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# Facilitating Conceptual Learning in Quantitative Chemistry

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# Facilitating Conceptual Learning in Quantitative Chemistry

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by

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ABSTRACT

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Traditional chemistry laboratory courses have a manual consisting of "step-by-step" experiments; instructions are given to complete experiments, requiring minimal information/concepts processing to be successful. This experience leaves students unprepared for the real-world, where critical thinking skills are needed to conduct research. This study focused on building analytical techniques, conceptual knowledge, and critical thinking skills used to solve research problems. A new quantitative chemistry laboratory manual was developed to transition students from traditional to inquiry-based experiments, requiring analytical method development. Data showed students having less difficulty using the new manual (0.8281 average difficulty) on method development exam questions and experiments, compared to the traditional manual (0.600 average difficulty). T-test showed significant difference between item difficulty, $p = 0.029$. Using null hypotheses, the new laboratory manual led to an increase in students' conceptual knowledge and research skills. They were able to use their knowledge and skills to successfully solve real-world related problems.

# DEDICATION

I would like to dedicate this work to the tireless educators in our society that have devoted their time, heart, and life to the advancement of the educational system and the students that their efforts impact. You are the foundation upon which our society stands and you do it with a relentless passion. You are all heroes in my mind; I applaud and admire you for all that you do!

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LIST OF ABBREVIATIONS

ZPD – Zone of Proximal Development

PBL – Problem-Based Learning

SALG – Student Assessment of Learning Gains

UV-Vis – Ultraviolet-Visible Spectrometer

AAS – Atomic Absorption Spectrometer

MSA – Method of Standard Addition

LOD – Limit of Detection

LOQ – Limit of Quantification

IRB – Institutional Review Board

HCl – Hydrochloric Acid

R.O. – Reverse Osmosis Water

# CHAPTER 1

# INTRODUCTION

In analytical chemistry, a foundation of proper research methods, critical thinking skills, and quantitative techniques is required to be able to solve problems faced by the industry. This foundation is based on the knowledge that students receive from chemistry courses taken during their college education. The chemistry program of study consists of multiple courses generally made up of lecture and laboratory classes.[1] Lecture courses provide students with the basic knowledge required for understanding chemistry, while the hands-on techniques are acquired in the laboratory courses. Laboratory courses are important because they give students the opportunity to add to the content learned in lecture through active experimentation and increase their conceptual knowledge of the material. The knowledge gained from both types of courses is equally important in the student's education because lecture helps introduce several concepts, while the laboratory course allows students to apply them and learn hands-on techniques. After graduation, if chemistry students are required to conduct their own research to solve problems in the industry, their ability to perform this task will be based on the foundation that they received in research methods, critical thinking skills, and quantitative techniques, which can be learned in a laboratory setting.

Educators accept the importance of laboratory courses because it gives students the opportunity to build their knowledge of concepts and gain valuable cognitive skills.[2] However, students not having these important cognitive skills, critical thinking and research methods, when they graduate from college is an area of concern.[3] The way to optimize student conceptual learning has been debated for many years. There are several factors that affect a student's ability to learn and retain that knowledge for future use. The primary concern that generally affects the student's learning ability (regardless of age, demographic background, *etc*.) is how the information can be presented in a way that will enhance the student's conceptual learning of the subject so they will get the most out of an instructor's curriculum and the teaching of that curriculum.[4]

## Traditional Laboratory Teaching

Traditional laboratory teaching is the general term used to describe experiments, curriculum, and/or teaching methods that use the structure of following a procedure, which typically provides step-by-step instructions, explaining how to conduct a laboratory experiment, *e.g.* "How to Extract Alum from Aluminum Cans".[5] With traditional experiments, students are told how to achieve the solution; their ability to get the experiment finished is not based on their understanding of the chemical reactions or concepts. Zhao describes it as, "Instead of reflecting the practice of scientific investigation, chemistry lab sessions are often designed for repetition and verification. Students follow the directions in the lab manual, perform the manipulations, record the data, and fill out a worksheet."[6]

Although these types of chemistry experiments can be a good method for teaching students the basic laboratory techniques (e.g. how to use a volumetric pipet), they do not require students to go through a process of understanding the underlying concepts involved in an experiment. All the student is required to do is to complete the experiment using the laboratory manual and memorize any reactions and/or equations that may appear on a future laboratory exam. Essentially, being able to follow a 'recipe' and memorize are often the only things needed for the students to achieve a high score in laboratory courses.

With the traditional curriculum for chemistry laboratories, students are required to only have minimal knowledge of the material. Bloom's Taxonomy can be used to understand what is usually required of students in traditional chemistry laboratory courses. In many cases, traditional laboratory curricula utilize the two bottom levels, knowledge (recalling of information) and comprehension (ability to summarize and discuss).[7] Since these are the only levels used, it can set students up for poor or difficult performance in future chemistry occupations that require much more than recalling information and being able to summarize/discuss. If students are to succeed, not only in their educational but also professional careers, they need to be able to use the four upper levels of Bloom's Taxonomy, application (apply knowledge to solve problems through designing and experimenting), analysis (identify/analyze trends and ability to organize ideas), synthesis (use concepts to create/design new ideas), and evaluation (assess theories/concepts and evaluating outcomes).[7] If laboratory curricula enabled students to operate in the four upper levels, it may facilitate their learning of the underlying concepts involved in the experiments, develop their critical thinking skills, use those skills to examine problems, and be able to develop their own methods of analysis to be able to solve any chemistry related problems. All of these are situations they will encounter in their professional careers and will be unprepared for using a traditional curriculum that only uses the first two levels.

## Inquiry-Based Teaching

Although inquiry-based teaching is an intellectual thought process that has been around for thousands of years, comparatively, its application into the world of science is a relatively new idea.[8] In the early 1900's, John Dewey criticized the educational methods of teaching science by a means of memorization and urged that instead science should be taught as a way of thinking, a process that must be gone through to reach the conclusion.[9] This realization helped raise awareness of the issues involved in how chemistry was taught and how it should be taught in the years to follow.[10,11]

Later, during the 1960's, Joseph Schwab indicated that science was being guided by a new vision of scientific inquiry.[12] Schwab's system categorized inquiry into two main groups, stable and fluid. He described stable as being inquiry that involves current understanding used to fill a space in a growing body of knowledge, while fluid inquiry involved the creation of new concepts that has the potential to revolutionize the world of science. Schwab applied these two basic concepts to a laboratory setting and developed three different levels of openness in teaching/instruction.[13] The most basic level consisted of using educational materials to pose questions and then providing methods for students to discover relationships for themselves. In the next level students are expected to use their knowledge to devise their own methods to answer questions posed to them. The final level, when provided phenomena, it leaves students to formulate their own questions, collect evidence, and generate explanations based on their findings.[6] These concepts marked a new beginning and the core tenets for inquiry-based curriculum for laboratory settings in the educational system.[14]

Inquiry is a basic term used to describe the teaching process involved in getting students to understand concepts, instead of just verifying them. Through inquiry, students are able to ask questions and develop a reasoning to make sense of what they are learning; this allows students to learn the underlying concepts involved in what is being taught.[15] This is accomplished through the type and amount of information given to students during their learning process; for this reason, all types of inquiry are not the same. Inquiry differs not only differ by the information provided to students, and the amount of guidance provided from the instructor, but it can also differ in complexity.

## The Four-Level Model of Inquiry

A four-level model was developed to classify the levels of activity that may be used in teaching a topic for experimentation.[16] The first two levels are the most basic and common techniques used in traditional teaching; they can also be considered traditional laboratory teaching, since a procedure is provided in most experiments. For the first level, confirmation inquiry, students conduct the experiment with a provided question and procedure; the results are known in advance. This level can be useful when the instructor wants to reintroduce a previously learned concept. At the second level, structured inquiry, the instructor provides a question and an experimental procedure. Students then develop their explanation of the results that they collected. A structured inquiry can be helpful to enable students to develop more open-ended inquiry abilities.[17]

The third level, guided inquiry, is where the instructor provides only the question. The students design the procedure and formulate their own explanation. This type of inquiry is usually only successful with repeated exposure until students are accustomed to being able to plan procedures. For the fourth level, open inquiry, students have the opportunity to formulate questions that are related to a main topic. Then, they are required to design and conduct their experiment, as well as analyze the results and draw their own conclusions. This level can help in developing scientific reasoning skills in the students.[17]

Usually the four level model is applied to a single topic with each level increasing the difficulty of critical thinking skills required to complete the task. An example of this is shown in

Table 1, the levels of inquiry in an effervescent antacid table activity. This type of inquiry can be considered a continuum as each level builds on the knowledge acquired on the previous task to help complete the next one.

**Table 1.** Levels of inquiry in an effervescent antacid tablet activity. [16, 17]

| Levels of inquiry in an effervescent antacid tablet activity | |
|---|---|
| **Inquiry Level** | **Description and Examples** |
| **1** | **Confirmation**—Students confirm a principle through an activity in which the *results are known in advance.*<br>"In this investigation you will confirm that the rate of a chemical reaction increases as the temperature of the reacting materials increases. You will use effervescent antacid tablets to verify this principle. Using the following procedure, record the results as indicated, and answer the questions at the end of the activity." |
| **2** | **Structured inquiry**—Students investigate a teacher-presented inquiry through *a prescribed procedure*.<br>"In this investigation you will determine the relationship between temperature and the reaction rate of effervescent antacid tablets and water. You will use effervescent antacid tablets and water of varying temperatures. Using the following procedure, record the results as indicated, and answer the questions at the end of the activity." |
| **3** | **Guided inquiry**—Students investigate a teacher-presented question using *student designed/selected procedures*.<br>"Design an investigation to answer the question: What effect will water temperature have on the rate at which an effervescent antacid tablet will react? Develop each component of the investigation including a hypothesis, procedures, data analysis, and conclusions. Implement your procedure only *when it has been approved by your teacher*." |
| **4** | **Open inquiry**—Students investigate topic-related questions that are *student formulated* through *student designed/selected procedures*.<br>"Design an investigation to explore and research a chemistry topic related to the concepts we have been studying during the current unit on chemical reactions. Implement your procedure only *when it has been approved by your teacher*." |

The four-level model can be a useful tool for teaching students on a single topic. By doing this, it allows students to apply concepts it in multiple ways and therefore increasing the depth of their knowledge of that topic.  However, going through all four levels for every topic can be time consuming. This is not desirable in a typical course laboratory setting where multiple topics must be covered in a set amount of time.

## The Mini-Journal Laboratory

The mini-journal laboratory manual modifies a traditional laboratory experiment into an inquiry-based experience.[9] The format of the mini-journal is similar to scientific literature, it consists of six sections, including abstract, introduction, materials and methods, results, discussion, and references. The students read the mini-journal (which details scientific research completed by the instructor) and construct questions needed to carry out their investigation. The students then collect and interpret their data, as well as communicate the results with their own mini-journals.[18]

This style of teaching in a laboratory setting is useful because it allows students to be engaged in the five essential features of inquiry; (i) engage in scientifically oriented questions, (ii) give priority to evidence, (iii) formulate explanations from that evidence, (iv) connect those explanations to scientific knowledge, and (v) communicate and justify their explanations.[16] Going through these steps engages the students in several learning techniques and forces them to utilize their knowledge to solve problems while building their critical thinking skills. This process helps to solidify the information they learned and gives them the opportunity to solve that problem in a scientific manner.

Research has shown that mini-journal laboratory manuals may be an effective teaching method.[18,19] It fosters student engagement in the topic while approaching it from a scientific point of view. However, it has been noted that mini-journal laboratory experiments can be difficult for the students. The transition from traditional labs to the mini-journal labs does not happen immediately and can be frustrating for students. Regardless of it being challenging in terms of transition, students enjoyed the mini-journal format and actually prefer it to the traditional experiment style.[6]

## The Learning Cycle

While inquiry-based learning has proven to be an effective teaching/learning tool, it does have its disadvantages.[20-22] The process can be time consuming to develop activities/experiments and students are required to put in more work than a traditional method, something some students are not willing to do. To get students interested in participating in inquiry-based learning, they must be engaged and excited about the experiment they are going to perform.[22] This can be done by designing a good inquiry-based experiment using the Learning Cycle approach. The first step in designing an inquiry-based problem is identifying the concept/principle that is the target of the experiment and then to write a problem statement/question to help engage the student's interest. Next write or let students develop a method of analysis that will be beneficial in collecting the appropriate data. After collecting the data, students should be encouraged to organize the data into a format, *i.e.* tables/figures, which will aid in the data interpretation process. Finally, the instructor asks questions that will lead the students to develop the target concept.[23]

In the Learning Cycle approach, it is desired that students develop their understanding of the concept based on what they observed during the experimentation process and their conclusions. To aid in this process, it is advised that pre-laboratory activities/lectures should be limited to instruction of safety in the laboratory and skills needed to complete the activity. It should not let the students know the concept they will learn, how to conduct the experiment, what they are going to observe, or what they should conclude about the experiment. It is the student's responsibility to figure out these things on their own, which will in turn help facilitate their learning of the material.[23]

It may be difficult and frustrating for students to figure out things on their own. It is the responsibility of the instructor to act as a facilitator to aid them in the learning process. It is important for the instructor to have a good attitude about the Learning Cycle experiments; the instructor's attitude directly affects students' attitudes.[24] To help instructors have a good attitude, they must be trained in inquiry-based teaching and feel comfortable with guiding students in these types of experiments.[25] If instructors are not trained, then it can be frustrating for the teacher and the students, and students are likely to develop wrong conclusions by inefficient strategies used to discover information.[26] For instance, if an instructor isn't familiar with successful problem solving techniques and inefficient in getting a good representation of the problem the students are attempting to solve, students may get confused and formulate wrong conclusions. It is essential for the trained instructor to develop the right questions to ask students about the experiment they conducted to help guide them in their understanding the material and to the correct conclusions.[25] Questions should be used that will guide the students to reflect on the experiment they completed and what the results mean. Questions like "*What did you do?"*, "*What did you observe?"*, and "*What does it mean?"* will cause students to focus on the experiment, what happened, and relate the data to the target concept. The inquiry tactic of using questions is one of the best instructional tactics for concept learning.[23]

The Five E's. Another form of the Learning Cycle is the Five E's, engage, explore, explain, elaborate, and evaluate.[22] The students must be Engaged, where experiment design will help promote curiosity and engage their attention, and to elicit their preconceptions. The students are encouraged to Explore, which will help guide students to collect and process the meaning of data. Then, the students are to Explain what they have learned through the exploration process. In order for this phase to be successful, the teacher/instructor cannot explain for them; instead their primary role is to provide small hints and provide guiding questions to help them construct their own knowledge and reasoning. After explaining, students are to Elaborate, where they will relate what they just learned to what they previously knew about the subject. This conceptual reorganization is essential in transitioning between understanding a concept and the ability to perform intellectual operations with that concept, leaving them being able to use their new understanding to solve novel problems. Lastly, students must Evaluate their learning and conceptual understanding. It is suggested that this can be accomplished following laboratory experiments with a traditional laboratory report so that students can assess their own learning during the writing process. In the instances of using exams, instead of giving lower-order, algorithmic questions, it may be better to let students evaluate their knowledge by giving higher-order, conceptual questions.[27]

When using the Five E's for laboratory experiments, it is important for the instructor to not explain to the student, but instead offer questions to help stimulate their understanding of the concept.[22] This process can be exasperating for students if they are not able to figure out the solution with the guiding questions given by the instructor.[23] Furthermore, the evaluating that takes place in a laboratory report is essential in student learning, but it is labor intensive for students to write long reports every week; this added onto the stress of dealing with other classes and can lead to student frustration.

Team Learning

While students can learn by working as an individual to gain knowledge in a subject, they can also learn by working in teams to accomplish a common goal.[15] Team learning can complement and strengthen pre-laboratory lectures and traditional experiments to help students gain more insight into the topic.[27] In teams, students are required to negotiate meaning and understanding in a discussion with their peers, leaving them with the ability to develop their knowledge and form conceptual understanding of the material.[28]

Furthermore, in the chemical industry or academic environment, scientists rarely work as an individual, rather they must be able to work within a group setting with individual responsibilities while working towards a common goal. Being able to work in a group, for students, will not only enable their learning in chemistry, but will also allow them to gain valuable skills while working within a group; such as, how to communicate among peers/colleagues with their ideas, how to be a part of a peer review process to critique and analyze a process, data, or concepts to be more productive for the team's common goal.

There are four areas that contribute to students being able to learn in teams. The first is group learning (collaborative and cooperative learning methods). Collaborative learning implies that knowledge is established through the interaction between people that involves prior knowledge; it results in the building of "critical thinking, problem solving, sense making, and personal transformation, the social construction of knowledge-exploration, discussion, debate and criticism of ideas."[29] Team learning incorporates cooperative learning aspects such as positive interdependence and face-to-face interactions, but it is necessary to ensure that there is a trained team leader who can hold each team member accountable and can facilitate decision making.[30]

The second area that aids in team learning is reciprocal teaching and explanatory knowledge.[31] In reciprocal teaching, tasks are created by examining and analyzing the strategies used by successful learners and then they are utilized to guide novices in the learning process. The explanatory knowledge is simply that students reflect on their learning with the group which develops communication skills as a part of their understanding. This shows that if students discuss the basis for their thoughts and reasoning, rather than memorizing the answers, they will create a deeper understanding of the material that establishes true learning.[32]

The third area that is critical to team learning is based on the Zone of Proximal Development (ZPD) from Lev Vygotsky.[33] ZPD simply explains that as a student learns, the level of challenge and the level of competence of that student must fall within the ZPD, so that the student will not become anxious (too challenging/difficult for the current abilities of the student) or bored (not challenging enough for their current abilities) as given in Figure 1. Given this, tasks must be designed to press the learner to a reasonable expectation of achievement based on their existing understanding of the subject.

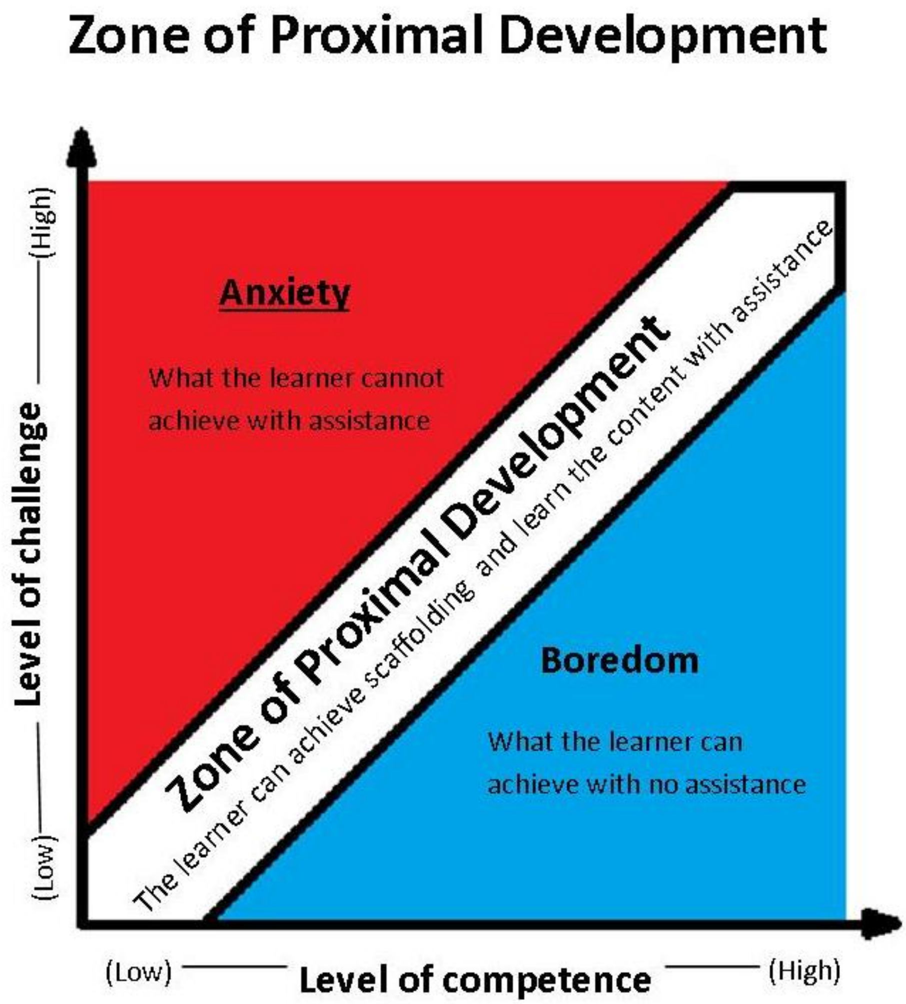


**Figure 1.** Diagram showing the Zone of Proximal Development, adapted from Lev Vygotsky.[34]

The fourth area to assist in team learning is studio instruction; this is where students must generate materials that represent their learning.[35] In a laboratory setting, this could be the procedure developed to analyze a compound, the experimental results obtained, and/or a laboratory report explaining their reasoning and data, basically anything that represents products of learning. If the students are completing a problem solving experiment, their solutions to that problem can even serve as a product of learning. These products of learning represent the students' understanding, which is then the object of study for the group, where they can critique and peer review, and ultimately learn from a collective understanding of the material.[36]

When designing a team learning experience for students, it is essential to make sure certain aspects are successfully integrated into the instruction/learning experience that will contribute to the student's learning.[36] The students must participate in the team rather than work through the experiment as individuals; they must see that being a part of the team is an integral part of the experiment, not as an option. The experiment must be challenging for the students; so, it will encourage students to work together and create an active learning experience. The instructor of the course, that assists with leading the groups, must be well trained and have knowledge of the material and guide the students in the learning process.[24] With these components in a well-designed team learning experiment, students will have a positive attitude towards the experience and it will increase their success in learning conceptual knowledge of the material.[31, 36]

## Problem Solving

When designing experiments, whether in team learning or individual learning, students are more excited and engaged in the experiment if there is a clearly defined goal.[37] One way of accomplishing this is to set up a problem solving experiment; these types of experiments allow students to see how content knowledge can be coupled with thinking strategies and give students the opportunity to increase their knowledge of the subject through problem-based learning (PBL). Through these problem solving situations, students get to use their current understanding to solve a problem which mirrors encounters they could experience during their professional careers.

Most of the problems students encounter in typical laboratory experiments are well-defined.[38] The initial conditions, the goals, the procedure for obtaining and evaluating the data, and all aspects of the experiments are clearly specified for the student. However, this type of experiment is not representative of what they will encounter after graduation. In contrast, problem solving experiments are usually less-defined, where the means of solving the problem is unknown and must be discovered using critical thinking skills; the ultimate goal for the instructor is for the students to advance towards a more expert-like understanding of the material as well as how to solve a problem.[39]

It is also important to be able to identify the general problem solving approaches and characteristics of successful problem solvers. People who are successful at solving problems tend to have a strong realization of basic facts and principles, can develop appropriate representations, have the ability to make logical connections among the different elements of the problem, and can verify the process at multiple points during the problem solving.[40, 41] Students tend to memorize the relevant specific information concerning the problem and try to apply it to the task, rather than applying more general skills they have been taught to make connections among the elements of the problem.[42]

It is the instructor's responsibility to help students have the proper understanding of how to solve a problem. This includes making sure that they understand and have a good representation of the problem they are attempting to solve and a good solution method. After they understand what problem solving concepts that are used by experts, they will then have the ability to use that knowledge to develop a plan to solve problems they will encounter in their future careers.[39]

## Problems to be Addressed

Inquiry-based learning involves students in the topic that is being studied and allows them to use their knowledge of the subject to complete the material. On the other hand, traditional laboratory style teaching does not require much knowledge other than knowing how to follow a procedure and fill out a piece of paper with the results; absolutely no scientific thought process or investigation has to be practiced in this type of method. Traditional experiments do not involve students in the learning process that is essential for them to build a foundation for their problem-solving skills.

In analytical chemistry laboratory courses, it is essential to build a proper foundation of research methods, problem-solving skills, and quantitative techniques. The traditional format of laboratory teaching only allows the development of quantitative techniques. Although, quantitative techniques are essential for executing a designed experiment, they are only a small part of the knowledge required to conduct research in the chemical industry.

If analytical laboratory classes were designed in an inquiry-based teaching format, students would be able to learn how to conduct scientific research while developing their problem-solving skills. However, this format does not leave any room for teaching any necessary quantitative techniques. Using the four-level model, an instructor could teach students quantitative techniques in the topic wanted for the inquiry-based learning at the first level and then work their way up to the more research-based level four. Given that this tactic can be time consuming and could take several class sessions to accomplish, it is not realistic for analytical laboratory courses in which several topics need to be covered to develop the students' analytical skills for post-graduation.

Since there are valuable skills to learn in both traditional and inquiry-based teaching, both types need to be taught to students. They could both be taught in a single laboratory course, which would provide the students with the opportunity to learn quantitative techniques and how to conduct research, while developing their problem-solving skills. Considering the findings from mini-journal research, students have a difficult time transitioning from traditional "cook-book" to inquiry-based experiments.[18,19] This occurs because students are used to the traditional format from previous chemistry laboratory experiences. Going from this type of procedural based experiment to not being able to have a step-by-step procedure can be intimidating and troublesome for them.[18] In order to successfully teach students all the skills required to make a good chemist, there must be a transition from traditional to inquiry-based teaching that can be implemented in analytical chemistry laboratory courses to relieve student frustration and increase their conceptual learning.

## Chemical Education Research Studies

When constructing a research project in chemical education, there are three fundamentals that must be considered in the initial stages of the research development process. A good research study must have a guiding research question which will direct the focus of the research, a theoretical framework upon which the study will be built, and a methodological framework that helps to investigate the guiding research questions. Each of these stages is discussed below.

### Guiding Question

Before deciding on a theoretical framework, a researcher must develop a guiding question for the research study to try to answer. In the process of developing a good guiding research question, it is important to frame the inquiry so that it addresses the central issue that is driving the investigation. First, the problem must be identified and the needs that are to be addressed. It must take into account the situation in which there is a problem, how that problem can be addressed, and if the problem is important enough to try to answer it through research. In other words, what is the problem and is it worth asking? To help find a worthwhile problem, it is suggested that the researcher think about ideas they are interested in, what aspects they would like to investigate, and if research on this topic would provide meaningful data not only to the researcher but also to the chemical education community.[43, 44]

In the process of refining the guiding question, it is essential to incorporate the variables that will be addressed and how they will be measured. It is better to collect data using tools/methods that have already been proven to be reliable and validated. So, it is imperative to choose a method that addresses the desired variable and provides data that pertains to the research question. If there is not a tool that will address the research question, then one must be developed, proven to be reliable, and validated before it can be used to collect data for the research. Another aspect of a good research question is ensuring that the researcher has a potential "take home" message in mind. In other words, the researcher must have an idea of what results are to be expected; this way, when the outcome of the research is stated, it clearly answers the research question. If the research study has the potential to provide this type of result that correlates to the original research question, then the researcher has developed a good researchable question.[43,44]

Theoretical Framework – Constructivism

During the process of developing a good guiding research question, it is important to choose an appropriate theoretical framework that will help address the guiding questions from the proper perspective.[43, 45] Many research studies in chemical education, that focus how students learn, concentrate on the theoretical framework called constructivism. Constructivism focuses on students making sense of the experiences they encounter in the study and how they learn during the process.[46] This is based on the idea that new knowledge is not discovered, instead it is actively constructed from concepts and models to make sense of the information presented. Furthermore, this knowledge can then be altered and modified based on new experiences. Constructivism heavily relies on the process by which students make sense of their experiences. The best way to measure the student's ability to make sense of what they encounter is not only from observations, but for the researcher to discuss the observations with the individual students to get their perspective on what they have learned.[46]

Constructivism has general assumptions about how people construct their knowledge. One is that individual, social, and/or cultural interactions play a role in knowledge construction. Another is the learning construction and the language used in that construction process must be useful, practical, and adaptive. With this, the learning and language serve to bring coherency to the individual's experiences and the knowledge base of the community.[47] Since individuals are always facing new experiences, it is inferred that even though knowledge has been constructed, it is constantly being altered and reconstructed from their previous knowledge and their new experiences. These qualities of constructivism are what make it an ideal framework to use for making changes to a current curriculum that is interested in seeing how students construct their knowledge and to use that knowledge in new situations; this makes it a good framework for the curriculum change that progresses from traditional experiments to a more inquiry-based course.

## Methodologies

Since constructivism deals with how knowledge is constructed and reconstructed, it is essential to ensure that the guided questions concerning the focus of the research relate to how students make sense of phenomena or concepts. In turn, it is also important to choose an appropriate methodology; this means that the strategy, plan of action, and the design of the course (use of particular methods and/or experiments) are consistent with the correct methodology that will address the problem being researched.[46] In order for the constructivism framework to be successful, it is imperative that the research is designed to aid in the understanding of concepts by the learners and a methodology is chosen that allows the researcher to reconstruct the cognitive process that the learner used to understand those concepts.[46]

Qualitative Methods. A useful method in deciphering how students construct their knowledge is qualitative methodology. This type of method is useful in providing descriptive data and gives details into a student's experience in the research. Qualitative data offers details and descriptions of what takes place in the study, so that the researcher can read and interpret the meaning, then to come to their own conclusions about the study. Some examples of qualitative methods involve interviews, observations, and/or document analysis to get insight into the student's perspective.[48]

Interviews are an important dialectic methodology that can help the researcher gain insight into how the student defines their own experience and how they feel about that experience. It is essential in interviews that all students are asked the same set of questions and the interviewer gives prompts for elaboration of details and examples of the student's personal views and ideas. The interviewer needs to have active listening skills and be able to ask questions in a way to encourage the interviewee to elaborate with examples and to think out aloud. This can help researchers gain awareness to how students process information, how they made sense of the material presented to them, and how they can use their knowledge on new ideas. [48]

When using the dialectic methodology, it is important to design a data collection strategy to get the most useful information out of the research study. The researcher can interview the student about instances, where students are given different choices/occasions and then they must decide which ones relate to the desired concept. This can be useful to see if they can recognize a concept out of the context from which they originally learned it. Students can also be interviewed about events; in doing so, they are given a series of events through demonstration, then they must interpret what they saw and provide an explanation for their observation. This type of interview allows the researcher to see if the students can use their knowledge to recognize concepts without being told that concept was involved in the demonstration. The think-aloud protocol involves the researcher giving a problem for the student to solve; this can be useful in seeing how they can apply their conceptual knowledge to new situations. All of these techniques activate the concepts and stimulate a conversation between the researcher and the student.[46, 48]

Observations can be used in qualitative research studies to help the researcher see how different students interpret the same situation. Observations can provide valuable information for researchers to understand the influence the setting has on how students are able to construct learning. All students may be in the same course, but they will all have different experiences based on how they interpret data and make sense of it. Observations can be used to gain insight into their individual experiences in the classroom.[48]

One of the other common ways of collecting data in qualitative research is document analysis. Some examples of the types of documents that can be analyzed include laboratory experiment reports and surveys.  These types of documents allow for researchers to get more perspective into how students are able to interpret material presented to them, how they feel about their own experiences, and their attitude towards the learning process. In particular, it can be insightful to employ surveys to obtain this type of qualitative information. There are several different types of surveys and one should be chosen to fit the needs of the research study. If there is not one that fits the needs of the research, then one can be developed that will measure the desired variables; it is important to note that the survey must be tested to give reliable and valid results. If time is an issue in the research study, it may be desirable for a researcher to adapt an existing survey instrument to fit the needs of their research.[48]

Using surveys can provide valuable data to the researcher, but caution should be taken depending on the type of survey chosen. For instance, self-reported learning surveys, like Student Assessment of Learning Gains (SALG), are chosen where students can give an account on how they feel about their own learning. This can be useful to see students attitudes towards their own learning experience, but students might think that they are greatly increasing their knowledge because of a new instructional tactic while, in reality, they may be not learning. Just because students believe they are increasing their knowledge of something does not mean that they are indeed learning more. Using this qualitative methodology along with the other methodologies may provide more convincing and accurate results.[48, 49]

There are several important factors which must be considered when developing a survey. It is crucial that the survey questions are worded correctly, with proper use of figures, and rating scales. To begin the survey development, the designer must decide if it is appropriate to pool the survey items together to get an overall measurement for the benefit of the research study. Pooling can be used in situations to increase precision of data collection, but should only be used if reliability or validity requirements are met with the results.[50]

Special attention must also be paid to the details in the survey. If there is a figure or chart as part of any question in the survey, then it must be easy for the students to read and understand what it represents. Concerning the text of the questions, they must be easy to read and to understand as well. The language used in the questions must be at the reading and vocabulary level of the students the survey was designed for. The designer must use caution with certain words that may confuse students or leave the question to where it is hard to answer. For example, questions that use "and" or "or" can be difficult for students to answer. If the survey question was "I increased my knowledge of the subject by using the laboratory manual and online journal articles.", then the question could be confusing and hard to answer for students that learn from one source but did not learn anything from the other. Furthermore, it is essential that questions are short and simple for easy understanding. If questions are too long, then it can make it difficult for students to process and answer correctly. In the same respect, if the overall test/survey is too long, then students may become fatigued and frustrated. It is better to have a concise test/survey with simple, straightforward questions to get more valuable perspectives from students.[50]

If the survey designer wants to have questions/statements that students must answer using a rating scale, then the scale must be easy to understand. For instance, if the scale was "Very

Important", "Important", "Unimportant", and "Very Unimportant", students may get confused and have a difficult time answering the questions. Something like "Very Unimportant" is not a common phrase for people to be familiar with; therefore, it will be hard for students to understand and pick an accurate answer. Another point to consider in a rating scale is the use of options like "Not applicable" or """Neither"; options like these do not force students to analyze the question being asked and will make it easy for them to answer with this type of option out of convenience and not put much thought into the survey. Another way of keeping students focused on a survey and keeping them focused is to have "wake up" questions. This can be accomplished by placing a question that will force them to carefully read it, like "No matter what, answer Strongly Agree to this statement" or "Circle the page number at the top of this page". Statements/questions like these can be placed during the point of a survey that students may get fatigued, or somewhere in the survey just to see if they are actually paying attention to the questions.[50]

Quantitative Methods. In general, more meaningful and convincing results are collected if more than one type of methodology instrument is used in a study, *i.e.* interviews and surveys. If it is appropriate for the research study, the researcher could choose to use quantitative methodologies as well. The data for quantitative methods are usually systematic, standardized, and easily presented, likely in a tabular or graphical format; an example of quantitative methods is to use exams or laboratory experiment grades to measure student performance. This makes it easy for the researcher to examine the data for trends and correlations between variables. It is essential to have a control group and a treatment group; for example, one group may be taught using the new instructional method, while the control group is taught using the current instructional method. When using quantitative data, it is important to remember that the method may provide data, but that data is the result of many contributing factors, so it can be hard to determine the effect of the variable that the research may be studying.[51]

In quantitative methods of research, it may be easier for the researcher to state a null hypothesis (an opposite hypothesis). This is commonly done in statistical analysis. For example, the hypothesis may be that a new instructional tactic will increase students' conceptual learning, while the null hypothesis may be that the new instructional tactic has no effect or did not decrease the student's conceptual learning. Since there are many factors that contribute to students' learning, it can be hard to prove that one variable helps. Instead it may be easier for the research to show that the new variable did not decrease the learning of the student. If the null hypothesis (new instructional tactic had no effect or did not decrease learning) is rejected, then the research is considered to support the hypothesis (new instructional tactic increased student learning). On the other hand if the null hypothesis is proven to be correct, then the research does not support the hypothesis.[51]

## Drawing Conclusions in Chemical Education Research

After the all data has been collected, using reliable and validated research instruments, conclusions must be drawn that attempt to answer the guiding research question. However, caution must be taken when going through the data in an attempt to deduce some meaningful results. One common mistake is that the wrong causality can be assumed when trying to decipher the data. For example, it might appear that a new instructional tactic led to the increases of students' knowledge, when in reality there are many factors that can aid in students' learning. An example of this would be that one laboratory section was taught using a new instructional tactic, while the other laboratory sections were not. Students in this “new instructional tactic” laboratory section met every week to study the material for the course, while the other laboratory sections students did not. By doing this, the “new instructional tactic” laboratory students ended up knowing the material better and scored higher on the final exam than the other laboratory sections. Without knowing that these students had study groups every week, it would be easy to assume that the instructional tactics used in that laboratory section was the cause of higher exam scores. These like factors must be ruled out, minimized, and/or controlled. Another common mistake is related to the self-reported learning surveys. As mentioned previously, this type of instrument cannot be used on its own due to the validity of stating its results as facts, *i.e.* just because students feel like they have learned more, does not mean that they really have. Easy misinterpretations may happen, but if careful attention is paid to processing the data, then it can avoid these types of pitfalls that can happen while drawing conclusions.[52]

## Research Aims

Determine if the transition from traditional “step-by-step” to inquiry-based experiments in quantitative analysis laboratory increases the student’s conceptual understanding of chemistry, to where they can apply analytical techniques to solve related real-world problems.

# CHAPTER 2

# EXPERIMENTAL PROCEDURES

To accomplish the research goal of a successful transition from traditional to inquiry-based teaching in analytical chemistry laboratory course, first a course was chosen to alter the laboratory curriculum. Of the two major analytical courses offered at most universities, quantitative and instrumental, the quantitative was chosen for the research study. The study was dependent on three components: chemical education aspects in designing the transitional curriculum/laboratory manual, the qualitative and quantitative methodologies used to collect student data, and development of the analytical components of the experiments.

## Chemical Education Aspects

The following question was developed and served as the guiding question of the research study:

> Does the transition from traditional to inquiry-based experiments in quantitative analysis laboratory increase the student's conceptual understanding of chemistry, to where they can apply analytical techniques to solve related real-world problems?

As the goals of the research were to design a curriculum that focuses on how students understand concepts and apply it to solve problems in quantitative chemistry, it was decided that the study should use a constructivism theoretical framework. This framework is useful in studies for seeing how students interpret the material presented to them, alter and use it whenever they are faced with problem solving experiments, and to discuss how they came to their conclusions to get their perspective on the learning process.

Some aspects of the research had variables that could not be controlled by the researcher, but may have an effect on the research results, like causality effects. The effects of causality that were foreseen are students having different lecture instructors, different laboratory instructors (getting different pre-laboratory lectures, etc.), study groups outside of class, and prior research background. The causality of different lecture and/or laboratory instructors was eliminated because there was only one lecture instructor and the control group and test group were taught by the same laboratory instructor. The potential of study groups outside of class could not be eliminated or minimized; if students wanted to get together to study, it was considered good that they wanted to learn. Notes were made about who usually studied with one another to see if those in the group created a trend during the data analysis process. As for prior laboratory background, this factor could not be changed either, but was recorded; this way, if those students outperform the others, this factor could be considered to attribute to their success and not necessarily due to the new laboratory manual.

As this study has variables which cannot be completely eliminated, the research study utilized a null hypothesis. The hypothesis was

> The transition from traditional "cookbook" to inquiry-based experiments in quantitative analysis laboratory does increase the student's conceptual understanding of chemistry. The students were able to successfully apply analytical techniques to solve related real-world problems.

There were two null hypotheses:

> The transition from traditional "cookbook" to inquiry-based experiments in quantitative analysis laboratory decreases or has no effect on the student's conceptual understanding of chemistry.

> The students were not able to apply analytical techniques to solve related real-world problems.

Since, the research involves two aspects, learning analytical chemistry concepts and then being able to apply them to solve problems, there will be two null hypotheses. If the research accepts both of the null hypotheses to be true, then the hypothesis will be considered to be incorrect. If only one of the null hypotheses are rejected and the other true, then the hypothesis will still be incorrect. Only when both null hypotheses are rejected, the hypothesis will be considered valid. In any of the three cases, meaningful conclusions can be drawn from the research that will add to the knowledge of teaching and learning in chemical education.

Design of the Quantitative Laboratory Manual

In a laboratory setting, students can learn the basic hands-on that involves laboratory techniques, the underlying concepts involved in the experiment, and the ability to apply their knowledge to solve problems. It was decided that it was desirable for students to learn both the laboratory techniques required to physically complete the experiment and the concepts involved in the experiment; this way, they may be available to use all of their knowledge to complete problem solving experiments where they must develop their own method of analysis to solve it.

Instead of developing a completely new laboratory manual, the current manual was altered to become a transitional curriculum. The laboratory manual had eight experiments which mostly consisted of different types of titrations. Of those experiments, one was eliminated due to the undesirable use of mercury chloride ("Analysis of Iron Oxide Ore Using Dichromate") and the other was removed for being too lengthy for most students to complete during allotted class time ("Determination of Copper in Copper Oxide Samples by Iodometric Titration"). The remaining six experiments were employed in the new transitional laboratory manual, see Table 2.

**Table 2.** List of experiments from the current manual and their use in the new manual.

| Experiment | Experiment Title | Kept or Discarded |
|---|---|---|
| 1 | Analysis of Impure Potassium Hydrogen Phthalate | Kept |
| 2 | Gravimetric Determination of Chloride | Kept |
| 3 | Analysis of Iron Oxide Ore Using Dichromate | Discarded |
| 4 | Determination of the Purity of Soda Ash | Kept |
| 5 | Determination of Copper in Copper Oxide Samples by Iodometric Titration | Discarded |
| 6 | Determination of Acetic Acid in Commercial Vinegar by Potentiometric Titration | Kept |
| 7 | Spectrophotometric Determination of Iron Via Its 1,10-Phenanthroline Complex | Kept |
| 8 | Determination of Fluoride Using a Fluoride Ion Selective Electrode | Kept |

The current laboratory manual had a few weeks at the end of the semester for students to complete make-up labs. This was reduced so that more experiments could be added to the transitional laboratory manual. In total, it was decided that the new manual could have ten experiments, instead of eight like the current manual. One traditional experiment was added, to both the current and new laboratory manual, as a beginning experiment to teach students basic laboratory techniques that they may not have encountered previously, i.e. the use of volumetric glassware and use of analytical balances.

Rearranging and Altering of Old Experiments. The order of experiments was rearranged to build upon previously learned techniques. The first four experiments were traditional experiments in respect to the basic format of introduction of concepts and development of quantitative techniques. The first experiment, "Volumetric Glassware Analysis", was taught as a confirmation level of inquiry. The other three traditional experiments, "Gravimetric Determination of Chloride", "Analysis of Impure Potassium Hydrogen Phthalate", and "Determination of the Purity of Soda Ash", were scheduled based on their structured level of inquiry.

The remaining structured experiments were altered to present problem solving situations where the quantitative techniques were presented. These experiments were set up as problems to solve to introduce students to PBL experiments and give them the opportunity to see how the previous concepts were applicable and the techniques used to solve them. Additionally, while many of the students in this course were chemistry majors, several were majors in other science fields; so, the "problem to be solved" section of the experiments were written to have different themes (environmental biology, forensics, pharmaceuticals, etc.) to relate to many scientific fields. This decision was made to help excite and Engage (Five E's) the students in the learning of the concepts in the experiments. Also, it was designed to help lay the ground work for the students to follow in learning how to read a "problem to be solved", what basic facts and principles relate to the problem, how the problem can be represented to be easily understood, and how to relate different concepts used to solve the problem; these steps were outlined during the pre-laboratory lecture given to students by the laboratory instructor to help them see the problem solving process.

The PBL experiments were designed to ensure that the students always fell within the ZPD and could successfully solve the problem without feeling overwhelmed and anxious about the learning process. To accomplish this, the beginning PBL experiments were more well-defined and gave students most of the procedure to Explore (Five E's) the problem. Progressively through the experiments/semester, the experiments became less-defined (fewer instructions) and the students were required to provide more information while Exploring to complete the assignments rather than being provided all the steps. This allowed students to apply knowledge from previous experiments to solve some aspects of the experiments they were working on. Basically, if the students were required to use a technique more than one time in the laboratory manual, they only received directions/procedure for the first time and had to rely upon their understanding of the concept for later uses; this ensured that students were challenged to use their current abilities to solve the problem they were facing and fell within their ZPD.

The PBL was also reflected in the pre-laboratory lecture by the instructor. During pre-laboratory lecture for the more well-defined experiments, the instructor read the "problem to be solved", described the key points for the students to focus on, and the concepts involved in addressing the problem. This reciprocal teaching was used to ensure the students had a good representation of the problem, what concepts were needed to solve it, and to guide them in the learning process. As the experiments went from well-defined to less-defined, it was also reflected in the pre-laboratory lecture. The "problem to be solved" was still read aloud but instructor did not discuss the key points and concepts involved; instead, students were left to reflect on the problem, decide what concepts were involved, and develop a plan based on their understanding of how to solve a problem. This allowed students to operate in two of the upper levels of Bloom's taxonomy, application (apply knowledge to solve problems through designing and experimenting) and synthesis (use concepts to create/design new ideas). If students had difficulty with the problem solving process, the instructor did not provide answers but gave them guiding questions to help them develop the answer on their own.

Addition of New Experiments. The last two experiments, nine and ten, in the transitional curriculum were less-defined PBL and only gave a problem for the students to solve with no procedure; these two experiments were guided levels of inquiry. For the students to complete the final experiments, they had to apply their analytical conceptual knowledge to new situations and use their critical thinking skills to develop a method of analysis. Both experiments paralleled the mini-journal experiments; the students were given journal articles and other reading materials relating to the experiments. These sources contained information about how those types of problems had been investigated previously. The sources were chosen based on the college reading level that the students could or should be able to read and interpret their meaning. However, none of the sources were a single answer on how to solve the problem; to solve the problem, students would need to be able to piece parts of the sources together or alter a single one to fit their needs. In addition to this, students were allowed to look at other sources, including but not limited to their laboratory manual, print journals, and online articles.

Of these two method development experiments, the ninth experiment was designed to be a team learning experience. Students were divided into groups and given a problem to be solved with no given procedure. While in groups, students could collaborate and draw upon the knowledge they gained previously in the course, as well as other resources, to develop the method of analysis to solve the current problem. This gave students the opportunity to learn through explanatory knowledge, be able to discuss the basis for their thoughts and reasoning with their partner, and create a deeper understanding of the material. It was essential to ensure that each group cooperated with their laboratory partner and transformed into a team, where they would develop a mutual trust between one another, commit to each other in accomplishing the task, accept accountability, and provide the proper attention to obtain quality results. To aid in this, three of the earlier experiments were completed as group experiments instead of individually. While the instructor could not guarantee each group would be successful, the instructor of the course was well versed in the subject matter of the experiment and had experience in problem solving and team-based learning to help guide the teams in their ability to solve the problem.

The tenth experiment was an independent method development experiment. From the team learning experiment, it was anticipated that students would develop the skills to analyze a problem and the steps they must take in order to solve it. By having the last experiment completed individually, it gave the students the opportunity to use their conceptual knowledge of the analytical techniques used in prior experiments, but it also allowed students to use their critical thinking skills to analyze the problem and how it would be best to solve it.

For both of these inquiry-based method development experiments, the students were required to turn in an outline of their proposed method. This served as a part of the studio instruction of the course, giving students the opportunity generate a material that represented their learning. For Experiment 9 it represented their collective understanding of the problem through peer review and critiquing with their laboratory partner. This outline was due two weeks in advance of the date was to be performed. This gave the instructor one week to review the outline, check its plausibility and any safety concerns, and draw attention to any areas that the students might still need to work on. In doing this, it was important for the instructor to only guide them to the correct solutions, not tell them the answers. The outlines were then returned, leaving students one week to make any necessary changes before coming back the next week to complete their experiment. While it would have been ideal for students to turn their outlines in earlier than this, it was not feasible given that students were still learning techniques that could be employed on the last two experiments. Furthermore, students were not only encouraged to make any changes they wanted during the week leading up to the experiment, but they were also encouraged to make any changes during the experimentation process. This was reflective of real-world situations where problems are ran into during the method development process and changes are made to account for unforeseen circumstances.

For both of the last two experiments, students were required to write formal scientific reports to Explain, Elaborate and Evaluate (Five E's) their developed methods; this was another form of studio instruction that had shown how they solved their problem and their reasoning in interpreting the data. In these reports, students were also encouraged to include information about how they thought their experiment would go, how it actually went, and then if they could make any changes for a second chance at it, what they would do differently. This was to determine if the students really had a good understanding of the concepts used in their experiment, determine if they were carried out correctly, and if there were any flaws in their previous analysis. The formal report also allowed students opportunity to operate in the upper level of Bloom's taxonomy, analysis (identify trends in their data and organize their ideas) and evaluation (assess theories/concepts and evaluating outcomes).

While it was hoped that the last two experiments would aid the students learning of analytical concepts, it was decided to also make them accountable for knowing the concepts before they attempted the last two experiments. This was accomplished by having students submit a summary report each week, in addition to just reporting their results in an Excel data sheet. In these reports students were required to Explain and Elaborate (Five E's) the concepts involved in the experiments and Evaluate their results. The summary reports served as another form of studio learning.

With the addition of the first, ninth, and tenth experiment into the new transitional laboratory manual, it left room for one more experiment to be added. Given these experiments, students were introduced to gravimetric analysis, acid-base titrations via color indicators and potentiometry, ion-selective electrodes, and UV-Vis spectrometry. It was decided to add additional instrumentation and concepts for the students to potentially employ in their method development experiments. Ultimately, the decision was made to develop an experiment that involved Atomic Absorption Spectrometry (AAS) and Method of Standard Addition (MSA), both being valuable techniques in analytical chemistry. Since it was a later experiment in the laboratory manual, the decision was made to include very few procedural details.

Since the new laboratory manual was designed to transition them to the ending experiments, it is essential that the students learn several techniques and concepts throughout the semester that are necessary for later experiments. It was hoped that by designing experiments to start with conformation and structured level of inquiry experiments, students would be forced to learn the underlying concepts that they would need on the guided inquiry experiments. Table 3 shows each experiment in the new transitional laboratory manual, what concepts students need to know before beginning the experiment, what concepts they learned because of the experiment, and level of the inquiry for the experiment.

**Table 3.** List of experiments, concepts needed, concepts learned, and level of inquiry.

| Experiment | Title | Concepts needed | Concepts learned | Type of Inquiry |
|---|---|---|---|---|
| 1 | Volumetric Glassware Analysis - Pipetting | - None | - How to use volumetric pipets/glassware<br>- When it is appropriate to use certain types of volume delivery glassware<br>- Accuracy and precision of glassware | Confirmation |
| 2 | Gravimetric Determination of Chloride | - None | - Gravimetric analysis | Structured |
| 3 | Analysis of Impure Potassium Hydrogen Phthalate | - When it is appropriate to use certain types of volume delivery glassware | - How to use/read a buret<br>- Acid/base titration<br>- Standardization | Structured |
| 4 | Determination of the Purity of Soda Ash | - How to use/read a buret<br>- How to use volumetric pipets/glassware<br>- Acid/base titration<br>- Standardization | - Polyprotic acid/base titration | Structured |
| 5 | Determination of Acetic Acid in Commercial Vinegar by Potentiometric Titration | - How to use/read a buret<br>- How to use volumetric pipets/glassware<br>- Acid/base titration<br>- Standardization | - Potentiometric titration<br>- Accuracy and precision of using potentiometry | Structured |

**Table 3** (Continued)

| Experiment | Title | Concepts needed | Concepts learned | Type of Inquiry |
|---|---|---|---|---|
| 6 | Determination of Fluoride Using a Fluoride Ion Selective Electrode | - How to use volumetric pipets/glassware<br>- How to use/read a buret<br>- When it is appropriate to use certain types of volume delivery glassware | - Calibration Curve<br>- Ion Selective Electrodes<br>- Linear dynamic range | Structured |
| 7 | Spectrophotometric Determination of Iron Via Its 1,10-Phenanthroline Complex | - How to use volumetric pipets/glassware<br>- How to use/read a buret<br>- When it is appropriate to use certain types of volume delivery glassware<br>- Calibration Curve<br>- Linear Dynamic Range | - UV-Vis Spectrometry<br>-Instrument Detection limit<br>-How construct calibration curve, determine standard solution concentrations, based on detection limit and linear range of instrument | Structured |
| 8 | Determination of Zinc using Atomic Absorption Spectroscopy | -Determine spike concentrations based on detection limit and linear range of instrument | - Method of Standard Addition<br>- Atomic Absorption Spectrometry (AAS) | Structured |
| 9 | Group Method Development Experiment | - Calibration Curve<br>- How construct calibration curve, determine standard solution concentrations based on detection limit and linear range of instrument<br>- UV-vis or AAS | - How to develop a method in a group setting | Guided |
| 10 | Independent Method Development Experiment | - Calibration Curve<br>- How construct calibration curve, determine standard solution concentrations based on detection limit and linear range of instrument<br>-UV-vis or AAS<br>- Titrations | - How to develop a method independently | Guided |

Current Manual Versus New Transition Manual. To see if a transitional curriculum was essential for students to be able to successfully complete an independent method development experiment, it was decided to employ a control group. For the control group, the students were given the current laboratory manual with some alterations. To see if the transition from traditional to inquiry-based experiments were necessary, the control group had the same experiment 10, Independent Method Development, as the test group.

From the current manual, the Analysis of Iron Oxide Ore Using Dichromate was removed for safety reasons. With the addition of Experiment 10 and Experiment 1 (Volumetric Glassware Analysis – Pipetting), this left the current laboratory manual with nine experiments. So both manuals would have the same number of experiments, the newly developed experiment for MSA on the AAS was added; this also ensured that the control group would be exposed to the same concepts as the test group before they attempted Experiment 10. However, the MSA on the AAS experiment was written in full procedural detail for the control group, whereas many details were left out of the transitional manual for the test group. Besides the details given to students in their experimental procedures, the biggest difference between the two manuals was Experiment 9. For the test group, this experiment was the Group Method Development; this experiment was a part of the new curriculum designed to enhance conceptual learning. The current manual needed to stay the same as much as possible for it to remain the control group. This circumstances lead to leaving in the Determination of Copper in Copper Oxide Samples by Iodometric Titration and making in Experiment 9.

The experiments were scheduled to be completed during the same weeks so that all students on both the control and test groups would be exposed to the same concepts at the same time. This was done so neither group would have an advantage nor disadvantage of when concepts were covered compared to when Experiment 10 would be completed. The list of experiments for the control group and test group is displayed in Table 4.

**Table 4.** Comparison of experiments between the control group and the test group.

| Experiment | Control Group (Current Manual) | Test Group (Transitional Manual) |
|---|---|---|
| 1 | Volumetric Glassware Analysis - Pipetting | Volumetric Glassware Analysis - Pipetting |
| 2 | Gravimetric Determination of Chloride | Gravimetric Determination of Chloride |
| 3 | Analysis of Impure Potassium Hydrogen Phthalate | Analysis of Impure Potassium Hydrogen Phthalate |
| 4 | Determination of the Purity of Soda Ash | Determination of the Purity of Soda Ash |
| 5 | Determination of Acetic Acid in Commercial Vinegar by Potentiometric Titration | Determination of Acetic Acid in Commercial Vinegar by Potentiometric Titration |
| 6 | Determination of Fluoride Using a Fluoride Ion Selective Electrode | Determination of Fluoride Using a Fluoride Ion Selective Electrode |
| 7 | Spectrophotometric Determination of Iron Via Its 1,10-Phenanthroline Complex | Spectrophotometric Determination of Iron Via Its 1,10-Phenanthroline Complex |
| 8 | Determination of Zinc using Atomic Absorption Spectroscopy | Determination of Zinc using Atomic Absorption Spectroscopy |
| 9 | Determination of Copper in Copper Oxide Samples by Iodometric Titration | Group Method Development Experiment |
| 10 | Independent Method Development Experiment | Independent Method Development Experiment |

## Qualitative and Quantitative Methodologies

Both qualitative and quantitative methods were employed. The qualitative methods employed were surveys and student interviews. Time was a factor in the research study, so a proven instrument was used, an adapted version of Student Assessment of their Learning Gains (SALG), see Appendix A. This version did not vary in the content of the questions, so there was no need to verify the adapted survey. From this template, some wording of sentences were changed, the “Not Applicable” was taken out as an option for students, and the number of questions were reduced to decrease student fatigue. The questions/statements were modified to be as short and concise as possible while still being straightforward. Additionally, there were two questions in regards to “keeping their attention” and making them read the instructions carefully. In general, the questions in the survey were designed to help gather information in regards to how they viewed their own learning “gains” of analytical chemistry concepts, how the curriculum design helped their learning, how the instruction of the teacher helped their learning, how their personal experiences helped their learning, and their overall attitude towards the class.

The research study employed some quantitative methodological aspects as well. For this data, the Experiment 10, Independent Method Development, was compared for the students’ unknown values compared to the true value. This experiment was also used qualitatively to see if students understood the concepts involved in the experiment. More quantitative data was gathered by using final exam scores from the control group and the test group. For the final exam, a series of method development questions (Appendix B), that involved higher-order, conceptual thinking, were employed to Evaluate (Five E’s) students and determine if they were able to understand the concepts covered in class well enough to answer to the questions.

The mixed methods of qualitative and quantitative method designs were chosen to be completed sequentially. They were administered in the following order:

1. Completing experiment and report for the Independent Method Development Experiment (Quantitative and Qualitative)
2. SALG Survey (Qualitative)
3. Final Exam (Quantitative)
4. Interviews (Qualitative)

By giving the surveys after the last experiment, students should be able to better recall their experience with that experiment and previous ones to answer the survey questions, than by having the interviews after the surveys and the final exam, it provided a good starting point and focus for the interview questions. By having the two types of methodologies sequentially, it allowed this research study to explore the students' experiences in relation to topics of interest from the previous data collections.

Out of the two types of analysis methods, the qualitative had priority over the quantitative methodology results. Qualitative played an important role in the research; when trying to develop students' analytical techniques, it was important to understand how they thought during the process of solving a problem and why they developed a particular method for analysis. For instance, a student may have made a poor grade on that last experiment, but through an interview or analysis of their Experiment 10 laboratory report it may be seen that even though their experiment did not go as planned, they may have had good reasoning behind their developed method. This happens in the real world and adjustments can be made in the next trial of an experiment. However, in the class room setting with only one attempt, the students may fail to get a good answer, but may provide reasoning as to why it fails and how to improve it; this could show that, ultimately, they understand the analytical techniques and concepts, and how to use them to solve real world problems.

## Analytical Components of the New Experiments

Four new experiments were developed for the current and transitional laboratory manuals:

1. Experiment 1 – Volumetric Glassware Analysis – Pipetting
2. Experiment 8 – Determination of Zinc using Atomic Absorption Spectroscopy
3. Experiment 9 – Group Method Development Experiment

4. Experiment 10 – Independent Method Development Experiment

The first experiment, Volumetric Glassware Analysis – Pipetting, was added to teach students how to use volumetric pipettes and analytical balances. In the experiment, students delivered three replicates of 10 mL of water via a graduated cylinder and volumetric pipette. The students compared the two delivery methods using accuracy and precision. This experiment also emphasized the importance of when is appropriate to use each method of delivery.

The second experiment, Determination of Zinc using Atomic Absorption Spectroscopy, was added to introduce students to another elemental analysis instrument and method of standard addition (MSA). For the development of this experiment, a calibration curve was constructed to determine the linear range for Zn on the Atomic Absorption Spectrometer (AAS), the detection limit determined, and a MSA was developed. For all of the studies, the glassware was cleaned using 10% nitric acid and then rinsed using Reverse Osmosis (R.O.) water. For the linear range study, nine 100 mL Class A volumetric flasks were used. The solutions were made to be 0, 1, 2, 3, 5, 10, 15, and 25 ppm Zn by delivering 0, 1, 2, 3, 5, 10, 15, and 25 mL, respectively, using a multi element standard, CCS-6 lot# F2-MEB415035 (Inorganic Ventures), containing 100.00ppm Ag, Cd, Co, $Cr^{3+}$, Cu, Fe, Hg, Mn, Ni, Pb, Ti, V, and Zn. The volumes were added using 1.000 mL Fisher Brand Finnpipette II and 5 mL, 10 mL, and 20 mL Class A volumetric pipets. The solutions were diluted to the mark using R.O. water. To determine the instrument detection limit, the blank solution was analyzed ten times and then the Limit of Detection and Limit of Quantification were calculated. For the MSA development, five 100 mL Class A volumetric flasks were used. To each flask, 0.025 mL of a 1000 ppm Zn single element solution, CGZN1-1 lot# F2-ZN02075 (Inorganic Ventures), was added using a 5-40 µL Finnpipette. The solutions were made to be 0, 1, 2, 3, and 5 ppm Zn by delivering 0, 1, 2, 3, and 5 mL, respectively, using a multi element standard, CCS-6 lot# F2-MEB415035 (Inorganic Ventures), containing 100.00 ppm Ag, Cd, Co, $Cr^{3+}$, Cu, Fe, Hg, Mn, Ni, Pb, Ti, V, and Zn. The volumes were added using 1.000 mL Fisher Brand Finnpipette II and 5 mL Class A volumetric pipets. The solutions were diluted to the mark using R.O. water. All solutions were analyzed on a Shimadzu Atomic Absorption Spectrometer AA-6300 (Japan) at 213.86 nm. Instrument operating parameters were 93 psi compressed air and 15 psi acetylene; integration time was set to 3 seconds per measurement.

The third experiment, Group Method Development Experiment, was developed to be a determination of phosphorous concentration in water samples. At this point, the students had worked with two different instruments that could be used for phosphorous determination, UV-Vis and AAS. The students were provided with information pertaining to phosphates in water sources and three articles describing analyses performed on those two instruments;[48] two articles for colorimetric determinations on UV-Vis and one for elemental analysis on AAS.[54,55,56]

The fourth experiment, Independent Method Development Experiment, was made to be a two part experiment; the students were given a hydrochloric acid (HCl) sample and had to determine the iron concentration and the percent HCl (acidity). For this experiment, students had already completed all of the other experiments and had several types of analytical techniques and instrumentation to use for developing their method. Students were provided with three articles pertaining to iron analysis on AAS, with one of those using a MSA.[57,58,59] Students were also allowed to use their laboratory manual which contained an experiment for the determination of iron using UV-Vis. Students were given the articles for the ninth and tenth experiments at the beginning of the semester and were encouraged to start reading through them to get practice reading scientific articles and to understand more those experiment. They were allowed to construct a method of analysis for each experiment using these articles, other sources found online or in print, and their laboratory manual.

# CHAPTER 3

# RESULTS AND DISCUSSION

## Development of Experiment 8

There were three parts to developing the eighth experiment, Determination of Zinc using Atomic Absorption Spectroscopy: the linear range for Zn on the instrument had to be determined, the LOD and LOQ had to be calculated, and the MSA had to be successful in determining the true concentration of the unknown.

### Determination of Linear Range

For the linear range study, the results demonstrated that the 10 and 25 ppm Zn solutions were too concentrated with absorbances higher than 1 (Table 5), Analysis using absorbance based spectrometers is kept within the 0-1.0 absorbance unit range; given the fact that A = -log T, an absorbance of 1.0 indicates a transmission of radiation of 10%, with 90% of the light being absorbed by the sample. Below this transmission level, the precision of the measurement drops off dramatically, increasing the error of each subsequent measurement. Furthermore, when graphed, it was apparent that these two solutions did not follow a linear calibration curve with deviation from Beer's Law, $A = \varepsilon bc$, as shown in Figure 2. Since, the 10 and 20 ppm Zn solutions had an absorbance of above 1.0 and they deviated from Beer's Law, they were considered to be out of the linear range of the instrument, therefore they were removed from the calibration curve.

**Table 5.** Linear range study using 0-25 ppm Zn.

| | Absorbance | | | |
|---|---|---|---|---|
| **Concentration** | **Trial 1** | **Trial 2** | **Trial 3** | **Avg.** |
| 0 | -0.0058 | -0.0001 | 0.0016 | -0.0014 |
| 1 | 0.1868 | 0.1839 | 0.1863 | 0.1857 |
| 2 | 0.3707 | 0.3626 | 0.3632 | 0.3655 |
| 5 | 0.8274 | 0.8252 | 0.8258 | 0.8261 |
| 10 | 1.3051 | 1.3068 | 1.2958 | 1.3026 |
| 25 | 1.5573 | 1.5566 | 1.5473 | 1.5537 |

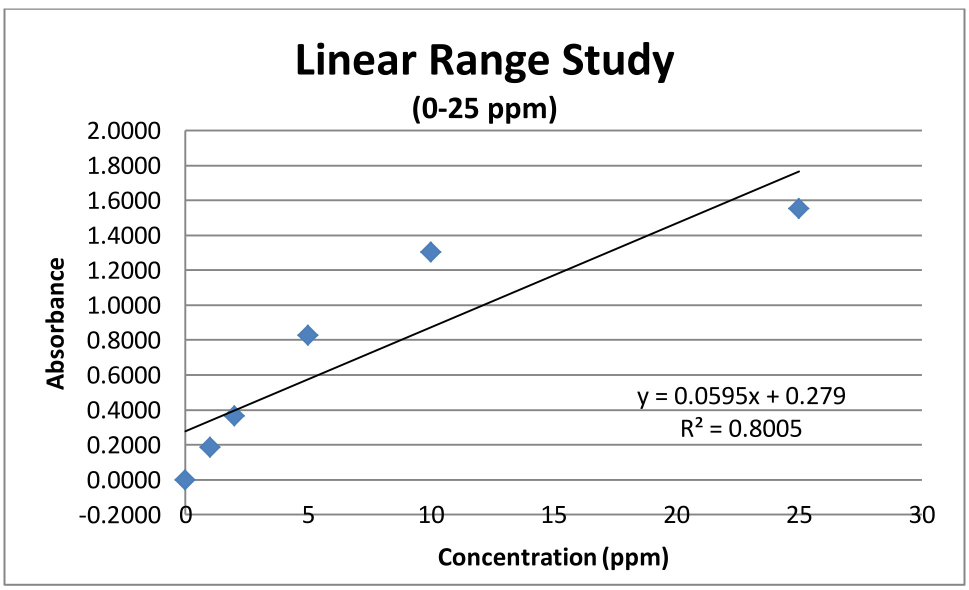


**Figure 2.** Linear range study showing the absorbances of 0, 1, 2, 5, 10, and 25 ppm Zn standards.

After excluding the 10 and 25 ppm Zn standards the remaining standards were graphed and their linearity was examined as shown in Figure 3. The 5 ppm calibration standard appeared to be slightly lower than expected. When comparing this graph to Figure 2 it became apparent that the 5 ppm Zn calibration standard may have deviated slightly from Beer's Law, even though it was under 1.0 absorbance. The 5 ppm concentration was too close to the linear range of the instrument to be determined accurately and, therefore had to be omitted. The linear range was set slightly below this concentration, at 4 ppm Zn when presented to the students, to be sure that it would be within the readable range of the instrument.

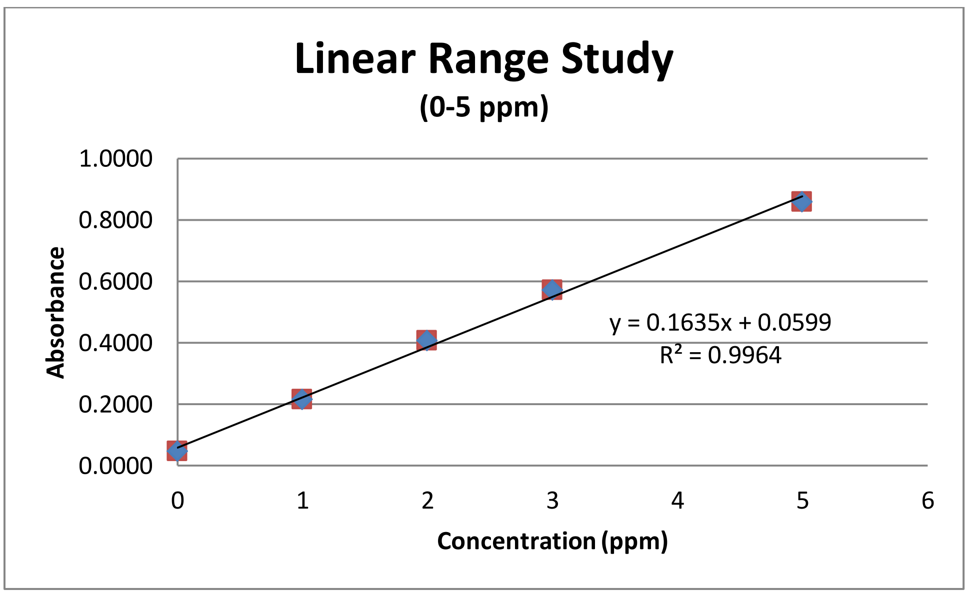


**Figure 3.** Linear Range Study showing the absorbances of 0, 1, 2, and 5 ppm Zn standards.

Determination of the Limit of Detection and Limit of Quantification

After determining the linear range of the instrument, the lower end of this range had to be determined by calculating the LOD and LOQ. The LOD is the lowest concentration of the analyte that can be distinguished from the background signal of the instrument. The LOQ is the lowest concentration of the analyte that can be determined with a high degree of confidence. The LOQ is used as the bottom end of the linear range of the instrument, because anything below this value is considered unreliable. To determine these values, a low concentration solution of the analyte or a blank solution is analyzed with several replicates and then calculated using these formulas:

LOD = (3s)/m

LOQ = (10s)/m

where (s) was the standard deviation and (m) was the slope of the calibration curve. The blank was analyzed ten times as shown in Table 6. The data from the linear range study, excluding the 5, 10, and 25 ppm Zn solutions, were graphed to determine the slope as shown in Figure 4. Using this data, the LOD was determined to be 0.0534 ppm Zn and the LOQ was determined to be 0.1781 ppm Zn. For this experiment, any solution (standard or unknown) that has a value below 0.1781 ppm Zn was considered unreliable.

**Table 6.** Absorbances of the blank solution

| **Analysis** | **Absorbance** |
|---|---|
| 1 | 0.0040 |
| 2 | -0.0029 |
| 3 | 0.0028 |
| 4 | 0.0039 |
| 5 | -0.0020 |
| 6 | -0.0034 |

**Table 6** (Continued)

| Analysis | Absorbance |
|---|---|
| 7 | -0.0030 |
| 8 | 0.0029 |
| 9 | 0.0034 |
| 10 | -0.0023 |

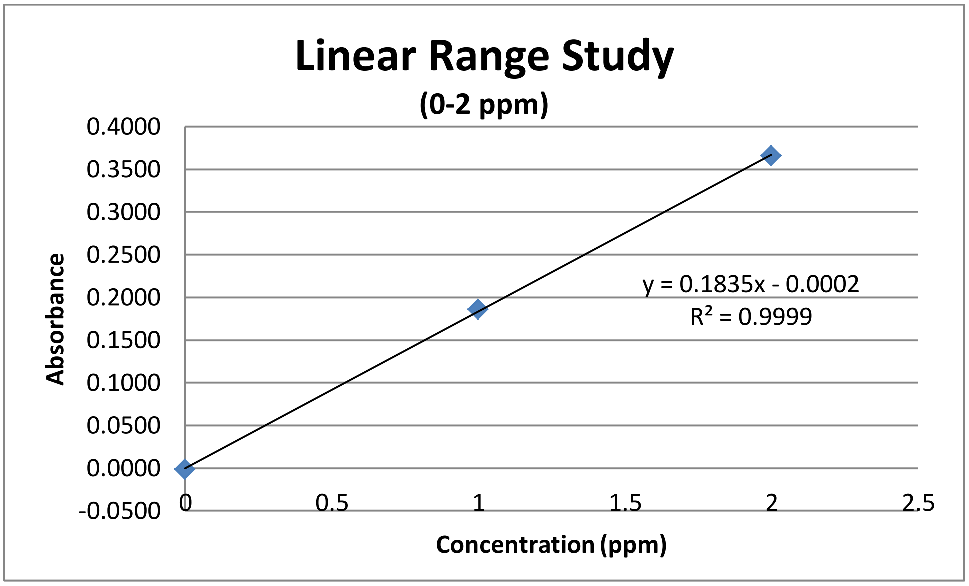


**Figure 4.** Concentration vs. Absorbance of the calibration standards (0-2 ppm)

<u>Method of Standard Addition</u>

When the solutions for the MSA were first made, the linear range of the instrument was not known. Spiked solutions were made with 0, 1, 2, 3, and 5 ppm spikes with the final diluted solution to have an unknown concentration of 0.25 ppm. The results of these solutions are displayed in Table 7.

**Table 7.** Method of standard addition results

| | Absorbance | | | |
|---|---|---|---|---|
| **Concentration** | **Trial 1** | **Trial 2** | **Trial 3** | **Avg.** |
| 0 | 0.0475 | 0.0470 | 0.0459 | 0.0468 |
| 1 | 0.2168 | 0.2134 | 0.2142 | 0.2148 |
| 2 | 0.4077 | 0.4081 | 0.4033 | 0.4064 |
| 3 | 0.5728 | 0.5714 | 0.5709 | 0.5717 |
| 5 | 0.8638 | 0.8535 | 0.8596 | 0.8590 |

The results were graphed in Figure 5 and, using the y=mx+b; the y-intercept was allowed to be non-zero to account for background noise of the instrument. With this, the unknown was calculated to be 0.3663 ppm Zn. However, after the linear range study was completed, the 5 ppm Zn was determined to deviate slightly from Beer's Law. Given this, the graph was replotted excluding the 5 ppm spiked solution as shown in Figure 6. The unknown Zn was calculated to be 0.2548 ppm Zn which was extremely close to the true value of 0.250 ppm Zn. The accuracy of the results with excluding the 5 ppm spiked solution further validated the decision to eliminate this solution. Given the results, the determination of Zn using MSA on AAS was considered valid and accurate.

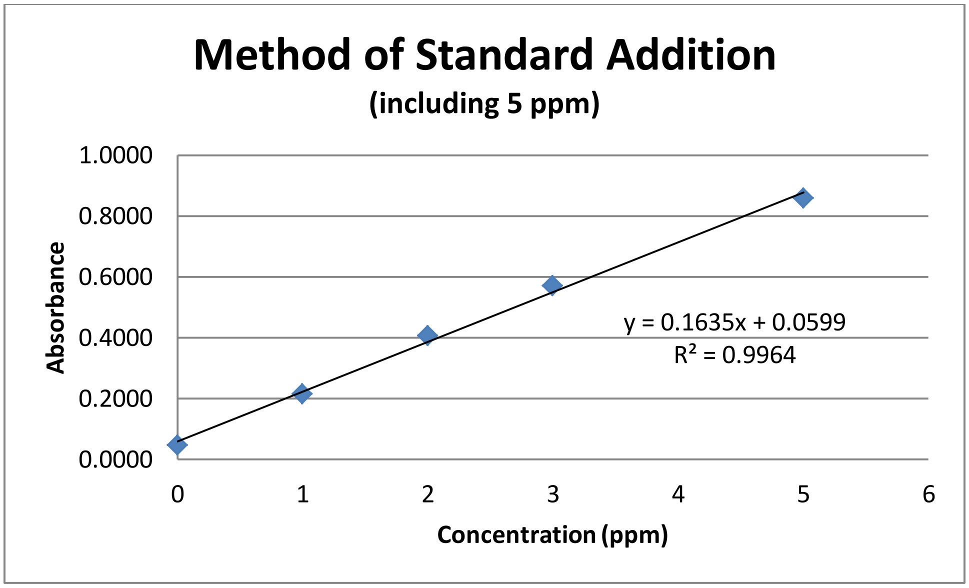


**Figure 5.** Method of standard addition including the 5 ppm spike.

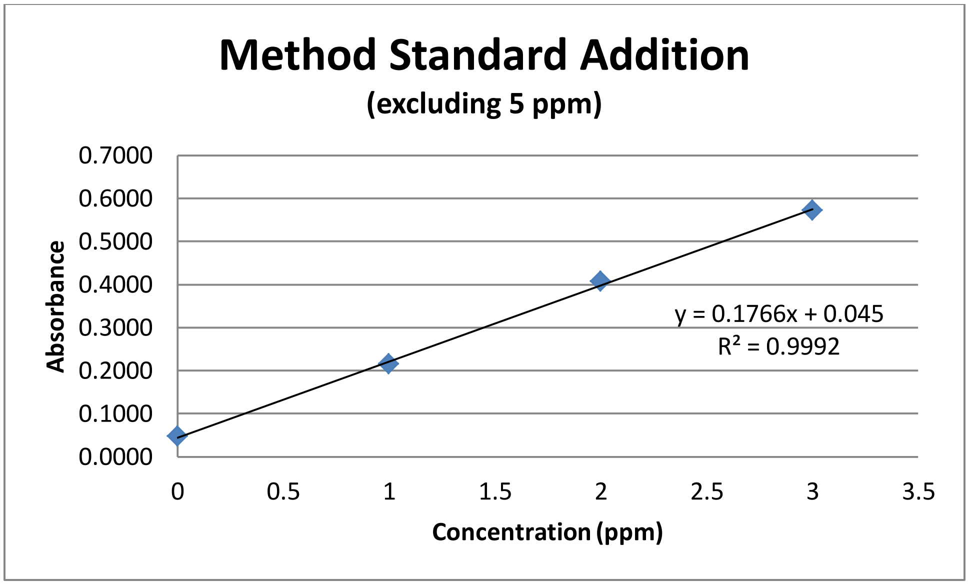


**Figure 6.** Method of standard addition excluding the 5 ppm spike.

## Student Data Collection

Before beginning any data collection from students, a request for approval was submitted to the East Tennessee State University Institutional Review Board (IRB). The IRB request was approved and data collection with the control group and test group could begin. A copy of the IRB approval letter is attached as Appendix C.

One control group and one test group were taught for the study in different semesters. For each of the groups, one of the existing Quantitative Analysis Laboratory courses were used; special laboratory sections were not created for the research study. Before the semesters began, students were not told that certain laboratory sections would be used in a research study. This was purposefully done so that the population of students signing up for the research study laboratory sections would represent a normal, random population of students. If students may have known about the difference between the research study sections beforehand, it may have affected their attitude about the course and whether or not they signed up for that particular laboratory section.

For the data collection, the sample populations of the control group and test group were small. The quantitative laboratory course only allows twelve students in each section. Only one section was taught as a control group and only one section was used as a test group. Of these two sections, five control group students and six test group students agreed to participate in the study and signed the IRB informed consent documents. It was hoped that the sample population would have been bigger but, due to unexpected issues and limited time frames, this was not a reasonable option. Since, the sample populations were small, statistical analysis would have been difficult to validate. The study focused more on the qualitative data to determine if the null hypotheses were true.

## Quantitative Methodologies

There were two parts of the quantitative data for this research study: the results of the independent method development experiment and the method development questions on the final exam.

### The Independent Method Development Experiment

The control group and test group were compared by their accuracy in determining the iron concentration and percent acidity by volume of their unknown sample. Both groups had some outliers, while others were closer to the true values of 10 ppm iron and 10% hydrochloric acid (HCl). The raw data may be seen in Table 8 and Table 9, and comparative graphs can be seen in Figure 7 and Figure 8.

**Table 8.** Comparison of unknown values for determining the concentration of iron.

| **Iron Concentration (ppm)** | |
|---|---|
| Unknown true value = 10.00 ppm | |
| **Control Group** | **Test Group** |
| 22.84 | 7.430 |
| 19.30 | 9.792 |
| 12.83 | 10.29 |
| 11.14 | 15.06 |
| 18.75 | 11.71 |
| | 3.930 |

**Table 9.** Comparison of unknown values for determining the weight percent of HCl.

| Weight Percent Acidity | |
|---|---|
| Unknown True Value = 10.00% HCl | |
| **Control Group** | **Test Group** |
| 7.440 | 12.36 |
| 11.58 | 12.72 |
| 13.94 | 12.46 |
| 11.80 | 13.35 |
| 10.88 | 12.79 |
| | 11.79 |

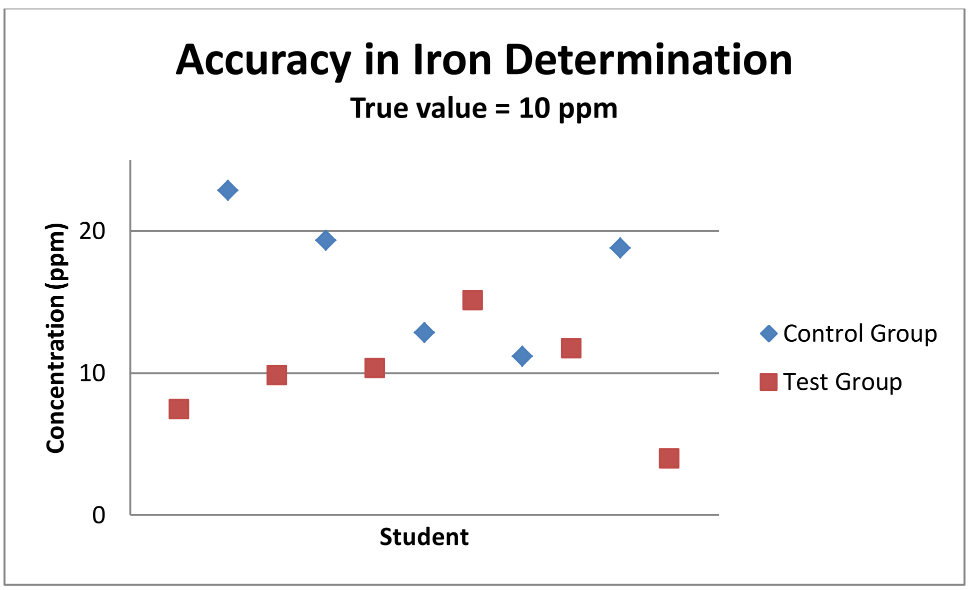


**Figure 7.** Comparison of unknown values for determining the concentration of iron.

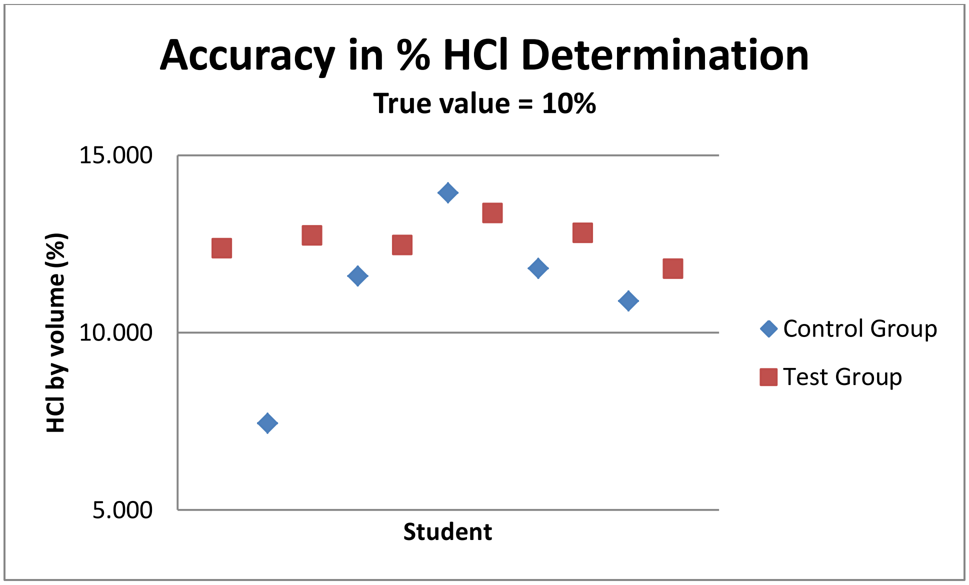


**Figure 8.** Comparison of unknown values for determining the weight percent of HCl.

For the iron concentration determination, students in the test group were able to be more accurate in their individual analysis of iron, with two test group student obtaining values extremely close to the true value. The test group also had a better average, 10.86 ppm iron with a standard deviation of 2.81 ppm, while the control group had an average of 16.97 ppm iron and a standard deviation of 4.85 ppm. This showed that the test group students had a better overall average and were far more consistent in determining the iron concentration.

For the determination of percent HCl in the unknown sample, the individual results had more variation than the iron. Overall, the test group had an average of 12.74% HCl and a standard deviation of 0.386 %. The control group had a class average of 11.13% HCl, but had a larger standard deviation of 2.36 %. One of the control group students obtained the most accurate value, but the control group also had a higher number of outliers, hence the higher standard deviation. For the test group, all of the HCl results were consistently above the true value.

Method Development Exam Questions

The method development questions on the final exam were employed to test the students' knowledge of the fundamentals concerning method development. Table 10 shows a summary of the exam questions, the level of Bloom's Taxonomy for each question, and the item difficulty for the control group and test group students. The exam questions with answers and grading rubric are in Appendix D. The item difficulty is based on the number of correct responses, including partially correct, divided by the total number of student responses. The higher the item difficulty (closer to 1.00), the easier the question was for that group of students; the lower the item difficulty (closer to 0.00), the harder the question for the group of students..

**Table 10.** Summary of exam questions and item difficulty for control group and test group.

| | | | **Item Difficulty Index (# Correct Responses /Total Responses)** | |
|---|---|---|---|---|
| **Question 5** | | **Level of Bloom's Taxonomy** | **Control Group** | **Test Group** |
| (a). | Define instrument detection limit and why it occurs. | Knowledge | 0.60 | 1.00 |
| (b). | Define linear range and why it occurs. | Knowledge | 0.60 | 1.00 |
| (c). | General steps in beginning to develop a method. | Comprehension | 0.60 | 1.00 |

**Table 10** (Continued)

| Question 5 | | Level of Bloom's Taxonomy | Item Difficulty Index (# Correct Responses /Total Responses) Control Group | Test Group |
|---|---|---|---|---|
| (d)(i). | Using good calibration curve to determine good unknown value. | Application | 1.00 | 1.00 |
| (d)(ii). | Using good calibration curve to determine bad unknown value. | Application Analysis | 0.80 | 0.75 |
| (e). | Using a bad calibration curve (high standard above linear range). | Application Analysis | 0.40 | 0.38 |
| (f)(i). | Identify matrix effects in a spectrum graph. | Evaluation | 0.20 | 0.75 |
| (f)(ii). | How matrix effects can be fixed? | Evaluation | 0.60 | 0.75 |

Overall, test group students had performed better than the control group on the lower order cognitive skill questions (a)-(c) that asked students to recall information already taught to them (a, b) and be able to summarize concepts involved in developing a method(c); the questions utilized Knowledge and Comprehension, respectively, from Bloom's Taxonomy. Comparatively between the two groups, this showed that the test group students were able to recall basic principles and summarize concepts better than the control group.

Questions (d)-(e) required students to look at and interpret data from calibration curves. Question (d)(i) was a simple straightforward question requiring an Application level of understanding; the question gave the students a calibration curve and a sample intensity, then asked them to solve for the unknown concentration. Both the control group and test group have a high difficulty index meaning the question was relatively easy for both groups.. Question (d)(ii) used the Application level too, as well as the Analysis level; both group found this question to be less difficult. This question used the same calibration curve as (d)(i), but gave the students a sample intensity that was above the high calibration standard, but within the linear range. When calibration curves were discussed in class, students were informed that any unknown values above the high calibration standard are assumed to be invalid; since the unknown value above the calibration curve, even if it is below the linear range, there is a possibility that the calculated unknown value could be slightly incorrect. If it's out of the calibration curve range and it should be diluted to be within the calibration curve to be an accurate calculated concentration. Some of the students overlooked this and solved for it anyway, not accounting for the fact that the sample should always be below the high calibration standard for more accurate and reliable results. Question (e) was considered Application and Analysis level questions as well. The question gave a different calibration curve and asked them to solve for a different sample intensity. However, this calibration employed a high standard that was above the linear range of the instrument. The students were given the linear range at the beginning of the question and this point on the curve starts to plateau instead of remaining linear. This was a more difficult question for all of the students (control and test groups).

The last set of questions, (f)(i)-(f)(ii), were about MSA. The students were shown a spectrum with the wavelength intensity peaks for the calibration standards and the unknown, with the unknown having an elevated background from the standards. The students had to determine that the unknown elevation was due to matrix effects and list different ways that this problem could be fixed. Both of these questions assess higher order cognitive skills and considered at the upper level of Bloom's Taxonomy since it involved student's ability to assess the spectrum of the analyzed solutions and to determine the problem and a solution to fix it. The test group students performed much better on these questions than the control group, even though many of the control group employed this method for their final method development experiment.

The average item difficulty index for the control group was measured at moderate difficulty (0.600) and the test group difficulty index is considered 0.828 indicating, on average, the assessment was easy. This demonstrates that the control group found these method development exam questions more difficult than the test group. Due to the low sample population for both the control group (five students) and test group (six students), a paired t-test was used to analyze the exam items. The t-test showed there was a significant difference between the item difficulty of the two groups, $p = 0.029$ ($p \leq 0.050$, significantly different).

## Qualitative Methodologies

There were three parts of the qualitative data for this research study: the methods developed for the independent method development experiment, the SALG surveys, and student interviews.

The Independent Method Development Experiment

The control group and test group were compared in the methods they developed. This was done to see if the students understood the concepts involved in their method and to also understand their results, *i.e.* obtained inaccurate results due to a poorly developed procedure. Table 11 shows a summary of the methods the students used to determine the iron concentration in a concentrated hydrochloric acid sample for the method development experiment. This table shows how many students used UV-Vis, AAS using a standard calibration curve, and AAS using MSA.

**Table 11.** Methods used by control group and test group students for iron determination.

| | **Control Group** | **Test Group** |
|---|---|---|
| **UV-Vis** | 2 | 3 |
| Added extra buffer | 0 | 2 |
| Added extra reducing agent | 0 | 0 |
| Matrix incorrect | 2 | 3 |
| Matrix correct | 0 | 0 |
| Over dilution of unknown | 2 | 1 |
| | | |
| **AAS - Standard Calibration** | 0 | 3 |
| Matrix incorrect | 0 | 0 |
| Matrix correct | 0 | 3 |
| Over dilution of unknown | 0 | 0 |

**Table 11** (Continued)

| | **Control Group** | **Test Group** |
|---|---|---|
| **AAS - MSA** | 3 | 0 |
| Over Dilution of unknown | 2 | 0 |
| | | |
| **Total Over Dilution of unknown** | 4 | 1 |

Of the students who used UV-Vis to determine the iron concentration, 2 of the 3 test group students remembered to add extra buffer due to the increased acidity of the unknown, while neither of the control group students considered this. It was important for them to remember, with the increased amount of HCl in the sample, to add more buffer (sodium acetate) in order to buffer all of the hydrochloric acid in the sample. Additionally, hydrochloric acid and iron react to form $FeCl_3$, where iron is in the Fe (III) oxidation state. For the UV-Vis method to be successful, all the iron must be in the Fe (II) form instead of the Fe (III) form in order to form the correct complex with 1,10-phenanthroline. To help account for this, extra hydroxylamine (reducing agent) was required. None of the students remembered the extra hydroxylamine. Since all of the iron was not converted in the correct oxidation state, it was impossible to analyze the iron concentration accurately.

Concerning the matrices of the UV-Vis solutions, the students who thought of adding extra buffer for the unknown, also remembered to add the same amount of buffer to the calibration standards to ensure the same buffer matrix. However, all of the students that used UV-Vis forgot to add some stock hydrochloric acid to the calibration standards to reduce matrix effects. The difference in matrices of the standards compared to the unknown may have led to some of the inaccurate results. Additionally, only one of the test group students over diluted their unknown to where it was below the LOQ, while both of the control group students over diluted. The unknown absorbances being lower than what the instrument could determine accurately definitely attributed to the lack of accuracy in these over diluted unknown samples.

All of the control group students who chose to use AAS used MSA. This method would have been suitable for overcoming the matrix effect from the high HCl concentration, except the concentration of iron was not taken into account. Of the three control group students, two students extremely over diluted their unknown. Many of the students completed a titration for the percent HCl first and heavily diluted their unknown sample. The students thought the diluted sample would still be good to use for the iron portion of the experiment, but they did not account for the heavy dilution of the iron. The diluted unknown samples were then further diluted by using MSA resulting in an iron concentration too low and indistinguishable from the background noise for the instrument to measure accurately. For the test group students that used AAS, all three chose to use a standard calibration curve. All three of these students remembered to account for the acid matrix of the unknown and added stock hydrochloric acid to the calibration standards. Of these three test group students, two were the most successful with the experiment as they were the closest to the true values (9.792 and 10.29 ppm iron).

For the determination of percent HCl in the unknown sample, Table 12 shows the number of students who chose to do a KHP titration using phenolphthalein indicator, potentiometry, or a soda ash titration using phenolphthalein indicator to determine the endpoint of acid-base titration for the HCl analysis. There did not appear to be any correlation to which analytical technique was used to their results.

**Table 12.** Methods used by control group and test group students for acidity determination.

| Titration | Control Group | Test Group |
| --- | --- | --- |
| KHP - Phenolphthalein indicator | 4 | 3 |
| Potentiometry | 0 | 3 |
| Soda ash - Phenolphthalein indicator | 1 | 0 |

It was unique that the control group students thought of using their unknown HCl sample to titrate pure sodium carbonate (from soda ash experiment). The student stated that it would save time in class to only do a one-step titration instead of a two-step titration, standardization and then determination of unknown. This was a creative way of thinking of this assignment, but unfortunately, this student ended up getting the farthest away from the true value, 13.94% HCl. The student had a unique idea but did not account for the increased acidity of the unknown and the amount of the pure sodium carbonate that was being titrated. The titration only required a small amount of the unknown HCl to change color. There was a lack of accuracy due to this, but it was still noted as being the most unique method performed. There was also a note made about a method developed by one of the test group students. The student developed a method of using a stock 20 ppm HCl solution to make a set of calibration standard solutions of varying percent acidities. Their plan was to use the potentiometer to determine the pH of each of the standard solutions and then make a calibration curve of concentration versus pH. Then using the potentiometer to determine the pH of the unknown, they could then use the calibration curve to calculate the unknown concentration. After more thought about this, the student abandoned the method and settled for a normal acid-base potentiometric titration. The student did not perform this method, but it was recorded as being a unique idea. While these two unique methods had their flaws, it was insightful to see how the students tried to apply the concepts they knew in a different setting and showed that they were trying to come up with something new instead of using a tested method.

SALG Surveys

The SALG survey data maybe important because it may give insight into how well the students believed they learned the material. While it does not provide evidence of the students' ability to solve problems, it does give insight into how they felt about the learning process. Furthermore, as both the control group and test group were given the same survey, it could help elucidate the differences between the groups, if any.

The survey questions were divided into six main categories: (1) how they viewed their own learning "gains" of analytical chemistry concepts/content, (2) their gains in research skills, (3) how the curriculum design helped their learning/performance, (4) how the course instruction by the teacher helped their learning, (5) how their personal experiences in the course helped their learning, and (6) their overall attitude towards the class. Figure 9 compares the average reported gains of the SALG surveys completed by the students from the traditional (control group) and inquiry-based (test group) laboratory manual formats.

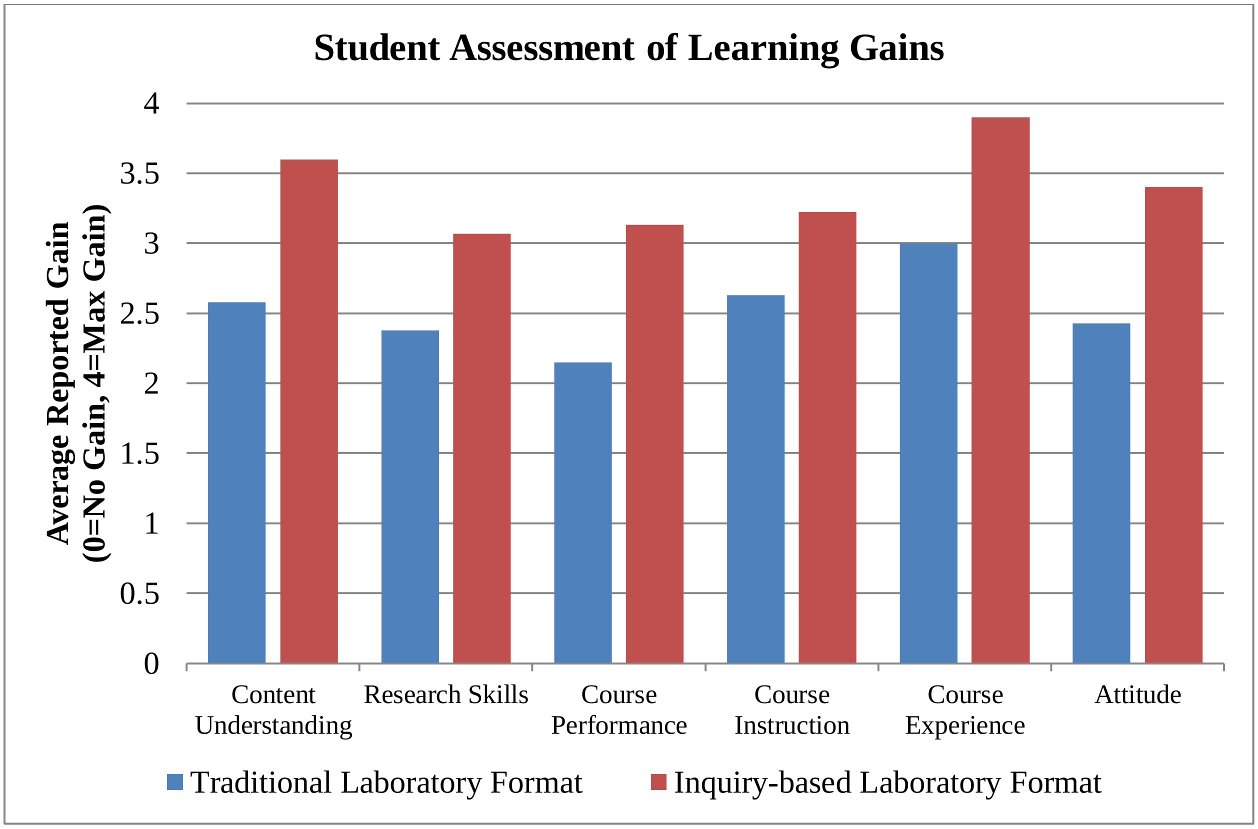


**Figure 9.** Average reported gain for the six sections of the SALG survey for traditional and inquiry-based laboratory manual formats.

"Gains" in Analytical Chemistry Concepts. When asked about their learning "gains" of analytical chemistry concepts, there was an obvious difference between the two groups. The control group had varying answers from a "little" to "great" gains with no clear trend, instead it was evenly distributed. The test group students had an overwhelming majority of students indicating that they had a "great" gain in their perception of how well they understood analytical chemistry concepts.

When the students were asked to comment on their understanding of these concepts as a result of the course, the students from the control group focused mostly on discussing their improvement in the need for precision accuracy and how their laboratory technique improved.

The control group also indicated the traditional laboratory manual could have explained concepts better and wished that there was more room for creativity. The test group students indicated that their knowledge of analytical equipment and analytical techniques had greatly improved, indicating they saw analytical concepts as more than just need for precision and accuracy. Some test group students also commented that they thought the course was the most constructive laboratory course they had taken at the university and their learning was a direct result of the curriculum design; they explained that the laboratory manual, by giving less instructions as the semester progressed, forced them to think critically because they had to know why they were doing what they did, as opposed to just going through a laboratory procedure step-by-step.

“Gains” in Research Skills. When asked about their learning “gains” in different research skills (reading journal articles, developing logical arguments, explaining results, etc.), the test group (inquiry-based) students consistently felt like they had greater “gains” than the control group students, except for “gains” in “developing an experiment to answer questions”, see Figure 10. This was the exact opposite of what was observed of the students while they were preparing their methods for experiment 10. All students were asked to turn in an outline of their developed method two weeks in advance for the instructor to review. Out of the test group students, only one student revised the initially developed method to utilize different analytical techniques. For the control group, half of the students changed their method from their original developed one. This showed insecurity in the first developed method and feeling like they needed to change it. Considering this, the test group students showed more confidence in their ability to develop a method, while the control group students were more insecure.

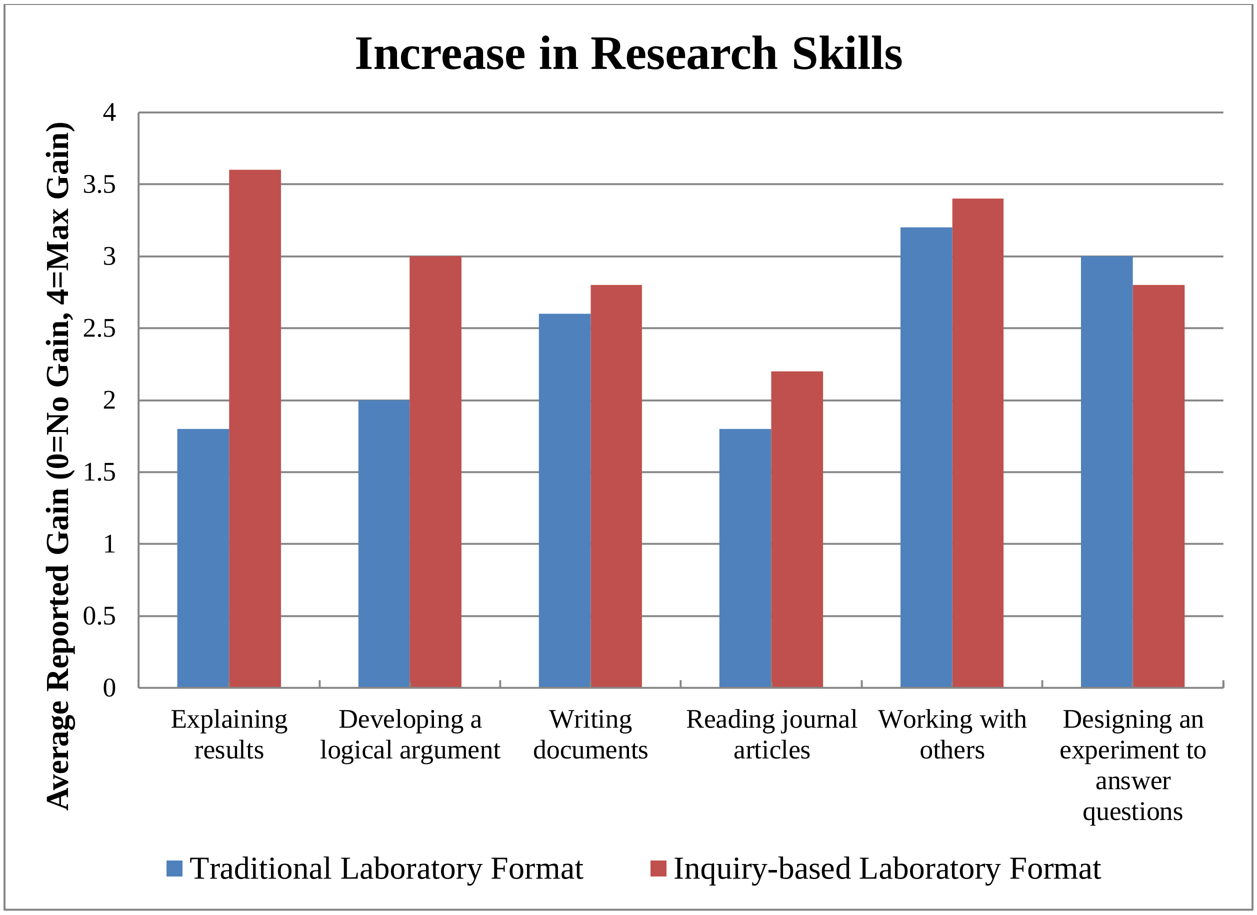


**Figure 10.** Average reported gain for the "Increase in Research Skills" section of the SALG survey for traditional and inquiry-based laboratory manual formats.

In reviewing the qualitative data from Experiment 10, Independent Method Development, the control group students consistently over looked small, important details when developing a method, yet they were more confident in their skills in designing a method. Conversely, the test group students outperformed the control group students in their knowledge and understanding of the concepts used in developing their methods by remembering these small, important details. Even though the test group students performed better in method development, according to these SALG survey results, they were less confident. The control group seemed to focus more on the general concepts than on the specifics of those concepts. By focusing less on the details, it appeared as though they thought the problem was easier that it really was, leaving them with a false sense of confidence. Many of the test group students were more aware of the details/specifics of the concepts and took the time to account for them in their methods. By seeing how difficult the problem was, because of these types of small/important details and how easily they could be overlooked, the test group students had a better understanding of the skills necessary to successfully solve the problem. The test group students' awareness of the increased difficulty of the problem may have led to them to have less confidence, even though they performed better on Experiment 10 with a class average of 10.86 ppm versus 16.97 ppm for the control group . This is also reflected in the final exam, where the test group had less difficulty than the control group. The final exam also showed that while many of the control group students used the MSA in their developed methods for Experiment 10, several of them found these questions more difficult and failed to get the MSA/matrix questions correct. The control group students confidence in their method development skills was not due to their knowledge of those concepts involved in their experiment, which left them lacking in their ability to solve the problem correctly.

Curriculum Design. The transitional curriculum course was designed to have students learn to use many different resources to understand the analytical concepts such as the laboratory manual, writing reports, journal articles, and other sources. Likewise, the control group was given all concepts in their laboratory manual, but they were also asked to write a final report for the last experiment and use journal articles and other resources while developing their method. Almost all of the test group students indicated that all of the resources greatly helped their learning (an average reported gain of 3, on a scale of 4 being great gain), while the control group students were more scattered in their answers (an average reported gain of 1.4) . Some of the control group students thought the writing of the report helped their learning while some others said that it was only moderate help or no help. Concerning the use of journal articles and other resources, the control group students mostly shared that they were of little to no help.

The students were asked to comment on how the design of the curriculum helped their learning. The control group expressed that they liked having detailed procedures to follow in the laboratory manual explaining exactly how to perform the experiments. Conversely, the test group specified that the lack of procedural details in the later experiments helped them learn because they had to understand what they were doing and the theory of why it worked, instead of just following directions. Many of them shared that, because of a lack of details, they were forced to look to at the outside sources (journal articles, *etc.*) to critically evaluate the concepts so that they would be able to complete the experiments. This helps account for the test group students expressing that the journal articles and other resources were of great help in their learning of the analytical concepts.

<u>Instruction and Teaching of the Course.</u> The results from this section of the survey were undiscerning as there was no overall trend in student answers, but there were some noteworthy individual question distinctions. One of the test group students indicated that the traditional experiments were of no help to them in learning analytical chemistry concepts. All of the test group students shared that the experiments where they had to provide more detail to complete the experiments, including the method development experiment, were of greater help in their learning. The control group had more mixed feeling about their instruction indicating that it was only moderate to great help. However, one control group student indicated that they enjoyed the

method development experiment, and expressed the desire to have more of these types of experiments with traditional experiments at the start of the semester to help teach the basics.

As for the instruction of the two groups, they were both treated very differently. The pre-laboratory lectures and instruction were designed to match the laboratory manual the students were given. For the control group, pre-laboratory lectures were very detailed and resembled the lectures they may have encountered in other chemistry courses. For the test group, the transitional laboratory manual provided less and less detail, especially on experiments where a concept was previously covered earlier in the semester. The pre-laboratory instruction mirrored this and the test group students received only the details concepts which were newly introduced. This difference could also be witnessed in answering student questions. The purpose of the transitional laboratory manual was to have students build on their knowledge, so when students asked questions, they were not given answers as they were in the control group; instead, students were asked questions in return to get them thinking of about the concepts involved in their question and they were guided until they came up with the correct answer themselves.

In both instances, the students enjoyed the instruction given from the laboratory instructor, especially during the pre-laboratory lectures where they could see practical applications. The test group students also stated that the student-instructor interactions were beneficial to their learning and enjoyed the instructor's "keen skill in answering your question, while making you come up with the answer on your own." They also thought that the instructional approach allowed them to review the concepts, learn from them, and use them to formulate solutions to their problem, which helped them understand the concepts more.

Personal Experiences. The control group had mixed feelings regarding personal experiences, ranging from a little help to a great help, on how their personal experiences in the helping their learning; this included interactions with the instructor and their peers. However, all of the students in the test group indicated that the interaction with the instructor and with their peers helped increase their learning. More notably, the test group students all indicated that participating in group work greatly helped them learn and the group method develop experiment aided their learning as well. Some of them also indicated that they developed team-working skills which played in a key role in helping them learn.

Overall Attitude Toward the Class. The last portion of the survey was designed to see how they perceived their own understanding and application of analytical chemistry, including working with complex ideas and their confidence in completing future research projects in a professional career. Everyone in test group shared that they had good or great gains in their understanding of analytical chemistry and its applications; while the control group students indicated a lower level of understanding with mixed opinions. When asked about their confidence in being able to complete research projects in a professional career, the test group had slightly more assurance than the control group. One of the control group students specifically indicated that they would have "more confidence with this if they had had an opportunity to do more method development experiments."

It was also noteworthy that the students were asked about their enthusiasm for analytical chemistry since taking this course. The control group had a wide array of opinions from no gain to great gain. However, the students in the test group were more weighted towards having good or great gain. The students were never asked what made them more enthusiastic about this subject because of their laboratory experience, but the transitional manual was designed to get students more interested in the subject and its applications. This was a part of the Five E's where the students must be Engaged to help promote curiosity and increase their attention. By more of the test group students indicating that they were more enthusiastic about the subject, it may have been one of the causes in the students believing they were successful in increasing their knowledge of analytical chemistry concepts.

### Student Interviews

Two students were interviewed from both the control group and test group; they were interviewed at the end of the semester so that they had all of their laboratory experiences to recall when interviewed. All of the students were asked the same basic set of questions regarding their opinions of their laboratory manual, how to improve that manual, what helped them learn more concepts in the class, if they enjoyed working in groups, and if they thought their laboratory manual helped prepare them for a professional chemistry or science related career.

Concerning the laboratory manual, the control group students both indicated that their manual resembled other manuals they had encountered in other laboratory courses; the experiments were the same basic setup of a procedure giving you step by step instructions on how to complete it. The two test group students both admitted that their manual was unlike anything that they had experienced before. Both students noticed the gradual transition of leaving out instructions and having to recall information learned on previous experiments to complete the later ones; they shared that this left them having to understand how things worked and why in order to complete the later experiments. One of the control group students indicated, "You couldn't just go in and have the instructions every single time about you had to do, so you really had to understand why a particular type of instrumentation or analysis worked because, as the semester went on, you got less and less instructions and you had to know exactly what you had to do and why. Otherwise, you were just in there wasting time. You had four hours to get your experiments done and you really needed to know what you were doing at every step in order to plan what you were going to do next. So, you not only needed to be good at the instrumentation and good at the analysis, but you needed to know how and why they worked at the same time."

To get an understanding of what helped the students learn in this class, they were asked what aspect of the course helped them learn more concepts and how confident they felt in their knowledge of those concepts. The answers varied between all of the students. One of the control group students stated that the detailed laboratory manual helped with understanding the material because they had not taken a chemistry course in a long time. Concerning their confidence, one control group student shared that they felt comfortable with the concepts, but not the specifics. The other control group student said that they learned more from the pre-laboratory lectures because the laboratory manual was a little "long-winded" with details and it made it hard to picture things in their mind. The student said, "I always transcribed from the lab manual into my notebook, seen it probably read over it twice, but it didn't really come together until I saw it in the pre-lab…when you explained everything it was more direct and it was, step by step and very quick, so it made it very concise and clear, whereas maybe I've read it directly and then tried to picture it in my mind, it still didn't come together. It was kind of long winded in the lab manual." This control group student attributed their confidence in learning the concepts to having to study for the final exam.

The test group students were also asked about what aspect of the course helped them learn more concepts and how confident they felt in their knowledge of those concepts. One said that they learned the most from the last two, group and independent, method development experiments and the way that the laboratory manual transitioned up to them. This student expressed that since less instructions were given, they had to know more about what was going on and know the procedure, which forced them to learn the concepts. This gave the test group student confidence in the concepts they had learned. The other test group student stated that since there were fewer instructions, “I had to research into why I was doing something, which helped me understand it more.” This student also felt like they did not have to rely on traditional “cookbook” experiments anymore, which increased their confidence in their conceptual knowledge. The varied answers between the students were expected because all students learn differently, but it does give insight into what students find helpful in helping them learn the material and their viewpoints on certain aspects of the course.

The students were asked which experiment helped them learn more concepts. All of the students stated that they learned the most from the Experiment 10, the independent method development experiment. The control group students both shared that they found the last experiment very difficult since it was unlike any of their previous traditional experiments, where they received a detailed procedure. Even with this added difficulty, they felt like in the process of researching to develop their own method of analysis, they learned more because they had to think about the concepts involved in the problem, how they fit together, and how to accomplish the problem effectively. The test group students also indicated that they learned more from the last experiment or experiments, since they also had Experiment 9 being a group method development experiment. As before, the test group students stated that these experiments helped them because they learned more about the concepts through having to research them to come up with their own method of analysis. Overall, the consensus from all of the students was that the last experiment greatly enhanced their conceptual knowledge.

Students were asked how they thought their manual could have been changed to help them increase their conceptual knowledge of analytical chemistry. The test group students agreed that they could not think of any changes that could be implemented to help them learn more and the transitional laboratory manual was designed well. Both of the control group students thought that since they learned more concepts because of Experiment 10, it would help if there were more experiments like those that forced students to interact with the material and allowed them to be more creative. One of these students recommended that there could be a gradual transition leading up to the last experiment, with each subsequent experiment building upon the next, leaving out more information, to get students to think more for their selves. The student said, “I think it would be a little bit easier if each subsequent experiment…it built up to that (Experiment 10). It seemed kind of sudden, like cookbook chemistry and then now you get to think for yourselves, as opposed to gradual thinking for yourself…leading into it.”

Both laboratory manuals have students working in groups for Experiments 5 (“Determination of Acetic Acid in Commercial Vinegar by Potentiometric Titration”), 6 (“Determination of Fluoride Using a Fluoride Ion Selective Electrode”), and 7 (“Spectrophotometric Determination of Iron Via Its 1,10-Phenanthroline Complex”); the test group had the additional Experiment 9 (Group Method Development). The control group and test group students were asked if they enjoyed working in groups and if working in a group increased their learning. The control group students had mixed feelings about it. One shared that they usually do not enjoy working in groups because of previous bad experiences; however, they did state that they worked well with their partner in this course and learned a lot from it. The other one said that they always enjoy working in groups and actually feel more comfortable whenever they have someone there to support them; this helps them learn more than they would on their own. For the test group, both students indicated that they enjoyed working in groups, especially in Experiment 9, group method development. They felt like they learned more because they could discuss ideas on what to do on their method and they ended up learning a lot from their partner. In all of the cases, it seemed that by having the students work in some of the middle experiments in a group, they were able to undergo the steps necessary to turn a group into team and make for more effective learning; this was reflected in how well the test group students were able to communicate and learn from each other to develop a method to complete experiment 9.

Lastly, students were asked if they thought the laboratory manual helped prepare them for a professional career in chemistry/science-related field. Both of the control group students thought that their manual was insufficient for this. One of the control group students enjoyed having the details in the manual, but thought that the laboratory manual could use more experiments that made students think more the material and how to solve problems. The other control group student stated:

> "To be out in a career where they're interested in method development and they want you to sort of think outside the box and improve things, I don't think that the current lab manual sort of pushes in that direction. It's like a cookbook, I think you can get decent results based on that, but I feel like the current lab manual could have prepared me for the job that I'm in now, as a lab technician, but not for a job that I wanted to work up to, as like a chemist or, you know, a higher level job. So, you can perform the techniques using the lab manual, but you can't do what's required for a better job, a more prestigious job."

After this, the student explained that more method development experiments in the manual would have helped with putting more thought into completing the experiments and would make for a better laboratory manual. Both of the test group students thought that their manual had prepared them for professional careers. They both shared that they felt like having to do research and think for their selves to solve problems, in the ending method development experiments, resembled real-world experiences.  One student shared:

> "..there at the end…you told us what we needed to do and then we had to come up with how to do it and you prepared us all semester for that. You held our hand there at the beginning, but then there at the end, you kind of just let us go play and figure it out, and use what we learned and what you taught us over the semester, to come up with our own methods and I felt like that was so important…when did our group techniques and individuals. I feel like that was such an integral part was being able to figure it out yourself, or with your partner, and that's exactly what it's like to work in industry. You know what you have, you know where you need to get, and you have to figure out the middle part and whether that means using primary sources, outside research, a friend, coworker, having to go find someone…who is over you and understands what's going on, you have to be able to do all that. I feel like those last two experiments especially, where was just, you know, this is what is have, this is where you need to get, figure it out. I felt like that was so integral. I feel like it was really representative of what working in industry would be like."

Both test group students shared this opinion of the transitional laboratory manual being representative of a professional career and because of it, they felt well prepared.

# CHAPTER 4

# CONCLUSIONS AND FUTURE WORK

## Conclusions

The research study focused on analyzing the qualitative and quantitative data to determine if the null hypotheses were valid and true. The null hypotheses focused on two statements:

> The transition from traditional "cookbook" to inquiry-based experiments in quantitative analysis laboratory decreases or has no effect on the student's conceptual understanding of chemistry.

> The students were not able to apply analytical techniques to solve related real-world problems.

When analyzing this data for the null hypotheses, the control group had less understanding of the conceptual material based on the control group students focusing on the "big picture" in their developed methods, rather than also looking at the small details like the test group students. Even though the test group students had a decreased confidence because of focusing on the small, important details, this showed that these students had an increased understanding of the conceptual material involved in the experiments; this, combined with test group students having less difficulty on the final exam questions (0.828 for the test group, 0.600

for the control group), showed the new transitional lab manual had an increase in student conceptual understanding. This made the first null hypothesis invalid.

The students' ability to successfully solve the Experiment 10 was used to determine if they were able to apply analytical techniques to solve real-world related problems. The test group students were able to obtain more accurate values for the iron analysis and had overall smaller standard deviations than the control group; the developed methods from the test group students were more consistent in determining the true concentration of the iron and percent acidity of the unknown sample. This may have been due to the test group students' ability to focus on more small, important details than the control group students. Even though both the control group and test group students were able to use analytical techniques to solve real-world problems, it appeared as though the increased conceptual understanding in the test group allowed them to solve the problem with more accuracy and precision than the control group. Since, the test group students were able to use analytical techniques to solve related real-world problems, the second null hypothesis was also determined to be invalid.

Since both null hypotheses were invalid, it proved the hypothesis to be valid and true:

> The transition from traditional "cookbook" to inquiry-based experiments in quantitative analysis laboratory does increase the student's conceptual understanding of chemistry. The students were able to successfully apply analytical techniques to solve related real-world problems.

The new transitional laboratory curriculum was able to increase the students' understanding of quantitative chemistry concepts. Their increased knowledge helped them to successfully solve problem faced in analytical chemistry.  To understand how the new transitional laboratory manual helped the test group students, the design of the manual was analyzed through the qualitative data to see what aspects of the curriculum design enabled their learning of quantitative chemistry concepts.

The qualitative data, surveys and interviews, showed how the Quantitative Laboratory students viewed their own learning, their confidence in the subject area and it gave insight into their thought process during the method development process. The data from student interviews and surveys indicated that the test group students felt more confident in the concepts they learned and felt that it was due to how the manual gradually transitioned them from the traditional experiments to the method development experiments. The students carried the confidence that they gained throughout the transitional curriculum into the last method development experiment. It was observed that the test group students were confident in preparing a method for this last experiment, and thought of it has having fun. The control group students looked at this experiment completely differently from a confidence standpoint; the students seemed anxious and stressed because of the assignment. These students even stated in their interviews that it was hard moving from all traditional experiments to a method development experiment. They felt like they learned more in having to prepare for the last experiment, but they wished they there had been some type of transition from the traditional experiments to the method development experiment.

The test group also showed more confidence in their developed methods, since a majority of the students used their originally developed method, instead of changing it. The control group students were more insecure in their ability to develop a method, with half of the students changing the analytical techniques from their original method to different ones. However, this increased confidence in the test group students was not reflected in the SALG survey results. This was believed to be due to a false sense of confidence. Upon inspection of the qualitative data from the Experiment 10, independent method development, the test group students performed better by remembering important details, which many of the control group students overlooked. The test group students had a greater understanding of the concepts involved in the experiments and were more aware of these types of small details and the difficulty in developing a method. The control group students looked more at the “big picture” and failed to notice these important aspects of their methods and thought the problem to be easier than what it really was. This gave the control group students a false sense of knowing the concepts well and therefore a false sense of confidence in their ability to develop a method. This was also reflected in the control group students’ performance on the final exam. Many of them used MSA in their developed methods, but several of them got those questions wrong. Their confidence in their method development abilities were not linked to their knowledge of the material or concepts.

It was believed the increased confidence in the test group and lack of confidence in the control group was also direct reflection of ZPD. The test group was able to learn because the level of challenge and competence fell within the ZPD; they were challenged to use the concepts they knew in a different way and they had the confidence to do it without being stressed. The control group students were also challenged, but had a harder time with the concepts and were not confident in their knowledge; this caused the students to become anxious and stressed while completing this assignment. Given this, the transitional manual seemed to have enabled students to fall within the ZPD by assigning tasks that were designed to press them to a reasonable expectation based on their existing conceptual understanding of the subject. The transitional manual only discussed a concept the first time it appeared in the laboratory manual. The next time it appeared, no details were given and student had to depend on their previous knowledge to complete that current experiment. It enabled students to build a scaffolding of their understanding of what that concept was and how it could be applied to something new. By not reviewing these concepts the next time they were used in the manual, the students had to depend on their scaffolding of the knowledge to build on it again and apply it to something new. This scaffold building process allowed to students to be ready and confident for the last, method development, experiment and allowed them to fall within the ZPD.

The scaffolding structure for basic principles of concepts was reflected in the final exam questions too. The test group students found the first three questions (a-c), which focused on recalling basic principles and being able to summarize concepts, less difficult than the control group. This showed that the test group students were able to build a good supporting foundation of knowledge for their conceptual understanding during the scaffold building process.

This transitional manual also utilized the Five E's as a part of the scaffolding building process. This was accomplished by assigning a summary report for every experiment. In this report, the students had to discuss the concepts involved in the completed experiment and their implications on the results they obtained. Since the experiments were set up as problem solving experiments, the students were Engaged by being able to see how particular concepts are applicable to real-world problems, this was mimicked in the pre-laboratory lecture as well as the laboratory manual. The students then had to Explore the problem by completing the experiment. In the summary report, they had to Explain what they learned during the experiment, Elaborate on concepts involved, and then Evaluate their data based on their understanding of the concepts. By enabling students to build a scaffolding of their conceptual knowledge on every experiment as the semester progresses, it allowed related concepts to build on each other to give students more understanding on what those concepts are and how they can be used to solve problems.

There were several experiments, four out of ten, that were to be completed as group assignments (three transitional experiments and then the group method development). In these groups, students had to learn to work as a team to accomplish their goal. During this process, students were able to negotiate meaning and understanding in a discussion with their laboratory partner; this enabled them to develop their knowledge and form an understanding of the concepts involved in those experiments. Students both the control group and test group indicated that, through collaborative learning, they were able to learn from their partner and increased their understanding of the concepts. They were able to discuss what they knew about the concepts and helped each other increase their understanding of it. The last group experiment allowed students to complete a problem solving experiment by developing their own method of analysis. During the method development process, the students had to discuss how they thought the problem would be solved and had to explain their reasoning for it. This process allowed students to critique each other and peer review, build upon the conceptual knowledge that they already had to come up with their method and ultimately increase their conceptual understanding. Both of the test group students that were interviewed indicated that being able to work in a group greatly increased their learning of analytical chemistry.

This constructivist type of learning (scaffold building) was encouraged throughout the transitional curriculum, something that completely was missing from the old one. There were several aspects that were considered when developing the transitional laboratory manual. The gradually transitioned from traditional to inquiry-based experiments (method development), manual utilized problem-based learning, the Five E's through summary reports, and team learning to allow students to operate within the ZPD throughout the semester and for the final independent method development experiment. This allowed the students to have more confidence in their knowledge of analytical chemistry concepts and increased their conceptual understanding; through this, the students were able to successfully develop their own methods of analysis to solve real-world related problems. Although the control group students were able to develop methods, the methods were not as successful in obtaining accurate and precise results. With the traditional curriculum, the students felt anxious and stressed over having to develop their method and felt that even though they learned more from this experiment it was difficult and a transition was needed for them to be more confident in their ability to solve these types of problems. All of the students agreed that the last, method development, experiment was the closest laboratory experience that they have experienced to how research is conducted in professional chemistry or science-related careers. They also all agreed that traditional curriculums did not prepare students for their professional careers.

So, why are laboratory classes still being taught with a traditional curriculum? The old traditional curriculum needs to be altered to facilitate conceptual learning and prepare students more for what they will experience in their careers once they graduate. One of the test group students put this into perfect terms:

> "Just having to understand why things work and understanding why you're doing something, other than just doing it, is such an important thing to be able to ask, especially in science and you get all of these chemistry majors who have just read a book their entire life that tells them exactly what to do. So, first of all, they're not asking questions. Second of all, they don't know why they're doing anything. Third, how in the hell are they a scientist? They are doing what everyone else does. They're not coming up with

> their own procedures. They don't understand why they're doing anything, they're just doing what they are told because they were told to do it, and that's not good science."

A traditional laboratory curriculum is not doing students any favors or teaching them on a level that they need to be able to operate on after graduation. This student and the success of this transitional curriculum puts things in perspective; how chemistry laboratories are taught needs to change if students are to be treated justly by getting an education that they will be able to use for the rest of their lives.

## Future Work

Due to time constraints, this research study was only completed using one control group and one test group. To increase sample population and be able to have more data to run more statistical test, this research could be carried out with more control groups and test groups with future quantitative laboratory sections at East Tennessee State University. The study could also be increased to an inter-academia level and be tested at other universities to see if it could help their students increase their understanding of analytical chemistry concepts.

Both groups were taught by the same laboratory instructor. Although this was intentional in the study to control as many variables as possible, there has been no data collected to see how students will do with a different laboratory instructor using the new transitional laboratory manual. The new manual was designed with the intention of it being taught by someone trained in inquiry teaching; many times, this is not always the case and teachers can sometimes be apprehensive and resist teaching style/tactic that they are unfamiliar with. It is recommended that anyone who wants to use this transitional laboratory manual in their own courses be familiar, preferably experienced, with inquiry teaching.

This type of transitional manual was helpful in facilitating conceptual learning for students in analytical chemistry. A transitional curriculum like this is unique and no research was found for similar research studies adapting this type of manual into a laboratory course. More research studies could be performed where a transitional curriculum is applied to other types of laboratory courses, *i.e.* organic.

One of the goals of the research was to have students successfully apply analytical techniques to solve real-world problems. Problems faced in professional careers can be unpredictable and difficult to solve; sometimes taking years of research and fail to finally be successful. The experiments for this new laboratory manual were designed to be related to the real-world and solvable on their current level of knowledge. There is a big difference between these two types of problems. Time was a factor for this research study, but it would be interesting to complete a long-term study to see if this laboratory manual was able to help students increase their conceptual knowledge to a point they were able to help them solve the more difficult problems they will be faced with in their future careers.

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APPENDICES

Appendix A
SALG Survey

## **Survey for CHEM 2221: Quantitative Analysis Laboratory Course**

This survey will be used in a research study to see students' opinions and feelings on the curriculum used in CHEM 2221.
This survey was developed from a validated and reliable survey called Student Assessment of their Learning Gains (SALG). The purpose and focus of this survey is to help determine if a course had enabled student learning.

There are a total of 50 questions and should take approximately 30 minutes to complete.

---

Semester (Spring/Fall) ______________ Year ______________
Lab Section_______________________

Please circle the most appropriate answer.

1. Which gender do you identify with? Male or Female

2. What is your expected grade in this laboratory class? A B C D F

3. Is Chemistry one of the following for you…

   Your Major Your Minor An Elective Course

I. <u>Your Understanding of the Class Content</u>
As a result of your work in this class, what GAINS DID YOU MAKE in your UNDERSTANDING of each of the following?

| | No Gains | A Little Gain | Moderate Gain | Good Gain | Great Gain |
|---|---|---|---|---|---|
| 1. The main concepts explored in this laboratory class | O | O | O | O | O |
| 2. The concepts explored in this class | O | O | O | O | O |
| 3. The following concepts that have been explored in this class… | | | | | |
| • UV-vis for elemental analysis | O | O | O | O | O |
| • Gravimetric Analysis | O | O | O | O | O |
| • Calibration Curves | O | O | O | O | O |
| • Titration Curves | O | O | O | O | O |
| • Analytical techniques (volumetric pipetting, need for precision and accuracy, etc.) | O | O | O | O | O |
| 4. How ideas from this class relate to ideas encountered in other classes within chemistry | O | O | O | O | O |
| 5. How studying quantitative chemistry helps people address real world issues | O | O | O | O | O |

6. Please comment on HOW YOUR UNDERSTANDING OF ANALYTICAL CHEMISTRY HAS IMPROVED as a result of this class.

7. Please comment on how THE WAY THIS CLASS WAS TAUGHT helped you learn analytical concepts.

II. <u>Increases in Your Skills</u>

As a result of your work in this class, what GAINS DID YOU MAKE in the following SKILLS?

| | No Gains | A Little Gain | Moderate Gain | Good Gain | Great Gain |
|---|---|---|---|---|---|
| 1. Engaging your curiosity and attention in the topic of the experiments | O | O | O | O | O |
| 2. Explaining what happened during the experiment process | O | O | O | O | O |
| 3. Relating what you learned during an experiment to your existing knowledge of that concept | O | O | O | O | O |
| 4. Recognizing a sound argument and appropriate use of evidence | O | O | O | O | O |
| 5. Developing a logical argument | O | O | O | O | O |
| 6. Writing documents in discipline-appropriate style and format | O | O | O | O | O |
| 7. Critically reading articles about topics raised in class | O | O | O | O | O |
| 8. Working effectively with others | O | O | O | O | O |
| 9. Designing an experiment to answer questions | O | O | O | O | O |

10. Please comment on what SCIENTIFIC INQUIRY SKILLS you have gained because of this class.

III. The Curriculum for the Course

DRAW a smiley face next to the word COURSE at the TOP of this page.

HOW MUCH of the following aspects of the curriculum HELPED YOUR LEARNING?

| | No Help | A Little Help | Moderate Help | Good Help | Great Help |
|---|---|---|---|---|---|
| 1. Pre-lab Lecture | O | O | O | O | O |
| 2. Writing Summary Reports | O | O | O | O | O |
| 3. Writing the Short and Formal Reports | O | O | O | O | O |
| 4. The format of the lab manual going from more to less details/information in the experiment procedure | O | O | O | O | O |
| 5. Using journal articles | O | O | O | O | O |
| 6. Using online materials (other than the ones provided by the teacher) | O | O | O | O | O |

7. Please comment on how the DESIGN OF THE COURSE CURRICULUM helped you learning.

## IV. The Instruction and Teaching of the Course

HOW MUCH of the following aspects of the instruction and teaching HELPED YOUR LEARNING?

| | No Help | A Little Help | Moderate Help | Good Help | Great Help |
|---|---|---|---|---|---|
| 1. The pace of the class | O | O | O | O | O |
| 2. How the class topics, activities, reading, and assignments fit together | O | O | O | O | O |
| 3. Traditional Experiments with full details on how to complete the assignment. | O | O | O | O | O |
| 4. Experiments where you had to provide more information to complete the assignment. | O | O | O | O | O |
| 5. Group Method Development Experiment | O | O | O | O | O |
| 6. Individual Method Development Experiment | O | O | O | O | O |
| 7. The Instructor's explanation of how the class activities related to each other | O | O | O | O | O |
| 8. The Instructor's explanation of why the class focused on the topics presented during lab. | O | O | O | O | O |

9. Please comment on how this class INSTRUCTIONAL APPROACH to this class helped your learning.

## V. Your Experiences in this Course

HOW MUCH of the following aspects of this class HELPED YOUR LEARNING?

| | No Help | A Little Help | Moderate Help | Good Help | Great Help |
|---|---|---|---|---|---|
| 1. Participating in discussion during lab | O | O | O | O | O |
| 2. Participating in group work during lab | O | O | O | O | O |
| 3. Interacting with the instructor | O | O | O | O | O |
| 4. Interacting with peers | O | O | O | O | O |

5. Please comment on how the PERSONAL EXPERIENCES you had in this class helped your learning.

## VI. Class Impact on Your Attitudes

Circle, or draw a small picture next to, the page number at the TOP of this page

As a result of your work in this class, what GAINS DID YOU MAKE in the following?

| | No Gains | A Little Gain | Moderate Gain | Good Gain | Great Gain |
|---|---|---|---|---|---|
| 1. Enthusiasm for this subject | O | O | O | O | O |
| 2. Interest in taking or planning to take additional classes in this subject | O | O | O | O | O |
| 3. Confidence that you understand and can do analytical chemistry | O | O | O | O | O |
| 4. Your comfort level in working with complex ideas in analytical chemistry | O | O | O | O | O |
| 5. Willingness to seek help from others (teacher, peers, TA) when working on analytical problems | O | O | O | O | O |
| 6. Confidence in completing an independent research project in a professional career | O | O | O | O | O |
| 7. Confidence in pursuing a professional career in chemistry or a science-related field | O | O | O | O | O |

8. Please comment on how this class CHANGED YOUR ATTITUDES toward analytical chemistry.

Appendix B
Method Development Questions on the Final Exam

5. You recently started a new job at an analytical services laboratory. Your first assignment is to determine the amount of Na in a sample using an Inductively Coupled Plasma – Optical Emission Spectrometer (ICP-OES) for the analysis. The IDL for the instrument is 0.005ppm and the LR is 10ppm.

(a) What does IDL stand for and why does it occur on an analytical instrument?
(**2 points**)

(b) What does LR stand for and why does it occur on an analytical instrument?
(**2 points**)

(c) When developing a method for determining the concentration of Na in the above problem, what general steps would you take in starting to develop a method of analysis on the ICP-OES? Assume at this point that the instrumentation setup of the ICP-OES is similar to the AA and you have already completed your research.
(**2 points**)

(d) If your graph of the data you obtained from the experiment look like Figure 1:

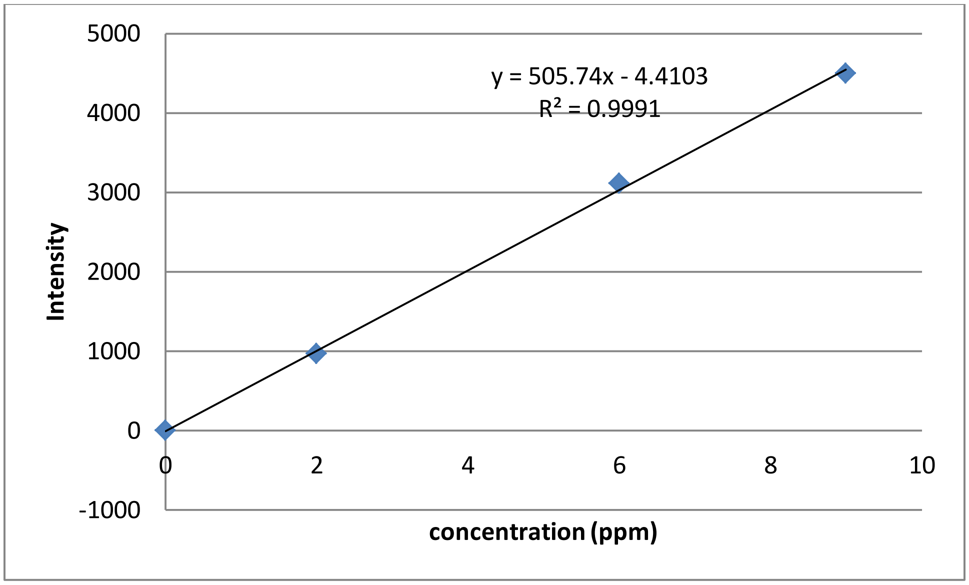


Figure 1 – Calibration Curve of Na on ICP-OES

(i). Can this graph be used to determine the correct Na concentration in the unknown sample if its intensity was found to be 1362? Why or Why not? If so, calculate the unknown concentration. (**3 points**)

(ii). Can this graph be used to determine the correct Na concentration in the unknown sample if its intensity was found to be 4985? Why or Why not? If so, calculate the unknown concentration. (**3 points**)

(e) If your graph of the data you obtained from the experiment look like Figure 2:

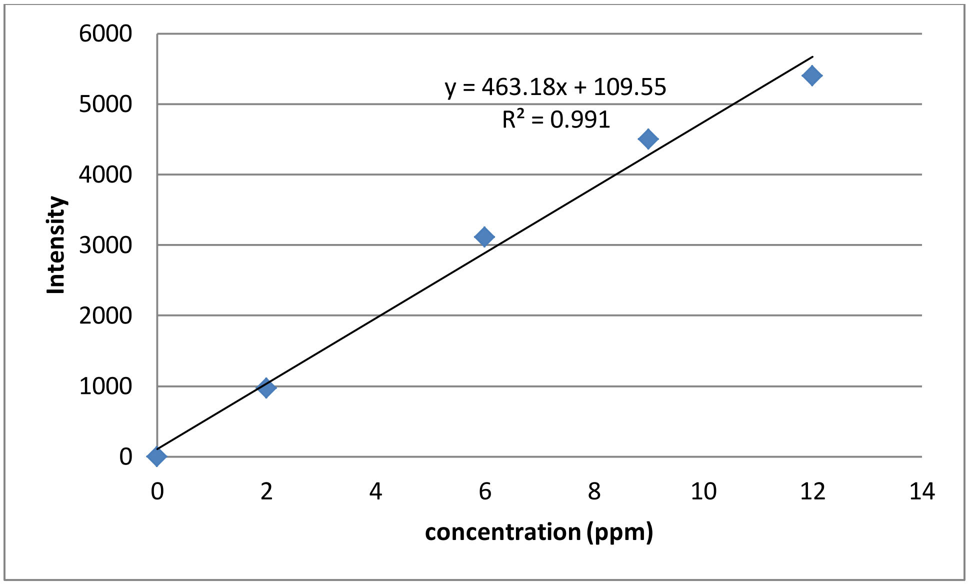


Figure 2 – Calibration Curve of Na on ICP-OES

(i). Can this graph be used to determine the correct Na concentration in the unknown sample if its intensity was found to be 1647? Why or Why not? If so, calculate the unknown concentration. **(3 points)**

(f) Use the following Figure 3 to answer the next two questions.

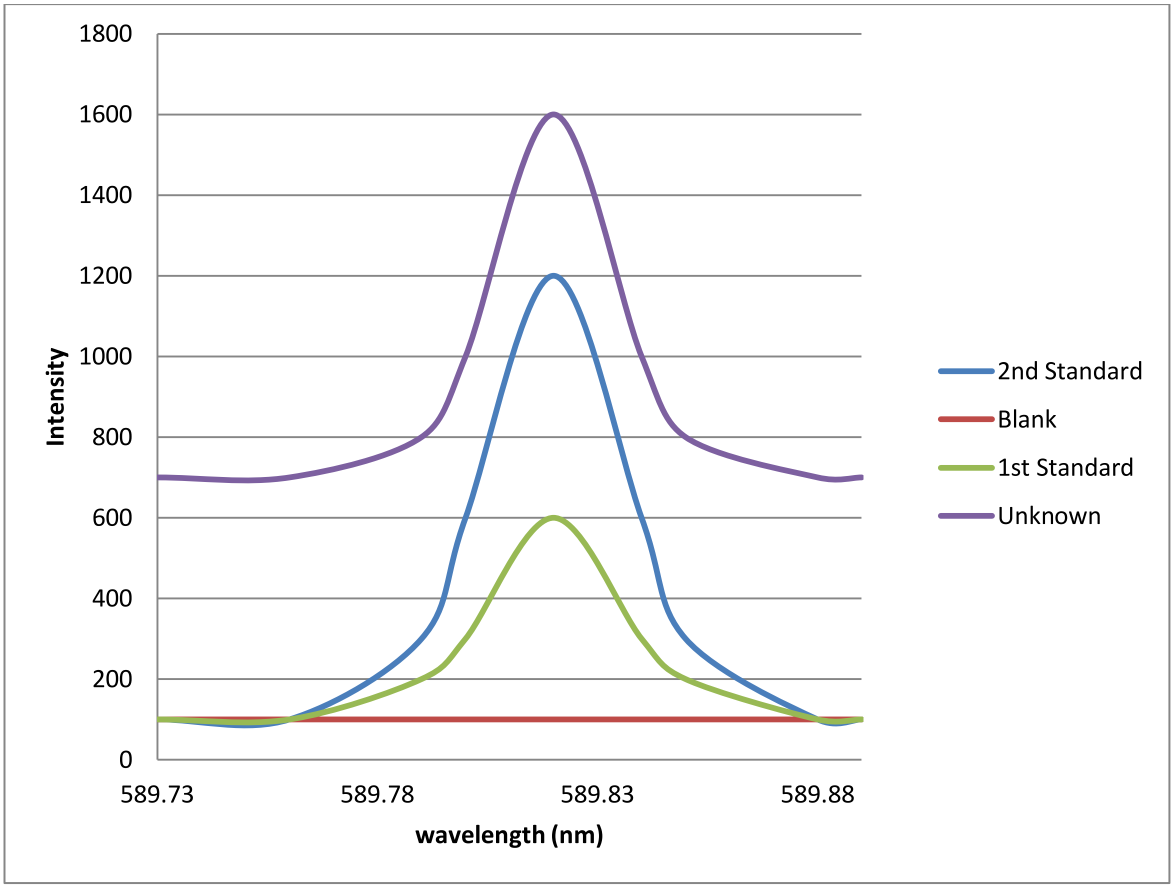


Figure 3 – Wavelength spectra of the calibration standards and unknown for Na at 589.82 nm

(i). Name two instances, that we have discussed in class, that the unknown sample would be elevated above the calibration standards as in Figure 3. (**2 points**)

(ii). How would you fix each situation you listed in 5(f)(i) to get better results? (**3 points**)

Appendix C
IRB Approval Form

East Tennessee State University
Office for the Protection of Human Research Subjects • Box 70565 • Johnson City, Tennessee 37614-1707
Phone: (423) 439-6053 Fax: (423) 439-6060

**IRB APPROVAL – Initial Expedited Review**

November 12, 2014

Sarah R Johnson

**Re:** Quantitative Chemistry Laboratory: A transition from traditional format to critical thinking and method development in the laboratory manual for CHEM 2221.
**IRB#:** c0814.14s
**ORSPA #:** n/a

The following items were reviewed and approved by an expedited process:

- xform New Protocol Submission; Informed Consent Document* (version 8/27/14, stamped approved 9/18/14); Survey; Interview Questions; Literature; CITI; CV

The item(s) with an asterisk(*) above noted changes requested by the expedited reviewers.

On **September 18, 2014**, a final approval was granted for a period not to exceed 12 months and will expire on **September 17, 2015**. The expedited approval of the study and requested changes will be reported to the convened board on the next agenda.

***Note: Requested changes were approved on 9/18/2014; however, final approval of this study was on hold due to pending issues and was granted & issued to PI on 11/12/2014.*

The following **enclosed stamped, approved Informed Consent Documents** have been stamped with the approval and expiration date and these documents must be copied and provided to each participant prior to participant enrollment:

- Informed Consent Document (version 8/27/14, stamped approved 9/18/14)

Federal regulations require that the original copy of the participant's consent be maintained in the principal investigator's files and that a copy is given to the subject at the time of consent.

*Accredited Since December 2005*

**Projects involving Mountain States Health Alliance must also be approved by MSHA following IRB approval prior to initiating the study.**

Unanticipated Problems Involving Risks to Subjects or Others must be reported to the IRB (and VA R&D if applicable) within 10 working days.

Proposed changes in approved research cannot be initiated without IRB review and approval. The only exception to this rule is that a change can be made prior to IRB approval when necessary to eliminate apparent immediate hazards to the research subjects [21 CFR 56.108 (a)(4)]. In such a case, the IRB must be promptly informed of the change following its implementation (within 10 working days) on Form 109 (www.etsu.edu/irb). The IRB will review the change to determine that it is consistent with ensuring the subject's continued welfare.

Sincerely,
Stacey Williams, Chair
ETSU Campus IRB

Appendix D

Answers and Grading Rubric for Method Development Questions on the Final Exam

5. You recently started a new job at an analytical services laboratory. Your first assignment is to determine the amount of Na in a sample using an Inductively Coupled Plasma – Optical Emission Spectrometer (ICP-OES) for the analysis. The IDL for the instrument is 0.005ppm and the LR is 10ppm.

(a) What does IDL stand for and why does it occur on an analytical instrument? (**2 points**)

**(1) – Instrument Detection Limit**
**(1) – The concentration of the analyte is too low for the instrument detectors to distinguish the true analyte concentration from the background noise of the instrument.**

(b) What does LR stand for and why does it occur on an analytical instrument? (**2 points**)

**(1) – Linear (Dynamic) Range**
**(1) – The concentration of the analyte is too high for the instrument to read correctly. The high analyte concentration saturates the instruments detectors and gives inaccurate results. Must obey Beer's Law.**

(c) When developing a method for determining the concentration of Na in the above problem, what general steps would you take in starting to develop a method of analysis on the ICP-OES? Assume at this point that the instrumentation setup of the ICP-OES is similar to the AA and you have already completed your research. (**2 points**)

**Any of the following:**

**(1) – Determine type of method to be used (MSA or normal calibration curve).**
**(1) – Determine number of standards (spike) to be used in calibration curve (MSA).**
**(1) – Decide on concentration of calibration standards (spikes) based on IDL and LDR.**
**(1) – Determine if sample preparation is needed before analysis.**

(d) If your graph of the data you obtained from the experiment look like Figure 1:

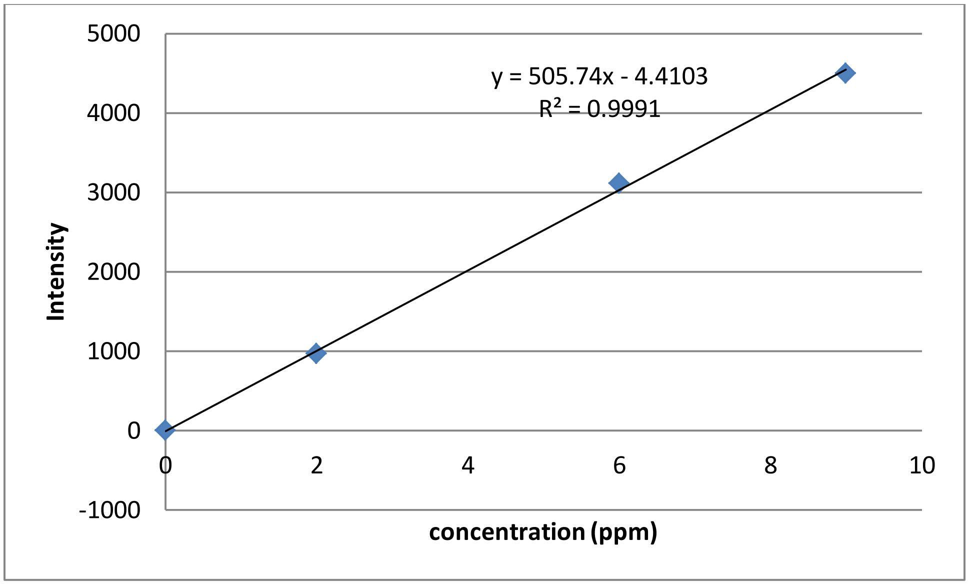


Figure 1 – Calibration Curve of Na on ICP-OES

(i). Can this graph be used to determine the correct Na concentration in the unknown sample if its intensity was found to be 1362? Why or Why not? If so, calculate the unknown concentration. (**3 points**)

**(1) – Yes**
**(1) – Calibration curve is good, linear, and all calibration standards are within the LDR of the instrument.**
**(1) – y= 505.74x – 4.4103**
**y=1362**

**x=2.702 ppm**

(ii). Can this graph be used to determine the correct Na concentration in the unknown sample if its intensity was found to be 4985? Why or Why not? If so, calculate the unknown concentration. (**3 points**)

**(1) – No**
**(1) – Intensity is above the high calibration standard. When unknown value is above calibration curve, even if it is below the linear range, it is considered unreliable because of the possibility of it being inaccurate. (The unknown sample should be diluted to be within the calibration curve).**
**(1) – Doesn't calculate value.**

(e) If your graph of the data you obtained from the experiment look like Figure 2:

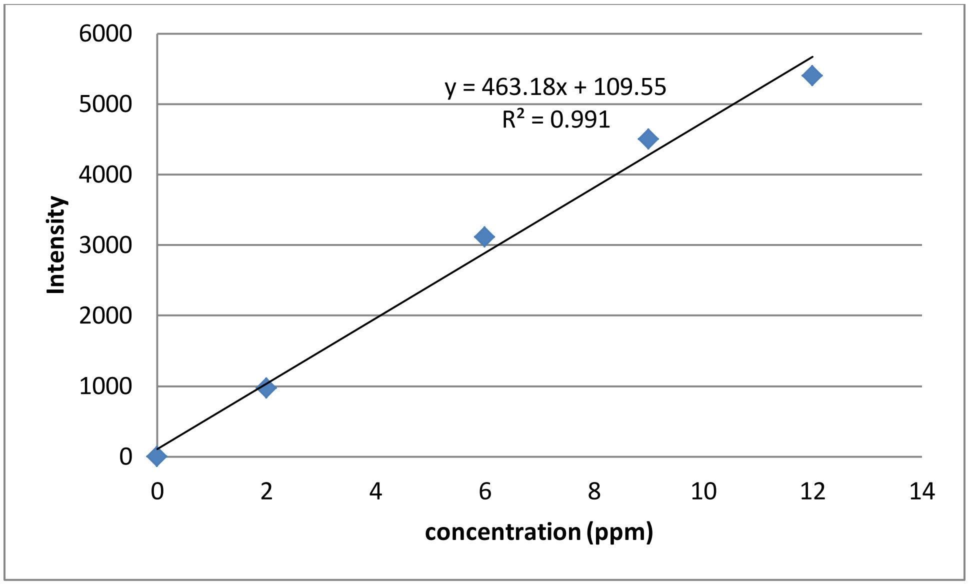


Figure 2 – Calibration Curve of Na on ICP-OES

(i). Can this graph be used to determine the correct Na concentration in the unknown sample if its intensity was found to be 1647? Why or Why not? If so, calculate the unknown concentration. **(3 points)**

**(1) – No**
**(1) – The high calibration standard is above the linear range of the instrument. Calibration curve doesn't follow Beer's Law.**

**(1) – Doesn't calculate value.**

(f) Use the following Figure 3 to answer the next two questions.

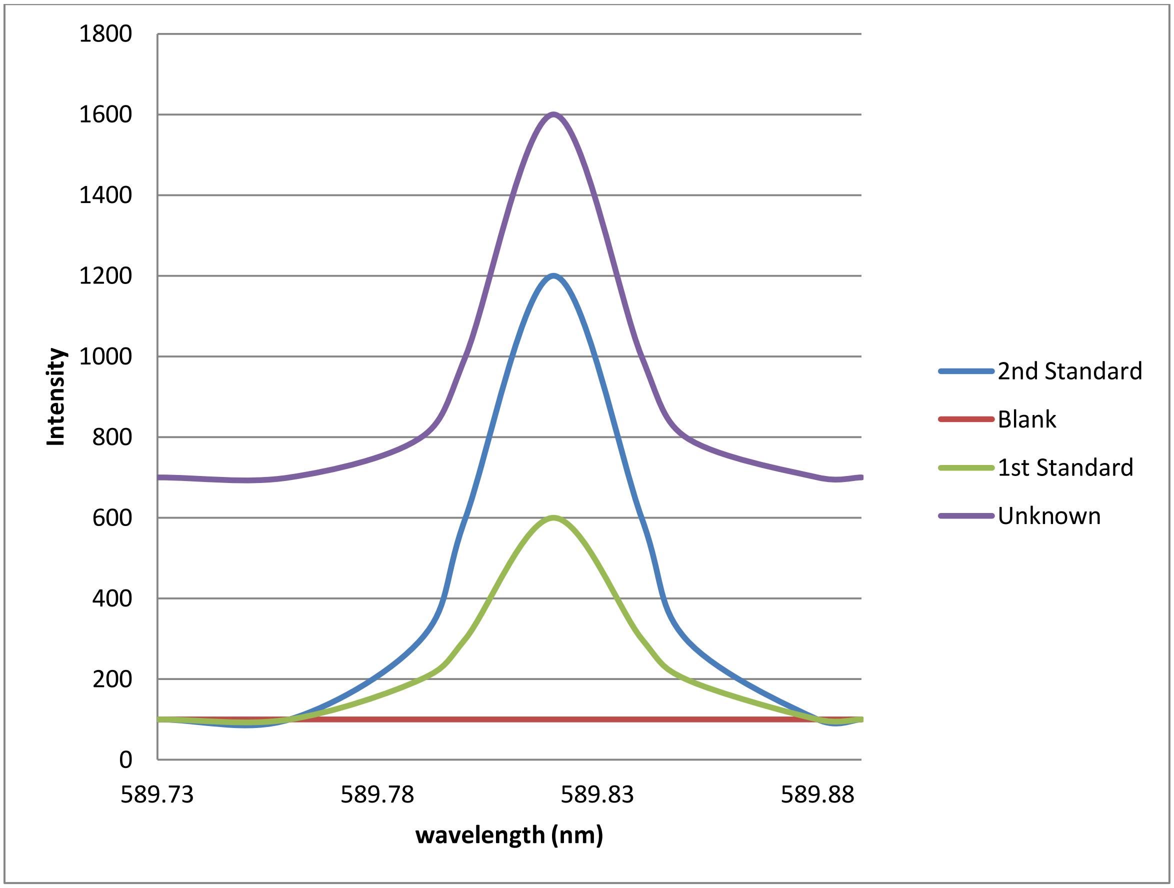


Figure 3 – Wavelength spectra of the calibration standards and unknown for Na at 589.82 nm

(i). Name two instances, that we have discussed in class, that the unknown sample would be elevated above the calibration standards as in Figure 3. (**2 points**)

**(1) – Calibration standards do not contain same matrix components as the unknown sample.**

**(1) – Calibration Standards and unknown sample matrix components are not in the same concentration (components are the same, but in different concentrations).**

(ii). How would you fix each situation you listed in 5(f)(i) to get better results? (**3 points**)

**(1) – Ensure matrix components of both calibration standards and unknown sample are the same and in the same concentrations.**

**(1) – Can try diluting the unknown sample, therefore diluting the sample matrix to better match the matrices of the calibration standards.**

**(1) – MSA**

# VITA

## SARAH REBECCA JOHNSON

| | |
|---|---|
| Education: | M. S. Chemistry, East Tennessee State University, Johnson City, Tennessee 2016<br>B.S. East Tennessee State University, Johnson City, Tennessee 2015<br>Pre-Pharmacy Studies, Eastern Kentucky University, Richmond, Kentucky 2006 |
| Professional Experience: | Chemist, Unimin Corporation, Spruce Pine, NC 2010-present<br>Graduate Teaching Assistant, East Tennessee State University, College of Arts and Sciences, Johnson City, Tennessee 2013-2016 |
| Additional Experience: | Event Coordinator and Creator, Color Me Chemistry 5k, East Tennessee State University, Department of Chemistry, Johnson City, Tennessee, 2014-present |
| Honors and Awards: | Outstanding Graduate Student of the Year, East Tennessee State University, Department of Chemistry, Johnson City, Tennessee, 2014<br><br>Outstanding Teaching Assistant of the Year, East Tennessee State University, Department of Chemistry, Johnson City, Tennessee, 2015 |