were developed within the course of Advanced Chemical Methods at SUNY College at Old Westbury. The goals were to intrinsically motivate students' desire to learn, improve their confidence in self-directed activities and enhance their problem-solving skills by providing them an opportunity to define a problem, implement self-designed experiments, analyze data and draw appropriate conclusions on their own. This article provides detailed information about the course, student's self-designed projects, challenges and assessments.

## ■ DESCRIPTION OF THE COURSE

Advanced Chemical Methods (CP 4800) surveys the theory and practice of modern analytical chemistry techniques and instrumental methods. This course is offered in the spring semester at SUNY College at Old Westbury; 3.3 h of lectures and a 4-h laboratory per week are scheduled. The lectures concentrate on the principles, applications and limitations inherent with various techniques, while the laboratories provide students opportunities to learn the practical aspects of techniques via hands-on experiments that include a series of manual-based laboratories held on a regular basis and one self-designed project conducted during the semester (see the syllabus in Supporting Information.). The characterization and quantification of organic and inorganic compounds are carried out with the aids of various analytical instruments, such as UV/visible, atomic absorption, fluorescence and NMR spectroscopy, GC/MS, HPLC, LC/MS, electroanalytical chemistry instruments, etc. This course is required for chemistry majors and is an elective for biochemistry majors. The prerequisites are Organic Chemistry II (with laboratories), Analytical Chemistry (with laboratories) and Physical Chemistry I.

## ■ DESCRIPTION AND EXAMPLES OF STUDENT'S SELF-DESIGNED PROJECTS

The learning objectives were, by completing self-designed projects, that students were able to (1) apply textbook knowledge to a real analytical chemistry problem in an independent manner through literature survey, problem identification, experimental design, sample preparation, measurements, results and discussion, and conclusion; and (2) produce a laboratory report written in a scientific format. Junior and senior undergraduates enrolled in Advanced Chemical Methods had already taken Analytical Chemistry. Their basic understanding of analytical instrumentations facilitated the development of their own projects. A semester-long schedule is given in Table 1. In week 1, students were informed about the project and instructed on commonly used chemistry search engines, such as Analytical Abstracts published by the Royal Society of Chemistry (RSC); SciFinder provided by the Chemical Abstract Service (CAS), a division of ACS; PubMed maintained by the United States National Library of Medicine (NLM) at the National Institutes of Health (NIH). For instance, students learned how to use keywords, molecular formula and chemical structures to find references in SciFinder. There were no specific journals or published papers assigned to students. Each student had to find his/her own references and develop an analytical project that involved the use of analytical instruments. A list of instruments available at the Chemistry and Physics Department was provided. In weeks 2−6, students developed initial project ideas based on personal interest, literature survey and facility availability. In week 7, students shared their project ideas in class. They had the complete freedom to select a topic, which might consist of organic/inorganic analysis, structure identification or a comparative study of different samples. Students could use or modify a published method for their samples but had to avoid simply repeating others' work. The instructor was responsible for instrument availability and project feasibility. In weeks 8−11, students might need to read more papers and recall their basic chemistry knowledge before finalizing a project. A needed list of chemicals, instruments, pipettes and glassware had to be provided by each student in week 11. In weeks 12−14, they executed experiments, analyzed data and drew appropriate conclusions. Students might work individually or in pairs. However, each individual had to write his/her own laboratory report independently. In week 15, each student gave a 10 min oral presentation about the project focusing on what he/she did in laboratories in addition to the introduction, problem statement and method description. A written report in scientific or ACS format was required and due in 2 weeks upon the completion of experiments.[15] The following sections should be included in the report: (1) title of the project, (2) authors and their affiliations, (3) abstract, (4) introduction, (5) experimental section (chemicals, instruments and methods), (6) results and discussion (results, data and error analysis, etc.), (7) conclusion, (8) acknowledgment and (9) references (in text numbered citations). Examples of self-designed projects are listed in Table 2. Three of them are briefly described. Some references students used are cited. An example of a student

**Table 1. General Schedule for Student-Designed Projects**

| Week | Assignment |
|---|---|
| 1 | Orientation: think about the project. |
| 2−6 | Literature survey: resolve an analytical project. |
| 7 | Informal presentation: share project ideas in class. |
| 8−11 | Procedure finalization: design experiments and provide a needed list. |
| 12−14 | Experiment execution: implement experiments and complete report in 2 weeks |
| 15 | 10 min oral presentation: present projects in class. |

**Table 2. Examples of Student-Designed Projects**

| No. | Project | Instrument Used |
|---|---|---|
| 1 | Determination of calcium in vitamin water | Atomic absorption spectroscopy |
| 2 | Determination of iron in cereal | Atomic absorption spectroscopy |
| 3 | Atomic Absorption Spectroscopy for Sea Water and Drinking Water Analysis | Atomic absorption spectroscopy |
| 4 | Determination of zinc in multivitamins by atomic absorption spectroscopy | Atomic absorption spectroscopy |
| 5 | Determination of phosphate ion concentration in colas | UV/vis absorption spectroscopy |
| 6 | Determination of loading ratio of porphyrin to silica | UV/vis and $^1$H NMR spectroscopy |
| 7 | Comparing fluoride ion concentrations in toothpaste brands | Fluoride electrode and pH meter |
| 8 | Cocaine in dollar bills | GC/MS |
| 9 | Determining the presence of 1,4-dioxine in hair dye | GC/MS |
| 10 | Determination and quantification of toluene in nail polish | GC/MS |
| 11 | Is there traceable dibutyl phthalate presence in water from plastic bottle? | GC/MS |



DOI: 10.1021/ed500502w
J. Chem. Educ. 2015, 92, 444−449

report can be found in Supporting Information (with the permission from student authors).

Project 1: To determine and compare zinc contents in multivitamins, two students used a flame atomic absorption spectrophotometer to examine the zinc amount in multivitamins of different brands. The absorbance of zinc atoms was taken at 214 nm. According to the modified literature method,[16] students employed a general method to make a calibration curve with $Zn^{2+}$ standard concentrations ranging from 2.00 to 20.00 ppm. In the experiment, "A blank was first aspirated into flame and its absorbance reading was set to zero. Zinc standards were analyzed from the lowest concentration to the highest before testing the unknowns from each multivitamins brand. Deionized water was aspirated before and after each unknown was measured". To ensure that the absorbance of multivitamin samples fell in the linear calibration range, students had to figure out a proper dilution factor through experiments for each unknown by comparing the absorbance to that of calibration standards. Zinc concentrations obtained were consistent with the values labeled on products within acceptable error ranges. Students, therefore, accurately determined zinc content in various multivitamins and achieved their goal.

Project 2: The goal was to compare fluoride contents in different brands of toothpaste by potentiometry. The significance of this project lay in the fact that fluoride is the most popular ingredient in toothpaste and has beneficial effects on the formation of dental enamel and bones.[17] Therefore, it is important to report fluoride amounts in toothpaste accurately. According to the method in the laboratory handout, the student weighted an appropriate amount of toothpaste of each brand and prepared sample solutions that could be measured using a fluoride selective electrode. He carefully described the procedure: "... Starting with the lowest concentration, pour solution into a 50 mL beaker... Gently insert the electrode into the solution. ... When the pH meter beeps, record the potential. ...". A linear calibration curve of potential (mV) via $-\log[F^-]$ with $[F^-]$ ranging from 0.10 to 2.00 ppm was constructed. The fluoride amount obtained was close to that labeled. The student also pointed out that experimental error might be introduced in toothpaste weighing and dissolution.

Project 3: Dibutyl phthalate (DBP) is one of the commonly used plasticizers that are added into plastics to make products more durable and flexible. Those compounds are known to leech off plastics and accumulate in the environment.[18] DBP is toxic to the reproductive system through contact with the skin and by ingestion. This safety concern has been raised with the increasing use of plastics. The goal was to detect possible contamination of DBP in bottled water of some popular brands in supermarkets. The student analyzed four brands of water bottles using the modified literature method of GC/MS.[19] Hexane was used in the standard preparation (0.1−10 ppb) and sample extraction because of its good solubility to DBP. With the use of GC/MS equipped with a capillary column and electron impact ion source, trace amounts of DBP were detected from all of the four water samples. The data obtained indicated the apparent risk of exposure to daily consumption of plasticizers. The accurate concentrations of DBP, however, were not obtained due to broad DBP bands on chromatograms. Within the laboratory period available for a self-designed project, the student was not able to work out a best experimental condition for quantitative analysis. However, she commented on how to improve results with other organic solvents and more diluted samples. Overall, the student went through the whole analytical process to confirm the existence of DBP in bottled water and achieved the project goal.

## ■ DISCUSSION

### First Learning Objective and Assessment

The first learning objective for students was to be able to apply textbook knowledge to a real analytical chemistry problem in an independent manner through literature survey, problem identification, experimental design, sample preparation, measurements, results and discussion, and conclusion. The instructor's hypothesis was that students would be motivated to learn more chemistry and improve their problem solving skills by conducting self-designed projects because they had to understand what they were doing before and while in the laboratory and be able to make progress toward completing the projects. This learning objective was accomplished by students' active participation in the projects. Although the effect of self-designed projects on lecture performance has not been evaluated, significant improvement has been seen in students' independence and confidence in their abilities to execute projects and solve problems. To assess the degree to which students met this learning objective, instructor's observations and students' self-evaluations were compiled.

Three characteristics differentiate inquiry-based activities from other chemistry laboratory instruction styles: induction, an undetermined outcome and the student's generated procedure.[20] In an inquiry-based learning style, students are more involved and responsible in determining procedural options. The benefits for students from inquiry-based activities in chemistry laboratories have been actively studied.[21,22] The instructor's strategy to improve problem solving skills was to get students to think for themselves when faced with a laboratory problem, rather than providing a solution. The two most challenging practices students encountered were seen in problem identification and experimental design.

For problem identification, the challenge started with students' lack of knowledge of chemical instrumentation, sample preparation and separation. To form an analytical question and develop a hypothesis that could be tested, students had to know the basic mechanisms and applications of various analytical techniques. This required knowledge had not been fully taught at the beginning of the course although students acquired some understanding of chemical instrumentation from an Analytical Chemistry course. They might have struggled with the uncertainty of carrying out an independent project, the unfamiliarity of literature search tools and instruments, and the limitation of facilities the department can offer. It was not uncommon for students to plan an overly ambitious project that might have required natural product separation and characterization, or from total synthesis, purification to detection. At this stage, they needed to figure out what they were able to do within a one-semester period. They might have had to read more references or looked at the instrument to get some sense on how it would work. Many students proposed several ideas before finalizing a decision. Each student had to rely on his/her own judgment because each project was different. They were making progress while thinking through all possibly involved processes. As reflected in self-evaluations, students became more confident about their ability to carry out self-designed projects. An appropriate instructional adjustment based on students' needs also helped



DOI: 10.1021/ed500502w
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keep them on track, such as holding regular informal discussions and providing introductions in regard to search tools and analytical instruments within the beginning period of the course.

Prelaboratory experimental design was the heart of this project. Self-inquiry-driven learning fostered the development of higher-order cognitive skills through the implementation of student-generated procedures. The assignment for each student was to develop a specific and detailed written procedure that could be followed while executing experiments, such as "A 50 mL beaker is washed with tap water, deionized water and acetone three times. Once the beaker is dried, its mass is recorded on an analytical balance". Students often got stuck in concentration calculation, filtration setup, buffer preparation, control examination, poorly organized information, etc. It often helped asking students to review basic concepts and operations they had learned, or to check if the numbers made sense. For example, something was probably wrong if a student calculated to weigh a 30.1234 g solid and dissolved it into a 10.00 mL solution. They might have had to modify or revise experimental designs accordingly based on the results obtained. Students gained problem solving skills by going through those practices with minimum input from the instructor. Many of them who were initially skeptical about their abilities became more confident and independent as the projects proceeded. In all of the cases, students showed more curiosity and a sense of responsibility for their own projects, leading to significant gains of basic science understanding and chemistry laboratory skills.

In addition, a scientific investigation requires planning and coordination of activity. To achieve the ultimate goal of self-inquiry in a research-based laboratory, students had to be able to organize and manage their activities, which were not typically required for traditional laboratory exercises. In self-designed experiments, students often encountered the problems that were usually taken care of by the instructor. For instance, acid digestion and heating might be needed in a sample preparation. An appropriate dilution had to be made based on concentration, method sensitivity and calibration linearity. A solvent selection depended on both sample solubility and instrumental method, e.g., an aqueous solution for reversed-phase HPLC and an organic solvent for GC/MS. A chemical reaction might be sped up by changing pH or temperature. Analytical instruments needed warming up for 20−30 min before measurements. A needed list of chemicals, instruments, pipettes, glassware, etc. had to be provided by students in advance. These investigating processes were valued as much as the results of students' efforts.

The improvement of students' ability to identify and solve problems is reflected in their self-evaluations (Figure 1 and Supporting Information). For instance, students were frequently confused by calculations involving unit conversion. In this project, however, they showed great confidence in improving their ability in calculation and standard preparation (4.6/5.0). They also self-reported improvement in the ability to do a literature search (4.2/5.0), select an instrument (4.4/5.0), design experiments (4.4/5.0), troubleshoot experiments (4.1/5.0) when something unexpected occurs, analyze data (4.5/5.0) and develop laboratory skills (4.6/5.0). A fair improvement was seen in students' ability to pursue a research topic (3.9/5.0). Students rated project difficulty on a scale of 1 (easiest) to 5 (hardest). An average of 3.7/5.0 for students' perception of project difficulty suggested that students were challenged by carrying out their own projects. Overall, self-designed projects

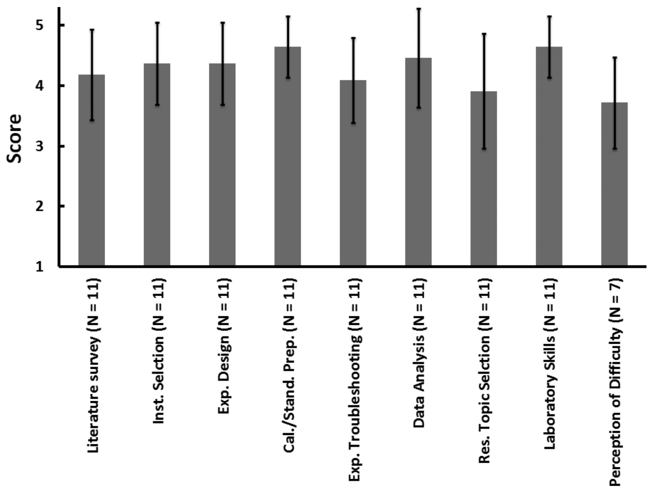


**Figure 1.** Students' self-evaluations and their perceptions of project difficulty. First eight columns indicate the improvement of students' abilities on literature survey, instrument selection, experimental design, calculation and standard preparation, experimental troubleshooting, data analysis, research topic selection and laboratory skills on a scale of 1−5 with 1 = deficient, 2 = insufficient, 3 = fair, 4 = good, and 5 = excellent. The last column represents students' perceptions of project difficulty on a scale of 1−5 with 1 = easiest, 2 = moderately easy, 3 = average, 4 = moderately hard, and 5 = hardest. Error bars represent one standard deviation from the mean.

received positive responses from students. A few excerpts included "It was a great idea. Since this is a senior level course, we as students should be expected to have some level of competence in independent analytical research."; "The self-designed project was essential for making sense of the class materials. ...". The negative comments were directed at the time commitment for selection and completion of a project. Students commented "...The only thing I would change is to maybe choose our topics a little later in the semester. I know we were delayed because of snow but before picking a project I would have liked to know more about what we are capable of doing."; "If we were able to run our experiment twice with different standards, it would be better."; "Need more time...".

The limited time allowed for self-designed projects was challenging for both students and the instructor. Conducting students' self-designed projects was much more time-consuming than carrying out a traditional instrumentation laboratory. Self-designed projects took time to set up and often required more time to optimize experimental conditions. It was a challenge for the instructor to encourage students' learning from self-inquiry while ensuring all of the required materials in the textbook were covered. Moreover, students ran experiments on different instruments at different locations simultaneously. The demands on the instructor were high in laboratories. Extra time out of classroom was normally needed. To better organize course materials and balance between regular manual-based laboratories and self-designed projects, each student was allowed to do only one project. To use laboratories efficiently without dropping necessary course materials, both manual-based and self-designed experiments were scheduled in the same laboratory session as needed (see syllabus in Supporting Information). Another option in the future is to expose students to a literature survey on certain topics in the course of Analytical Chemistry for extra credit, so that they will be better prepared for self-designed projects since many students taking


DOI: 10.1021/ed500502w
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Analytical Chemistry were enrolled in Advanced Chemical Methods.

### Second Learning Objective and Assessment

The second learning objective for students was to be able to produce laboratory reports written in a scientific format. Students were instructed on a commonly used method of organization for a laboratory report, e.g., an ACS format.[15] To assess the degree to which students met this learning objective, the instructor's evaluations on selected sections of laboratory reports were compiled (Figure 2 and Supporting Information).

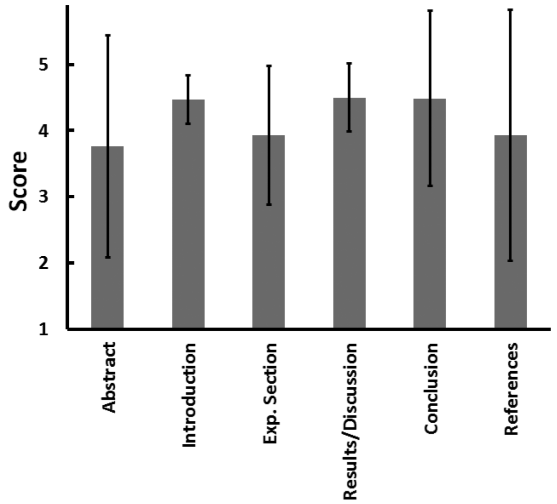


**Figure 2.** Instructor's evaluation of laboratory reports ($N = 14$) on a scale of 1−5 with 1 = no achievement, 2 = unsatisfactory, 3 = fair, 4 = good, and 5 = excellent. Error bars represent one standard deviation from the mean.

Students were scored well for Introduction (4.5/5.0), Results and Discussion (4.5/5.0) and Conclusion (4.5/5.0). The score from Introduction revealed that students did a fairly thorough job on the literature review and created their own goals for the projects. For Results and Discussion and Conclusion, scores pointed toward students' understanding of the projects. They effectively executed experiments, collected data and derived appropriate conclusions on their own. The outcome was moderately well on Abstract (3.8/5.0), Experimental section (3.9/5.0) and Reference (3.9/5.0). Those data indicated that students were well prepared for the work but were somewhat unfamiliar with a scientific writing format. For instance, the following information was often missing from the written document: chemical purity, in text numbered citations, argumentation and purpose in abstract, because this information was usually not required in a regular laboratory report. Although many undergraduates have participated in research projects with faculty members, they did not have much experience writing their own papers. As a result, their abstracts might not have been informative enough in terms of the scope, purpose, results and contents of the work. The instructor's assessments on items related with research training somewhat matched the lower score from students' self-evaluation on the improvement of their ability to pursue a research topic (3.9/5.0). This assessment revealed the areas that students did not participate in or did not pay attention to as much as the instructor would have liked. Additional instruction would likely enhance a student's understanding and success, proposing a future direction in regard to teaching and learning effort.

## ■ CONCLUSIONS

In conclusion, incorporation of students' self-designed projects into a chemical instrumentation curriculum is beneficial, but comes with challenges. Self-designed projects encourage students to go through every aspect of analytical chemistry in a way that authentically models the scientific process. This self-inquiry-based exploration strengthens students' independence and self-confidence in their abilities to execute projects and solve problems. The author believes that this learning experience enriches students' intellectual growth as scientists and as competent life-long learners, and will become an important part of their education.

## ■ ASSOCIATED CONTENT

### Ⓢ Supporting Information

Course syllabus, self-evaluation form, scoring rubric and a student report (used with permission). This material is available via the Internet at http://pubs.acs.org.

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### Notes

The authors declare no competing financial interest.

## ■ ACKNOWLEDGMENTS

The author thanks the support from Faculty Development Grant at SUNY Old Westbury, SUNY Research Foundation's Enhancing STEM Research Experiences Pilot Program, Chemistry and Physics Department at SUNY Old Westbury and CP 4800 students from spring 2013 and spring 2014 (Adil N. Afridi, Jeannine E. Angeloch, Anique Baig, Matthew Carey, Aileen Espinal, Kisuk Jin, Pierre Laguerre, Rashmeet Nagpal, Michael Quinones, Madeeha Rahat, Penelope Riascos, Fanny Torres, Weiqiao Wesley, Oznur Zaim and Ruixue Zhang). The author also thanks Mr. Jianli Zheng for his great support in preparations of chemicals, glassware and instruments. The valuable inputs by my colleagues, Dr. Judith Lloyd and Dr. Duncan Quarless, are greatly appreciated.

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