PART I. STUDENT SUCCESS IN INTENSIVE VERSUS TRADITIONAL INTRODUCTORY CHEMISTRY COURSES

PART II. SYNTHESIS OF SALTS OF THE WEAKLY COORDINATING TRISPHAT ANION

by

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ABSTRACT

PART I. STUDENT SUCCESS IN INTENSIVE VERSUS TRADITIONAL INTRODUCTORY CHEMISTRY COURSES

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Part I. Intensive courses have been shown to be associated with equal or greater student success than traditional-length courses in a wide variety of disciplines and education levels. Student records from intensive and traditional-length introductory general chemistry courses were analyzed to determine the effects, of the course format, the level of academic experience, life experience (age), GPA, academic major and gender on student success in the course. Pretest scores, GPA and ACT composite scores were used as measures of academic ability and prior knowledge; *t*-tests comparing the means of these variables were used to establish that the populations were comparable prior to the course. Final exam scores, total course points and pretest-posttest differences were used as measures of student success; *t*-tests were used to determine if differences existed between the populations. ANCOVA analyses revealed that student GPA, pretest scores and course format were the only variables tested that were significant in accounting for the variance of the academic success measures. In general, the results indicate that students achieved greater academic success in the intensive-format course, regardless of the level of academic experience, life experience, academic major or gender.

Part II. Weakly coordinating anions have many important applications, one of which is to function as co-catalysts in the polymerization of olefins by zirconocene. The structure of tris(tetrachlorobenzenedialato) phosphate(V) or "trisphat" anion suggests that it might be an outstanding example of a weakly coordinating anion. Trisphat acid was synthesized and immediately used to prepare the stable tributylammonium trisphat, which was further reacted to produce trisphat salts of Group I metal cations in high yields. Results of the ^{35}Cl NQR analysis of these trisphat salts indicate only very weak coordination between the metal cations and the chlorine atoms of the trisphat anion.

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PART I. STUDENT SUCCESS IN INTENSIVE VERSUS TRADITIONAL INTRODUCTORY CHEMISTRY COURSES

CHAPTER 1

INTRODUCTION

Intensive courses are courses that are equivalent to semester- or quarter-long courses but are offered in a compressed format and are widely available in many disciplines. They include summer courses, interim courses, weekend courses, courses that may extend over an entire semester but are offered only once per week, and block-scheduled courses in K-12 education. More than 200 complete college-level programs are available in accelerated or intensive format.[1] While many educators are skeptical of the effectiveness of intensive courses, the majority of studies comparing student success in intensive versus traditional courses concludes that intensive courses are at least as effective as traditional courses.[2] Various studies have investigated the factors affecting student success in the intensive versus traditional format, but none of the studies have focused on intensive chemistry courses.

Diversity of Intensive Course Offerings

Student success in intensive courses has been documented in a wide variety of disciplines, students, and institutions. This success has been measured in various ways: course grades, exam grades, pretest-posttest differences and scores on standardized tests.[2-15] High school students have been shown to achieve higher grades in block-scheduled classes in all disciplines at a private school,[3] in various public high school classes[4-5] and in foreign language courses in an Austrian high school.[7] Student success in intensive-length, summer physics courses for gifted high school students, measured by scores on a standardized test, was found to be equivalent to that achieved in traditional-length classes.[6] Adult learners in the intensive version of an English as a second language program (ESOL) in the United Kingdom exhibited lower attrition rates and greater achievement rates, again on standardized tests, than students in the traditional-length version of the same program.[8]

A meta-analysis of the early research of both high school and college-level intensive courses published by Scott and Conrad[2] in 1991 indicated that student success in the intensive courses, assessed via course grades, scores on final exams and/or scores on standardized tests, was equal to or greater than that of students in the traditional courses, regardless of the level of the student, the format of the intensive course, or the discipline. This review compared the results of more than thirty studies of intensive courses in fifty-two different disciplines, including courses in the humanities, social sciences, physical and life sciences and various vocations. Some examples include English, philosophy, foreign language, history, psychology, economics, sociology, political science, algebra, calculus, statistics, biology, physics, architecture, pharmacy, business administration and home economics. Half of the courses studied showed significant differences between the intensive and traditional format with greater student success in the intensive version. The remaining courses showed no significant differences between the two formats with the exception of one undergraduate-level education course, which found a significant difference favoring the traditional format. Other studies have supported the general idea of the greater effectiveness of intensive courses in economics[9] and information systems.[10] A comparison of unit test scores in a variety of information systems courses showed that achievement levels of students in intensive, weekend-format courses were comparable to that of students in traditional, semester courses.[10] Student achievement in three-week and 14-week economics courses was assessed using scores on the standardized RTUCE (Revised Test of Understanding of College Economics), different versions of which were given as pretests and posttests. Students in the three-week course scored an average of 10.5% higher on the RTUCE that the students in the traditional-length course.[9]

In 2001, Geltner and Logan[11] published a report entitled *The Influence of Term Length on Student Success* for Santa Monica College, in which they analyzed student success in courses from fifty different disciplines that were offered in traditional format (16 weeks) and two compressed, or intensive, formats (six and eight weeks) from 1998 through 2001. Student success in this study was evaluated by comparing average course grades among the various formats but without controlling for any differences in the student populations. They found significant differences in student success between both of the compressed formats and the traditional format in forty-five of the fifty disciplines, all favoring the compressed formats. There were no significant differences in course grades between the compressed and traditional formats in the remaining five disciplines (child development, cosmetology, fashion, nursing and physiology). These data are in agreement with the conclusions reported ten years earlier by Scott and Conrad,[12] that "intensive courses seem to be effective alternatives to traditional-length classes regardless of format, degree of intensity, discipline, or field of study."

While Scott and Conrad limited their review to studies of high school and undergraduate-level college courses, similar results have been found for graduate level courses. Student success has been found to be higher in intensive versions of graduate courses in education,[13] journalism[14] and educational psychology.[15] Williams[13] found no differences in the scores on objective tests of graduate students in elementary and secondary education in any of three formats: intensive-format courses occurring over four weekends, traditional-length, evening courses or eight-week summer courses. Seamon[15] found that graduate students in the intensive version of an educational psychology course scored higher on the posttest than did graduate students in the traditional version of the same course.

All the previous studies mentioned were limited to comparing success in individual courses; however, studies of entire accelerated programs also support these conclusions.[1,17,18] Meyer,[17] in a study of the characteristics of graduates of an accelerated registered nursing program at St. Louis University, reported that students chose that particular program, in part, due to its high NCLEX-RN (nursing licensure exam) pass rate as compared to the pass rate of students in the traditional program at the same institution. Jonas and coworkers[18] used standardized testing to compare the success of graduates of intensive versus traditional undergraduate business programs. Graduates of the intensive program scored significantly higher on the Major Fields Assessment Test, or MFAT, than did graduates of the traditional program.

While there are no comparative studies available, accelerated courses are commonly used to provide continuing education experiences for a number of professions; nurses, physicians, K-12 educators and others are required to complete specific numbers of continuing education hours to maintain licensure or certification. College-level educators also take advantage of workshops and short courses. The format of many of these professional continuing education programs qualifies them as intensive courses.[19-22]

A Closer Look at Student Success in Intensive Courses

Student Success in Intensive Courses in Science and Mathematics:

At Santa Monica College, Geltner and Logan[11] demonstrated that the student success rate, as determined by the percentage of passing grades above a D, was significantly higher in the intensive-format courses than in the traditional-format courses in many different science disciplines. The withdrawal rate of the intensive courses was also found to be significantly lower for these same classes. The list below shows success rates (intensive/traditional) and withdrawal rates (intensive/traditional) for courses in the various science disciplines:

- Astronomy: success rates, 71%/64%; withdrawal rates, 10%/14%.
- Biology: success rates, 81%/67%; withdrawal rates, 9%/19%.
- Chemistry: success rates, 82%/70%; withdrawal rates, 11%/18%.
- Computer Science: success rates, 76%/61%; withdrawal rates, 12%/21%.
- Geology: success rates, 89%/74%; withdrawal rates, 4%/13%.
- Microbiology: success rates, 86%/70%; withdrawal rates, 8%/17%.
- Physics: success rates, 80%/71%; withdrawal rates, 12%/17%.

Masat[23] found that students in six-week courses in the programming language, BASIC, had average final grades that showed no significant differences from the final grades for students in the traditional, 16-week courses. Students in a three-week version of the same course had significantly higher final grades than either of the longer formats. A comparison of student success in two intensive versus traditional mathematics courses, calculus and differential equations, at the University of Minnesota, found that there were no significant differences in student grades, once differences in GPA were taken into account.[24] Students taking biology courses in a weekend college version of the intensive format achieved higher exam and course grades and "regularly out-performed their weekday counterparts".[25] An extremely intensive statistics class, consisting of eight classes of eight hours each, was found to prepare students adequately for further courses by both students and faculty in a qualitative, interview-based evaluation.[26]

Retention of Knowledge:

A common criticism of intensive courses is that students are unlikely to retain the knowledge learned in such a short period of time. While only a handful of studies have evaluated the retention of learning from intensive courses, the available data tend to counter this criticism. Scott and Conrad[2] reported the results of three longitudinal studies in their review. High-school students in blocked classes significantly outperformed students in traditional classes on standardized achievement tests in four different subjects given immediately after the relevant classes. Six months later, the scores for the students in the blocked classes had dropped by a larger factor than for the students in the traditional classes, but the means for the two groups of students were not significantly different. That is, although the intensive-format students no longer exhibited enhanced knowledge, they retained the same amount of knowledge as the traditional-format students. College students in an intensive business administration course earned slightly, but not significantly, lower scores on a cognitive achievement test administered at the end of the course than their counterparts in a traditional class. When the achievement test was re-administered nine months later, the scores for the students from the intensive course were slightly, but not significantly higher. Pretest-posttest difference scores for students in a college-level, intensive earth science course were no different from those of students in the traditional course even when retested three months or four and one-half months after the end of the course.

Caskey[27] investigated the success rates for college students in courses that were prerequisites for other courses. Specifically, the study investigated student success in progressing from Accounting I to Accounting II and from Developmental Algebra to Intermediate Algebra. No significant differences were found between the resulting grades in the second courses for students who took the prerequisite course in the intensive or in the traditional format. Logan and Geltner[28] found no differences in English II grades between students who had taken the intensive version of English I or the traditional-length version of English I at Santa Monica College. A study[9] comparing the retention of knowledge in economics students in intensive and traditional-length courses used a standardized posttest to evaluate student success. While students in the intensive course scored 11% higher (a statistically significant difference) on the posttest than students in the traditional course, no significant differences were found in posttest scores when the students were retested in a subsequent economics course nor were significant differences found for the grades of the subsequent course.[9] Seamon[15] found that graduate students in intensive educational psychology courses achieved higher test scores than in the traditional versions of the courses but that there was no significant difference in the retention of knowledge between the two groups of students after three years.

The final example is one that does not compare intensive to traditional courses but rather points to the effectiveness of intensive courses both for learning and for retention and application of that learning in an authentic situation. Third-year medical students were offered a one-week intensive course in dementia to replace the traditional coverage of the diagnosis and treatment of this disease within a larger course. Physicians who took the one-week intensive course on dementia were found, six years later, to be far more confident in their diagnoses of dementia and more aware of community resources for patients and their families than physicians from the same graduating class who had not taken the intensive course but received instead the traditional education related to dementia.[16]

Factors Affecting Student Success in Intensive Courses

Characteristics of Students in Intensive Courses:

A 1996 survey of summer (intensive) students at West Chester Community College found that more than 80% of the students graduated from high school more than 5 years earlier, more than 70% are working with approximately 40% working full-time, and fewer than 10% are taking a summer course to replace a failing grade.[29]

The 2000 report by Logan and Geltner[28] investigated students enrolled in Spring (traditional) and Summer (intensive) courses. Their findings indicate that the ethnic mix of the students was not significantly different for the two types of courses, but that students who had chosen to enroll in intensive courses had significantly higher GPAs (2.9) than students enrolling in traditional courses (2.7). However, it is unlikely that such a small difference in mean GPA, while statistically significant, represents a meaningful difference in student academic ability. Of potentially greater importance is the observation that students enrolled in intensive courses had significantly lower withdrawal rates than students enrolled in traditional courses. In fact, those students who had enrolled in both traditional and intensive courses at Santa Monica College had both higher course GPAs and lower withdrawal rates when enrolled in the intensive courses.

Motivation:

It seems obvious that students who are more highly motivated in their academic endeavors are more likely to succeed in either intensive or traditional courses. In a study of adult graduate students taking an intensive course in public relations management, students responded to a survey designed to evaluate various factors including levels of both intrinsic and extrinsic motivation. The results suggested that neither form of motivation is correlated with age or gender but both are correlated with academic performance.[14] Motivation can also be measured using the Need for Cognition (NFC) scale developed by Cacioppo and Petty.[30] Seamon's study[15] found no difference in the NFC scores of the graduate students in intensive and traditional educational psychology courses while finding greater student success, as measured by exam scores, in the intensive format. These results suggest that motivation alone does not account for the enhanced student success in intensive courses.

Academic Experience:

While it seems logical to conclude that students with more academic experience would be able to succeed in intensive courses more readily than less experienced students, there is little evidence to support this. A comparison of grades for students who enrolled in intensive summer courses over a two-year period showed no differences in GPA between the first intensive course term and the second. That is, an additional year of college experience did not result in higher grades in the intensive courses.[28] A study of student success in an economics course was measured by scores on a standardized test (RTUCE or Revised Test of Understanding of College Economics).

The results indicated that the number of completed semester hours was not a factor in determining student success in either format.[9] One study did report that adult students were more likely to succeed (complete the program) in accelerated or traditional programs if they had prior academic experience.[1]

Life Experience:

Life experience has been shown to have both a positive and a negative impact on success in the intensive format. Geltner and Logan[11] analyzed student success in intensive (six week) and traditional (16 week) courses by age of student (Figure 1). Their data

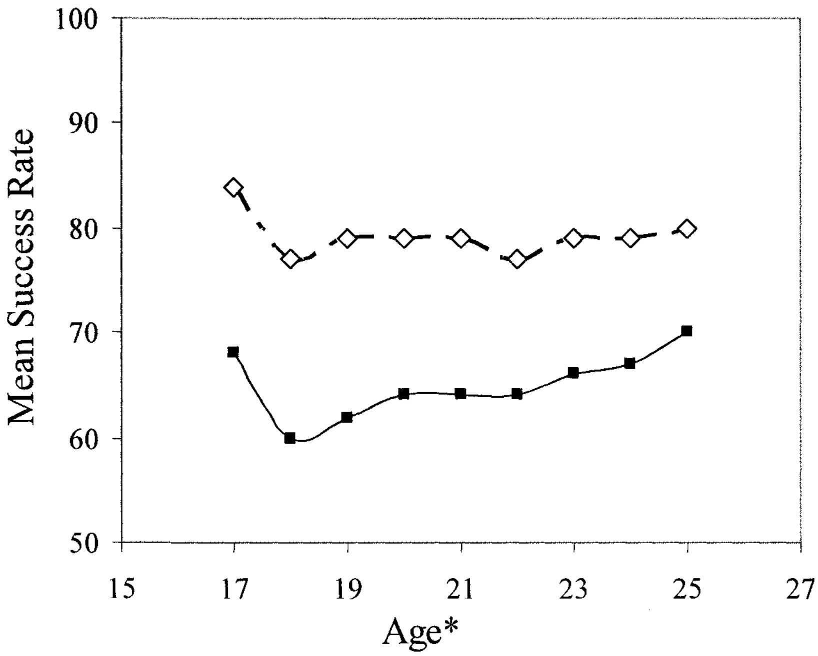

Figure 1. Student success rate (%) vs. age in intensive (◇) and traditional courses (■). *The lowest age point represents ≤ 17 years and the highest age point represents ≥ 25 years.[11]

indicate that age has a greater effect on student success in traditional courses than it does in intensive courses, but results of other studies do not agree necessarily with Geltner and Logan's data. Several studies have concluded that older students were particularly likely to succeed in intensive courses as compared to traditional courses and are more likely to persist in accelerated programs.[1,27]

Grade Point Average:

Grade point average, or GPA, is an obvious predictor of future student success in either intensive or traditional courses. However, in a large study at Santa Monica College, students with GPA scores lower than 3.0 improved their GPA more frequently when taking intensive courses than students with GPA scores above 3.0.[28] A follow-up study[11] at the same institution identified the following GPA categories and determined that the student success rates were significantly different between intensive and traditional courses in all cases. The enhanced student success rates, intensive versus traditional for the various GPA ranges were as follows:

- GPA range: 3.5 to 4.0; enhanced student success rate: 5%
- GPA range: 3.0 to 3.49; enhanced student success rate: 8%
- GPA range: 2.5 to 2.99; enhanced student success rate: 10%
- GPA range: 2.0 to 2.49; enhanced student success rate: 15%
- GPA range: 1.5 to 1.99; enhanced student success rate: 17%
- GPA range: 1.0 to 1.49; enhanced student success rate: 15%
- GPA range: 0.5 to 0.99; enhanced student success rate: 14%
- GPA range: 0.0 to 0.49; enhanced student success rate: 1%

Enhanced student success rates for traditional courses were greater for students in the middle GPA ranges than for students in the highest two GPA ranges. Students on academic probation experienced an enhanced student success rate of 17% in intensive courses versus traditional courses.[11]

Other studies have verified that GPA is an important predictor of student success and persistence[1] in undergraduate economics[9] and in graduate educational psychology[15] courses. These same studies have also shown that GPA is not more strongly correlated to success in intensive courses than it is in traditional courses.[1,9,15]

Gender:

Very few studies of student success in intensive courses have studied gender as a potential factor; the few that have present somewhat conflicting results. Geltner and Logan[11] found that female students tended to achieve higher success (higher grades) than the male students in both formats. Both male and female students had higher success rates in the intensive course format with the difference between success rates in intensive and traditional being slightly higher for male students, 16%, than that of female students, 13%. Van Scyoc[9] also found that male students added more to their knowledge than did female students in the intensive format as compared to the traditional format, as measured by differences in pretest and posttest scores. However, Kanun and coworkers[24] found that gender had no effect on student success in either format. Fall[14] suggests that, while gender may not be a factor in achievement, it may be an important factor in student satisfaction with different course formats. In a study of intensive courses in public relations management by Fall,[14] female students were found to be more satisfied when the course included opportunities for individual expression while male students tended to be less affected by the course presentation method and to rely more on test scores to determine their level of satisfaction with a course. To summarize, it seems that both male and female students achieve greater academic success in the intensive format but look to different qualities in the course for satisfaction.

Reasons for Greater Student Success in Intensive Course

Less Fragmentation, More Satisfaction:

In 1995, Fallon's study[3] concluded that the improvement in learning outcomes, including grades on semester exams, of high school students attending one class daily was due to increased opportunities for student-teacher interaction, increased involvement of the students, fewer distractions for the students and instruction methods specifically tailored for the longer classes. Other proponents of block-scheduling claim that the traditional high school schedule leads to fragmentation of learning, a problem alleviated by longer class periods and fewer subjects studied during a term.[5] Schools that have moved to block scheduling have observed improvements in student attitudes and behavior as well as academic success.[4]

New students at a private high school with more than thirty years of blocked-scheduling experience reported that "their grades improved, they were able to concentrate on a subject, they learned the subject in depth and they were able to ask teachers many questions".[3] Teachers at the same school reported that they spent less time grading, spent more time preparing for classes, and reported greater overall satisfaction with their teaching when compared with traditional class scheduling at other schools.[3] The results of a qualitative study of a foreign language program in an Austrian high school "clearly indicate both a higher variety and a richer qualitative interaction in the intensive course…".[7]

Logan and Geltner[28] interviewed college students and faculty at Santa Monica College about intensive courses in more than fifty disciplines and found that both students and faculty reported that a greater degree of cohesion developed in intensive classes, often making them memorable experiences. In a separate study, students in a college-level intensive computer programming course listed these reasons for their greater student success: they were totally involved in the course; they always had their minds on the course; the course was more coherent; they received more attention from the instructor; and, finally, the faster pace was never boring.[23] Students gave high satisfaction ratings for the very intensive statistics course described earlier.[26] However, a study by Reynolds[31] reported that, in graduate-level cohort programs, group cohesion was not influenced by term length. That is, students in both traditional-length and intensive courses developed a strong sense of group cohesion when in cohort groups but not in non-cohort courses of either format. In addition, Fall's work[14] suggests that gender, employer reimbursement, and experience with other intensive courses may be important variables for older students' satisfaction and achievement in intensive courses.

The Spacing Effect:

Early opponents of the use of block scheduling and accelerated courses cited the spacing effect as a reason these formats could not be successful.[2] The spacing effect is the name given the observed phenomenon that longer spacing between repeated training sessions improves recall. If the spacing effect is real, it must be reconciled somehow with the overwhelming evidence for success in intensive courses. Two examples of the types of studies that led to the development of this concept are described here.

One study of the spacing effect compared the ability of students to recall memorized Spanish vocabulary when presented with pairs of English and Spanish words. Training sessions were repeated either one day or 30 days apart. Recall within three months of the training and recall eight years later was found to be twice as high in the groups with longer spacing between training sessions.[32] However, this training was not part of a course (the subjects were not students in a Spanish language program) and was not tied to any larger purpose, such as a course grade or prerequisite learning.

The second study found that drilling twice a day for five days was less effective than spending the same total amount of time drilling once a day for ten days and concluded that course instructors should "make extensive use of spaced (weekly or monthly) reviews".[33] Again, this study did not involve learning within a course. The spacing effect could well be different if authentic learning was being measured rather than rote memorization. It is not possible to drill students in the understanding a course concept. On the other hand, perhaps the spacing effect is not an important factor when there are fewer distractions, when only one course is being studied at a time, as is typical for students in intensive courses.

Learning Cycles:

Learning cycles describe the cyclical nature of the steps thought to be involved in learning and are based, in part, on Piaget's theory of cognitive development. Piaget's theory, as described by various educators,[34-36] holds that students have preconceived ideas of how the world works. These preconceptions are in place even when they have little experience with the particular concept or object being studied. Students are not empty buckets waiting to be filled with knowledge. Learning can only occur when students make observations that counter these preconceived ideas and force them to reexamine and modify them. Using Piaget's terminology, students become disequilibrated when they make observations that do not fit their current understanding and must adjust their thinking, or accommodate, to then assimilate these new observations and become equilibrated once again. Learning involves a series of disequilibrations, accommodations, and assimilations. According to Bunce,[35] effective teaching places the student at the center of the process, enabling the student to learn by interacting with the physical environment and with peers and teachers. Good teaching emphasizes process over facts, accepts individual differences in students, and provides for these differences.

Piaget's last two stages of cognitive development, concrete-operational and formal operational, are particularly relevant to learning at secondary and post-secondary institutions. According to Herron,[34] students at the concrete-operational level are unable to either understand or create visualizations of abstract concepts. However, they are able to learn if provided with concrete examples or visualizations of the abstract concept. These visualizations enable the students to make the observations necessary to become disequilibrated and for the learning process to begin.

Piaget's theory is closely related to the educational theory, constructivism, in which students are described as constructing their own knowledge by looking for meaning or patterns in the physical environment or events. If these new observations do not fit the student's existing cognitive construct, the learner must adjust his/her mental constructs. The learner's constructs remain viable only as long as they fit reality. For teaching to be effective, it must include experiences that will challenge the existing mental constructs of the students. Learning occurs when the students alter their mental constructs to include these new experiences.[35-38]

Libby,[39] who uses constructivist ideas in teaching organic chemistry, cautions that learning occurs in a series of small steps. Attempting to achieve large steps runs the risk of disequilbrating the student to such an extent that they choose to memorize rather than understand. He also emphasizes that the social aspects of students developing concepts together is very important, and that continuity is of primary importance. Libby[39] reported that use of constructivist techniques enabled his students to think more like organic chemists instead of memorizing reaction pathways, while Caprio[40] stated that use of constructivist techniques resulted in students taking on more of the responsibility for their learning in anatomy and physiology.

Learning cycles, as developed by Karplus,[41] Bybee[46] and Kolb[50] describe a teaching approach that is consistent with Piagetian ideas and reflects the current understanding of how the brain functions in a learning situation.[51,52] There are several types of learning cycles described in the literature. Karplus[41] described a three-stage cycle of exploration, concept invention, and concept application. During exploration, students explore materials and/or ideas that raise questions that cannot be resolved using their existing understanding (disequilibrations). This leads to the need for concept invention to answer the questions raised during the exploration phase (accommodation). The last phase provides practice for the learner to become familiar with the newly invented concept and typically involves an extension of the original exploration (assimilation). Several researchers have reported success in teaching a variety of high school science courses, as well as college biology courses, using learning cycles[42-44] and have even advocated creating learning cycle texts.[43,45]

Bybee[46] created a five-stage learning cycle referred to as the 5E model: engage, explore, explain, elaborate and evaluate. The middle three stages are very similar to the Karplus model.[41] However, in the 5E model, an "engage" stage is added at the beginning of the activity to gain the interest of the students and an "evaluate" stage is added at the end, in which both students and teacher test the explanation developed in the middle stage. The 5E learning cycles are frequently used to teach science to elementary and middle school students.[47-49]

Learning cycles, as described by Zull,[51] Kolb[50] and Atherton[52] consist of four stages: concrete experience, reflection on the experience, development of an abstract hypothesis related to the experience, and testing the hypothesis, which leads to a new concrete experience. When these learning cycles repeat, they deepen and refine a learner's understanding of a concept, forming a learning spiral. Zull[51] connects the stages of this learning cycle with how the brain receives, processes and retains knowledge. In a gross oversimplification of the complexity of the brain, the cerebral cortex consists of sensory, integrative and motor regions. Sensory regions receive the signals from the five senses, which are then converted into meaning by neurons in the integrative regions that search for previously known patterns or detection of new patterns (disequilibrations). Once the pattern is established (accommodation), the integrative regions then determine a plan of action to be carried out by the motor region (assimilation). A concrete experience enters the brain via the sensory region, is sent to the back integrative cortex for reflection, where, combined with some problem-solving activity by the front integrative cortex, it becomes a hypothesis to be acted upon by the motor region.

Teaching via learning cycles is quite compatible both with the constructivist epistemology and with Piaget's theory of cognitive development. The concrete experience or exploration that is common to all the learning cycles provides the observations necessary for the learners to begin constructing their knowledge, provided that the experience is sufficient to disequilibrate the learners. The assimilation and accommodation processes are accomplished via the reflection, hypothesis-developing and hypothesis-testing steps in Kolb's learning cycle,[50] and with the concept development and concept testing stages in the three-stage learning cycle introduced by Karplus.[41] With each new concrete experience, the cycle continues, potentially forming a series of connected learning cycles, called a learning spiral.[21]

Characteristics of Quality Intensive Courses:

In 1989, Breckon[53] published eight principles and guidelines for effective teaching in compressed formats that contain elements of learning cycle methodology and constructivist principles. These guidelines included greater preparations, precourse assignments, variety in the classroom, physically comfortable class meetings, prepared visuals, active student involvement, postcourse or end-of-course papers/projects, and an emphasis on essay rather than objective exams. The precourse assignments and active student involvement are examples of concrete experiences. Projects, papers and essay exams provide opportunities for reflection and development of abstract hypotheses.

Students are in favor of applying learning cycles and constructivist ideas as well. Scott[54,55] completed a comparative study of the learning experiences of undergraduate students in intensive and traditional length courses in literature and marketing. Extensive student interviews and questionnaires resulted in the following conclusions regarding the attributes of high-quality intensive courses from the students' points of view. The instructor should exhibit enthusiasm, be knowledgeable in both the discipline and in pedagogy, be willing to learn from the students, and be student-oriented. The teaching methods should involve active learning, student interaction and discussion, and applied or experiential learning, all hallmarks of the use of learning cycles in the classroom.

Intensive Chemistry Courses

Of the literature evaluated, only one published study compared student success in intensive and traditional-length chemistry courses, but did not investigate any specific factors related to student success in the chemistry courses.[11] At Middle Tennessee State University, Introductory General Chemistry, CHEM 1010, is the first semester of a two-semester sequence and is regularly offered during the Fall term as a traditional, 15-week course and again in the Summer as a three-week, intensive course. The course instructor describes the course, in either format, as consisting of lecture interspersed with worked examples followed by students actively working similar examples and/or asking questions. The balance of lecture and interactive question-and-answer time is approximately 75%/25% in both formats.

Students taking this course in either format have varied backgrounds and varied interests. Some have taken a chemistry course in high school while others have not; some have strong math backgrounds while others have quite weak math backgrounds. They represent at least forty different majors, mostly in the physical and life sciences with approximately one-fourth of the students majoring in non-science-related fields. Introductory general chemistry students also differ greatly in their levels of prior academic and life experiences; they range from first-semester students to postbaccalaureate students and from current high-school students taking college courses to those who graduated from high school more than a decade ago.

This work aims to document how student success differs, if at all, between the two course formats of Introductory General Chemistry, what factors may be associated with student success in the two formats, and what factors may be appropriate to use as predictors of student success in intensive chemistry courses.

CHAPTER 2

EXPERIMENTAL

The idea for this research arose from the instructor's observation that students in the intensive version of the introductory general chemistry course seemed to earn higher grades than the students in the traditional course sections. Was this difference statistically significant? If so, what could account for greater success in the accelerated course? Could the summer-session students simply be better students than the fall-term students? Could they be more experienced students, either in terms of academic experience or age? Introductory general chemistry is part of the curriculum for many different science-related majors. Could the students' choice of academic major be a factor? Is student gender a factor? Many of these early questions developed into the hypotheses that were tested in this study. To test these hypotheses, the instructor developed a series of questions to use as a pretest and posttest and collected student records information over four years, for four traditional and four intensive courses.

Definitions of Terms

Following is a list of terms and definitions used in this study:

- "Traditional course" is used for the standard 15-week course taught during the Fall term at Middle Tennessee State University (MTSU), which consisted of three class-hours of lecture and a single two-hour laboratory session per week.

- "Intensive course" represents the same course taught during the three-week Summer term (also known as the interim session) at MTSU, which consisted of 14 class days with a three-hour lecture and a two-hour laboratory session each day.

- The term "Group" is used to represent the sets of students from classes taught in the two different course formats.
  - Group I refers to students from the traditional course sections (Fall term).
  - Group II refers to students from the intensive course sections (Summer term).
  - Students self-selected into the two Groups.
- The term "categories" refers to subsets of the full data set based upon specific student characteristics, which are described below.
  - The "academic experience categories" represent five categories within each of the two course format Groups based upon the number of completed semester hours at the beginning of the term in which the introductory general chemistry course was taken. These categories are: 0-30, 31-60, 61-90, 91-120, and greater than 120 completed semester hours.
  - The "life experience" categories are three categories within each of the two course format Groups based upon the number of years since high school graduation at the beginning of the term in which the introductory general chemistry course was taken. The categories are: minus one to two, three to five, and greater than five years since high school graduation.
  - The "academic majors" categories divide each of the two course format Groups into three categories based upon the departments (physical sciences, life sciences and other) under which the students' declared majors are administered.
  - The "gender" categories represent the male and female populations within each of the two course format Groups.
- The term "pretest," refers to a set of 24 questions related to material that all students would be expected to know at the completion of the course and was administered on the first day of class. These questions were developed by the course instructor, a full professor with more than 25 years of teaching and research experience.
- The term "posttest" refers to the identical 24 questions as the pretest, this time embedded within the 100-question final exam.
- "Pretest-posttest difference" is the posttest score minus the pretest score and is available for all students who completed both the pretest and the final exam.

Experimental Hypotheses

The experimental hypotheses were developed from the initial observations made by the course instructor:

- **Hypothesis One**: Students will achieve greater success in intensive introductory chemistry courses than in the traditional-length versions of the same course.

- **Hypothesis Two**: Students with greater academic experience will achieve greater student success in intensive versus traditional introductory general chemistry courses than students with less academic experience.

- **Hypothesis Three**: Students with greater life experience will achieve greater student success in intensive versus traditional introductory general chemistry courses than students with less life experience.

- **Hypothesis Four**: Students majoring in the physical or life sciences will achieve greater academic success in intensive introductory chemistry courses than students with non-science majors.

- **Hypothesis Five**: Gender has an effect on student success in intensive or traditional course formats.

Basic Assumptions

An important assumption was that the instruction for the two Groups was equivalent in ways other than the number of weeks the course lasted. The two Groups shared the same instructor, text, number of contact hours, course objectives, course requirements, classroom presentation methods, grading scale and exams. The unit tests, which covered the material from two chapters each, and the final exam, which was cumulative, were matched for the Fall and Summer terms of a given academic year.

A second assumption was that the existing knowledge of the students in the two Groups was comparable and showed no significant difference in ACT scores, pretest scores or GPA scores. For those students for whom only SAT scores are available, these scores were converted to ACT composite scores using the conversion tables provided by the College Entrance Examination Board.[56] A *t*-test was used to support this assumption, with the result that the two Groups were found to have no significant differences (all $p > 0.05$) in the means of the ACT composite scores ($p = 0.130$) or the means of the pretest scores ($p = 0.075$). The mean GPA scores for the two Groups were found to be significantly different but as this difference was very small (0.1 GPA units), it is unlikely to be meaningful in determining differences in student achievement in the course.

Data Collection

Student academic records, including course data and student records information were collected for students registered for sections of CHEM 1010 taught by one instructor from four academic years in the Fall terms (traditional course format, Group I) and the Summer terms (intensive course format, Group II) from Fall 2000 to Summer 2004. Students were not randomly assigned to these groups but were free to enroll in the two Groups. Enrollment in the four traditional course sections ranged from 43 to 52 students for a total of 161 students. Withdrawal prior to the final exam reduced this total to 136; these students comprise the population of Group I. Enrollment in the four intensive course sections ranged from 24-30 students for a total of 96 students. Withdrawal prior to the final exam reduced this total to 95; these students comprise the population of Group II. Throughout the data analysis, the students were identified by numerical codes and not by student names.

The course data analyzed in this study included pretest and posttest scores (24 total points possible, not included in the course grade), scores for five unit tests (100 points possible for each), final exam scores (200 points possible), and total course points (800 points possible) for each student. The total-course-points measure was comprised of the scores of four of the five unit tests (the lowest score was dropped), the final exam score, the laboratory grade (200 possible points) and a possible 15 extra credit points resulting from class attendance. The student records information included ACT or SAT scores, year of high school graduation, gender, current GPA, academic major and the current number of credit hours completed. ACT and/or SAT scores were not available for many of the students enrolled in the intensive courses, as the university did not require these tests for transfer or non-traditional students, who comprised a higher percentage of the Summer session students. The data for GPA and number of credit hours were collected at the beginning of the term during which the student was registered for the introductory general chemistry course. The pretest was given during the first class period, so pretest scores were missing for any students who did not attend the first class. The years since high school graduation data were determined by comparing the calendar year of the course to the year of high school graduation. For example, a student who graduated in 2000 and took the introductory general chemistry course in the Fall term of 2000 was considered to be at zero years since high school graduation. A high school graduate from the year 2000 who enrolled instead in the Summer session of 2001, was considered to be at one year since high school graduation. For the statistical analyses, the academic majors were grouped into three categories - physical sciences, life sciences and other- based on the department under which the program was administered. The academic major groups were coded as "1", "2" and "3," for physical sciences, life sciences and other, respectively. Similarly, student gender was coded as "0" for male and "1" for female and course grades were coded 0 through 4 for grades F through A.

Data Analysis

Descriptive Statistics

Data for students who took only the first of the five unit tests were not included in any part of the analysis or discussion. Students who did not take the final exam were considered to have withdrawn late in the term and their data were not included in the statistical analysis, either. Means and standard deviations of the full data set were determined for all measures except academic major and gender.

Two-Tailed Comparison of Means, *t*-Tests

Each hypothesis was tested by carrying out a two-tailed *t*-test of the student academic records for the appropriate category. The *t*-distribution statistic and the degrees of freedom (*df*) were used to determine the probability (*p*) that two means are the same. For this study, the primary criterion for significant differences in the data is set at $p \leq 0.05$ (95% confidence level). Six of the eighteen different measures for which data were available were chosen as the most useful; these were then divided into two categories. The first category consisted of measures of the general academic ability and/or prior knowledge of the students related to the course: GPA, ACT composite score and pretest score. The second category was comprised of final exam scores, total course points, and pretest-posttest differences, which were used as measures of the level of student success in the introductory general chemistry course.

A two-tailed test of proportions was carried out on the withdrawal rates of the two groups. The resulting *z* statistic was used to determine the probability that the two proportions represented the same population.

Analysis of Covariance

Two multivariate ANCOVA analyses were used, in part, to test the five hypotheses. Final exam scores, total course points and pretest-posttest difference scores, which are indicators of student success in the course, were chosen as dependent variables. The data for academic major (coded 1, 2 or 3), gender (coded 0 or 1) and Group (I or II), were included in the test as fixed-factor variables. Two slightly different sets of covariate factors were used. The first set, GPA, pretest scores, number of years since high school graduation and the number of semesters completed, omitted the ACT composite scores to maximize the population size, *N*, for each category and is referred to as the full data set. A second data set was created to evaluate the possible relationship of ACT composite scores with the dependent variables. This second set of covariates included all of the covariate factors listed for the full data set plus the ACT composite scores, which were not available for all students. The data for this smaller population was called the reduced data set. A significance level of $p \leq 0.05$ was chosen to identify factors making a significant contribution to the variance in the data in either data set.

Testing the Individual Hypotheses

Hypothesis One: Students will achieve greater success in intensive introductory chemistry courses than in the traditional-length versions of the same course.

Hypothesis One was evaluated by carrying out a *t*-test comparing the means of Group I and Group II for the entire data set and by using the results of the two ANCOVA analyses. Comparison of the means of the three measures of prior knowledge were used to determine if the students in the Groups were comparable while a comparison of the means of the three measures of student success were used to determine relative levels of success. A comparison of the withdrawal rates for the two groups was also carried out.

Hypothesis Two: Students with greater academic experience will achieve greater success in intensive versus traditional introductory general chemistry courses than will students in traditional introductory general chemistry courses.

Five different categories were created from the variable number of completed semester hours to approximate years of academic study: zero to 30, 31 to 60, 61 to 90, 91 to 120, and greater than 120 completed semester hours. This hypothesis was tested by carrying out *t*-tests of the means of Group I and Group II data within each of the categories. Comparison of the means of the three measures of prior knowledge were used to determine if the students in the Groups were comparable while a comparison of the means of the three measures of student success were used to determine relative levels of student success. The results of the ANCOVA tests were also used in the evaluation.

Hypothesis Three: Students with greater life experience will achieve greater success in intensive versus traditional introductory general chemistry courses than will students in traditional introductory general chemistry courses.

Three categories were identified to test the effect of life experience on student success. The first category, minus one to two years since high school graduation, includes high school students concurrently taking college courses and recent high-school graduates. The second category, three to five years since high school graduation, is made up of slightly older, but still traditional-age college students, while the third, greater than five years, is comprised of non-traditional, returning adult students. This hypothesis was tested using *t*-tests of the means of Group I and Group II data within each of the categories. Comparisons of the means of the three measures of prior knowledge were used to determine if, within each category, the students in the two Groups were comparable, while a comparison of the means of the three measures of student success were used to determine relative levels of success within each category. The results of the ANCOVA tests were also applied to this hypothesis.

Hypothesis Four: Students majoring in the physical or life sciences will achieve greater success in the intensive courses than students with non-science majors.

The various academic majors were divided into three categories, physical sciences, life sciences and other, as determined by the departments in which the programs were offered. The physical sciences category was made up of students with these majors: aerospace, chemistry, concrete industry management, engineering technology, environmental science technology, geoscience, industrial technology, mathematics, and computer science. Students in the following majors comprised the life sciences category: animal science, biology, nursing, nutrition and food science, plant and soil science, pre-dental, and science (a major chosen by pre-health professional students at this university). The final category in the academic major categories, dubbed “other”, is very diverse as it included students in 24 different majors such as art, English, entrepreneurship, and psychology. Any “undeclared” or “non-degree” students were also included in the “other” category. Hypothesis Four was tested via *t*-tests comparing the means of the Group I and Group II data within each of the categories. Comparison of the means of the three measures of prior knowledge were used to determine if the students in the Groups were comparable within each category while a comparison of the means of the three measures of student success were used to determine relative levels of success within each category. The results of the ANCOVA tests were also used in evaluating this hypothesis.

Hypothesis Five: Gender has an effect on greater student success in intensive versus traditional course formats.

This final hypothesis was tested by comparing the means using *t*-tests of the Group I and Group II data within the categories of male students and female students. Comparison of the means of the three measures of prior knowledge were used to determine if the students in the Groups were comparable within each category while a comparison of the means of the three measures of student success were used to determine relative levels of success within each category. The results of the ANCOVA analyses were also used to test this hypothesis.

CHAPTER 3

RESULTS AND DISCUSSION

General Description of the Data

The general academic ability and the level of prior knowledge related to the course was assessed using GPA, ACT composite scores and pretest scores; none of these are without drawbacks. While GPA scores were available for all students, they are not likely to be valid measures of ability for students who have completed very few semester hours, such as students in their first term at college. ACT composite scores have been established as valid measures of academic ability, but were not available for all the students in the data set as these were not required by the university for older students or for transient students currently enrolled at other universities. The pretest scores were better indicators of prior knowledge related to the course than academic ability. In this study, the combination of ACT, GPA and pretest scores were used to determine if two populations were comparable in terms of prior knowledge and academic ability.

Student success was evaluated by final exam scores, total course points and pretest-posttest differences. Final exam scores were measures of student success at the end of the course and reflected cumulative knowledge, while the total-course-points measure reflected student success throughout the course. Pretest-posttest difference showed the improvement in knowledge of the course content. However, students who entered the course with higher levels of background knowledge may have had lower pretest-posttest difference scores than those who entered the course with little chemistry knowledge but were successful in the course.

Only 136 of the 161 initially-enrolled students completed the four traditional sections of the course but, of all the tests, only the final exam was taken by all 136 students. These totals reflected the instructor's grading practice of dropping the lowest grade of the five unit tests. The mean scores for the unit tests decreased as the terms progressed with a slight increase for the last test: test one (73%), test two (71%), test three (64%), test four (60%) and test five (64%). The mean final exam score was 61% while the mean of the total-course-points measure was 71%. The mean pretest score of 9.2 out of 24 (38%) was improved by 8.2 points for a mean posttest score of 17.4 out of 24 (72%). Students in Group I had a mean GPA of 2.8, which is considerably higher than the mean course GPA of 1.9. ACT scores were available for 126 of the 136 students (92.6%); the mean ACT composite score was 22.2. These students graduated from high school an average of 3.5 years ago and have completed an average of 49 semester hours of college credits. The mean of 0.55 for gender indicated a distribution of slightly more female (55%) than male students (45%).

A total of 96 students were enrolled in the four intensive sections of the course with one student withdrawing late. Only one test, the final exam, was taken by all 95 students, again reflecting the instructor's policy of dropping the lowest unit test grade. The mean scores for the unit tests decreased as the term progressed with a slight increase for test five: test one (79%), test two (77%), test three (71%), test four (66%) and test five (71%). The mean final exam score is 73% while the mean for total course points is 78%. The mean pretest score of 9.9 out of 24 (41%) was improved by 10.4 points for a mean posttest score of 20.3 out of 24 (85%). Students in Group II had a mean GPA of 2.9, which was somewhat higher than that Group's mean course GPA of 2.5. ACT scores were available for 59 of the 95 students (62%); the mean ACT composite score was 21.3 for this Group. Group II students graduated from high school an average of 5.0 years ago and have completed an average of 85 semester hours of college credits. The gender mean of 0.41 indicated a somewhat higher distribution of male students (59%) than female students (41%).

During the terms, the number of students taking tests in either course format decreased, reaching a low for test five, and then increasing for the required final exam. One difference between the two Groups was immediately obvious; there were many more late withdrawals from the traditional sections (Group I) than from the intensive sections (Group II). Table 1 lists the number of students taking each unit test and the final exam for the two Groups. 25 students, or 16% of the total number of traditional students, did not complete the course; almost half of these (48%) were non-science majors. Only one student (~ 1%), a physical science major, did not complete the course in the intensive sections. The results of a statistical test of proportions indicates that the withdrawal rates for the two Groups were significantly different ($z = 3.73, p < 0.0001$).

Hypothesis One: Students will achieve greater success in intensive introductory chemistry courses than in the traditional-length versions of the same course.

Analysis of the Full Data Set

t-Test

The *t*-test results of the comparison of the means of the full data set appear in Table 2. The means of the Group I and Group II data showed statistically significant differences for all five unit tests, the total test scores, the final exam, the total course

Table 1. Numbers of Test-Takers during the Term

Unit test	Group I	Group II
Test one	161	93
Test two	158	94
Test three	149	94
Test four	143	91
Test five	131	89
Final exam	136	95

points, the posttest, the pretest-posttest difference, GPA, number of completed semester hours, years since high school graduation, and gender. In other words, the means of all measures, except the pretest and ACT composite scores, were found to be significantly different between the two Groups.

The means of the student success measures (final exam, total course points and pretest-posttest difference) of the two Groups were among the measures that were significantly different. Two of the three prior knowledge measures, ACT composite scores and pretest scores, showed no significant differences, while GPA was found to be significantly different. This difference, while statistically significant, may not be meaningful as the mean scores differ by only 0.16 GPA units. It appears that student

Table 2. Comparison of the Means (*t*-Test) of the Full Data Set

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I		Group II				
	Mean	SD	Mean	SD	*t*	*df*	*p*
Test one (136, 93)	72.9	12.7	78.9	11.9	3.66	227	0.000*
Test two (134, 94)	70.7	14.5	76.7	13.3	3.21	226	0.002*
Test three (134, 94)	64.2	15.8	71.2	16.2	3.27	226	0.001*
Test four (130, 91)	60.2	16.3	65.8	15.2	2.61	219	0.010*
Test five (131, 89)	63.6	19.2	70.8	15.0	2.97	218	0.003*
Total Tests (136, 95)	278	48.4	302	45	3.79	230	0.000*
Final Exam (136, 95)	122	28.7	145	28	6.27	230	0.000*
Course Total (136, 95)	565	89.2	627	83	5.34	230	0.000*
Course Grade (136, 95)	1.86	1.03	2.53	0.94	5.00	230	0.000*
Pretest (132, 89)	9.20	2.94	9.91	2.79	1.79	219	0.075
Posttest (135, 94)	17.4	3.81	20.3	2.9	6.31	226	0.000*
Pretest-Posttest Difference (131, 88)	8.12	3.75	10.5	3.5	4.79	216	0.000*
GPA (136, 95)	2.78	.58	2.94	0.56	2.16	230	0.032*
# Semester Hrs (136, 95)	49.8	36.0	85.2	49.9	6.28	230	0.000*
Years Since HS (136, 95)	3.54	5.85	5.03	3.67	2.12	230	0.035*
ACT (126, 59)	22.2	3.77	21.3	3.7	-1.52	162	0.130
Gender (136, 95)	0.55	0.50	0.41	0.49	-2.061	230	0.040*

*indicates statistically significant difference success was greater in the intensive course (Group II) than in the traditional course (Group I). However, there were many possible confounding variables that could have influenced student success beyond course format, some of which are discussed below.

The means of the unit tests (test one, test two, etc.) and the final exam for both Groups are plotted in Figure 2. The test means were consistently higher for Group II than for Group I. The test data for the two Groups followed a very similar trend (until the final exam) that most likely reflected the nature of the course material. The larger difference between the means of the two Groups for the final exam may reflect factors that were different in the two course formats. First, the longer, traditional term may have given the students more time to forget the material from the early units compared to the

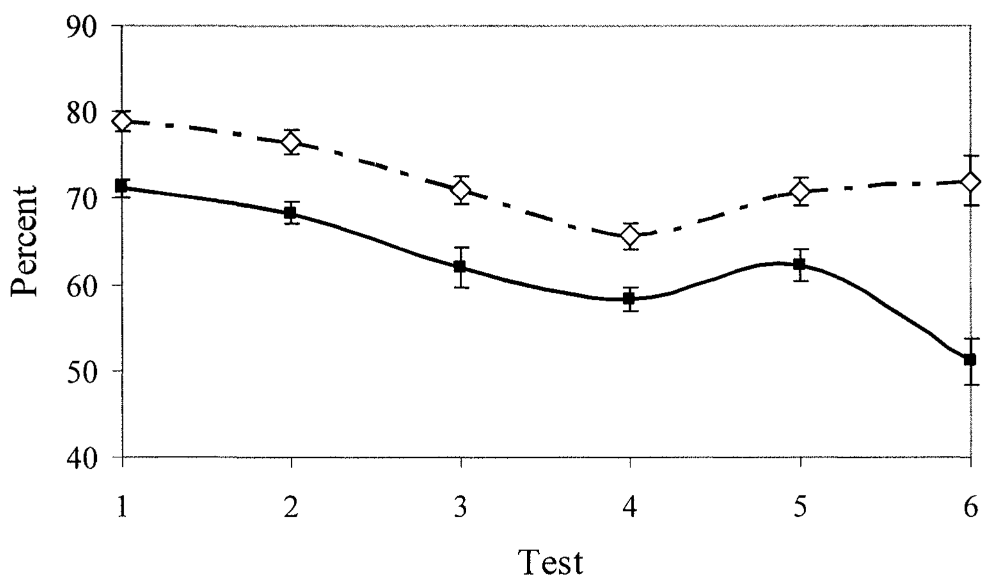

Figure 2. Unit test means and final exam (test #6) means (as percentages) of the traditional course format group (Group I,■) and the intensive course format group (Group II,◇).

three-week, intensive course. Second, students in the traditional-length course were typically enrolled in three to four additional courses that may have had projects due or exams scheduled near the time of the final exam for the chemistry course, while students in the intensive course typically were enrolled in only the one course. It is likely that the students in the intensive course had fewer distractions and, potentially, had more time available to study for the course.

ANCOVA Analysis

The results of the ANCOVA analysis of the full data set appear in Tables 3 and 4. These data are described for each independent variable in the paragraph below. Any student for whom a specific measure was unavailable, typically the pretest-posttest difference, was omitted from the analysis, reducing the population sizes from 136 to 130 for Group I and from 95 to 88 for Group II.

Table 3. Means of the Dependent Variables: Full Data Set

	Group I (*N* = 130)		Group II (*N* = 88)	
Dependent Variable	Mean	*SD*	Mean	*SD*
Final Exam	122	28	147	28
Course Total	567	90	633	82
Pretest-Posttest Difference	8.12	3.75	10.53	3.47

Table 4. Effects of Covariates in the Full Data Set

	Final Exam[a]			Course Total[b]			Pretest-Posttest Difference[c]		
Co-variate	Mean Sq.	*F*	*p*	Mean Sq.	*F*	*p*	Mean Sq.	*F*	*p*
Pretest	10300	24.5	0.000*	85400	25.7	0.000*	775	102	0.000*
GPA	53900	128	0.000*	658000	198	0.000*	697	92.0	0.000*
# Sem-ester Hours	592	1.41	0.237	4260	1.28	0.259	0.105	0.014	0.906
Years Since HS	255	0.606	0.437	2290	0.689	0.408	6.06	0.800	0.372
Group	9880	23.5	0.000*	56800	17.1	0.000*	167	22.0	0.000*
Major	604	1.44	0.240	8710	2.62	0.075	10.6	1.40	0.249
Gender	466	1.11	0.294	1710	0.514	0.474	20.9	2.75	0.099

*indicates significant difference

[a] $R^2 = 0.583$

[b] $R^2 = 0.636$

[c] $R^2 = 0.518$

The Group II (intensive format) means were significantly higher than the Group I (traditional format) means for final exam scores ($F = 18.8$, $p < 0.000$), total course points ($F = 23.5, p < 0.000$) and pretest-posttest difference ($F = 23.5, p < 0.000$). The following factors were found to be significant in contributing to the variance of the final exam scores for both Groups: GPA ($F = 128, p < 0.000$), pretest scores ($F = 24.5, p < 0.000$), and Group ($F = 23.5, p < 0.000$). The same factors were found to be significant in contributing to the variance of the total course points of both groups: GPA ($F = 198, p <= 0.000$), the pretest scores ($F = 25.7, p < 0.000$), and Group ($F = 17.1, p < 0.000$); and for pretest-posttest difference: GPA ($F = 92.0, p < 0.000$), pretest scores ($F = 102, p < 0.000$), and Group ($F = 22.0, p < 0.000$). Four of the tested factors were not found to be significant in explaining the variance of any of the three dependent variables: the number of completed semester hours, years since high school graduation, academic major and gender. The R-squared values for the analysis related to final exam score was 0.583, indicating that these three factors - pretest, GPA and Group - accounted for approximately 58% of the variance in the final exam score data. The R-squared values were 0.636 for total course points and 0.518 for pretest-posttest difference indicating that the same three factors accounted approximately 63% and 51% of the variance in those data, respectively.

Analysis of the Reduced Data Set

The results of the ANCOVA analysis of the reduced data set (the population sizes of the two Groups were reduced to only those students with ACT composite scores and pretest-posttest differences) are in Table 5 and 6. These data are described below for each independent variable.

The Group II means were significantly higher than the Group I means of the final exam scores ($F = 5.21, p > 0.024$), the total course points ($F = 2.22, p > 0.139$) and the pretest-posttest difference ($F = 5.30, p > 0.023$). Four factors were found to be significant in contributing to the variance of all three dependent variables: pretest scores, GPA, the years since high school graduation, and ACT composite scores. A fifth factor, Group was found to be significant in contributing to the variance of two of the three dependent variables, final exam scores and pretest-posttest differences. The results for the variance of the final exam scores were: pretest scores ($F = 5.49, p > 0.021$), GPA ($F = 66.8, p > 0.000$), the years since high school graduation ($F = 10.8, p > 0.001$), ACT composite scores ($F = 17.3, p > 0.001$), and Group ($F = 5.21, p > 0.024$). The results for total course points were: pretest scores ($F = 9.50, p > 0.002$), GPA ($F = 107, p > 0.000$), the years since high school graduation ($F = 7.92, p > 0.006$), and ACT composite scores ($F = 11.8$, $p > 0.001$). The results for pretest-posttest difference were: pretest scores ($F = 97.9, p > 0.000$), GPA ($F = 50.4, p > 0.000$), the years since high school graduation ($F = 5.41, p > 0.021$), ACT composite scores ($F = 9.84, p > 0.002$) and Group ($F = 5.30, p > 0.023$).

Table 5. Means of the Dependent Variables in the Reduced Data Set

	Group I (*N* = 99)		Group II (*N* = 52)	
Dependent Variable	Mean	*SD*	Mean	*SD*
Final Exam	123	29	145	27
Course Total	570	91	625	74
Pretest-posttest Difference	8.08	3.81	10.35	3.41

Table 6. Effects of Covariates in the Reduced Data Set

	Final Exam[a]			Course Total[b]			Pretest-Posttest Difference[c]		
Co-variate	Mean Sq.	*F*	*p*	Mean Sq.	*F*	*p*	Mean Sq.	*F*	*p*
Pretest	1979	5.49	0.021*	26900	9.50	0.002*	620	97.9	0.000*
GPA	24100	66.8	0.000*	304000	107	0.000*	319	50.4	0.000*
# Semester Hours	1080	2.98	0.087	6480	2.28	0.133	7.82	1.23	0.269
Years Since HS	3910	10.8	0.001*	22500	7.92	0.006*	34.3	5.41	0.021*
ACT	6225	17.3	0.001*	3360	11.8	0.001*	62.4	9.84	0.002*
Group	1880	5.21	0.024*	6290	2.22	0.139	33.6	5.30	0.023*
Major	178	0.494	0.612	3470	1.22	0.297	12.5	1.98	0.142
Gender	106	0.294	0.588	88.1	0.031	0.860	1.81	0.286	0.594

*indicates statistically significant difference

[a]$R^2 = 0.644$

[b]$R^2 = 0.684$

[c]$R^2 = 0.611$

Three of the tested factors were not found to be significant contributors to the variance in final exam scores or pretest-posttest differences: the number of completed semester hours, academic major and gender. These same three variables plus Group were not found to contribute significantly to the variance of the total course points. The significant factors were found to account for approximately 64% of the variance in final exam scores, 68% of the variance in total course points and 61% of the variance in the pretest-posttest differences.

Course Grade and GPA

Group (traditional or intensive course format) was found to be a statistically significant factor in determining the variance in the final exam scores, total course points and the pretest-posttest differences, the three indicators of student success in the course in all but one instance (total course points in the reduced data set). Other aspects of the data also pointed to greater success in the intensive course. The overall mean GPA was very close for the two groups, 2.8 for Group I and 2.9 for Group II, but the mean course GPA of 2.5 for Group II was higher than the mean course GPA of 1.9 for Group I. Students in the intensive course earned grades in this course that were slightly lower than the average course grades they had earned in the past, while students in the traditional course format earned much lower grades in this course than the average grades they had earned in the past.

Summary

The *t*-test results indicated that students in the two Groups were comparable in two of the three preparedness measures, ACT composite scores and pretest scores, and were different in all three of the student success measures. Students were less likely to withdraw from the intensive-format courses and achieved grades in the intensive course that were closer to their typical grades than did students in the traditional-format courses. Group was found to be a significant factor in determining the variance of all three student success measures in the full data set and two of the three student success measures in the reduced data set. Based on the *t*-test and ANCOVA results, one can conclude that student success was different in the two course formats and that the intensive course format resulted in greater student success than the traditional course format.

Hypothesis Two: Students with greater academic experience will achieve greater success in intensive versus traditional introductory general chemistry courses than students with less academic experience.

Group II (intensive) students had, on average, completed approximately 70% more semester hours than Group I students (Table 2). To test the hypothesis concerning the effect of academic experience on student success, five separate categories were created. Each category represented a year of full-time academic work, except for the final category: zero - 30 completed semester hours, 31 - 60 completed semester hours, 61 - 90 completed semester hours, 91 - 120 completed semester hours, and greater than 120 completed semester hours. The majority of Group I students (69.5%) were found in the two categories with the least academic experience, zero to 30 and 31 to 60 completed semester hours. Approximately 20% of Group I students were in the third category of academic experience (61 to 90) and only a very few (4 - 7%) were found in the fourth (91 to 120) and fifth categories (greater than 120 completed semester hours). Group II students were evenly distributed among the first 4 categories (16 - 18%) of academic experience with a larger representation in the fifth category (28%) of academic experience (Table 7).

Table 7. Student Distribution by Number of Completed Semester Hours

# Completed Semester hours	Group I (136)		Group II (96)	
	%	*N*	%	*N*
0 - 30	34.6	47	16.8	16
31 - 60	33.8	46	17.9	17
61 - 90	20.6	28	17.9	17
91 - 120	4.4	6	17.9	17
>120	6.6	9	29.4	28

Zero to Thirty Completed Semester Hours

Results of the *t*-test for the category with 0 to 30 completed semester hours are in Table 8. The mean of one measure was significantly higher for Group I, the number of completed semester hours. This was puzzling, as it was expected that the Group I students would have been first-term college students and the Group II students would have completed a year of college. However, means of the number of completed semester hours suggested that the Group I students, within this category, had completed more than a semester's worth of courses and were possibly second-year college students while the Group II students were novice college students. The following means were significantly higher for Group II: test one, test two, test three, test four, total test scores, the final

Table 8. *t*-Test Results: Zero to 30 Completed Semester Hours

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I		Group II				
	mean	SD	mean	SD	*t*	*df*	*p*
Test one (47, 16)	72.7	14.1	82.4	9.2	2.57	61	0.013*
Test two (46, 15)	71.1	16.1	84.9	10.7	3.11	59	0.003*
Test three (47, 16)	65.9	14.1	77.0	15.0	2.68	61	0.010*
Test four (45, 13)	60.6	15.5	70.6	16.7	2.02	56	0.048*
Test five (45, 15)	65.0	15.5	73.1	12.3	1.51	58	0.137
Total Tests (47, 16)	282	47	320	40	2.84	61	0.006*
Final Exam (47, 16)	122	29	157	24	4.42	61	0.000*
Course Total (47, 16)	573	84	662	74	3.77	61	0.000*
Pretest (46, 16)	9.85	2.84	10.9	2.6	1.28	60	0.207
Posttest (46, 16)	17.6	3.7	21.1	2.2	3.64	60	0.001*
Pretest-Posttest difference (45, 16)	7.53	3.71	10.3	2.6	2.69	59	0.009*
GPA (47, 16)	2.74	0.69	3.00	0.75	1.23	61	0.222
# Semester Hrs (47, 16)	16.7	5.0	10.8	9.1	-3.26	61	0.002*
Years Since HS (47, 16)	0.68	1.35	3.94	4.51	4.46	61	0.000*
ACT (46, 7)	22.8	4.1	22.9	4.3	0.02	51	0.985
Gender (47, 16)	0.64	0.49	0.75	0.45	0.81	61	0.421

*indicates significant differences

exam, total course points, the posttest, the pretest-posttest difference, and years since high school graduation. The means of these measures were not found to be significantly different: test five, the pretest scores, GPA, ACT composite scores, and gender.

The results above indicated that Group I and Group II data were not significantly different for the three prior knowledge measures, pretest, GPA and ACT composite scores. These data can be used to support the claim that the students in two Groups within this category were comparable in their level of prior knowledge for the course. The mean scores of Group II were significantly higher than those of Group I for all three student success measures, final exam scores, total course points, and pretest-posttest differences.

Thirty-One to Sixty Completed Semester Hours

The results of the *t*-test for this category are given in Table 9. The means of the following measures were significantly higher for Group II: the final exam, the posttest and the number of completed semester hours. The means of the number of completed semester hours, while different, are not as puzzling for this category, as they both indicate that students have completed the equivalent of 1.5 years of college. The means of the following measures were not found to be significantly different: test one, test two, test three, test four, total test scores, the total course points, the pretest scores, the pretest-posttest differences, GPA, years since high school graduation, ACT composite scores, and gender.

Table 9. *t*-Test Results: 31 - 60 Completed Semester Hours

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (45, 17)	72.3	11.9	76.3	13.8	1.14	60	0.257
Test two (44, 17)	70.8	14.8	73.9	13.2	0.77	59	0.446
Test three (45, 17)	62.7	18.7	71.7	11.3	1.85	60	0.069
Test four (45, 16)	62.2	15.3	62.6	12.8	0.07	59	0.942
Test five (43, 17)	65.4	21.0	69.0	12.9	0.66	58	0.509
Total Tests (46, 17)	277	56	294	42	1.16	61	0.253
Final Exam (46, 17)	123	34	142	26	2.06	61	0.044*
Course Total (46, 17)	526	151	614	72	1.75	61	0.085
Pretest (44, 15)	8.95	3.22	9.13	1.92	0.20	57	0.840
Posttest (45, 17)	17.8	4.2	20.2	2.9	2.18	60	0.033*
Pretest-Posttest Difference (43, 15)	8.81	4.02	11.1	3.1	1.97	56	0.054
GPA (46, 17)	2.82	0 .53	3.00	0.57	1.13	61	0.264
# Semester Hrs (46, 17)	43.4	8.0	49.6	9.4	2.59	61	0.012*
Years Since HS (46, 17)	3.24	5.59	1.82	1.01	-1.03	61	0.305
ACT (37, 16)	22.2	3.7	21.4	3.6	-0.66	51	0.512
Gender (46, 17)	0.57	0.50	0.53	0.51	-0.25	61	0.803

*indicates significant differences

Within the 31 to 60 completed semester hours category, Groups I and II were comparable in prior knowledge as there were no significant differences in the means of the GPA, ACT composite scores, and pretest scores. However, only the means of the final exam scores were significantly different among the student success measures. The other two measures of student success, pretest-posttest difference and total course points were not shown to be significantly different. The mean scores of Group II were higher than the mean scores of Group I for all the student-success related measures, but not always significantly so. Perhaps the significantly higher final exam scores for Group II were more influenced by the shorter time between the first class and the final than by any effect of the intensive course format.

Sixty-One to Ninety Completed Semester Hours

The results of the *t*-test for this category appear in Table 10. The means of the following measures were significantly higher for Group II: test one, test five, the final exam, the total course points, and the posttest. The means of the following measures were not found to be significantly different: test two, test three, test four, the total test scores, pretest, pretest-posttest differences, GPA, the number of completed semester hours, years since high school graduation, ACT composite scores, and gender.

The students in Groups I and II of the 61 to 90 completed semester hours category appeared to be comparable in terms of prior knowledge related to the course, having no significant differences between the means of the GPA, ACT composite scores, and pretest scores. Two of the three student success measures, final exam scores and the total course points, were significantly higher for Group II.

Table 10. *t*-Test Results: 61 - 90 Completed Semester Hours

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (28, 16)	72.0	11.9	80.1	13.3	2.07	42	0.044*
Test two (28, 17)	69.5	13.9	73.5	14.7	0.90	43	0.372
Test three (26, 17)	62.0	16.0	70.0	17.9	1.53	41	0.134
Test four (26, 17)	58.3	19.3	65.2	17.3	1.20	41	0.237
Test five (28, 17)	61.6	18.3	73.9	14.4	2.37	43	0.022*
Total Tests (28, 17)	274	46	299	55	1.66	43	0.104
Final Exam (28, 17)	121	26	144	30	2.63	43	0.012*
Course Total (28, 17)	557	81	624	99	2.45	43	0.018*
Pretest (27, 15)	9.56	2.22	10.1	3.0	0.63	40	0.532
Posttest (27, 17)	16.9	3.7	19.6	3.0	2.56	42	0.014*
Pretest-Posttest Difference (27, 15)	7.30	3.12	9.53	4.14	1.98	40	0.055
GPA (28, 17)	2.74	0.55	2.84	0.64	0.57	43	0.575
# Semester Hrs (28, 17)	74.3	9.3	75.6	8.7	0.45	43	0.654
Years Since HS (28, 17)	4.89	3.90	5.35	4.05	0.38	43	0.707
ACT (15, 13)	20.8	2.7	21.5	3.7	0.57	23	0.577
Gender (28, 17)	0.54	0.51	0.35	0.49	-1.18	43	0.243

*indicates significant differences

Ninety-One to One Hundred Twenty Completed Semester Hours

Table 11 contains the t-test results for this category. None of the means of the measures of the Group I and Group II data were found to be significantly different for this category. The Group I and II populations appeared to be comparable in prior knowledge related to the course, showing no significant differences between the means of the ACT composite scores, GPA, and pretest scores. Surprisingly, Groups I and II, within this category, showed no significant differences in the student success measures.

Greater than One Hundred Twenty Completed Semester Hours

The results of the *t*-test for this category appear in Table 12. The means for the Group I were significantly lower for the following measures: test five, total test scores, the final exam, the total course points, the pretest scores, and the posttest scores. The mean for years since high school graduation of Group I was significantly higher than that of Group II. The means of the following measures were not found to be significantly different: test one, test two, test three, test four, the pretest-posttest difference, GPA, the number of completed semester hours, ACT composite scores, and gender.

Within this final academic experience category, Groups I and II appeared to be comparable in prior knowledge related to the course with no significant differences between the means of the GPA and ACT composite scores. However, the pretest scores were significantly different, as the mean of Group II, is significantly higher than that of Group I. The posttest mean for Group II was also significantly higher than that of Group I. Within this category, the means of two of the student success measures, final exam

Table 11. *t*-Test Results: 91 - 120 Completed Semester Hours

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (6, 17)	77.9	6.6	77.7	12.8	-0.04	21	0.967
Test two (6, 17)	73.2	8.4	74.8	14.8	0.24	21	0.809
Test three (6, 17)	70.7	9.5	69.3	17.4	-0.18	21	0.862
Test four (4, 17)	58.0	11.7	66.2	12.3	1.21	19	0.240
Test five (6, 15)	68.5	10.0	71.7	14.3	0.49	19	0.631
Total Tests (4, 17)	291	30	299	44	0.42	21	0.631
Final Exam (6, 17)	120	24	142	32	1.49	21	0.152
Course Total (6, 17)	582	60	618	86	0.92	21	0.367
Pretest (4, 15)	7.33	1.97	9.33	2.85	1.57	19	0.139
Posttest (6, 17)	17.0	3.5	20.2	3.2	2.07	21	0.051
Pretest-Posttest Difference (6, 15)	9.67	3.27	11.4	4.1	0.92	19	0.369
GPA (6, 17)	2.79	0.42	2.97	0.50	0.78	21	0.446
# Semester Hrs (6, 17)	102	8	104	9	0.47	21	0.641
Years Since HS (6, 17)	5.17	2.93	5.24	2.33	0.06	21	0.954
ACT (4, 12)	23.3	2.5	19.7	3.2	-2.04	14	0.061
Gender (6, 17)	0.33	0.52	0.18	0.39	-0.78	21	0.446

*indicates significant differences

Table 12. *t*-Test Results: Greater than 120 Completed Semester Hours

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (9, 27)	75.6	15.8	78.6	10.8	6.36	34	0.529
Test two (9, 28)	69.6	11.3	77.2	11.6	1.73	35	0.093
Test three (9, 27)	64.1	12.8	69.3	17.8	0.80	34	0.429
Test four (9, 28)	53.7	20.0	65.6	16.6	1.78	35	0.083
Test five (9, 25)	51.3	13.2	68.0	18.6	2.47	32	0.019*
Total Tests (9, 28)	270	36	301	45	1.85	35	0.072
Final Exam (9, 28)	112	17	144	28	3.22	35	0.003*
Course Total (9, 28)	543	61	622	83	2.63	35	0.013*
Pretest (8, 28)	6.75	3.24	10.0	3.1	2.57	34	0.015*
Posttest (9, 27)	16.2	3.5	20.4	3.2	3.38	34	0.002*
Pretest-Posttest Difference (8, 27)	9.88	4.05	10.48	3.43	0.42	34	0.676
GPA (9, 28)	2.91	0.38	2.93	0.43	0.13	35	0.895
# Semester Hrs (9, 28)	145	18	144	23	-0.07	35	0.942
Years Since HS (9, 28)	14.5	17.6	7.29	3.07	-2.54	35	0.016*
ACT (5,11)	20.0	3.7	21.6	4.1	0.72	14	0.484
Gender (9, 28)	0.11	0.33	0.32	0.48	0.12	35	0.228

*indicates significant differences

means, and total course points, were significantly higher for Group II. The means of the third measure of student success, the pretest-posttest differences, were not found to be significantly different.

Summary of Results for the Academic Experience Categories

All five categories were shown to be comparable in terms of prior knowledge related to the course in at least two of the three measures. In all of the categories, Group II had higher means for all measures of student success but these differences were not consistently significant. Three of the categories showed significant differences in at least two of the three student success measures, one category showed significant differences in only one of the student success measures while another category did not show any significant differences in the student success measures. Figures 4 - 6, respectively, show the means of final exam scores, total course points and pretest-posttest difference for the five academic experience categories, with error bars to indicate which of these differences were significant.

The trends for two of the three student success measures, total course points and pretest-posttest differences, showed that the increase in student success associated with the intensive course format diminishes with increased academic experience. At first glance, it appears that the final exam mean data contradict this as the two categories with the largest differences in final exam scores are the least and most experienced populations. However, the most experienced category, greater than 120 completed semester hours, showed significant differences in pretest means, so some of the difference in final exam scores may well be due to the greater preparedness of intensive-student population (pretest mean = 10.0) as compared to the traditional-student

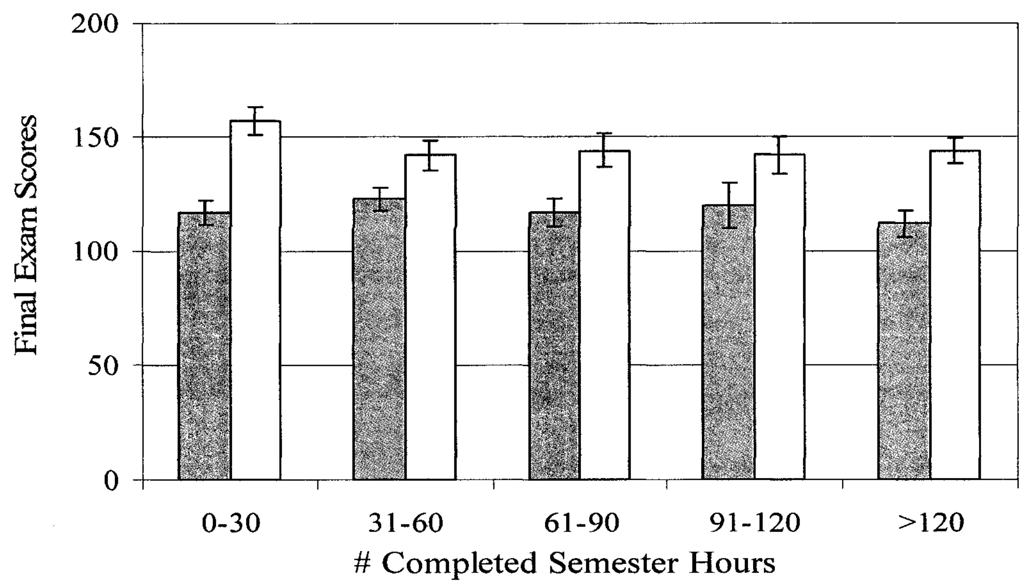

Figure 3. Comparison of final exam score means ± SE among the academic experience categories of Group I (traditional, shaded) and Group II (intensive, clear) students.

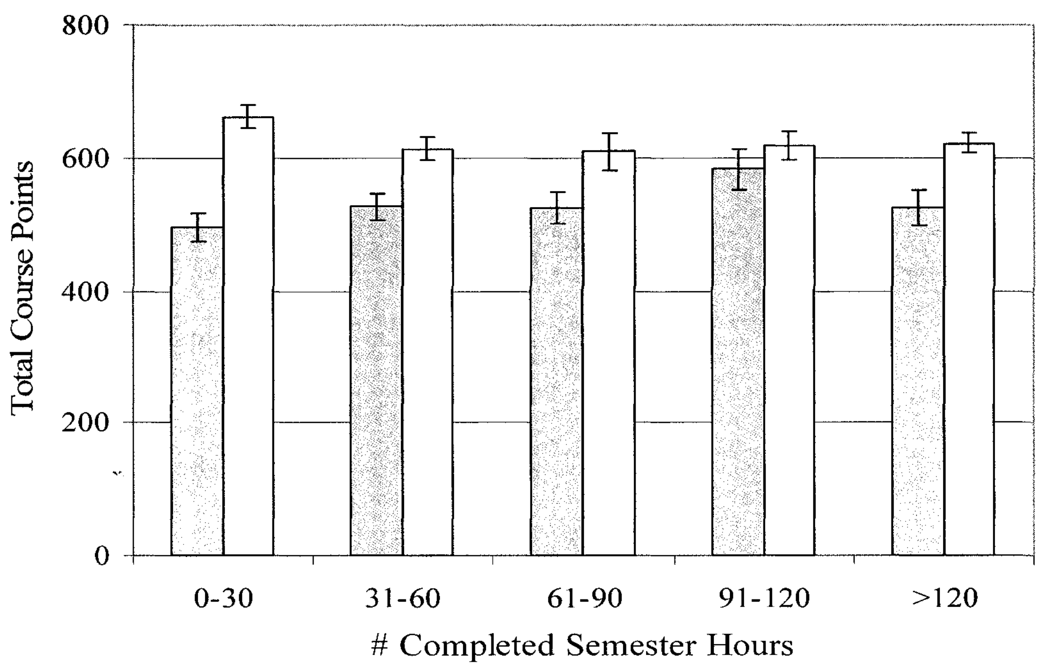

Figure 4. Comparison of total course point means ± SE among the academic experience completed categories of Group I (traditional, shaded) and Group II (intensive, clear) students.

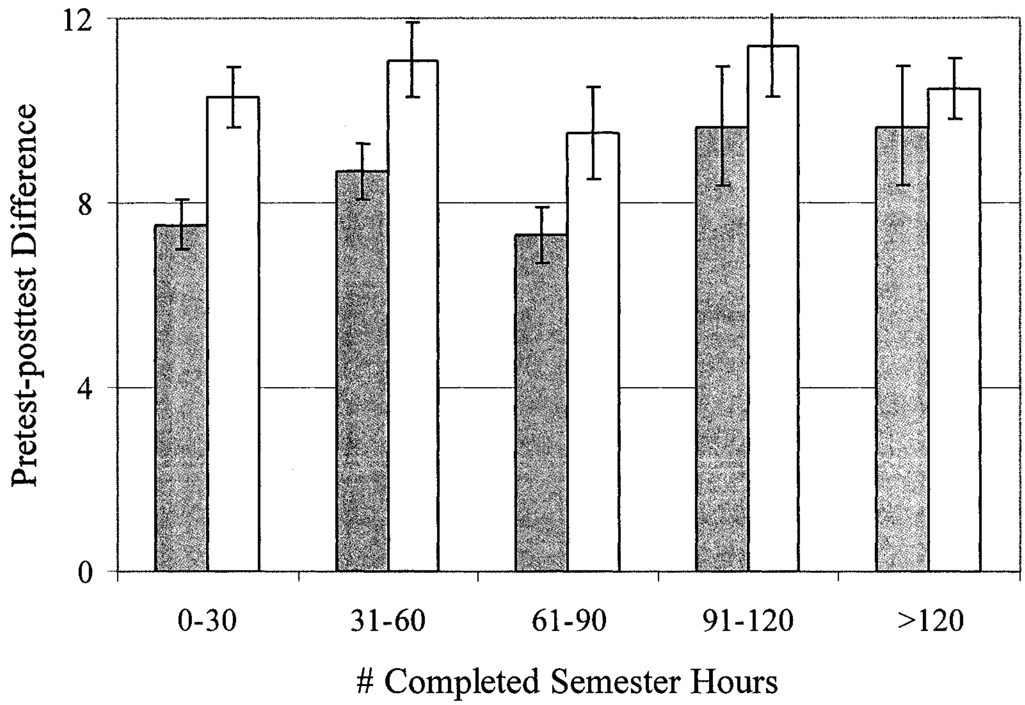

Figure 5. Comparison of pretest-posttest difference means ± SE for the academic experience categories within Group I (traditional, shaded) and Group II (intensive, clear) students.

population (pretest mean = 6.75) rather than any effect due to the intensive course. Both Groups achieved the same pretest-posttest differences within this most experienced category, suggesting that they added similar amounts of new introductory general chemistry knowledge. Therefore, enhanced student success was *not* associated with the intensive course format for this most academically experienced category of students.

The ANCOVA results of the full data set (Table 4) and the reduced data set (Table 6) indicated that the number of semester hours was not a significant factor in determining the variance any of the three measures of student success. Together, the *t*-test and ANCOVA data did *not* support the hypothesis that students with more academic experience achieved greater success in the intensive course format than did students with less academic experience. The number of completed semester hours appears to have had a somewhat negative effect on student success in the intensive course format so Hypothesis Two must be rejected. The data did indicate that students in the three less academically experienced categories (all but the highest two categories for number of completed semester hours) achieved significantly greater success in the intensive course format than in the traditional format.

Hypothesis Three: Students with greater life experience will achieve greater success in intensive versus traditional introductory general chemistry courses than students with less life experience.

Another major difference between the two Groups was the extent of life experience of the students, as estimated by the number of years since high school graduation at the time of the course. The mean of the years since high school graduation values (Table 2) of the students in Group II is 5.0 years, 1.5 times higher than that for the students in Group I (3.5 years). The range of values was also larger for Group I (minus one to 45 years) than for Group II (one to 20 years. It may be more appropriate to compare the median number of years since high school graduation of the two Groups. The median value of the years since high school graduation for Group I was two years while the median value for Group II was four years.

Three categories were created for these data: minus one to two years since high school graduation (traditional-age, beginning college students), three to five years since high school graduation (slightly older, but still traditional-age college students) and greater than five years since high school graduation (nontraditional-age college students). The distribution of students among these three categories is shown in Table 13. More

Table 13. Student Distribution by Years since High School Graduation

Years since High School Graduation	Group I (136)		Group II (95)	
	%	*N*	%	*N*
-1 to 2	64.6	84	32.6	31
3 to 5	22.3	28	33.7	32
> 5	18.5	24	33.7	32

than half of the Group I population was in the youngest category while Group II students were evenly distributed among the three categories.

Minus One to Two Years since High School Graduation

The results of the *t*-test of the data from this category appear in Table 14. The means of Group I were significantly lower than Group II for the following measures: test one, test two, test, the total test scores, the final exam, the total course points, the posttest scores, the pretest-posttest differences, and years since high school graduation. The means of the following measures were not found to be significantly different: test four, test five, the pretest scores, GPA, the number of completed semester hours, ACT composite scores, and gender.

In this, the youngest category, there were no significant differences between the two Groups in terms of GPA, ACT composite scores and pretest scores, indicating that the two Groups were comparable in terms of prior knowledge for the course. The means

Table 14. *t*-Test Results: Minus One to Two Years since High School Graduation

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I		Group II				
	mean	SD	mean	SD	*t*	*df*	*p*
Test one (83, 31)	72.7	13.1	80.0	11.1	2.72	112	0.007*
Test two (81, 30)	70.6	15.6	78.6	11.4	2.57	109	0.012*
Test three (83, 31)	64.5	17.2	73.2	11.1	2.82	112	0.006*
Test four (81, 29)	61.3	14.4	63.5	14.0	0.68	108	0.499
Test five (77, 30)	67.5	15.7	71.3	11.1	1.20	105	0.235
Total Tests (84, 30)	279	52	305	35	2.50	113	0.014*
Final Exam (84, 31)	122	30	147	26	4.05	113	0.000*
Course Total (84, 29)	568	94	633	66	3.53	113	0.001*
Pretest (80, 29)	9.4	2.9	10.0	2.2	0.87	107	0.387
Posttest (81, 31)	17.7	3.8	20.4	2.5	3.67	110	0.000*
Pretest-Posttest Difference (78, 29)	8.03	3.81	10.5	2.9	3.11	105	0.002*
GPA (84, 31)	2.81	0.60	2.94	0.58	1.05	113	0.294
# Semester Hrs (84, 31)	33.1	20.5	39.6	24.1	1.45	113	0.150
Years Since HS (84, 31)	0.75	0.77	1.58	0.50	5.55	113	0.000*
ACT (81, 24)	22.5	3.8	21.8	4.0	-0.74	103	0.459
Gender (84, 31)	0.60	0.49	0.52	0.51	-0.76	113	0.451

*indicates significant differences

of all three measures of student success (final exam scores, total course points and pretest-posttest difference) were found to be significantly higher for Group II.

Three to Five Years since High School Graduation

The *t*-test results for this category are in Table 15. The means of Group II were found to be significantly higher than Group I for the following measures: test five, the final exam, the total course points, the pretest scores, the posttest scores, and the number of completed semester hours. The means of the following measures were not found to be significantly different: test one, test two, test three, test four, the total test scores, the pretest-posttest difference, GPA, years since high school graduation, ACT composite scores, and gender.

Within this category, the two Groups were comparable in academic ability as judged by the lack of significant difference in their GPA and ACT composite scores. However, the pretest scores did show a significant difference with Group II exceeding Group I (10.5 and 8.90, respectively), indicating that Group II students had better academic preparation for the course material. Two of the measures of student success showed significant differences (the means of the final exam scores and total course points), while the means of the pretest-posttest difference were not significantly different. The higher pretest scores for Group II may have made it more difficult for these students to achieve a high pretest-posttest difference. However, even though they started with a greater pretest score, they were able to achieve a slight, but not significantly higher pretest-posttest difference. Overall, these results suggested that Group II students achieved greater success than did Group I students in this category.

Table 15. *t*-Test Results: Three To Five Years since High School Graduation

Measure ($N_{Group I}$, $N_{Group II}$)	Group I		Group II				
	mean	SD	mean	SD	*t*	*df*	*p*
Test one (35, 32)	69.5	12.4	75.6	12.8	-2.00	65	0.049*
Test two (29, 32)	69.5	13.5	73.8	14.0	1.22	59	0.226
Test three (29, 32)	65.3	16.8	67.3	16.8	0.47	59	0.640
Test four (27, 30)	58.3	12.9	64.0	12.5	1.69	55	0.097
Test five (28, 28)	60.2	16.8	68.9	11.2	2.28	54	0.027*
Total Tests (29, 32)	274	41	290	43	1.42	59	0.162
Final Exam (29, 32)	120	28	138	29	2.40	59	0.020*
Course Total (29, 32)	566	80	603	83	3.15	59	0.002*
Pretest (30, 28)	8.90	2.81	10.5	2.7	2.71	55	0.008*
Posttest (29, 32)	17.6	3.6	19.7	3.2	2.40	59	0.020*
Pretest-posttest Difference (29, 28)	8.55	3.55	9.36	3.13	0.91	55	0.368
GPA (29, 32)	2.76	0.47	2.84	0.44	0.66	59	0.510
# Semester Hrs (29, 32)	73.2	39.9	102	42	3.24	59	0.002*
Years Since HS (29, 32)	3.93	0.91	4.13	0.75	1.08	59	0.285
ACT (20, 24)	21.3	3.8	20.6	2.8	-0.67	42	0.508
Gender (29, 32)	0.45	0.51	0.38	0.49	-0.57	59	0.569

*indicates significant differences

Greater than Five Years since High School

The *t*-test results for this category are in Table 16. The means for Group I are significantly lower than Group II for the following measures: test one, test three, test five, the total test scores, the final exam scores, the total course points, the posttest, the pretest- posttest difference, GPA and the number of completed semester hours. The means of the following measures were not found to be significantly different: test two, test four, the pretest scores, ACT composite scores, number of years since high school graduation, and gender.

This final category consisted of data from the oldest students in the population. The two Groups appeared to be comparable in terms of ACT composite scores and pretest scores, but the mean GPA of Group II was significantly higher than that of Group I. ACT scores were only available for a subset of the students in this category, but the available data showed that these two populations are comparable. The ACT composite scores of 21.8 and 21.6 for Groups I and II, respectively, may have been statistically different, but these differences were not likely to result in any meaningful difference in academic ability. All three measures of student success showed significant differences between the two Groups, with Group II having the higher mean in all cases.

Summary of Results for the Academic Experience Categories

The differences between the means of the final exam scores for Group I and Group II among the life experience categories are illustrated in Figure 6. Figures 7 and 8 show the means of the total course points and pretest-posttest differences, respectively. The error bars show that the majority of these differences were significant. In particular,

Table 16. *t*-Test Results: More Than Five Years since High School Graduation

Measure ($N_{Group I}$, $N_{Group II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (24, 31)	73.8	13.0	81.3	11.1	2.30	53	0.026*
Test two (24, 32)	72.5	11.7	77.9	14.1	1.51	54	0.137
Test three (22, 31)	61.4	14.5	73.1	19.3	2.40	51	0.020*
Test four (22, 32)	61.5	18.6	69.6	18.2	1.49	52	0.118
Test five (22, 30)	65.9	13.7	74.5	15.8	2.05	50	0.046*
Total Tests (24, 32)	279	46	312	54	2.42	54	0.019*
Final Exam (24, 32)	120	27	152	29	4.13	54	0.000*
Course Total (24, 32)	566	84	645	95	3.28	54	0.002*
Pretest (23, 32)	8.48	3.26	9.34	3.32	0.96	53	0.341
Posttest (24, 31)	16.2	3.9	21.0	2.9	5.15	53	0.000*
Pretest-Posttest Difference (23, 31)	7.91	3.94	11.7	4.0	3.45	52	0.001*
GPA (29, 32)	2.69	0.62	3.04	0.65	2.07	54	0.043*
# Semester Hrs (24, 32)	80.2	39.1	113	45	2.82	54	0.007*
Years Since HS (24, 32)	12.9	10.0	9.28	2.92	-1.94	54	0.058
ACT (5, 10)	21.8	2.5	21.6	4.9	-0.09	13	0.933
Gender (24, 32)	0.50	0.51	0.34	0.48	-1.17	54	0.247

*indicates significant differences

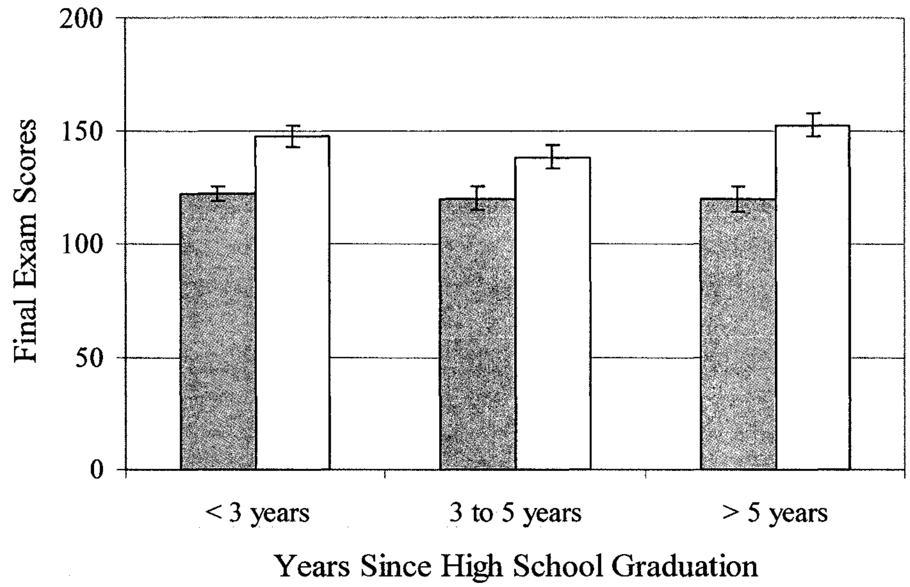

Figure 6. Comparison of the means of the final exam scores (± SE) among the life experience categories for Group I (traditional, shaded) and Group II (intensive, clear) students.

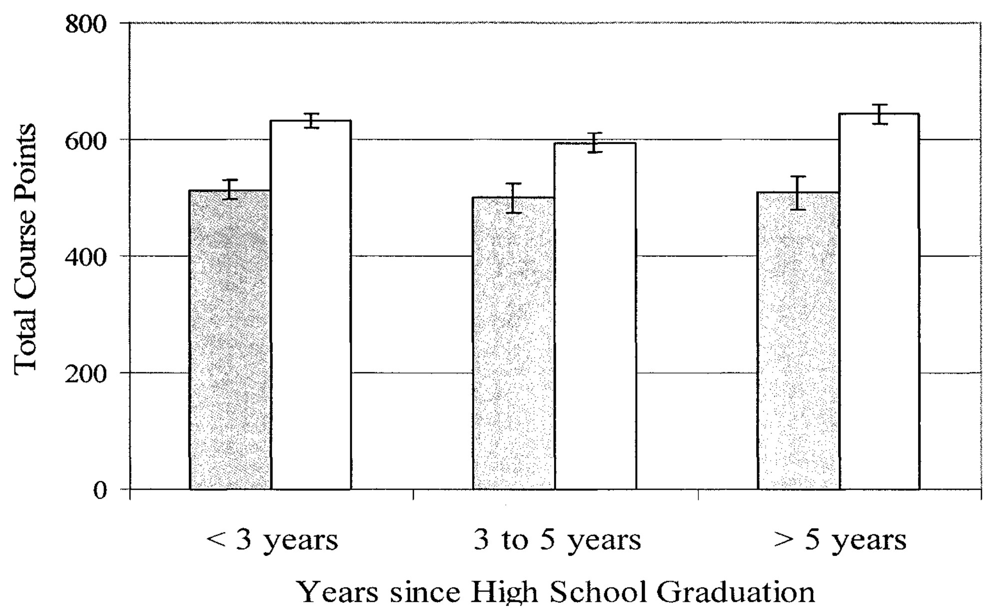

Figure 7. Comparison of total course points (± SE) among the life experience categories for Group I (traditional, shaded) and Group II (intensive, clear).

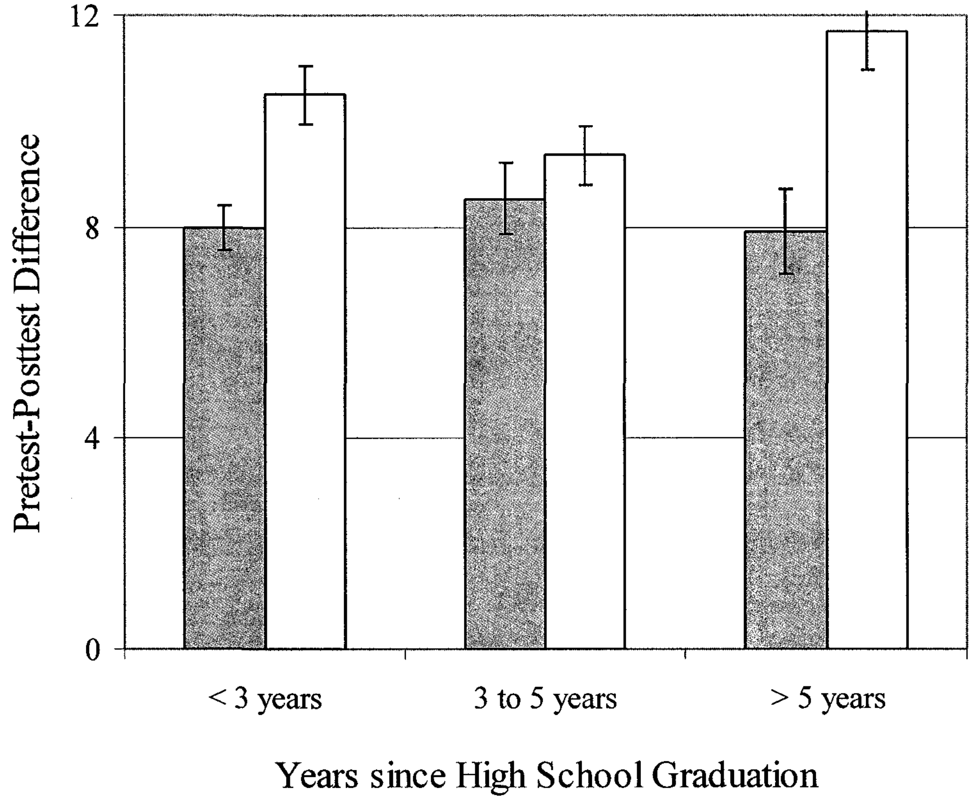

Figure 8. Comparison of the means of pretest-posttest differences (± SE) among the life experience categories for Group I (traditional, shaded) and Group II (intensive, clear).

the youngest and oldest groups showed larger differences than the middle group (three to five years since high school graduation) for all three measures. For the middle group of students, approximately 21 - 25 years of age, the format of the course appears to have had less impact on their success than it did for the youngest and oldest students. The results of the ANCOVA analysis for the full data set (Table 4) indicated that the number of years since high school graduation was not significant in determining the variance of any of the three measures of student success. However, the results for the analysis of the reduced data set (Table 6) indicated just the opposite, that the number of years since high school was significant in determining the variance of all three student success measures. The full data set excluded the effect of ACT scores but included more data points, and was considered to be more valid. However, it was possible that the number of years since high school graduation was a significant factor in the full data set but the effect of the ACT composite scores masked this effect.

Within the life experience categories, the majority of the measures indicated that students in Group II achieved greater success than the students in Group I. A possibly confounding factor was the significant difference between mean overall GPA scores of the two Groups in the greater than five years since high school graduation category. This small difference in mean overall GPA (0.35 units) might have indicated that these two populations were not comparable in terms of academic ability and that these differences in academic ability might have accounted for the some of the enhanced student success seen in Group II of this oldest student category.

These contradictory results made it difficult to state conclusively that academic experience led to greater success in either course format. The ANCOVA results also presented an unclear picture; analysis of the reduced data set indicates that number of years since high school graduation was significantly associated with student success while the analysis of the full data set indicated that this variable was not significantly associated with student success. Presumably, the full data set results were more valid since the populations were greater. However, it is possible that these contradictions may have been due to contradictory associations between life experience and student success. The means of the three student success measures in the three life experience categories within Group I were not found to be significantly different (Figures 6 - 8), indicating that life experience had no effect on student success in the traditional course format. The means of the student success measures for Group II were different for the three life experience categories. Students in the youngest and oldest categories experienced greater success in the intensive format course than did the students in the middle category. The seemingly contradictory ANCOVA analyses and the *t*-test results might be explained by a positive association between life experience and student success in the intensive course format and a lack of any correlation between life experience and student success in the traditional course format.

Hypothesis Four: Students Majoring in Life or Physical Sciences Will Achieve Greater Student Success in an Intensive versus Traditional Introductory Chemistry Courses than Students in Non-Science Majors.

The students in the two Groups represented 40 different declared majors, which were combined into three categories: life sciences, physical sciences and non-science majors (Table 17). More Group I (traditional format) students majored in life sciences (45.6%) than in the physical sciences (36%) or the non-science majors (18.5%). More Group II (intensive format) students majored in physical sciences (50.5%) and non-science majors (26.3%) than life sciences (23.2%). See Table 18 for a complete listing.

Table 17. Distribution of Students into Categories by Academic Major

Category	Code	Group I (136)		Group II (95)	
		N	%	*N*	%
Physical sciences	1	49	36.0	48	50.5
Life sciences	2	62	45.6	22	23.2
Other	3	25	18.4	25	26.3

Table 18. Academic Majors in the Two Experimental Groups, Traditional and Intensive

Major	Group I	Group II
Accounting	0	1
Aerospace	34	34
Agribusiness	2	0
Animal science	12	3
Art	2	0
Biology	12	2
Business	1	2
Chemistry	3	0
Computer science	3	3
Concrete industry management	0	4
Criminal justice	1	0
Early childhood education	1	0
Engineering technology	4	0
English	2	0
Entrepreneurship	1	0
Environmental science technology	2	0
Family and consumer services	0	1
Finance	1	0
Geoscience	0	1
History	1	0
Information systems	2	1
Industrial technology	7	5
Interdisciplinary studies	1	0
Liberal studies	1	0
Mass communications	3	1
Mathematics	0	1

Table 18, continued.

Major	Group I	Group II
Music	1	0
Non-degree	0	12*
Nursing	`8	7
Nutrition and food science	6	3
Office management	1	0
Physical education	1	4
Plant and soil science	4	5
Political science	2	0
Pre-dental	1	0
Psychology	2	0
Recording industry	1	1
Science	18	2
Social work	2	0
Undeclared	7	3
University studies	4	0

*These students may also be transient students.

Physical Science Majors

The *t*-test results for this category appear in Table 19. The means for Group I were found to be significantly lower than Group II for the following measures: test five, the final exam, the total course points, the posttest, the pretest-posttest difference, the number of completed semester, and the number of years since high school graduation. The means of the these measures were not found to be significantly different: test one, test two, test three, test four, the total test scores, the pretest scores, GPA, ACT composite scores and gender.

Table 19. Comparison of Means for Physical Sciences Majors

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (49, 47)	75.1	12.9	77.7	10.9	1.02	94	0.312
Test two (48, 48)	71.5	13.1	75.2	13.4	1.33	94	0.185
Test three (49, 47)	65.9	14.9	70.8	15.5	1.58	94	0.118
Test four (46, 48)	59.8	15.1	63.2	15.6	1.08	92	0.284
Test five (47, 45)	63.0	19.6	70.2	13.5	2.03	90	0.045*
Total Tests (49, 48)	282	44	297	46	1.54	95	0.127
Final Exam (49, 48)	126	27	143	28	3.06	95	0.003*
Course Total (49, 48)	572	83	618	81	2.84	95	0.006*
Pretest (47, 46)	9.83	2.75	9.37	2.78	-0.80	91	0.424
Posttest (47, 48)	18.0	3.6	20.1	3.0	3.13	93	0.002*
Pretest-Posttest Difference (46, 46)	8.00	3.47	10.8	4.0	3.58	90	0.001*
GPA (49, 48)	2.80	0.58	2.94	0.47	1.31	95	0.194
# Semester Hrs (49, 48)	59.2	45.4	101	37	5.02	95	0.000*
Years Since HS (49, 48)	2.94	4.57	5.40	3.34	3.02	95	0.003*
ACT (39, 30)	22.9	3.9	21.5	3.4	-1.55	67	0.125
Gender (49, 48)	0.20	0.41	0.10	0.31	-1.36	95	0.177

*indicates significant differences

These results indicated that the two Groups were comparable in terms of prior knowledge related to the course: pretest scores, GPA, and ACT composite scores were not significantly different between Group I and Group II. The student success measures, final exam scores, total course points and pretest-posttest differences were all found to be significantly higher for Group II data.

Life Science Majors

The *t*-test results for this category appear in Table 20. The means for Group I were found to be significantly lower than those of Group II for the following measures: test one, the final exam, the total course points, the posttest, the pretest-posttest difference, and the number of completed semester hours. The means of the following measures were not found to be significantly different: test two, test three, test four, test five, the total test scores, the pretest scores, GPA, number of years since high school graduation, ACT composite scores, and gender.

The results for the life sciences category were quite similar to those of the physical sciences category: all three measures of prior knowledge related to the course, pretest, GPA, and ACT composite score, were not found to be significantly different between the two Groups, while all the three student success measures, final exam, total course points and pretest-posttest difference, were found to be significantly higher for Group II.

Other Majors

The *t*-test results for this category appear in Table 21. The means for Group I were found to be significantly lower than Group II for the following measures: test one,

Table 20. Comparison of Means for Life Sciences Majors

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (61, 21)	71.4	12.3	78.7	12.6	2.34	80	0.022*
Test two (62, 22)	70.8	15.3	72.7	12.5	0.52	82	0.604
Test three (59, 22)	64.3	15.8	69.3	17.2	1.25	79	0.216
Test four (59, 20)	62.8	14.5	64.4	14.7	0.44	77	0.663
Test five (60, 21)	65.2	18.8	72.2	13.0	1.58	79	0.117
Total Tests (62, 22)	279	49	296	45	1.44	82	0.154
Final Exam (62, 22)	121	28	140	30	2.72	82	0.008*
Course Total (62, 22	568	87	612	87	2.01	82	0.048*
Pretest (60, 19)	8.80	2.83	9.84	2.06	1.48	77	0.143
Posttest (62, 21)	17.2	3.7	20.05	2.73	3.19	81	0.002*
Pretest-Posttest Difference (60, 18)	8.38	4.03	10.5	2.4	2.10	76	0.039*
GPA (62, 22)	2.74	0.58	2.78	0.64	0.24	82	0.810
# Semester Hrs (62, 22)	43.0	24.0	70.9	36.4	4.05	82	0.000*
Years Since HS (62, 22)	3.21	4.23	4.77	3.82	1.53	82	0.131
Act Composite Score (47, 17)	21.43	3.32	21.65	3.52	0.23	62	0.817
Gender (62, 22)	0.87	0.34	0.73	0.46	-1.56	82	0.123

*indicates significant differences

Table 21. Comparison of Means for Other Majors

Measure ($N_{Group\ I}$, $N_{Group\ II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (25, 25)	70.9	12.0	81.5	13.1	2.98	48	0.005*
Test two (23, 24)	6.4	14.4	83.6	11.6	4.25	45	0.000*
Test three (25, 25)	59.1	16.0	73.5	16.9	3.10	48	0.003*
Test four (23, 23)	55.1	16.1	72.6	13.5	3.98	44	0.000*
Test five (24, 23)	60.8	20.1	70.8	19.5	1.71	45	0.093
Total Tests (25, 25)	262	51	318	43	4.15	48	0.000*
Final Exam (25, 25)	112	31	155	26	5.28	48	0.000*
Course Total (25, 25)	536	97	657	81	4.77	48	0.000*
Pretest (24, 24)	8.79	3.32	11.0	3.1	2.38	46	0.021*
Posttest (24, 25)	16.3	4.3	21.0	2.9	4.46	47	0.000*
Pretest-Posttest Difference (23, 24)	7.65	3.65	10.1	3.2	2.44	45	0.019*
GPA (25, 25)	2.77	0.53	3.09	0.62	1.98	48	0.053
# Semester Hrs (25, 25)	49.5	37.8	66.6	69.4	1.09	48	0.283
Years Since HS (25, 25)	5.68	10.92	4.56	4.17	-0.48	48	0.634
Act Composite Score (19, 11)	22.2	4.0	20.0	4.65	-1.37	28	0.183
Gender (25, 25)	0.44	0.51	0.72	0.46	2.05	48	0.046*

*indicates significant differences

test two, test three, test four, the total test scores, the final exam, the total course points, the pretest, the posttest, the pretest-posttest difference, and gender. The means of the following measures were not found to be significantly different: test five, GPA, number of completed semester hours, ACT composite scores, and number of years since high school graduation.

The results for this last category were similar to those of the other two categories in the academic major category. Two of the three measures of prior knowledge related to the course (ACT and GPA mean scores) showed no significant differences with only the pretest means being significantly higher for Group II. All three measures of student success had significantly higher means for Group II than Group I. In fact, the differences in two of the student success measures (final exam and total course points) for this category were larger than the differences seen for either the life sciences or physical sciences categories.

Summary of Results for the Academic Majors Categories

Figures 9 through 11 show the means of the student success measures of Group I and Group II among the academic major categories. Figure 9 shows the means of the final exam scores, while Figures 10 and 11 indicate total course points, and pretest-posttest difference, respectively. Each clearly shows greater student success for Group II, the intensive course format group; the error bars indicate these differences are all significant.

The results of the both ANCOVA tests (Tables 4 and 6), suggest that a student's choice of academic major does not play a significant role in determining the variance of the student success measures. The significant differences in the means of the three

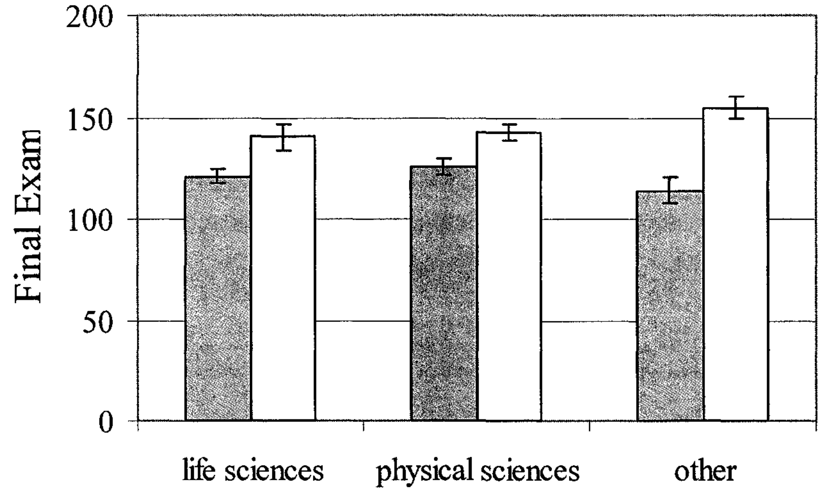

Figure 9. Comparison of final exam means among the three categories for academic major for Group I (traditional, shaded) and Group II (intensive, clear).

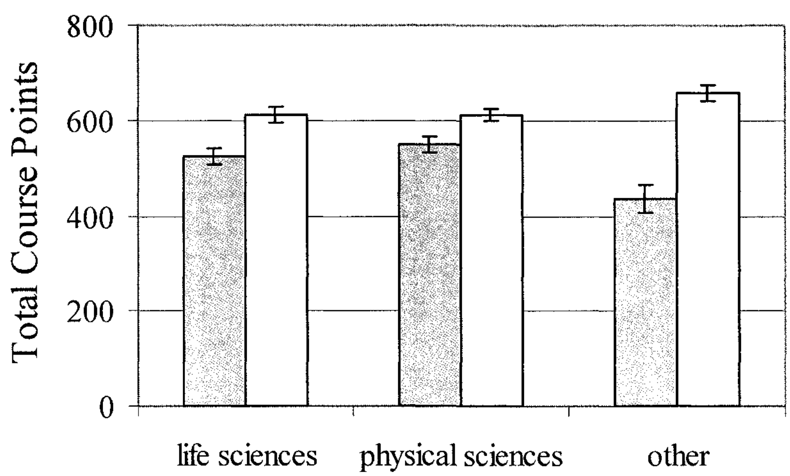

Figure 10. Comparison of total course points means among the three categories for academic major for Group I (traditional, shaded) and Group II (intensive, clear).

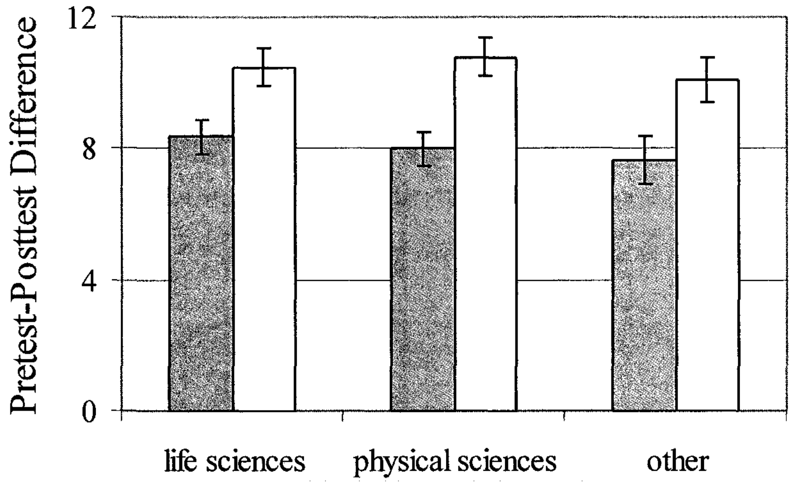

Figure 11. Comparison of pretest-posttest difference means among the three categories for academic major for Group I (traditional, shaded) and Group II (intensive, clear).

academic major categories, Group II students performed better than Group I, indicating a significant instructional effect. Interestingly, the non-science majors (the "other" category) had larger differences for two of the student success measures, final exam scores and total course points, than either the life sciences or physical sciences category (Figure 9 and 10). While the intensive course is associated with improved student success for all three categories, the greatest effect was evident for the non-science majors.

Hypothesis Five: Gender Has an Effect on Student Success in Intensive or Traditional Course Formats.

The final category, gender, compares student achievement between male and female students. Group I, the traditional format, was composed of 46% male students and 54% female students (while Group II, the intensive format, was composed of 59% male students and 41% female students (Table 2). This disparity in gender between the two

Groups was likely to have been linked to the choice of academic majors. Group I had more life sciences majors, while Group II had more physical science than life science majors (Table 17). Gender distribution among the academic majors within the two Groups is shown in Table 22. There were far more male than female students among the physical sciences majors and far more females than males among the life sciences majors.

Table 22. Gender Distribution among the Student Major Categories

Academic Major Category	Group I		Group II	
	Male	Female	Male	Female
Physical Sciences	36 (63%)	10 (14%)	41 (77%)	5 (14%)
Life Sciences	8 (14%)	52 (71%)	6 (11%)	12 (34%)
Other	13 (23%)	11 (15%)	6 (11%)	18 (51%)

Male Students

The *t*-test results for this category appear in Table 23. The means for Group I were significantly lower than those of Group II for the following measures: test two, test three, the total test scores, the final exam scores, the total course points, the posttest, the pretest-posttest difference, GPA, number of completed semester hours, and ACT composite scores. The means of the following measures were not found to be significantly different: tests one, test four, test five, the pretest, and number of years since high school graduation.

Table 23. Comparison of Means of Male Students Category

Measure ($N_{Group I}$, $N_{Group II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (62, 55)	74.0	12.9	78.1	11.2	1.86	115	0.066
Test two (59, 56)	69.1	14.1	75.4	12.9	2.49	113	0.014*
Test three (60, 55)	63.9	15.2	70.3	14.6	2.32	113	0.022*
Test four (57, 56)	57.8	17.7	63.1	14.9	1.74	111	0.084
Test five (58, 51)	63.2	19.9	69.1	15.6	1.68	107	0.097
Total Tests (62, 56)	276	48	297	42	2.58	116	0.011*
Final Exam (62, 56)	121	30	143	28	4.13	116	0.000*
Course Total (62, 56)	556	92	619	79	2.94	116	0.000*
Pretest (59, 53)	9.32	3.55	9.85	2.94	0.85	110	0.397
Posttest (59, 56)	17.4	3.8	20.3	3.1	4.38	113	0.000*
Pretest-Posttest Difference (57, 53)	8.04	3.70	10.49	3.93	3.34	108	0.001*
GPA (62, 56)	2.67	0.61	2.89	0.52	2.12	116	0.036*
# Semester Hrs (62, 56)	59.8	42.3	96.9	44.0	4.66	116	0.000*
Years Since HS (62, 56)	3.84	6.57	5.38	3.65	1.55	116	0.124
ACT (46, 33)	22.8	4.0	21.0	3.52	-2.01	77	0.048*

*indicates significant differences

The means of two of the prior knowledge related to the course measures, GPA and ACT composite scores were found to be significantly different. However, Group I had the higher mean ACT composite score while Group II had the higher mean GPA. The pretest means were not found to be significantly different. From these results, it seemed possible to conclude that the male students within Group I and Group II were comparable in terms of prior knowledge related to the course. The means of all three student success measures for the male students were significantly higher for Group II than Group I.

Female Students

The *t*-test results for this category appear in Table 24. The means for female student in Group I were found to be significantly lower than those of Group II for the following measures: tests 1, test two, test three, test four, test five, the total test scores, the final exam, the total course points, the posttest, the pretest-posttest difference, and the number of completed semester hours. The means of the following measures were not found to be significantly different: the pretest scores, GPA, ACT composite scores, and number of years since high school graduation.

The female student category showed no significant differences for the means of the three measures of prior knowledge related to the course, so it appeared that the female students in both populations were comparable in terms of prior knowledge related to the

Table 24. Comparison of Means of Female Students Category

Measure ($N_{Group I}$, $N_{Group II}$)	Group I mean	Group I SD	Group II mean	Group II SD	*t*	*df*	*p*
Test one (74, 38)	71.9	12.5	80.1	12.9	3.24	110	0.002*
Test two (74, 38)	71.9	14.8	78.7	13,8	2.35	110	0.021*
Test three (74, 39)	64.4	16.4	72.4	18.4	2.37	111	0.020*
Test four (73, 35)	62.0	15.1	70.2	15.1	2.62	106	0.010*
Test five (73, 38)	63.9	18.8	73.2	14.0	2.67	109	0.009*
Total Tests (74, 39)	280	50	309	49	2.96	111	0.004*
Final Exam (74, 39)	122	28	149	29	4.81	111	0.000*
Course Total (74, 39)	572	87	639	90	3.85	111	0.000*
Pretest (73, 36)	9.11	2.35	10.0	2.6	1.80	107	0.076
Posttest (74, 38)	17.4	3.8	20.4	2.6	4.42	111	0.000*
Pretest-Posttest Difference (73, 35)	8.19	3.75	10.6	2.7	3.40	106	0.001*
GPA (74, 39)	2.87	0.54	3.01	0.62	1.32	111	0.191
# Semester Hrs (74, 39)	41.6	27.5	68.4	53.4	2.55	111	0.001*
Years Since HS (74, 39)	3.29	5.77	4.54	3.68	-0.07	111	0.947
ACT (60, 25)	21.8	3.6	21.6	4.00	-1.22	83	0.224

*indicates significant differences

course. The means of all three measures of student success were significantly higher for female students in Group II.

Summary of Results for the Gender Categories

The means of the three student success measures for the gender categories are shown in Figures 12, 13 and 14. In each case there were no significant differences between male and female students' means within a Group. Male and female students achieved similar student success in the traditional format course and also in the intensive format course. Male and female students both achieved enhanced student success in the intensive course format. The error bars in the figures clearly show that these differences were significant.

According to the ANCOVA analyses of both the full and reduced data sets (Table 4 and Table 6, respectively), gender made no significant contribution to the variance of any of the student success measures. From these results, along with the results of the individual *t*-tests, it can be concluded that gender played no significant role in student success in either the intensive or traditional course format. Both male and female student achieved similar student success in the traditional course format and greater student success in the intensive course format. Thus, hypothesis five, that gender has an effect on student success in intensive introductory chemistry courses, must be rejected.

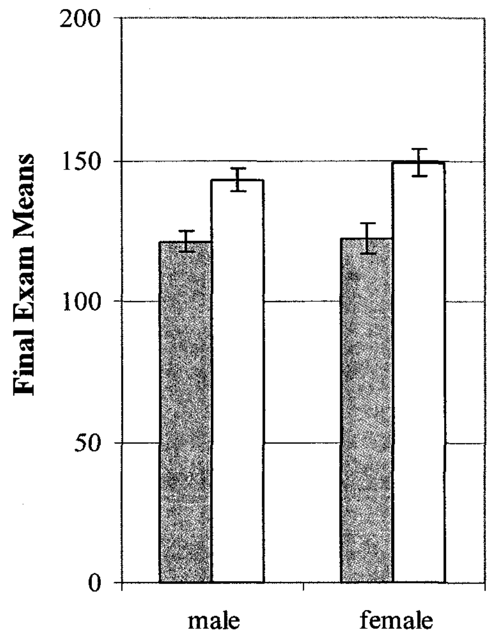

Figure 12. Comparison of final exam means between the genders for Group I (traditiona, shaded) and Group II (intensive, clear).

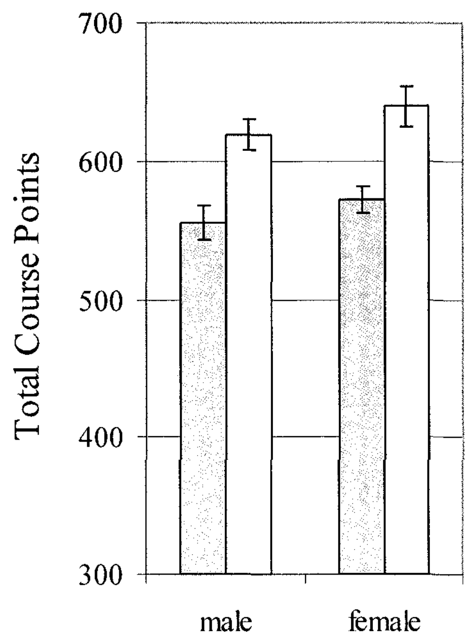

Figure 13. Comparison of total course points means between the genders for Group I (traditional, shaded) and Group II (intensive, clear).

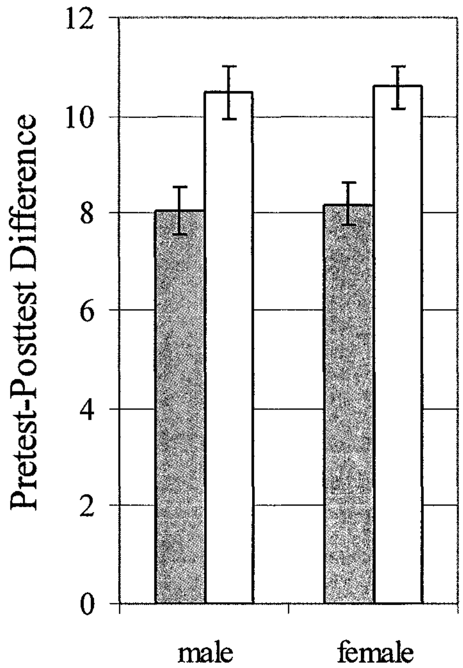

Figure 14. Comparison of pretest-posttest difference means between the genders for Group I (traditional, shaded) and Group II (intensive, clear).

CHAPTER 4

CONCLUSIONS

The results of this study support the body of literature related to the superior nature of intensive-format courses in effecting student learning as compared to traditional-length courses. Introductory general chemistry can be added to the long list of disciplines and educational levels in which enhanced student success in intensive format courses has been demonstrated. The impact of such factors as academic experience, life experience, academic major and gender on student success in the two course formats is described under the individual hypotheses.

Experimental Hypotheses

Each of the five experimental hypotheses was tested by comparing the means of various academic and course records for the students in the two Groups. ANCOVAs and *t*-tests were applied to the entire data set, and various categories created specifically to test the individual hypotheses. For each hypothesis, two sets of measures were compared: one set consisted of measures indicating student academic ability and/or prior knowledge related to the course while the other set was comprised of measures of student success in the courses. The five experimental hypotheses were:

- Hypothesis One: Students will achieve greater success in intensive introductory chemistry courses than in the traditional-length versions of the same course.

- Hypothesis Two: Students with greater academic experience will achieve greater student success in intensive versus traditional introductory general chemistry courses than students with less academic experience.

- Hypothesis Three: Students with greater life experience will achieve greater student success in intensive versus traditional introductory general chemistry courses than students with less life experience.

- Hypothesis Four: Students majoring in the physical or life sciences will achieve greater academic success in intensive introductory chemistry courses than students with non-science majors.

- Hypothesis Five: Gender has an effect on greater student success in intensive or traditional course formats.

Students performed better in the intensive course format than in the traditional format.

In general, students achieved greater success in the intensive course format. The means of the student success measures were consistently and, in most cases, significantly higher for the intensive format course in these comparisons: the entire data set, the academic experience categories, the life experience categories, the academic major categories and the gender categories. This is in agreement with the majority of the documented research on student success in undergraduate-level intensive courses. This greater success may be due to fewer distractions, greater motivation on the part of the students, increased engagement in the course by the students or it may be simply that the intensive format facilitates the completion of learning cycles by the students.[2,11-12,28]

Students with greater academic experience did not achieve greater academic success in the intensive versus traditional course than students with less academic experience.

Academic experience did not enhance student success in the intensive course format. In fact, students with greater levels of academic experiences achieved similar levels of success in both course formats, while students at lower levels of academic experience (equivalent to three years or less of full-time college enrollment) achieved greater success in the intensive course format. These results support those of Logan and Geltner,[28] who reported that while students improved their GPA when taking initial intensive courses, they did not continue to increase their grades upon taking subsequent intensive courses. That is, they continued to earn higher grades in intensive courses than they had in traditional courses but their grades in intensive courses remained steady and did not show further increases as the students earned more credit hours. The results in this study are consistent with those of another study that found the number of completed semester hours were not significant in determining student success.[9]

Students with greater life experience did not achieve greater student success in intensive versus traditional introductory general chemistry courses than students with less life experience.

Life experience was not found to have any clear effect on student success with the youngest (~17 - 20 years of age) and oldest (> 24 years) subgroups experiencing a greater effect from the intensive course format than the middle subgroup (20 - 24 years of age). This result is not entirely consistent with the finding by Geltner and Logan[11] at Santa Monica College that age appears to have a greater effect on student success in traditional courses than intensive courses, nor is it consistent with findings by other researchers that older students are more likely to succeed in intensive programs.[1,27] The Santa Monica study examined age as a factor in student success in intensive versus traditional courses in all disciplines; there may be differences in disciplines that require specific skills that are easier to learn at a younger age, such as mathematics.

Students majoring in the physical or life sciences did not achieve greater academic success in an intensive introductory chemistry course than students with non-science majors.

Choice of academic major had no effect on student success in the intensive or traditional course format. Students in physical science majors, life science majors, and non-science majors all achieved greater student success in the intensive course format. There were no studies of this nature reported in the literature. The closest data were those reported by Geltner and Logan[11] concerning student goals. In their results, student success rates were significantly higher in the intensive course format, regardless of the students' goals (completion of an Associates Degree, transfer, certification, pre-employment skills, etc.). In the current work, larger differences in student success associated with the intensive course format were seen in the data from students in non-science fields as compared to students in science fields. The three-week, intensive course format may provide for greater engagement of these non-scientists with the subject matter, causing them to become motivated to learn chemistry, which is not required for their programs of study as it is for students in the physical and life sciences.

Gender had no effect on greater student success in intensive versus traditional course formats.

No gender-related differences related to student success in the intensive or traditional course format were observed. Both male and female students achieved greater student success in the intensive course format. This is in agreement with previous conclusions in the literature that both male and female student achieve greater success in the intensive format while differing in their criteria for satisfaction with a course.[9,11,24]

Intensive Courses Facilitate the Completion of Learning Cycles

What can account for greater success in the intensive course format? It is certainly non-intuitive; common sense would lead one to think that having many weeks to learn the concepts of introductory general chemistry would be superior to cramming the course into three weeks. However, students in intensive courses have fewer distractions in the form of other courses, which may allow them to focus more time on the course. While learning cycles were not specifically included in the teaching methods used in the course, the longer class periods of the intensive course may facilitate the inadvertent completion of learning cycles and may afford students sufficient time to construct their knowledge of the course material in a way that is closer to the constructs being evaluated on unit tests and exams. If, as Zull[51] contends, learning cycles reflect how the brain functions, then a learning cycle happens any time learning occurs, whether or not the learning cycle was specifically designed by the class instructor.

Traditional courses meet, for example, three times a week for approximately one hour. It is unlikely that each class experience can allow each student to complete a learning cycle, especially when the instructor was not specifically implementing learning cycle activities in the class presentation. In fact, an experienced, well-prepared lecturer can give a presentation that lulls the students into believing that they understand the concepts presented. In the absence of any disequilibrations, no learning cycle begins. The students then must shift their mental gears for the next class, which may well be in an entirely different discipline and then repeat this process for the four to five different courses they are generally taking. Two days later, when they are back in the first class, if they have not reviewed their notes, completed the homework, read the chapter, or otherwise studied the material from the previous class meeting, they still may not have begun, let alone completed, a learning cycle.

In the intensive introductory general chemistry course in this study, each day represents a week in the semester course. The much longer meeting times allow for more students to complete learning cycles within a class. These lengthy classes force the students to apply what was presented earlier that same day, providing immediate opportunity for disequilibrations and the start of a learning cycle. Smaller class sizes, as seen in these intensive introductory chemistry courses, promote the types of social interactions that facilitate concept development. Students are more likely to chat during the class breaks and are more likely to have the opportunity to talk with each other during the laboratory sessions that happen after class each day. With fewer other classes to serve as distractions and with tests and other assignments occurring at frighteningly short intervals, students are more likely to make the time to do the homework and reading to prepare for the next day's class meeting, thus enabling the completion of learning cycles. The nature of the intensive format promotes greater continuity and social interaction - two characteristics that are of primary importance to learning [39]- far better than does the traditional format.

The fact that all types of students, regardless of GPA, earned higher grades in intensive classes[11,28] supports the notion that they are studying and learning more effectively in the intensive situation. In the current study, students in the intensive courses withdrew from the course at a far lower rate than did students in the traditional course. This may indicate a greater engagement with the subject or a greater motivation to complete the course successfully.[14,15] Greater levels of engagement or motivation appear to be typical of students in intensive courses, as previously reported by Geltner and Logan.[11,28]

How might the spacing effect[32,33] apply here? Perhaps the decreased spacing in repeated presentation of material explains why the increase in student success seen in intensive courses disappears over time, reducing the enhanced student learning in the intensive to the same level as that found in students in the traditional courses.[2,9,15,16,27,28]

Implications for the Classroom

The overwhelming amount of evidence points to the superiority of intensive-format courses for all but the very least academically successful students and, at least in the current study, the most academically experienced. These data support the notion that institutions should offer more intensive courses to a broader range of students. Specifically:

- Advisors should recommend that more students take intensive courses. Students on academic probation may be able to restore their academic standing by succeeding in one or more intensive courses, where they have the chance to be more successful. Non-science majors should be encouraged to take science courses in the intensive format where they are likely to achieve greater academic success.

- Educational institutions and science departments should consider offering more intensive courses, especially for some of the more challenging, upper-level courses that are rarely offered in the intensive format.

- Institutions should consider offering intensive courses on a year-round basis, rather than only during interim or summer sessions, as is commonly done. This could be done by dividing traditional semesters into two or three shorter sessions.

- Institutions planning new programs should consider offering those programs in accelerated format, in which all the courses are offered as intensive courses. Care should be taken when designing these programs to help students avoid the intensive course fatigue reported by students taking multiple intensive courses either simultaneously or in rapid succession.[12,14]

- Faculty development specialists should consider planning intensive courses for faculty who wish to learn new technological, pedagogical or other skills.

Recommendations for Future Studies

While this study was the first to focus on comparing student success between intensive and traditional chemistry classes, one other study[11] included chemistry as one of fifty different disciplines evaluated. This work needs to be corroborated by additional studies, particularly those with more complete records for ACT scores to elucidate the potential significance of life experience as a variable in student success in the intensive versus traditional course formats.

Student success in higher-level chemistry courses, both undergraduate and graduate, should be studied using intensive and traditional formats, where they exist, to see if intensive courses prove to be superior or at least comparable in effecting student learning at the more challenging levels of chemistry. It is possible that students choosing intensive courses may be more motivated; this hypothesis should be tested by the use of the Need for Cognition (NFC) instrument[30] or other measures of motivation[15] in planned studies. As this present work was a post-analysis of student data, no additional testing of the students' motivation was possible. The effectiveness of intensive courses and traditional courses in establishing long-term retention of knowledge should also be thoroughly evaluated.

The attitudes of students and faculty in intensive courses should be studied to investigate the possibility that intensive courses create greater cohesion, both among students and between students and faculty, than in traditional courses and, consequently, lead to more meaningful course experiences. The workplace of a modern scientist is not that of a lone researcher; the ability to form teams and work collaboratively is a valuable skill, which may be fostered by the formation of coherent groups in intensive classes. Any factors that facilitate the formation of coherent and productive student groups should then be adapted for use in the traditional course format.

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PART II. SYNTHESIS OF SALTS OF THE WEAKLY COORDINATING TRISPHAT ANION

CHAPTER 1

INTRODUCTION

Weakly coordinating anions (WCA) have the potential for important uses as industrial co-catalysts, ionic liquids, and electrolytes; consequently many different examples of possible anions have been proposed, synthesized and tested.[1-6] A weakly coordinating anion must have particular characteristics of size, charge, nucleophilicity and stability. The structure of the tris(tetrachlorobenzenedialato) phosphate(V) or "trisphat" anion (Figure 1) suggests it is an unusually weakly coordinating anion that may prove to be useful as a co-catalyst for olefin polymerization.[7] There is no single standard for the comparison of weakly coordinating anions, but rather an array of methods is in use, each yielding a different ranking for the various anions.[2,8-9] One of these methods, Nuclear Quadrupole Resonance (NQR) Spectroscopy, is uniquely able to provide a measure of the coordinating ability of anions containing atoms with quadrupolar nuclei, including chlorine, and thus to verify the nature of trisphat as a weakly coordinating anion.[10-11] This work, which is a continuation of the research begun by Pooja Marella,[7] focuses on the synthesis and NQR analysis of Group 1 metal cation salts of the trisphat anion.

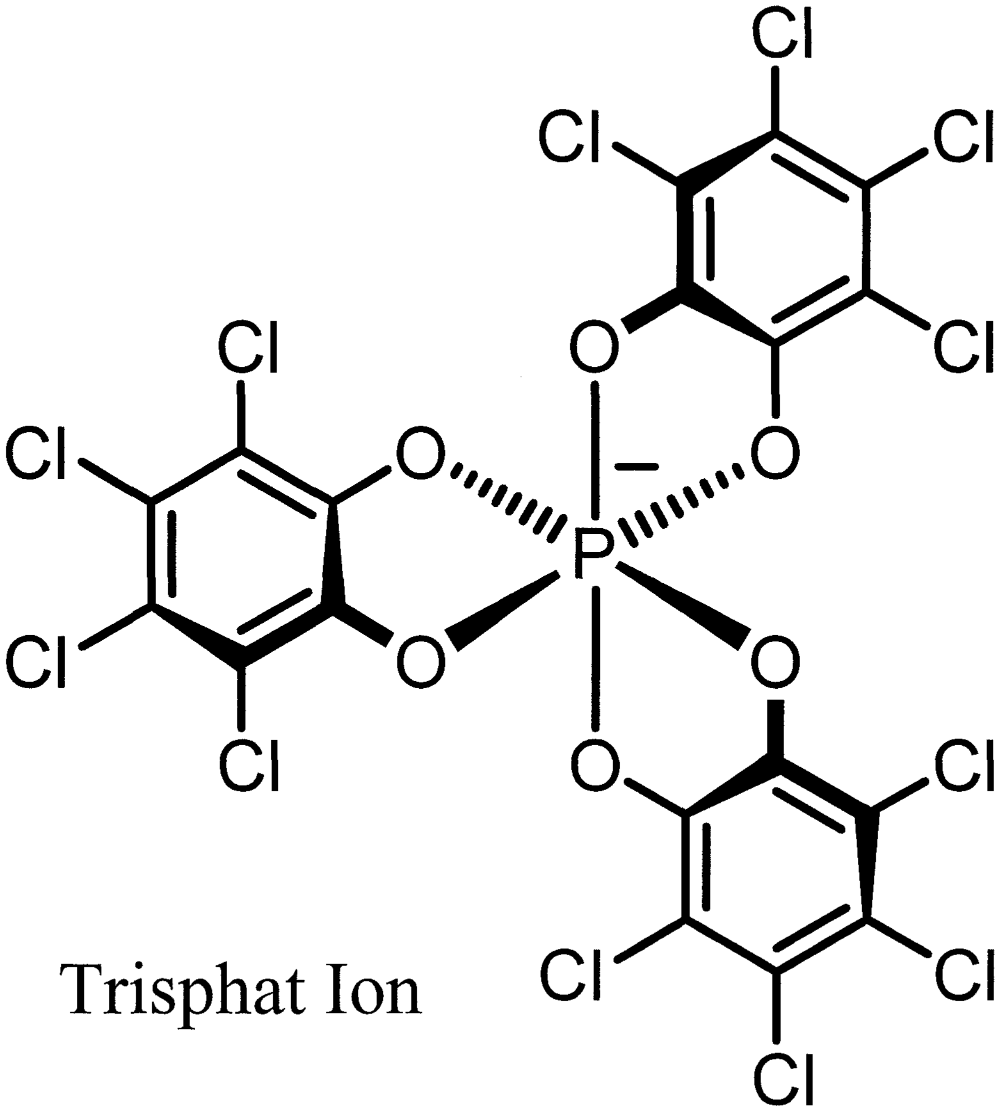

Figure 15. The structure of the trisphat ion, showing one enantiomer.[12]

Weakly Coordinating Anions

Characteristics of Weakly Coordinating Anions

According to Krossing and Raabe[1], the design of a weakly coordinating anion must include the "delocalization of the negative charge over a large area of non-nucleophilic and chemically robust moieties." Any nucleophilic sites on the anion must be somehow rendered inaccessible. Reed[2] describes the qualities of potentially useful weakly coordinating anions as "low nucleophilicity, chemical inertness, solubility, leaving group lability and weak coordination." Finally, Wulfsberg[8,10] states that weakly coordinating anions should be as nonbasic as possible. These anions should have both a low charge and a large size over which the charge is dispersed. A common approach to making an outstanding weakly coordinating anion is to start with a readily available weakly coordinating anion, such as methanesulfonate or tetraphenylborate, and replace the surface hydrogen atoms with fluorine atoms. Products substituted with the hard base, fluorine, however, might be expected to coordinate more strongly to hard acid catalysts than those containing softer bases, such as chlorine, bromine or iodine. This might lead one to propose that an iodinated large anion would make a better weakly coordinating anion for use with hard acid catalysts.

Types and Applications of Weakly Coordinating Anions

The types of weakly coordinating anions vary with the needs of each specific application. The following is not intended to be a comprehensive treatment of the weakly coordinating anion literature but rather a brief overview of these anions and their uses, both current and potential. The most relevant application of trisphat as a weakly coordinating anion is likely to be as a co-catalyst in olefin polymerization.

Weakly Coordinating Anions and Olefin Polymerization

One very important application of a weakly coordinating anion is to act as a co-catalyst, or counter-ion, with a hard acid cation such as zirconocene, $[(C_5H_5)_2Zr(CH_3)]^+$, in olefin polymerization. This application requires the weakly coordinating anion to be both chemically inert and a good leaving group, allowing it to be readily displaced from the cation by the alkene monomer.[1-2, 5-6,11] Examples of anions used for this purpose include many boron-based anions such as $[B(C_6F_5)_4]^-$ (Figure 2), $[B\{C_6H_3(CF_3)_2\}_4]^-$ and others with larger perfluoroalkyl groups replacing the trifluoromethyl groups.[1] A general scheme of how these anions ideally work in olefin catalysis appears in Figure 3.

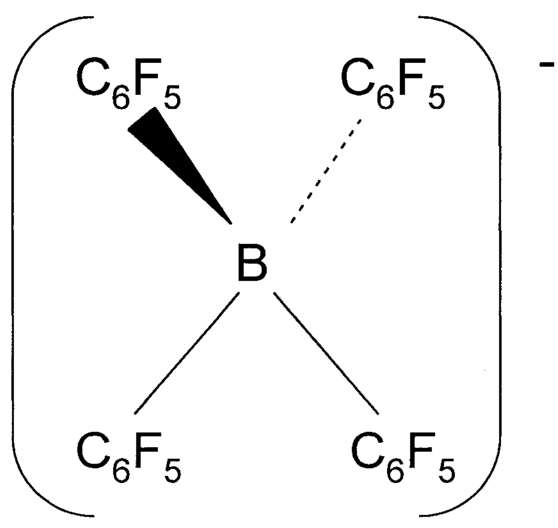

Figure 16. The perfluorotetraphenyl borate ion $[B(C_6F_5)_4]^-$.

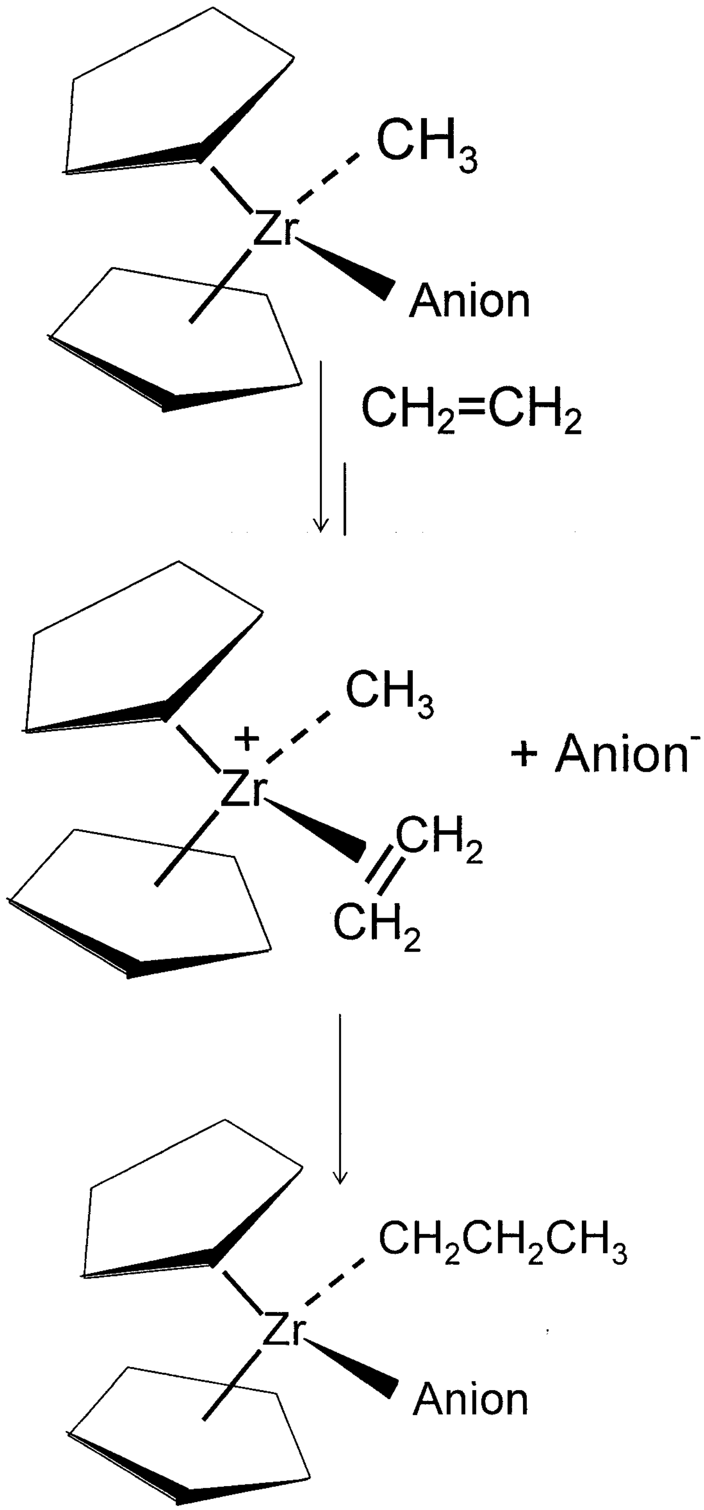

Figure 17. Ideal role of a weakly coordinating anion in olefin catalysis.[13]

Better, more stable co-catalysts can be made from these borate-based anions by forming dimeric borates (Figure 4) as reported by LaPointe and coworkers[6], or by other methods of expanding the borate ion.[14-17] These enlarged anions disperse the single negative charge over more atoms, resulting in weaker ion-pairing with the cationic catalysts.[14-17]

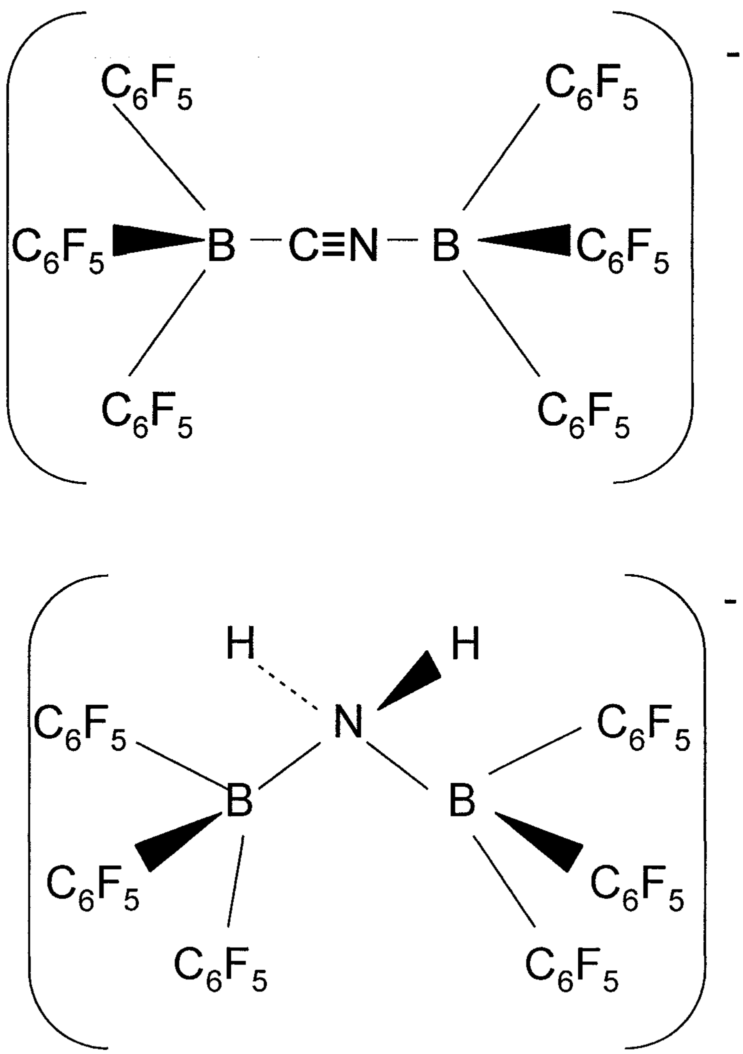

Figure 18. Examples of dimeric (expanded) borate ions.[17]

Another boron-containing group of weakly coordinating anions that function as co-catalysts is the carborane family of compounds. Carboranes, derivatives of the icosahedral $CB_{11}H_{12}^-$, are exceedingly inert, possessing no lone pair or π electrons and exhibiting delocalized σ bonding.[2] The hexahalocarboranes (Figure 5), $[CB_{11}H_6X_6]^-$ (X = Cl, Br, or I.), are among the most weakly coordinating anions yet identified and have been shown to have some ability to stabilize important cationic species.[1-2, 4, 17]

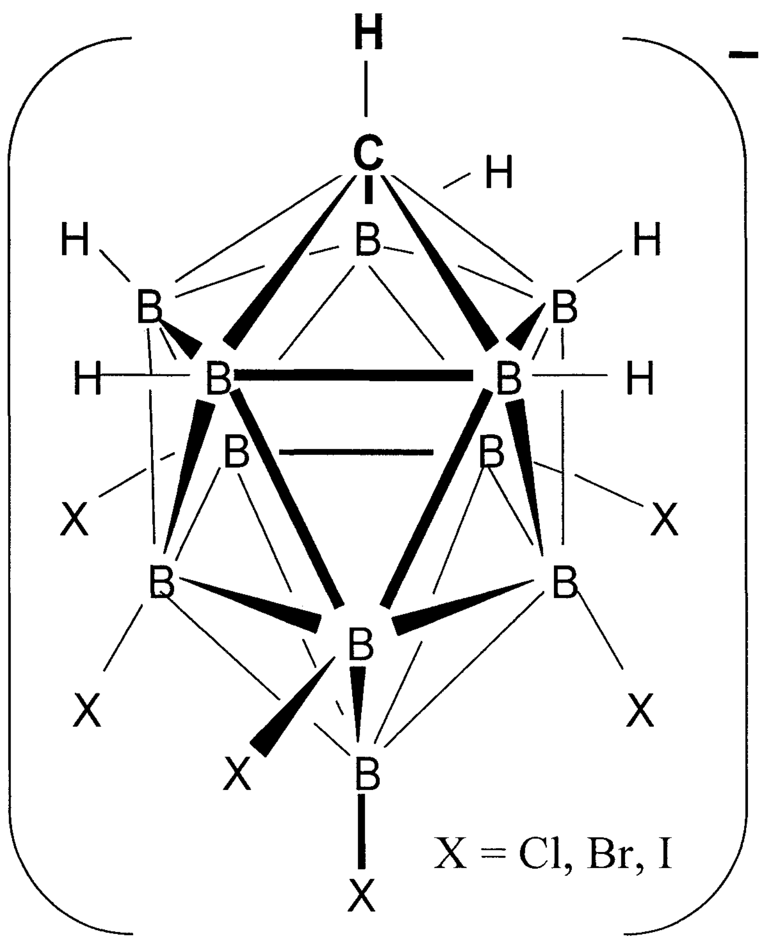

Figure 19. The hexahalocarborane ion.[17]

Alkoxy and aryloxy metallates (Figure 6) form another category of weakly coordinating anions. These contain central atoms such as B, Al, Nb, Ta, Y and La, that are coordinated to perfluorinated -OR and -OAr groups. In general, compounds exhibiting the general formula, $[M\{OC(CF_3)_3\}_4]^-$, are easier and safer to synthesize than the more standard co-catalyst anion, $[B(C_6F_5)_4]^-$, and function at least as well in olefin polymerization.[1,18]

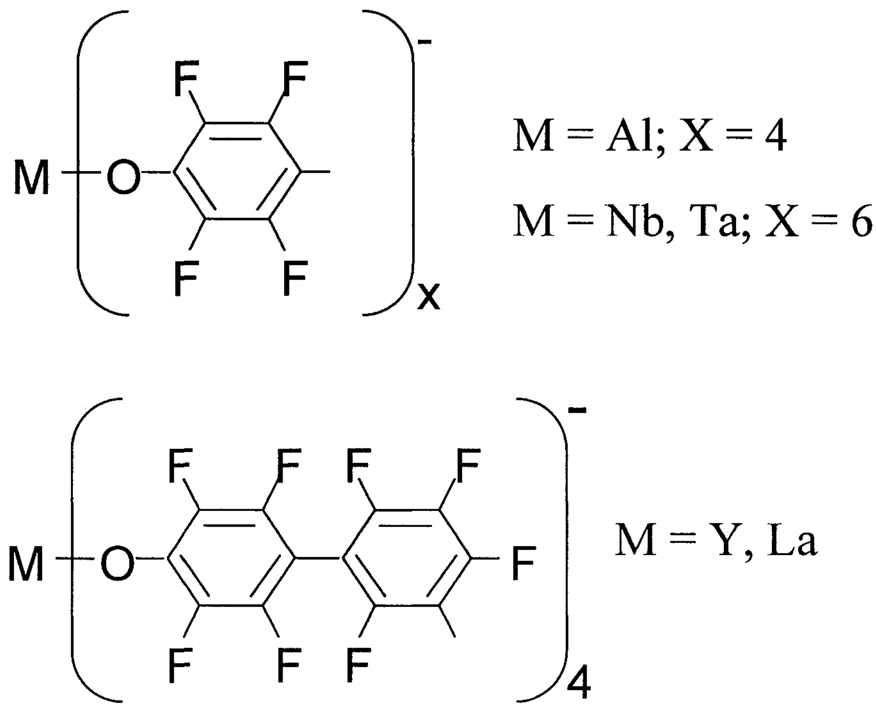

Figure 20. Examples of perfluorinated aryloxy metallates.[18]

A Selection of Examples of Other Weakly Coordinating Anions and Applications

Compared to traditional counter-ions, use of weakly coordinating anions renders the cation more accessible or "naked." Catalyst cations such as Li^+ and Ag^+, used in organic reactions, can be made effective at far lower concentrations and much safer to use by coupling them with one of the borate, carborane or aluminum alkoxide anions mentioned above instead of the traditional, and potentially explosive, perchlorate ion [1-2]. Carbaalanate ions such as $[(AlH)_8(CCH_2t\text{-Bu})_6]^{2-}$ (Figure 6) have been shown to stabilize aluminum hydride cations, making them safer and easier to use.[3]

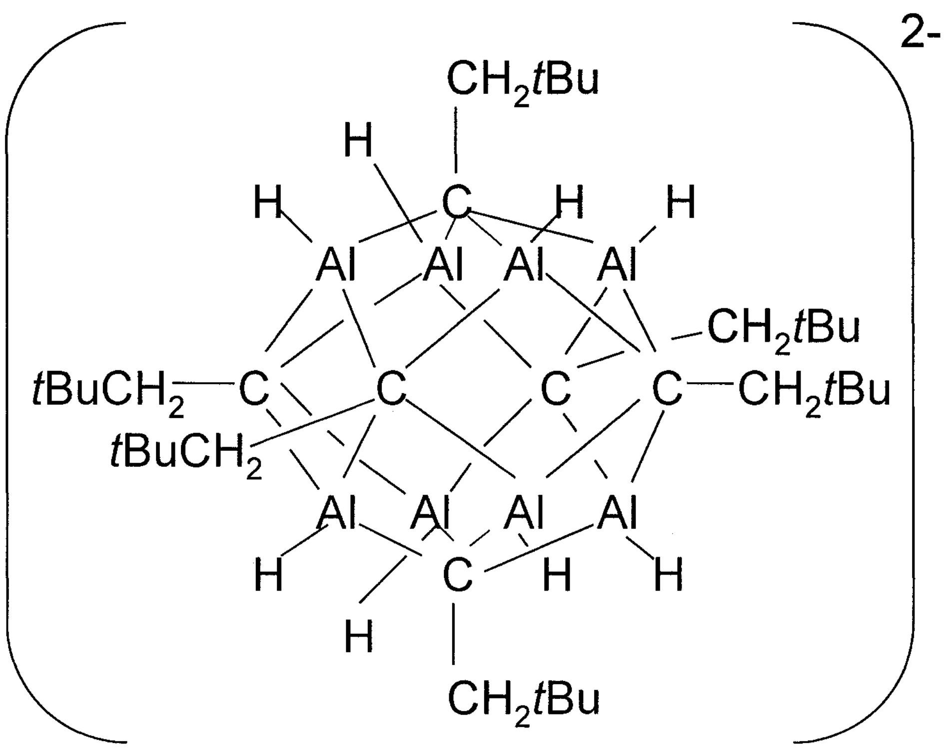

Figure 21. The carbaalanate ion, $[(AlH_8(CCH_2t\text{-Bu})_6]^{2-}$.[3]

Weakly coordinating anions can also enhance conductivity in lithium-ion batteries, due to their weak ion-pairing. However, larger anions increase viscosity, which tends to decrease conductivity. Therefore, lithium-ion batteries require smaller weakly coordinating anions to enhance conductivity, such as those found in the salts, $Li[Im(BF_3)_2]$ and $Li[2\text{-}MeIm(BF_3)_2]$ (Im = imidazolate), which are among the candidates to replace the more typical $Li[PF_6]$ salt.[1,18]

When weakly coordinating anions are partnered with unsymmetrical large cations, the resulting compounds may be ionic liquids if their melting points are below 100 °C. Ionic liquids may replace volatile organic solvents in industrial processes and provide a "greener" alternative.[19] Examples include cations such as 1-ethyl-3-methyl imidazolium or quaternary pyridinium ions paired with $[AlCl_4]^-$, $[PF_6]^-$, or $[B\{(C_6H_3(CF_3)_2\}_4]^-$ and the compound, $LiAl[OC(Me)(CF_3)_2]_4$, which has a melting point of 45 °C.[1,19]

Nuclear Quadrupole Resonance Spectroscopy and Weakly Coordinating Anions

Evaluating Weakly Coordinating Anions

There is no definitive measure for comparing various weakly coordinating anions, but rather a variety of measures that yield slightly different rankings for weakly coordinating anions. The most commonly used method is bond length determination from X-ray crystallography, which is discussed below. Other measures include various spectroscopic tools, conductivity measurements, and thermochemical data.[2,8-9] A commonly used spectroscopic method is the IR analysis of the CO stretching frequency (νCO) of $FeCp(CO)_2Y$ where Y is the weakly coordinating anion in question. Increasing νCO is associated with increasing cationic character of the Fe complex and is indicative of weak coordination. A second spectroscopic method is the NMR analysis of silyl species, i-Pr_3SiY, where Y is the weakly coordinating anion in question. The magnitude of the downshift in ^{29}Si NMR chemical shift correlates inversely to the degree of coordination to the anion.[2]

The most commonly used method of determining the degree of coordination between two atoms is to measure the distance between them by X-ray crystallography. If this distance is larger than the sum of the single covalent or ionic radii, but still smaller than the sum of the van der Waals radii, the interaction is deemed a weak secondary bond. However, van der Waals radii are not easily determined and the existing values may not be accurate in all situations.[8] Wulfsberg[8] suggests that when secondary bonding is indicated by X-ray crystallographic data, it should be verified by other means. One means of doing so is NQR spectroscopy, which is uniquely capable of detecting the subtle changes in electron density associated with weak coordination to NQR active nuclei.[8]

Brief Discussion of Nuclear Quadrupole Resonance Theory

Atomic nuclei with spins ≥ 1 have nonspherical nuclei and exhibit an electric quadrupole moment. This is in contrast with NMR-active nuclei that have spins of $+\frac{1}{2}$ and have magnetic dipole moments. In NQR, the electric moment of the quadrupolar nucleus interacts with the electric field gradient produced by the atom's electronic environment to generate a series of energy levels that are unique to the compound containing the quadrupolar nucleus and even to the specific crystal structure of the compound. Transitions between these energy levels reflect changes in the angular momentum orientation of the nuclei ("flipping" of the nuclei) are accomplished by exposing the quadrupolar nuclei to photons of electromagnetic energy in the radio frequency range.[8,13,21]

NQR frequencies are incredibly sensitive to the electronic environment of the quadrupolar nucleus[8,21]. According to Wulfsberg[8], "NQR frequencies can readily be measured to within a few kHz, but are shifted by their environment over a range of a few or many MHz." A classic example is CCl_4: there are 16 different chlorine positions in the crystal structure of solid CCl_4, which are reflected in its 16 unique ^{35}Cl NQR frequencies.[8]

The limitations of NQR spectroscopy are not trivial. The technique is restricted to compounds containing quadrupolar nuclei and, although most naturally occurring elements have at least one quadrupolar isotope, this isotope must be abundant to be useful. The fact that any samples must be available in gram-scale quantities for NQR analysis presents a even more serious drawback.[8]

Use of NQR to Evaluate the Extent of Coordination

The use of ^{127}I NQR spectroscopy has shown that there is significant coordination (bonding) between iodine atoms and Ag^+ in the compound, $[Ag(CH_2I_2)_2]PF_6$, but very little bonding between these iodine atoms and Hg atoms in the compound, $C_5Cl_5HgCl{\bullet}CH_2I_2$.[23]

Wulfsberg and coworkers[10] investigated the degree of coordination between Ag^+ and various halogen atoms in monosubstituted methanesulfonate anions, $CH_2XSO_3^-$ (X =

Cl, Br, or I), using NQR spectroscopy. According to the Hard-Soft Acid-Base Principle, Ag^+, a soft acid cation, is expected to coordinate more strongly with I, a very soft base, than with Br and even less strongly with Cl, a borderline base.[24] Potassium salts were also investigated. The K^+ ion, a hard acid cation, is expected to coordinate preferentially with the sulfonate oxygen atoms rather than the halogens. The results of ^{127}I, $^{79,81}Br$ and ^{35}Cl NQR spectroscopy of these salts showed decreases in NQR frequency for the Ag^+ salts as compared to NQR frequencies of the parent dihalomethanes. These decreases followed the expected pattern, with the iodinated compounds having the largest decrease and the chlorinated compounds the smallest. The NQR frequencies of the K^+ salts were very close to those of the parent dihalomethanes, suggesting, as predicted, that there was no detectable coordination between the K^+ and the halogen atoms.[10]

Wulfsberg and coworkers[9] also completed an NQR study of the coordinating abilities of trichlates (compounds including the trichloromethanesulfonate anion) and various chloroacetates. Each anion was paired with a series of Lewis acid cations, from the nonacidic $(CH_3)_4N^+$ through the very strongly acidic R_3C^+, and the ^{35}Cl NQR frequencies of the resulting ionic or covalent compounds were measured or obtained from the literature. Despite the fact that all of these compounds involve coordination via an oxygen atom, ^{35}Cl NQR frequencies differ sufficiently to elucidate certain trends in NQR frequencies. For covalent compounds involving both trichlates and chloroacetates, the average NQR frequencies increase with increasing electronegativity of the atom coordinated to the oxygen (Figure 8). Figure 9 shows that, for ionic trichlates and chloroacetates, the average frequencies decreased with increasing radius of the cation.

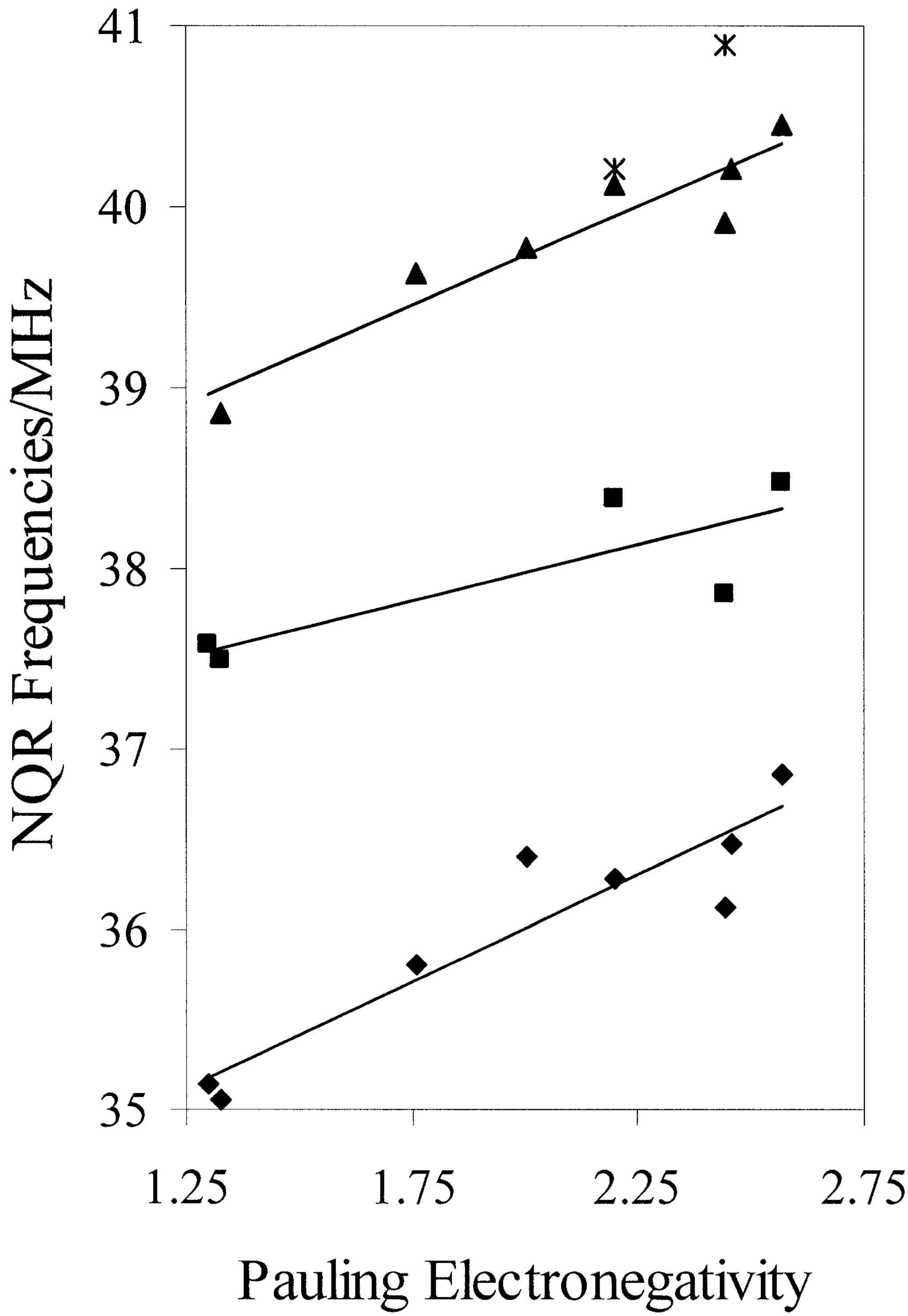

Figure 22. Average ^{35}Cl NQR frequencies of the covalent esters, acids and complexes of anions versus the Pauling electronegativity of the atom covalently bonded to oxygen: $Cl_3CSO_3^-$ (✱), $Cl_3CCO_2^-$ (▲), $Cl_2CHCO_2^-$ (■), and $ClCH_2CO_2^-$ (◆).[9]

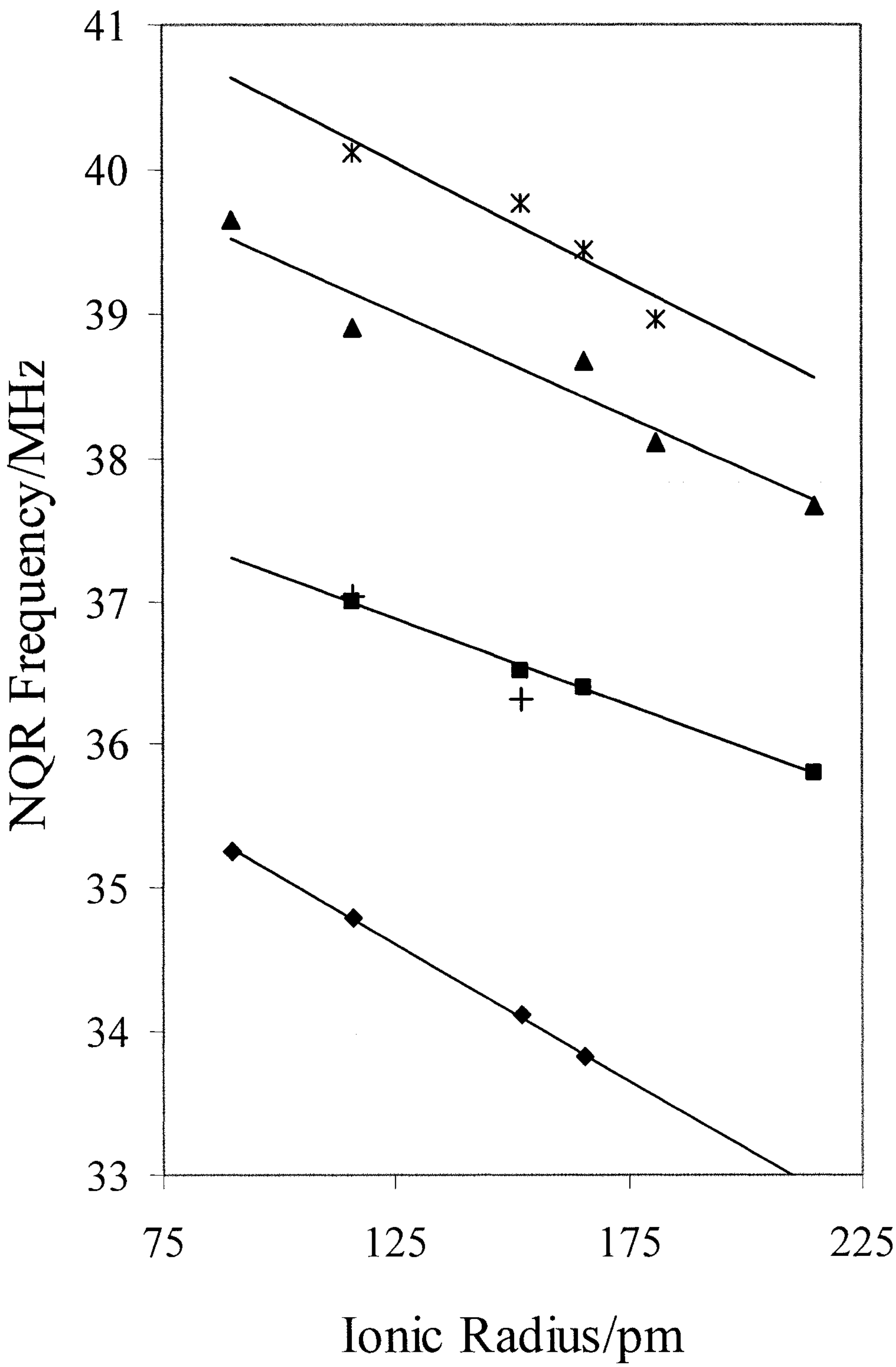

Figure 23. Average [35]Cl NQR frequencies of the ionic salts of anions versus the radius of the cation: $Cl_3CSO_3^-$ (✱), $ClCH_2SO_3^-$ (+), $Cl_3CCO_2^-$ (▲), $Cl_2CHCO_2^-$ (■), and $ClCH_2CO_2^-$ (◆).[9]

The range of NQR frequencies (spectral width) for a given anion paired with various cations sheds light on the coordinating ability of that anion. A broad range indicates that the anion is coordinating more strongly to some of the cations while a narrow range indicates that the anion is coordinating very weakly to all the cations. A truly noncoordinating anion, if one existed, would have the same NQR frequency with every cation. Hence, the slopes of the plots of NQR frequencies versus cation radius (Figure 8) can give insight into the relative coordinating ability of different anions.[9]

Trisphat Anion

Brief History of Trisphat Anion

Lacour[22] and co-workers synthesized trisphat acid as one example of the category of hexacoordinated phosphate ions for use as a chiral resolving agent. In 2004, these researchers[26] reported the details of a large-scale synthesis of tributylammonium trisphat that avoided the instability issues of trisphat acid by linking the synthesis of the acid to immediate conversion to the tributylammonium salt. This method combined a solution of phosphorus(V) chloride and tetrachlorocatechol under an inert atmosphere at 70 °C for 14 hours. After concentrating the resulting product under vacuum, and still working under an inert atmosphere, dichloromethane, hexane and tributylamine were added, causing the precipitation of solid, racemic tributylammonium trisphat. Once this product is formed, no special conditions are required for its stability. This synthesis gave, with a 60% yield, a product with a melting point of 305 °C, ^{31}P NMR δ -79.8 ppm, ^{13}C NMR δ 142.6, 123.6, 114.7, 54.0, 26.2, 20.4, 13.7 and IR (KBr) wave numbers (all in cm^{-1}) 3171.7(w), 2964.7(m), 2877.5(w), 1447.1(s), 1387.1(m), 1300.0(w), 1234.6(m), 989.4(s), 825.9(s), 716.9(m), and 673.4(s). Following Lacour's earlier procedure,[22] but stopping short of forming the tributylamine salt to form trisphat acid, Marella[7] found that using freshly opened phosphorus(V) chloride and recently resublimed tetrachlorocatechol resulted in a product of trisphat acid that could be used quickly in subsequent reactions.

Marella[7] synthesized trisphat salts either by combining solid cesium or rubidium carbonate with freshly prepared solid trisphat acid and grinding them together in a small volume of distilled water, or by reacting Cs_2CO_3 or Rb_2CO_3 and trisphat in aqueous solution. In all cases, the resulting solid was recovered by filtration and dried under vacuum.

A summary of ^{31}P NMR data for compounds involving four-, five- and six-coordinate phosphorus atoms gave the following ranges: compounds with six-coordinate phosphorus atoms, such as trisphat salts, exhibit the largest chemical shifts, -80 to -300 ppm; five-coordinate phosphorus atoms have chemical shifts between -25 and -66 ppm; while four-coordinate compound chemical shifts fall in the range of +40 to -35 ppm. Marella[7] used these ranges not only to determine if a trisphat synthesis has been successful, but also to help diagnose what other, related, products might have formed in an unsuccessful synthesis.

Marella[7] also reported that while the trisphat anion readily decomposes in aqueous or organic solution (it undergoes acid solvolysis), cesium trisphat is only sparingly soluble in water and thus is not subject to hydrolysis. The electrostatic potential map of the trisphat anion (Figure 10, produced by Spartan computational

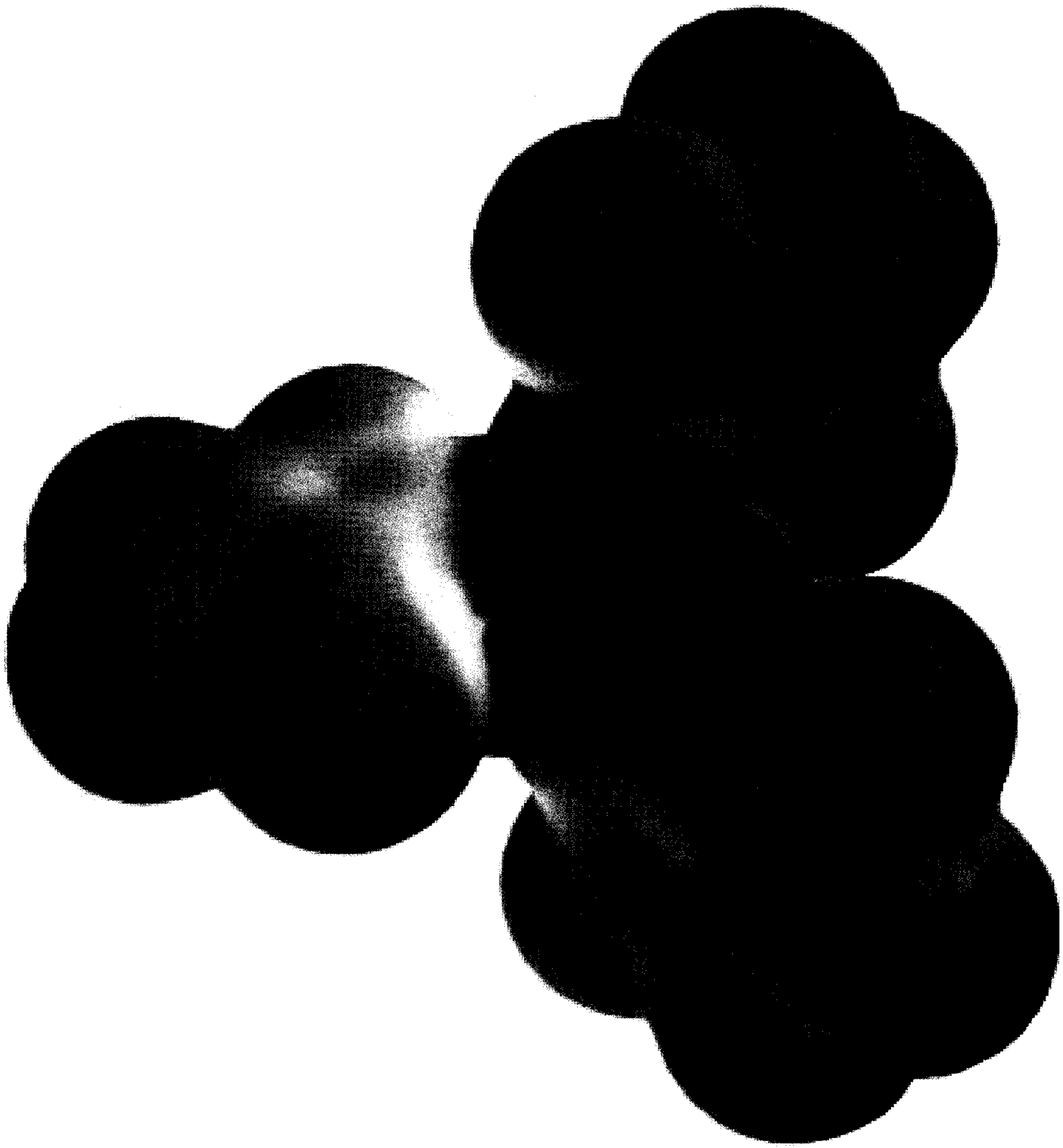

Figure 24. Electrostatic potential map of trisphat ion shows negative regions in red and positive regions in blue. Note: the phosphorus is not visible as it is surrounded by "red" oxygen atoms.

software) shows that the spaces between the aromatic rings, or clefts in the trisphat ion, are only slightly larger than the radius of the Cs^+ ion. It is possible that there is insufficient room for a water molecule to physically contact a cesium ion buried within a trisphat ion to initiate dissolution. The structure of cesium trisphat was only partially resolved by X-ray crystallography due to unexpected complexities in its structure, including the Cs^+ ion acting as a bridge between two trisphat moieties.[7]

Trisphat Anion's Potential as a Weakly Coordinating Anion

Weakly coordinating anions, as a chemical species, share the following characteristics: low charge, large size, charge delocalization over the large size, close to non-basic and non-nucleophilic, chemically stable and soluble in the solvent system for the given application. The trisphat anion clearly has a large size, low charge, an extensive π system for charge delocalization and the presence of electron-withdrawing chlorine atoms bonded to the aromatic rings (Figure 1, Figure 10). Compared to the fluorine atoms in the more standard weakly coordinating anion, perfluorotetraphenyl borate ion, the surface chlorine atoms in trisphat are less negatively charged and, presumably, less coordinating. The trisphat salts of the tributylammonium, potassium, rubidium and cesium cations are soluble in acetonitrile, acetone and dichloromethane but not water or chloroform. The size of the trisphat ion's cleft makes it unlikely that zirconocene would be able to fit into it to coordinate with the oxygen atoms near the central phosphorus atom.[7]

Taking all these facts into consideration, it appears that the trisphat anion has the potential to be a useful weakly coordinating anion for use as a co-catalyst with zirconocene. Recently, Lee[25] reported the successful use of the trisphat anion as a co-catalyst with zirconocene to catalyze the polymerization of carbodiimide monomers.

Since the trisphat acid synthesis is highly sensitive to the presence of water, and since trisphat salts are also highly reactive, specialized techniques and equipment are necessary when working with these substances. To learn the basic, but nontrivial, skills of working with these highly sensitive compounds, this work was begun by testing the synthesis of the antimony analog to trisphat, tristibate (Figure 11), which is more stable. When the appropriate conditions were determined and the skills mastered, these techniques were applied to the synthesis of trisphat.

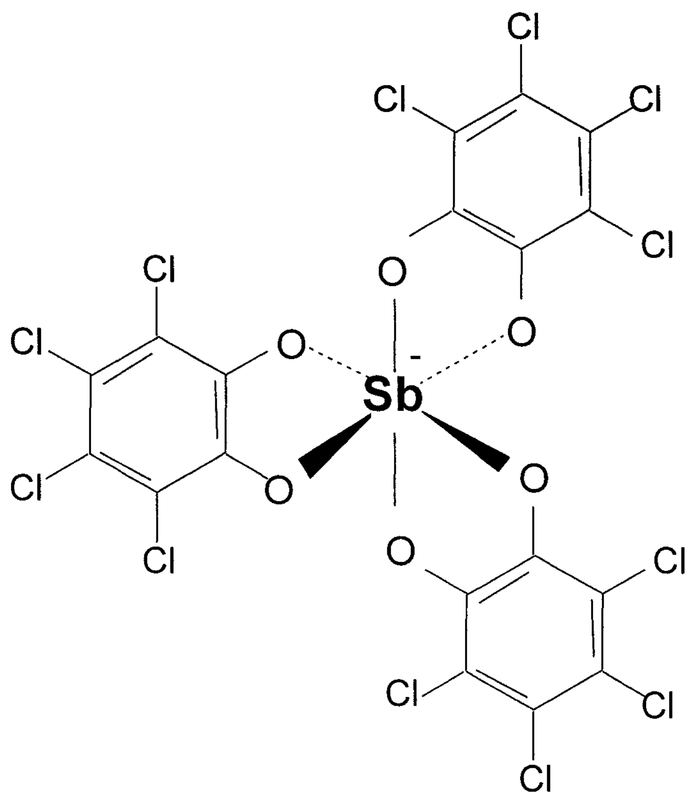

Figure 25. Structure of one enantiomer of the tristibate anion.

CHAPTER 2

EXPERIMENTAL

Methods

Spectra for ^{1}H, ^{13}C and ^{31}P NMR were measured using 300 MHz (ECX, JEOL, Peabody, MA) and 500 MHz (ECX, JEOL, Peabody, MA) instruments. Spectra for ^{35}Cl NQR were measured at 77 K using an NQR spectrometer (Decca Instruments, Walton-on-Thames, Surrey, England). Spectra for IR were measured in KBr pellets using a FTIR (model 1760X, Perkin Elmer, Wellesley, MA) spectrometer.

Materials

Antimony (V) chloride, 1.0 M solution in dichloromethane was 99% pure from Aldrich. Phosphorus pentachloride, sealed under nitrogen gas (5 gram bottles, 98%, Lot no. A106449701, Acros Organics, New Jersey) was used immediately upon opening in the synthesis reaction. Dry toluene was obtained in sealed bottles (99.8% anhydrous Acroseal™, Lot no. B0507409-5G-A, Acros Organics, New Jersey) capped with a septum and removed using a syringe. Tetrachlorocatechol monohydrate (99%, Lot no. 00006880, Lancaster, Pelham, NH) was obtained in 100 gram bottles, dehydrated and purified via vacuum sublimation at 180-190 °C. Tributylamine (98.5% pure, from

Aldrich) was distilled under vacuum over KOH pellets into a round bottom flask and stored sealed with a septum. Potassium hydroxide, cesium hydroxide, and sodium hydroxide were from Fisher Scientific Company. Hexane was from Fisher Scientific Company. Diethyl ether was > 99.9% pure from Aldrich. Acetone-d_6 (99.9 atom % D), chloroform-d (99.8 atom % D) and acetonitrile-d_3 (99.6 atom % D) were obtained in 0.75 mL ampoules from Aldrich.

First Synthesis of $H[Sb(O_2C_6Cl_4)_3]$, Tristibic Acid

A Schlenkware apparatus was assembled as follows: a 3-neck, 250-mL round bottom flask containing a magnetic stirring bar was fitted with a thermometer, a condenser plus gas inlet tube, and a septum. This apparatus was purged five times with argon to remove any air; a flow of argon was maintained through the apparatus for the duration of the reaction; this flow was increased during any additions to the apparatus and maintained at a constant slow flow during long reaction times. The flask was placed in a silicone oil bath on a stirring hot plate. A syringe was used to add 5.0 mL of 1.0 M $SbCl_5$ in dichloromethane (Aldrich) to the flask through the neck fitted with the septum, followed by 10.0 mL of dry toluene. Working within a glove bag purged five times with argon, freshly sublimed tetrachlorocatechol was ground to a fine powder; 4.0 grams of this were transferred to a solid addition funnel. The solid addition funnel replaced the septum in the three-necked flask and the contents of the funnel were added gradually to the flask over ten minutes. The funnel was then rinsed twice with 1.5 mL portions of dry toluene, after which the reaction mixture was heated to approximately 85 °C under continuous argon flow for about 22 hours before being removed from the heat and allowed to cool to room temperature. Twice during the reaction the slurry became too dry to stir and additional toluene was added through the septum (15 mL each time).

The product was dried in the reaction flask under vacuum, and was transferred to a vial within a glove bag purged five times with argon. An odor of toluene was evident so the product was further dried by being transferred to a watch glass and placed in a vacuum desiccator for three hours, at which time the toluene odor was no longer detected. Approximately 2.93 grams of dark-green and gray material were recovered (3.41 mmol of tristibic acid; 68% yield). Small dark crystals were observed under the microscope as well as needle-like crystals indicating the presence of unreacted tetrachlorocatechol. The ^{13}C NMR spectra yielded peaks at the following chemical shifts: 140 ppm, 123.9 ppm, 118.9 ppm plus additional minor peaks at 199.2 ppm, 128.4 ppm and 125.5 ppm.

Preparation of $K[Sb(O_2C_6Cl_4)_3]$, Potassium Tristibate

A 0.471 gram sample of the $H[Sb(O_2C_6Cl_4)_3]$ prepared above was dissolved in 5.0 mL of ethanol to form a yellow-brown solution. This solution was titrated with 0.0478 M ethanolic KOH using phenolphthalein as the indicator. This solution gradually became reddish due to oxidation of the tetrachlorocatechol so the titration was considered complete when the solution no longer turned pH paper red (20.8 mL of 0.0478 M KOH), resulting in a calculated equivalent weight of 474 g/mol. To verify this result, a second sample of 0.124 grams was dissolved in 5.0 mL of ethanol and titrated under argon with 0.0478 M KOH using phenolphthalein as the indicator. 5.47 mL of the KOH were needed to reach the equivalence point, yielding the same equivalent weight of 474 g/mol, far lower than the calculated molar mass of 858.8 g/mol. The low equivalent mass is likely the result of the presence of unreacted tetrachlorocatechol.

The potassium salt from both titrations was recovered by rotary evaporation in a 40-50°C water bath yielding a green-brown, crystalline product. The ^{13}C NMR spectrum showed only two of three expected peaks: 140.3 ppm and 118.8 ppm.

Second Synthesis of $H[Sb(O_2C_6Cl_4)_3]$, Tristibic Acid

The same procedure as described for the first synthesis was followed except that the volume of toluene was increased. 5.0 mL of 1.0 M $SbCl_5$ in dichloromethane was transferred to the Schlenkware apparatus under a flow of argon followed by the addition of 15.0 mL of dry toluene. 4.0 grams of tetrachlorocatechol were added through a dry addition funnel, which was then rinsed with a total of 5.0 mL of dry toluene. The reaction flask was maintained at a temperature between 75 °C and 90 °C for approximately 20 hours, at which time the pH of the gas outflow had reached a value of 2. Upon cooling, crystals formed in the product mixture. These were collected in two rounds of vacuum filtration (more crystals formed in the first filtrate) and washed with ice-cold toluene for a total mass of 2.36 g (2.75 mmol, 55% yield) of tristibic acid. The ^{13}C NMR spectrum had peaks at 140 ppm, 123.9 ppm, 118.9 ppm and additional weak peaks at 34 ppm, 22.4 ppm and 14.1 ppm.

Preparation of $Cs[Sb(O_2C_6Cl_4)_3]$, Cesium Tristibate

A sample containing 0.502 g of the $H[Sb(O_2C_6Cl_4)_3]$ from the second synthesis were dissolved in approximately 5 mL of ethanol to which a drop of phenolphthalein was added. This sample was titrated with a standardized CsOH solution (0.096 M, ethanolic) under argon. A muddy, brown precipitate formed, making it necessary to monitor the progress of the titration using pH paper. 6.66 mL of the 0.096 M CsOH were needed to reach the equivalence point, yielding an equivalent weight of 786 g/mol, closer to but still less than the calculated mass of 858.8 g/mol for $H[Sb(O_2C_6Cl_4)_3]$.

Synthesis of Trisphat Acid and Tributylammonium Trisphat

The entire synthesis was carried out under argon: all apparati were dried and purged 5 times with argon prior to use and all transfers were completed under high argon flow.

A fresh bottle of PCl_5 powder was opened and found to be not free-flowing. A small sample was dissolved in $CDCl_3$ for NMR analysis and found to have a major peak at -80 ppm and a very minor one at 0. ppm and, therefore, was determined to be uncontaminated and usable. 5.93 grams (28.5 mmol) of PCl_5 was transferred to an oven-dried 3-necked 500-mL round bottom flask along with a magnetic stirrer. Two of the necks were closed off with stoppers and the third was fitted with a septum. 24.22 grams (76.2 mmol) of tetrachlorocatechol were transferred to a dry addition funnel (following the procedure described in the synthesis of $H[Sb(O_2C_6Cl_4)_3]$ above), which was capped prior to removal from the glove bag.

A Schlenkware apparatus was assembled as follows using a 3-necked round bottom flask: a condenser topped with a gas inlet tube for argon was fitted to the center neck and a thermometer to the left neck, leaving the neck with the septum on the right. Approximately 75 mL of dry toluene were added through the septum to dissolve the PCl_5. A very small amount of bubbling was observed, indicating that the toluene was not completely dry. The solution was gently heated (50 °C) until a clear yellow solution formed, at which time the very gradual addition of the tetrachlorocatechol was begun. Each small addition of tetrachlorocatechol was accompanied by vigorous bubbling until about ¾ was added, at which point the rate of bubbling slowed. The temperature was maintained at approximately 50 °C until the pH of the outflow gas was greater than 1 (about 24 hours).

The solution was cooled to room temperature, and the Schlenkware apparatus rearranged to connect the condenser to a high vacuum pump via three traps, two consisting of a dry ice/isopropanol slurry, and the third of liquid nitrogen. The other two necks of the flask were closed off with stoppers. The solution was subjected to high vacuum distillation for approximately 30 minutes to remove toluene. This step was followed by 4 hours of high vacuum distillation at 100 °C to remove unreacted tetrachlorocatechol. At the end of the vacuum distillation, the product appeared to be pure white with some evidence of needle-shaped crystals at the top of the flask, indicating that some unreacted tetrachlorocatechol had sublimed.

At this point, while still under an active flow of argon gas, 150 mL of dry dichloromethane plus 156 mL of hexane were added to the white product in the round bottom flask through the septum and allowed to stir for 15 minutes, resulting in the formation of a white-gray suspension. A solution containing 7.1 mL of freshly distilled tributylamine in 60 mL of dichloromethane was added through the septum, causing a pink-gray color to appear in the suspension. After stirring overnight at room temperature, the product, a pure, white, crystalline substance, was separated from the clear, pale, pink-brown liquid by vacuum filtration. The crystals were dried under high vacuum for 4 hours, resulting in a mass of 19.058 grams (20.0 mmol, 82.5% yield). NMR spectra of the crystals were obtained in acetonitrile-d_3. The ^{31}P spectrum gave a single peak at -80.6 ppm and the ^{13}C spectrum yielded these peaks: 141 ppm (doublet), 122.7 ppm, 113.8 ppm (doublet), 54 ppm, 26 ppm, 20 ppm, 14 ppm, and 0.3 ppm. The IR spectrum (KBr) gave the following peaks (all in cm^{-1}): 3172(m), 2962(w), 1442(m), 1389(s), 1234(s), 988(m), 822(s), 721(m), 671(s). This material, presumably tributylammonium trisphat, was used for the synthesis of the M^+ trisphat$^-$ salts as described below.

Synthesis of Cesium Trisphat

A 1.909 gram sample (2.00 mmol) of tributylammonium trisphat was dissolved in approximately 10 mL of acetonitrile to form a violet-colored solution. Upon addition of 0.62 g (2.0 mmol) of a 50% aqueous cesium hydroxide solution and 5 minutes of stirring, a gray-white precipitate formed. The solvent was removed by rotary evaporation at 40 °C and the tributylamine removed under high vacuum (including heating the flask with a heat gun), leaving 1.831 grams of product, 2.03 mmol (101% yield). The product was dissolved in approximately 30 mL of acetonitrile to form a brown solution for recrystallization over cobalt(II) chloride under dynamic house vacuum. When crystals failed to appear after a week, the sample volume was reduced by rotary evaporation, causing the sample to precipitate. The precipitate was redissolved in acetonitrile and placed in a desiccator and subjected to dynamic house vacuum. Uniform, transparent and colorless crystals formed 5 - 7 days later and were removed, dried and stored under argon in a septum-sealed ampoule at -20 °C. NMR spectra of the crystals were obtained in acetone-d_6: The ^{31}P spectrum showed a major peak at -80.6 ppm and two very minor peaks at 0. ppm and -79.7 ppm. The ^{13}C spectrum yielded the following peaks: 141 ppm (doublet), 122.7 ppm, 113.8 ppm (doublet), and 0.3 ppm.

Synthesis of Rubidium Trisphat

A 1.916 gram sample (2.01 mmol) of tributylammonium trisphat was dissolved in approximately 10 mL of acetonitrile forming a violet-colored solution. Upon addition of 0.48 g (2.3 mmol) of a 50% aqueous rubidium hydroxide solution and 5 minutes of stirring, a purple precipitate formed. The solvent was removed by rotary evaporation at 40 °C and the product was heated for 60 minutes under high vacuum (including heating the flask with a heat gun), but tributylamine was not observed to be distilling from the solid. The result was 1.967 grams of product, which is 2.30 mmoles if it is rubidium trisphat (115% yield), or 1.89 mmol (94.2% yield) if it is tributylamine-rubidium trisphat, $(C_4H_9)_3NRb[P(O_2C_6Cl_4)_3]$, as is suggested by the ^{13}C NMR spectrum. The product was dissolved in approximately 10 mL of acetonitrile to form a brown solution for recrystallization over cobalt(II) chloride under dynamic house vacuum. After about 10 days, a "sludgy" solid appeared in the bottom of the flask; this solid was collected and stored under argon at -20 °C as "pre-crop I". The liquid was transferred to a second flask and was again placed over cobalt(II) chloride under dynamic house vacuum. Three days later, uniform, pale brown, transparent crystals were filtered off, dried and stored under argon in a septum-sealed ampoule at -20 °C (crop I). NMR spectra of both samples were obtained in acetone-d_6. Pre-crop I yielded a major peak at 80.6 ppm and a minor peak at 0. ppm in the ^{31}P spectrum and the following peaks for its ^{13}C spectrum: 142.1 ppm (doublet), 122.3 ppm, 113.6 ppm (doublet), 0.3 ppm. Crop I gave the same peaks as pre-crop I plus these ^{13}C peaks: 53.5 ppm, 20.0 ppm, 13.7 ppm. The ^{35}Cl NQR spectrum of crop I yielded the following frequencies (in MHz, with the signal to noise ratio in parentheses): 38.147(2), 37.683(2), 37.622(2), 37.604(2), 37.483(2), 37.381(3), and 37.194(2).

Synthesis of Potassium Trisphat

A 1.926 gram sample (2.02 mmol) of tributylammonium trisphat was dissolved in approximately 10 mL of acetonitrile, forming a violet-colored solution with some precipitate. The precipitate was removed by vacuum filtration prior to the addition of 0.218 g (2 mmol) of a 50% aqueous potassium hydroxide solution and five minutes of stirring, which resulted in the formation of a white precipitate. The solvent was removed by rotary evaporation at 40 °C and the product was heated for 60 minutes under high vacuum (including heating the flask with a heat gun) with no visible sign of tributylamine distillation), leaving 1.805 grams of product. This corresponds to 2.23 mmoles if the product is potassium trisphat (110% yield), but it is 1.82 mmoles (90.1% yield) if it is tributylamine-potassium trisphat, $(C_4H_9)_3NK[P(O_2C_6Cl_4)_3]$, as is suggested by its ^{13}C NMR spectrum. The product was dissolved in approximately 5 mL of acetonitrile to form a brown solution for recrystallization over cobalt(II) chloride under dynamic house vacuum. Two crops of colorless crystals were collected, dried and stored under argon in a septum-sealed ampoule at -20 °C. NMR spectra were obtained in acetone-d_6. The ^{31}P spectra show a single peak at -80.6 ppm for crop I, and a major peak at -80.1 ppm plus additional, minor peaks for crop II (0. ppm and 239 ppm). The ^{13}C spectra show these peaks for both crops I and II: 142.1 ppm (doublet), 122.3 ppm, 113.6 ppm (doublet), 53.3 ppm, 20.0 ppm, 13.7 ppm, and 0.3 ppm.

Synthesis of Sodium Trisphat

A 3.85 gram sample (4.03 mmol) of tributylammonium trisphat was dissolved in approximately 20 mL of acetonitrile forming a violet-colored solution with some precipitate. The precipitate was removed by vacuum filtration prior to the addition of 0.324 g (4 mmol) of a 50% aqueous sodium hydroxide solution and five minutes of stirring, which resulted in the formation of a pink-brown precipitate. The solvent was removed by rotary evaporation at 40°C, and the product was heated for 60 minutes under high vacuum (including heating the flask with a heat gun) with no visible evidence of tributylamine distillation), leaving 3.886 grams of product (now blue-green). This mass corresponds to 4.91 mmoles if it is sodium trisphat (123% yield), but it is 3.98 mmoles (98.7% yield) if it is tributylamine-sodium trisphat, $(C_4H_9)_3NNa[P(O_2C_6Cl_4)_3]$, as is suggested by its ^{13}C NMR spectrum. A 1.80-gram sample of the product was dissolved in approximately 5 mL of acetonitrile for recrystallization over cobalt(II) chloride under dynamic house vacuum. After 24 hours, the blue-green color was replaced by a brown color. Two crops of brown and black crystals were harvested, dried and stored under argon in a septum-sealed ampoule at -20 °C. NMR spectra were obtained for crop I in acetone-d_6. The ^{31}P spectrum gave a single peak at -80.6 ppm. The ^{13}C spectrum gave the following peaks: 142.1 ppm (doublet), 122.2 ppm, 113.6 ppm (doublet), 53.3 ppm, 20.0 ppm, 13.7 ppm, and 0.3 ppm. The ^{35}Cl NQR spectrum of crop I gave the following frequencies (in MHz, with the signal to noise ratio in parentheses): 38.009(2), 37.409(2), and 37.735(2).

Synthesis of Silver(I) Trisphat

Operating under red light only, 1.8 grams of tributylamine-sodium trisphat (2.27 mmol) and 0.39 grams of silver nitrate (2.30 mmol) were placed in a mortar and ground together using a pestle until a dark solid formed. A few drops of deionized water were added to the mixture, forming a gummy solid that did not change with the addition of more deionized water. The product, after vacuum filtration, produced a crystalline substance with a mass of 1.84 grams (2.1 mmol). This mass corresponds to 2.10 mmoles if it is silver trisphat (114% yield), but corresponds to 1.73 mmoles (94.2% yield) if it is tributylamine-silver(I) trisphat, $(C_4H_9)_3NAg[P(O_2C_6Cl_4)_3]$, as is suggested by its ^{13}C NMR spectrum . This sample was stored at -20 °C in a sealed amber glass ampoule under argon. When viewed under the microscope, this sample was found to contain mainly white crystals with trace amounts of a darker crystalline material present. NMR spectra were obtained in acetone-d_6. The ^{31}P spectrum showed a single peak at -80.6 ppm. The ^{13}C spectrum gave the following peaks: 141.6 ppm (doublet), 122.7 ppm, 113.8 ppm (doublet), 53.7 ppm, 27.8 ppm, 20.0 ppm, 13.2 ppm, and 0.3 ppm. The ^{35}Cl NQR analysis yielded no detectable frequencies.

Stability of Trisphat Salts

The samples of the trisphat salts in the NMR tubes were kept for several weeks and their ^{31}P NMR spectra were re-obtained on a second and third date to determine the stability of the salts in acetonitrile-d_3. This can be monitored by the appearance or increase in the relative magnitude of a peak at 0. ppm, which is associated with the trisphat hydrolysis product, a four-coordinate phosphorus compound.[12,7] The samples were stored in the NMR tubes and capped with teflon seals. The tube containing the silver salt was wrapped in aluminum foil for storage. All samples were stored at room temperature. The results are as follows:

- After 31 days, the spectrum of tributylammonium trisphat gave only a single peak at -80.6 ppm.

- After 28 days, the spectrum of the cesium trisphat product showed a number of very small peaks in addition to the major peak at -80.6 ppm, but none of those peaks were at 0. ppm. (Note: The minor peak at 0. ppm that was initially present was no longer evident.)

- After 27 days, the spectrum of tributylamine-rubidium trisphat product showed a number of very small peaks in addition to the major peak at -80.6 ppm, but none of those peaks were at 0. ppm. (Note: The minor peak at 0. ppm that was initially present was no longer evident.)

- After 26 days, the spectrum of tributylamine-potassium trisphat product showed a number of very small peaks in addition to the major peak at -80.6 ppm, but none of those peaks were at 0. ppm. (Note: The peaks at 239 ppm and 0. ppm that were initially present were no longer evident.)

- After 25 days, the spectrum of tributylamine-sodium trisphat product gave only a single peak at -80.6 ppm

- After 21 days, the spectrum of tributylamine-silver(I) trisphat product gave a very small peak (barely above the background) at 0. ppm in addition to the major peak at -80.6 ppm.

CHAPTER 3

RESULTS AND DISCUSSION

Synthesis of $H[Sb(O_2C_6Cl_4)_3]$

$$SbCl_5 + 3\ H_2O_2C_6Cl_4 \rightarrow H[Sb(O_2C_6Cl_4)_3] + 5\ HCl \quad (1)$$

The synthesis of tris(tetrachloro-benzenediolato) antimonic(V) acid or "tristibic" acid (Reaction 1, above) was carried out as a training procedure in preparation for the more sensitive trisphat anion synthesis. In the first attempt, a dichloromethane solution of $SbCl_5$, further diluted in toluene, was reacted with tetrachlorocatechol at 85 °C in an argon atmosphere for approximately 22 hours, during which additional toluene was added periodically to maintain the liquid consistency of the reaction mixture. The product, after removal of toluene under vacuum desiccation, was obtained in an apparent 68% yield. Visual inspection of the product crystals demonstrated the presence of unreacted tetrachlorocatechol, which indicated that the calculated percent yield was anomalously high. The ^{13}C NMR analysis of the product showed minor peaks at 142 ppm, 123.9 ppm and 118.9 ppm, which match those expected for tetrachlorocatechol[12] but also those deduced for tristibic acid[13] so the NMR spectrum was inconclusive. The second attempt followed the same procedure as the first but with the addition of a larger volume of toluene at the beginning of the reaction. This time the product was collected in a 55%

yield by vacuum filtration and dried under vacuum. There was minimal visual evidence of unreacted tetrachlorocatechol.

Preparation of $M[Sb(O_2C_6Cl_4)_3]$

$$H[Sb(O_2C_6Cl_4)_3] + MOH \rightarrow M[Sb(O_2C_6Cl_4)_3] + H_2O \quad (2)$$

The product of the first synthesis of tristibic acid was titrated with ethanolic potassium hydroxide (Reaction-2, above, where M = K) in an effort to determine the equivalent mass of the presumed tristibic acid and to form the potassium tristibate salt. The presence of unreacted tetrachlorocatechol, which turns red upon oxidation, interfered with viewing the phenolphthalein end point during the first attempt. A second attempt, carried out under argon, yielded an equivalent mass of 474 g/mol, far lower than the calculated 858.8 g/mol, most likely due to the presence of much lower equivalent weight tetrachlorocatechol. The product of the second synthesis of tristibic acid was titrated with ethanolic cesium hydroxide under argon (Reaction 2, above, where M = Cs) and yielded an equivalent mass of 786 g/mol, much closer to the calculated mass, suggesting only minor contamination of the tristibic acid.

Synthesis of Tributylammonium Trisphat

$$PCl_5 + 3H_2O_2C_6Cl_4 \rightarrow H[P(O_2C_6Cl)_3] + 5HCl \quad (3)$$

$$Bu_3N + H[P(O_2C_6Cl_4)_3] \rightarrow Bu_3NH[P(O_2C_6Cl_4)_3] \quad (4)$$

Due to the instability of the trisphat acid, the synthesis of the acid was combined with the immediate conversion of the acid to the tributylammonium salt. All steps were carried out in an argon atmosphere. There was some concern as to the purity of the PCl_5 used in Reaction-3, but the ^{31}P NMR spectrum verified that it had not hydrolyzed to phosphonic acid, which would be indicated by a ^{31}P NMR peak near 0. ppm.[7] Solid PCl_5 was slowly dissolved in anhydrous toluene with gentle heating followed by gradual addition of tetrachlorocatechol and gentle heating for approximately 24 hours. After removal of the toluene by room temperature vacuum distillation and removal of any unreacted tetrachlorocatechol by high temperature vacuum distillation, the dichloromethane and hexane solvents for Reaction 4 were added. After addition of redistilled tributylamine and ~12 hours of stirring at room temperature, the product was collected by vacuum filtration and dried under high vacuum to give the tributylammonium trisphat product in a 82% yield (Table 1), which was considerably higher than the 60% yield reported by Lacour.[12] The ^{31}P and ^{13}C NMR spectra and IR spectra all confirmed that the product was tributylammonium trisphat (Tables 2 and 3).

Synthesis of Trisphat Salts

In order to determine the extent of the coordinating ability of the trisphat anion, various salts of the trisphat ion were synthesized. A summary of the yields of the various syntheses appears in Table 1.

Synthesis of Cesium Trisphat

$$Bu_3NH[P(O_2C_6Cl_4)_3] + CsOH \rightarrow Cs[P(O_2C_6Cl_4)_3] + Bu_3N + H_2O \qquad (5)$$

Tributylammonium trisphat was dissolved in acetonitrile and reacted with an aqueous solution of cesium hydroxide (Reaction 5). The solvent was removed by rotary evaporation and the tributylamine under high vacuum. During the high vacuum step,

Table 25. Summary of Yield Data for Trisphat Salt Syntheses

		Reactant		Yield		
Equation	Product	g	mmol	g	mmol	% yield
3, 4	Tributylammonium trisphat[1]	24.22	76.2	19.058	20.0	82.5
5	Cesium trisphat	1.909	2.00	1.831	2.03	101
6 (M is Rb)	Tributylamine-rubidium trisphat	1.916	2.01	1.967	1.89	94.2
6 (M is K)	Tributylamine-potassium trisphat	1.926	2.02	1.805	1.82	90.1
6 (M is Na)	Tributylamine-sodium trisphat	3.850	4.03	3.886	3.98	98.7
7	Tributylamine-silver(I) trisphat	1.800	1.84	1.840	1.73	94.2

[1]Tetrachlorocatechol is the limiting reactant

Table 26. Summary of ^{31}P Nuclear Magnetic Resonance Spectra for Trisphat Salts

	^{31}P NMR Chemical Shifts (ppm)	
Substance	Minor peak(s) (if present)	Major peak(s)
Trisphat acid[26]		-80.6
Bu_3NH trisphat		-80.6
Cs trisphat (Figure 12)	0, -79.7	-80.6
Bu_3N:Rb trisphat	0	-80.6
Bu_3N:K trisphat	239.9, 0	-80.6
Bu_3N:Na trisphat (Figure 14)		-80.6
Bu_3N:Ag trisphat (Figure 16)		-80.6

Table 27. Summary of ^{13}C Nuclear Magnetic Resonance Spectra for Trisphat Salts

Substance	^{13}C NMR Chemical Shifts (ppm)							
	Assigned to Trisphat			Assigned to Tributylamine				Other
Bu_3NH trisphat[26]	142.6	123.6	114.7	54.0	26.2	20.4	13.7	
Tributylamine[26-27]				54.3	30.3	21.0	14.3	
Bu_3NH trisphat	141(d)	123	114(d)	54	26	20	14	
Cs trisphat (Figure 13)	141(d)	122.7	113.8					0.3
Bu_3N:Rb trisphat	141.6	122.7	114	53.7	29.4	20.4	13.4	0.3
Bu_3N:K trisphat	142.1(d)	122.3	113.6(d)	53.3	28	20	13.3	0.3
Bu_3N:Na trisphat (Figure 15)	142.1(d)	122.7	113.9	53.5	29	20	13.4	0.3
Bu_3N:Ag trisphat	141.6	122.7	113.8	53.7	27.8	20	13.2	0.3

(d) = doublet

droplets of tributylamine that condensed near the top of the round-bottom flask were clearly visible and were vaporized by the use of a heat gun.

The very high yield (~100%) was surprising and suggested that perhaps the tributylamine had not been entirely removed. However, after recrystallization from acetonitrile, the ^{31}P NMR showed the chemical shift associated with trisphat (Figure 12, Table 2) and the ^{13}C NMR spectral data included no peaks associated with tributylamine (Figure 13, Table 3).

Synthesis of Other Group I Cation Trisphat Salts

$$Bu_3NH[P(O_2C_6Cl_4)_3] + MOH \rightarrow Bu_3NM[P(O_2C_6Cl_4)_3] + H_2O \quad (6)$$

Tributylammonium trisphat was reacted separately with solutions of RbOH, KOH and NaOH following the same steps as for CsOH, above (Reaction 6, M = Rb, K or Na). However, no droplets of tributylamine were observed during the high vacuum step at the end of each of the processes and the end results give higher than 100% yield for metal(I) trisphat compounds. Additionally, the ^{31}P NMR data indicated that the trisphat moiety was intact in these salts (Figure 14, Table 2) while the ^{13}C NMR data for all three metallate trisphat salts suggested that tributylamine was part of these compounds (Figure 15, Table 3). Recalculations of the yields based on the assumption of a single equivalent of tributylamine being coordinated to the metal ion in each of these compounds resulted in still very high yields (90-99%) for the presumed tributylamine-metal trisphat salts: tributylamine-rubidium trisphat, tributylamine potassium trisphat, and tributylamine-sodium trisphat (Table 1).

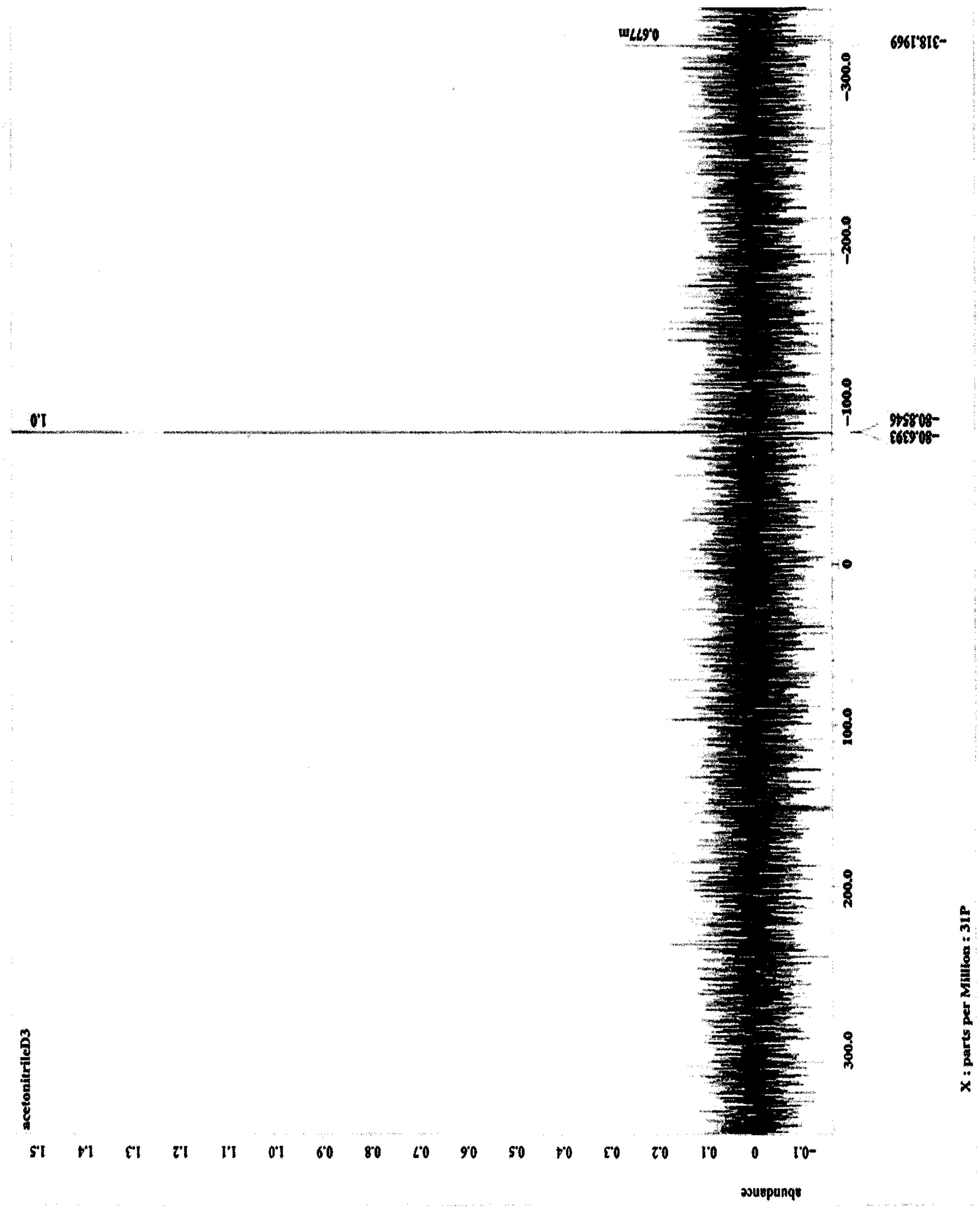

Figure 26. ^{31}P NMR Spectrum of cesium trisphat.

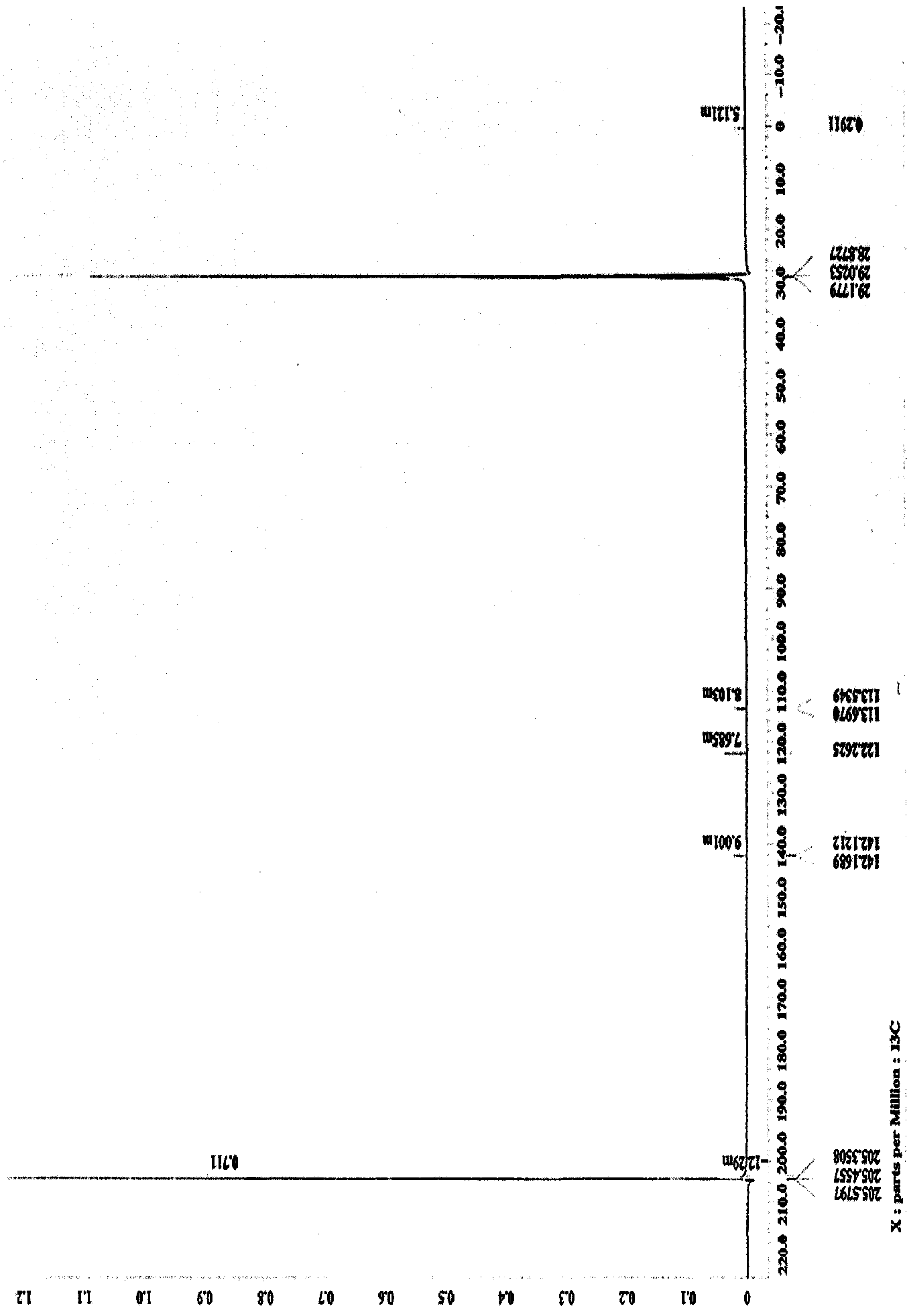

Figure 27. ^{13}C NMR Spectrum of cesium trisphat in acetone-d^6 solvent.

Figure 28. ${}^{31}P$ NMR Spectrum of tributylamine-sodium trisphat.

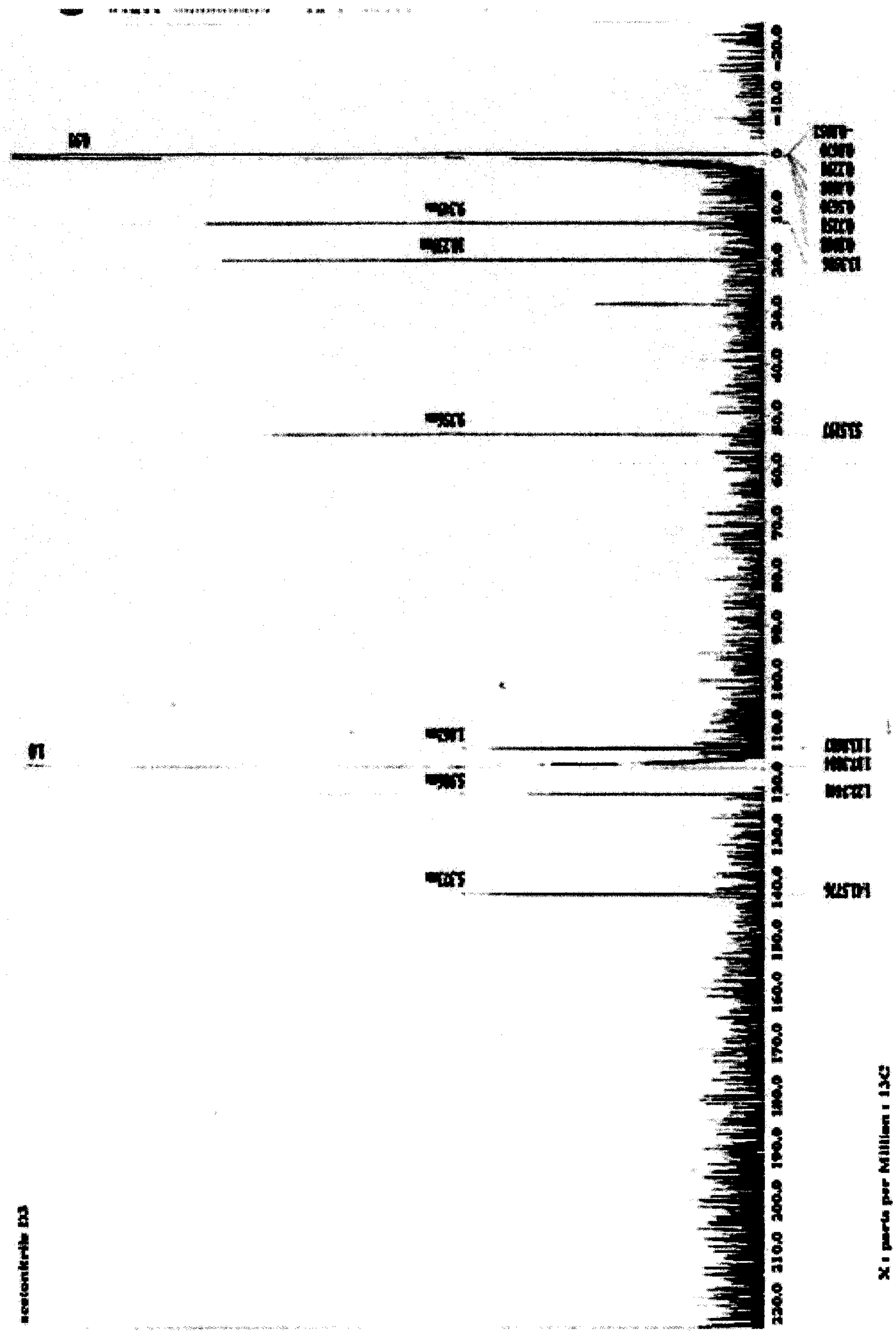

Figure 29. ^{13}C NMR Spectrum of tributylamine-sodium trisphat in $CDCl_3$ solvent.

Synthesis of Silver(I) Trisphat

$$Bu_3NNa[P(O_2C_6Cl_4)_3] + AgNO_3 \rightarrow Bu_3NAg[P(O_2C_6Cl_4)_3] + NaNO_3 \qquad (7)$$

Tributylamino-sodium trisphat was reacted with silver nitrate by grinding the two solids together under red light (to guard against the photosensitivity of the silver salt) followed by vacuum filtration. The resulting solid represented a 114% yield if it was assumed to be silver(I) trisphat. However the ^{13}C NMR data for this product suggested that tributylamine was part of the compound, while ^{31}P NMR data indicates that trisphat was present (Tables 2 and 3, Figure 16). A recalculation based on these data gave 94% yield, assuming the product was tributylamine-silver(I) trisphat.

Nuclear Quadrupole Resonance Spectra

The ^{35}Cl NQR spectral analyses were performed on those samples for which there were no available data, the sodium, rubidium and silver trisphat salts. The NQR signals for sodium and rubidium were quite weak with a signal to noise ratio of only two, while no NQR signal was observed for the silver trisphat salt. The ^{35}Cl NQR frequencies for the sodium and rubidium trisphat samples are listed in Table 4 along with those of other trisphat salts that had been determined previously[28].

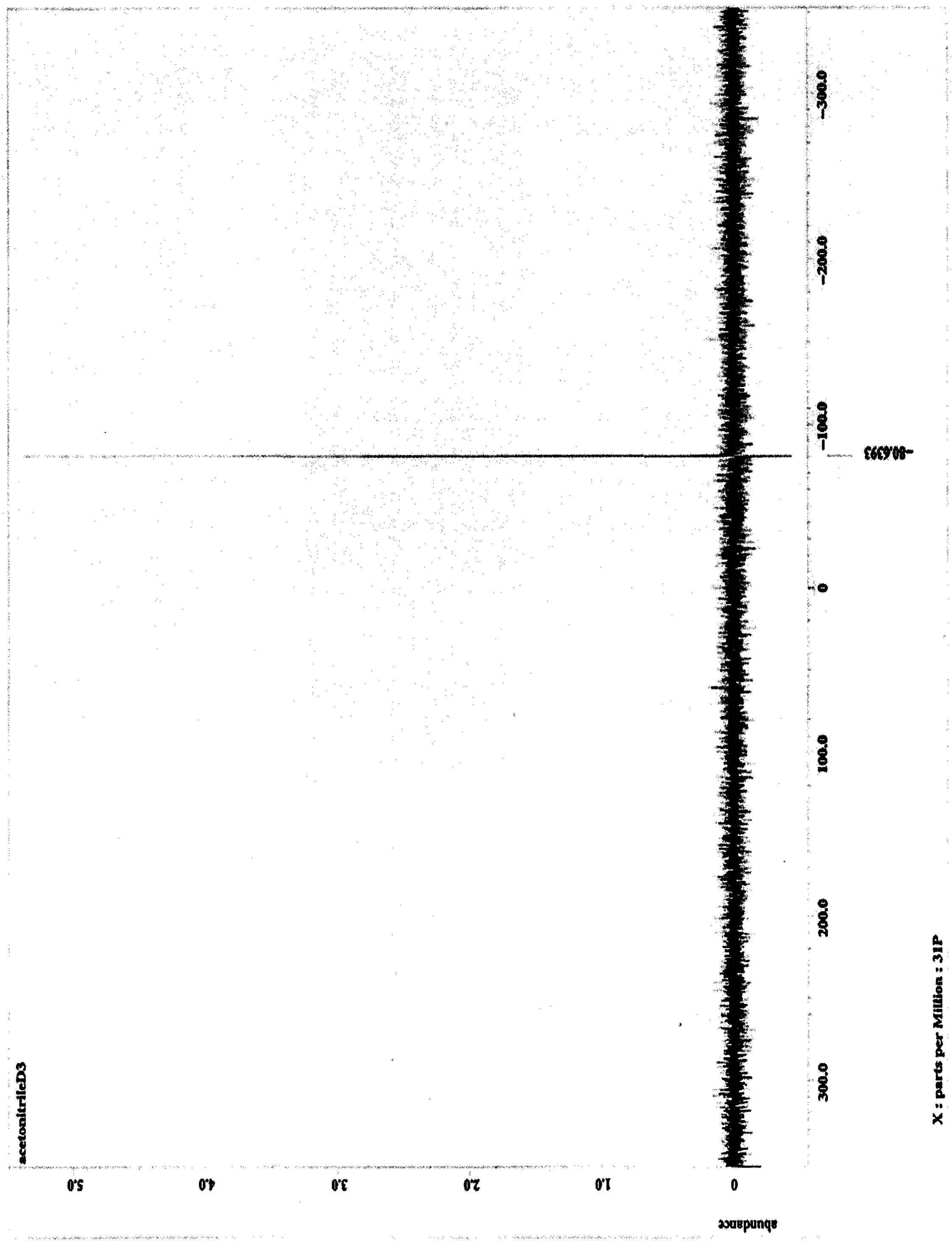

Figure 30. ^{31}P NMR Spectrum of tributylamine-silver(I) trisphat .

Table 28. ^{35}Cl Nuclear Quadrupole Resonance Frequencies of Trisphat Anion in Salts

Cation	Frequency (MHz) (S/N)[1]		Average (MHz)
$CH_3CN:Cs^+$	37.613(3)	37.539(5)	37.292
	37.318(3)	36.700(3)	
$CH_3CN:Rb^+$	37.713(3)	37.653(4)	37.555
	37.558(2)	37.297(2.5)	
$Bu_3N:Rb^+$	38.147(2)	37.683(2)	37.588
	37.622(2)	37.604(2)	
	37.483(2)	37.381(3)	
	37.194(2)		
$CH_3CN:K^+$	37.700(2)	37.643(2)	37.521
	37.554(1.5)	37.189(1.5)	
$Bu_3N:Na^+$	38.009(2)	37.735(2)	37.718
	37.409(2)		

[1]S/N is the signal to noise ratio

CHAPTER 4

CONCLUSIONS

Syntheses of Trisphat Salts

All the syntheses were successful and had good yields. The synthesis of tributylammonium trisphat incorporated the changes suggested by Marella[7] (freshly-opened PCl_5 and freshly-sublimed tetrachlorocatechol), and resulted in a greater than 80% yield, which was considerably higher than that reported previously.[12] The conversions of tributylammonium trisphat to the various M^+ trisphat salts had very high yields (Table 1). All syntheses resulted in products that, based upon the ^{31}P NMR spectra (Table 2), appeared to contain six-coordinate phosphorus atoms with minimal evidence of hydrolysis (or solvolysis) products. Based on observations made during the syntheses and the ^{13}C NMR data, all the metal trisphat salts, except for cesium trisphat, included tributylamine in the product, presumably coordinated to the M^+ ion. In fact, if the tributylamine was not included as part of these products, the data would suggest yields greater than 100%, which is impossible.

Trisphat as a Weakly Coordinating Anion

In Figure 15, the average ^{35}Cl NQR values were plotted versus cationic radius along with the NQR data for the various chloroacetate and chloromethanesulfonate

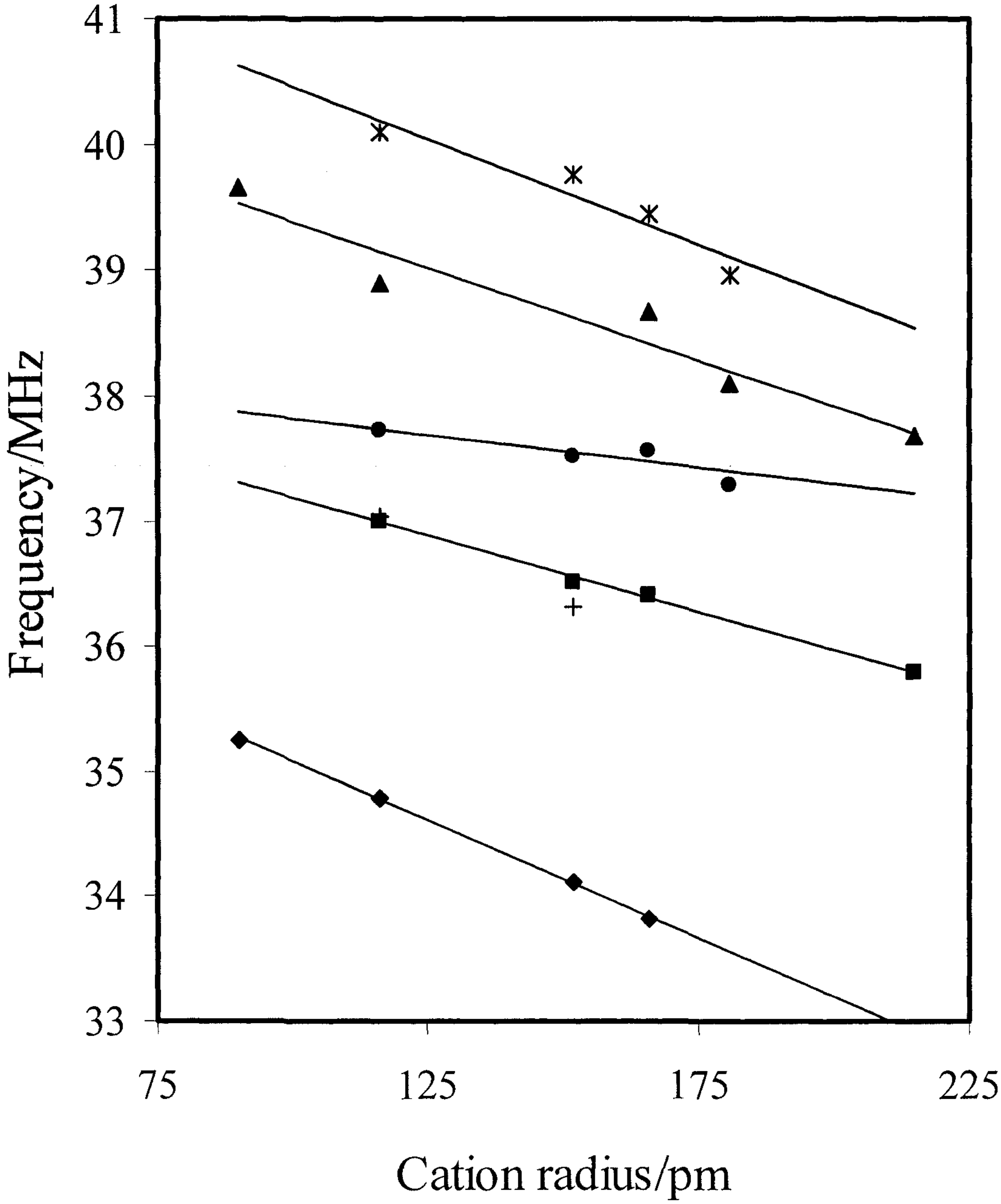

Figure 31. Average ^{35}Cl NQR frequencies versus cation radius, $Cl_3CSO_3^-$ (✱), $Cl_3CCO_2^-$ (▲), trisphat$^-$ (●), $Cl_2CHCO_2^-$ (■), $ClCH_2SO_3^-$ (+), $ClCH_2CO_2^-$ (◆).[11]

(trichlates) anions previously discussed in the Introduction.[11] The slope of the line for the trisphat ion is the shallowest of all the anions included in the figure.

The ^{35}Cl NQR data for the trisphat salts indicated an extremely narrow spectral width of 0.426 MHz; a value narrower even than the 0.8 MHz range typically observed for chlorine atoms in covalent compounds such as CCl_4.[8] This very narrow spectral range suggests that the chlorine atoms in the trisphat anion were coordinating only very weakly to the various cations. Examination of the plot of average ^{35}Cl NQR frequencies versus the radius of the cation (Figure 17) shows that the trisphat anion had the shallowest slope (and thus the narrowest spectral width) of all the anions tested thus far. These data support the assertion that the trisphat anion has the potential to be an important weakly coordinating anion.

The lack of a detectable NQR frequency for tributylamine-silver(I) trisphat was disappointing as, without it, it was not possible to test the hypothesis that the chlorine atoms of the trisphat anion, since borderline bases, are more likely to coordinate to a soft acid cation such as Ag(I). Having this information could confirm or refute the notion that the coordination of the trisphat anion is proceeding according to the hard-soft acid-base principles and would enable more general predictions of its behavior.

Recommendations for Future Studies

It is important to realize that the ^{35}Cl NQR data alone provides insufficient evidence to prove that the trisphat anion is an unusually weakly coordinating anion. Data from X-ray crystallographic studies are needed to support this assertion; the previous attempt to obtain the structure was incomplete and indicate a very complex structure.[7] An important next step would be to determine the crystal structure and examine the metal-oxygen and metal-chlorine bond distances to determine if these distances are consistent with weak coordination.

The product of the tributylammonium trisphat synthesis is a racemic mixture; it is possible that the presence of the two enantiomers contributes to the complexity of the crystal structure. Perhaps resolving the enantiomers prior to synthesis of the trisphat salts would help resolve the crystal structure issue.

Finally, and perhaps most importantly, the trisphat anion's ability to act as a weakly coordinating anion should be evaluated by using it as a co-catalyst for olefin polymerization or in other potential uses involving weakly coordinating anions, continuing the work recently completed by Lee,[25] who showed that the trisphat anion was capable of supporting zirconocene catalysis of the polymerization of carbodiimide.

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Appendix

INTRODUCTORY GENERAL CHEMISTRY 111

Pre-TEST

This test is to survey your current knowledge. Please do your best. You are not expected to know all of this material at this time.

Please DO NOT WRITE on this test booklet

Read all answers carefully *then* mark your answer on the answer sheet.

Multiple Choice

1. What type of reaction does the following equation represent?

   $BaCl_2 + K_2SO_4 \longrightarrow 2\,KCl + BaSO_4$

   A. double replacement B. decomposition C. single replacement D. combination

2. Which substance is ionic?

   A. NF_3 B. CF_4 C. K_2S D. OF_2

3. Solve this equation for T: PV=nRT

   A. V=nRT/P B. T=PVnR C. T=nR/PV D. T = PV/nR

4. Give the formula for tetraphosphorus trisulfide

   A. PO_4S_3 B. P_4S_2 C. K_4S_3 D. P_4S_3

5. What two factors would be expected to DECREASE the rate of a chemical reaction?

   A. decrease in temperature, decrease in reactant concentration
   B. decrease in particle size, use of a catalyst
   C. decrease in temperature, use of a catalyst
   D. decrease in particle size, increase in surface area

6. Calculate the pH of a solution that is 4.3×10^{-4} M in H_3O^+.

   A. 2.53 B. 3.37 C. 3.68 D. 2.47

7. The surface tension of water results from:

   A. hydrogen bonding B. covalent bonds between water molecules
   C. ionic bonds between water molecules D. nonpolar covalent bonds in water molecules

8. Which of the following is a physical change?

   A. the rusting of iron
   B. the reaction of zinc with hydrochloric acid
   C. evaporation of water
   D. the inability of gold to react with water
   E. the burning of paper

9. What weight of NH_4Br crystals would be required to make 400 g of 15% solution?

   A. 120 g B. 54 g C. 6.0×10^3 g D. 60 g

10. An atom has a nucleus containing 16 protons and 16 neutrons. The atomic number, mass number and symbol of this element are, respectively:

   A. 32, 16, S B. 16, 16, S C. 32, 72, Ge D. 16, 32, S

Fall 2004

COURSE SYLLABUS
INTRODUCTORY GENERAL CHEMISTRY 1010

I. **INSTRUCTOR:** Dr. Linda A. Wilson **OFFICE:** DSB 219

OFFICE HOURS: **1:15-3:00 TTH, 3-4 W** **PHONE:** 898-2489
(And BY APPOINTMENT) **e-mail:** Lwilson@mtsu.edu (best!!)

II. **COURSE DESCRIPTION:**

Chemistry 1010 is an introductory course in chemistry requiring no previous courses in chemistry. The main topics to be discussed include: matter and measurement, atoms, molecules, elements, periodic table, chemical bonds, chemical reactions, gases, liquids and solids, solutions, reaction rates, equilibria, acids and bases, oxidation and reduction, and radioactivity. *The next chemistry course to be taken depends on the student's major: either Chem 1020 or Chem 1110.*

III. **TEXTS: (required)**

Chemistry for Today: General, Organic and Biological Chemistry By: Seager and Slabaugh. 5th edition.

Lab Manual: *Chem 1010 Laboratory Manual, T. Melton and L. Scheich.*, 3rd edition.

Lab goggles are required. **There will be goggles for sale at the first lab meeting ~$4.75.**

A **calculator (TI-30 XE is recommended)** is necessary for homework, tests and labs (with log function and exponential notation). Wal-Mart, K-Mart or the bookstore sells fairly inexpensive ones. ******No graphing or programmable calculators, PDA's or cell phones will be permitted for use during tests (If you have one of these, obtain an inexpensive scientific calculator for use on exams or do the calculations manually).**

***You will need to purchase **6 of the scan sheets (#4521**, full page size) for computer grading of your tests.

IV. **TIPS: See attached "How to be Successful in Chemistry"**

a) **Read assignment** before coming to class. Ask questions about topics that you don't fully understand.

b) **Study for this course as we cover each chapter**. USE MY STUDY GUIDES!! If you wait until just before the test to study (such as the day or two before the test), you will find that there is far too much material to study in a short period of time.

c) **Answer the in-chapter questions and the questions and problems at the end of each chapter**. (SEE MY STUDY GUIDES) **Learning Objectives** are listed at the end of each chapter. These point out what you are to learn. Use these to check yourself to see if you are learning by working the appropriate problems and questions at the end of the chapter. The *answers* to the even-numbered questions and problems are at the end of the book in Appendix B and the in chapter Learning Checks in appendix C. There is also a study guide that accompanies the text that has *worked out solutions* to the even-numbered problems. It is available in the bookstore and is on reserve in the Reserves room of the library under Chem 1010/1020.

d) **Take good notes.**

e) If you need help **I will be glad to help**. Come to my office, see me after class or make an appointment. (Please don't come by just before class.)

V. **EXAMS:**

There will be five exams during the semester worth 100 points each. NO MAKE-UP EXAMS WILL BE GIVEN. The highest 4 grades will be counted. If a student misses an exam (for any reason including illnesses, funerals, university functions--I may let you take it *early*) then that will be the grade dropped. There will also be a comprehensive final exam worth 200 points. Turn off and put away all cell phones and planners during the exams (and classes!).

INSTRUCTOR: DR. LINDA A. WILSON PROFESSOR OF CHEMISTRY

INTRODUCTORY GENERAL CHEMISTRY 1010: FALL 2004

Tentative Schedule

AUG	30	M	Introduction, Ch 1
SEP	1	W	Ch 1
	3	F	Ch 1
	6	M	HOLIDAY!!!
	8	W	Ch 1,2
	10	F	Ch 2
	12	Su	****LAST DAY DROP/NO GRADE**
	13	M	Ch 2
	15	W	Ch 2, 3
***	17	F	**EXAM # 1 (Ch 1,2 & math review)**
	20	M	Ch 3
	22	W	Ch 3
	24	F	Ch 3
	27	M	Ch 4
	29	W	Ch 4
OCT	1	F	Ch 4
	4	M	Ch 4
	6	W	Ch 5
***	8	F	**EXAM # 2 (Ch 3,4)**
	11	M	Ch 5
	13	W	Ch 5
	15	F	Ch 5,6
	16-19		FALL BREAK !!!!
	20	W	Ch 6
	22	F	Ch 6
	25	M	Ch 6 ****LAST DAY DROP with W**
	27	W	Ch 6
***	29	F	**EXAM # 3 (Ch 5,6)**
NOV	1	M	Ch 7
	3	W	Ch 7
	5	F	Ch 7
	8	M	Ch 7
	10	W	Ch 8
	12	F	Ch 8
	15	M	Ch 8
	17	W	Ch 9
***	19	F	**EXAM # 4 (Ch 7,8)**
	22	M	Ch 9
	24	W	Ch 9
	25-26		THANKSGIVING HOLIDAY!!!
	29	M	Ch 10
DEC	1	W	Ch 10
	3	F	Ch 10
	6	M	Ch 10
	8	W	EXAM # 5 (Ch 9,10)
	9	Th	Study Day, No Classes!
DEC	13	M	FINAL EXAM--7:00 -9:00 AM COMPREHENSIVE--NOTE TIME !!!

CHEMISTRY 1010 LAB SCHEDULE FALL 2004

Dates	Experiment
Aug 30-Sept 1	Exp 1: Laboratory Safety
Sept 6-8	Labor Day - NO LABS
Sept 13- 15	Exp 2: Density and Significant Figures
Sept 20-22	Exp 3: Paper Chromatography
Sept 27-29	Exp 6: Hydrates
Oct 4-6	Exp 4: Molecular Models
Oct 11-13	Exp 5: Synthesis of Alum
Oct 18-20	Fall Break - NO LABS
Oct 25-27	**Mid-term Exam**[1]
Nov 1-3	Exp 7: Hardware Model: Mole, Limiting Reactant, and Theoretical Yield
Nov 8-10	Exp 8: Production of Hydrogen
Nov 15-17	Exp 10: A Series of Copper Reactions
Nov 22-24	Exp 11: Measurement of pH of Solutions
Nov 29-Dec 1	Exp 12: Titration of Vinegar
Dec 6-8	**Final Exam**[1]

Notes

1. The Mid-Term and Final exams are hands-on. Come prepared to do simple experiments. Bring your safety glasses. The mid-term covers experiments one through six; final covers experiments seven, eight, and ten through twelve.

Summer 2004

COURSE SYLLABUS
INTRODUCTORY GENERAL CHEMISTRY 1010

I. **INSTRUCTOR:** Dr. Linda A. Wilson **OFFICE:** DSB 219

OFFICE HOURS: TBA **PHONE:** 898-2489
(And BY APPOINTMENT) **e-mail:** Lwilson@.mtsu.edu (Best!!)

II. **COURSE DESCRIPTION:**

Chemistry 1010 is an introductory course in chemistry requiring no previous courses in chemistry. The main topics to be discussed include: matter and measurement, atoms, molecules, elements, periodic table, chemical bonds, chemical reactions, gases, liquids and solids, solutions, reaction rates, equilibria, acids and bases, oxidation and reduction, and radioactivity. *The next chemistry course to be taken depends on the student's major: either Chem 1020 or Chem 1110.*

III. **TEXTS:**

Chemistry for Today: General, Organic and Biological Chemistry By: Seager and Slabaugh. 4th edition.

Lab Manual: *Chemistry 1011 Laboratory Manualm 2nd edition* By T. Melton and L. Scheich

Lab goggles are required. **There will be goggles for sale at the first lab meeting ~$4.75.**

A **calculator** is necessary for homework, tests and labs (with log function and exponential notation). Wal-Mart, K-Mart or the bookstore sells fairly inexpensive ones. **No graphing or programmable calculators, PDA's or cell phones will be permitted (If you have one of these, obtain an inexpensive scientific calculator for use on exams or do the calculations manually).**

***You will need to purchase **6 of the scan sheets** (#4521, full page size) for computer grading of your tests.

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a) Read assignment before coming to class. Ask questions about topics that you don't fully understand.

b) Study for this course as we cover each chapter. USE MY STUDY GUIDES!! If you wait until just before the test to study, you will find that there is far too much material to study in a short period of time (such as the day or two before the test).

c) Answer the in-chapter questions and the questions and problems at the end of each chapter. (SEE MY STUDY GUIDES) Learning Objectives are listed at the end of each chapter. These point out what you are to learn. Use these to check yourself to see if you are learning by working the appropriate problems and questions at the end of the chapter. The *answers* to the even-numbered questions and problems are at the end of the book in Appendix B and the in chapter Learning Checks in appendix C. There is also a study guide that accompanies the text that has *worked out solutions* to the even-numbered problems. It is available in the bookstore and is on reserve in the Reserves room of the library under Chem 1010/1020.

d) Take good notes.

e) If you need help **I will be glad to help**. Come to my office, see me after class or make an appointment. (Please don't come by just before class.)

V. **EXAMS:**

There will be five tests during the semester worth 100 points each. NO MAKE-UP TESTS WILL BE GIVEN. The highest 4 grades will be counted. If a student misses a exam (for any reason including illnesses, funerals, university functions--I may let you take it *early*) then that will be the grade dropped. There will also be a comprehensive final exam worth 200 points.

Turn in Exam 8:45
Return 9:05

PROFESSOR: DR. LINDA WILSON

INTRODUCTORY GENERAL CHEMISTRY 1010
SESSION I, SUMMER 2004

Tentative Schedule May 17-June 4

LECTURE 7:30-8:55
BREAK 8:55-9:10
LECTURE 9:10-10:35
BREAK 10:35-10:50
LAB 10:50-1:00 (*lab starting time may vary*)

MAY	17	M	a) INTRODUCTION AND CH 1 b) CH 1 LAB: EXP 1 Lab Safety p 1, EXP 2 Density, p 11
	18	TU	a) CH 2 b) CH 2 LAB: EXP 3 Chromatography, p 21
	19	W	a) CH 2, 3 **(Last day, Drop no grade)** b) CH 3 LAB: Exp 6 Hydrates, p41
	20	TH	a) **EXAM # 1 CH 1, 2, math review** b) CH 3, 4 LAB: EXP 5, Synthesis of Alum p 35
	21	F	a) CH 4 b) CH 4 LAB: Exp 4 Molecular models p 27
	24	M	a) **EXAM # 2 CH 3, 4** **(Last day, Drop with W)** b) CH 5 LAB: Exp 7 Hardware Model p 49 (will not be on mid-term but will be on lab final)
	25	TU	a) CH 5 b) CH 5,6 LAB: **LAB EXAM OVER EXP 1, 2, 3, 4, 5, 6 (Be Prepared to do Experiments!!)**
	26	W	a) CH 6 b) CH 6,7 LAB: EXP 8, Hydrogen, p 65
	27	TH	a) **EXAM # 3 CH 5, 6** b) CH 7 LAB: EXP 10 Copper Reactions p 87
	28	F	a) CH 7, 8 b) CH 8 LAB: EXP 11 pH, p 95
	31	M	HOLIDAY!
JUN	1	TU	a) **EXAM # 4 CH 7, 8** b) CH 9 LAB: EXP 12 Titration, p 103
	2	W	a) CH 9 b) CH 9,10 LAB: **EXAM OVER EXP 7, 8, 10, 11, 12 (BE Prepared to do experiments)**
	3	TH	a) CH 10 b) CH 10 c) CH 10
	4	F	**EXAM # 5 CH 9, 10** (one hour) **FINAL EXAM (COMPREHENSIVE)** (two hours)

HOW TO BE SUCCESSFUL IN CHEMISTRY CLASS

Set aside at least two hours between classes to keep up with your chemistry. Regular, short periods of study are better than a long cram session before the exams. Repetitive study on things to be learned also contributes to understanding. Just like a basketball player must practice often, the good student must also practice often. It might be a good idea to join a study group with other chemistry students. Make flash cards for the things to be memorized, but make an effort to **learn concepts** not just memorize facts.

1. Read and study the chapter before the material is covered in lecture. Read each section separately, make sure you fully understand it before going on. Work example problems from the text and similar problems from the end of the chapter. You will find that later chapters build on information learned in preceding chapters. Learn concepts and how to apply them, just memorizing definitions is not enough! ***Use my study guide.***

2. Have questions in mind on topics that you did not fully understand.

3. Ask questions ***during the lecture*** about topics that were not clear to you. If you didn't fully understand something, likely about half the class has the same problem. Asking questions do not make you look stupid, however ***not*** asking questions is stupid.

4. Take good notes, especially on the topics that you have difficulty with. During the lectures, ***become an active mental participant***. Think along with the instructor. Just being present during the lecture won't help much.

5. After class, take time to expand on the notes taken in class.

6. When problems are worked in class, don't just copy what is written on the board but instead *write down the thinking process* that is used to solve the problem.

7. Answer questions and work problems (between classes) as we proceed through the chapter. It aids understanding. In addition, there is far too much material to learn in the day or two before the exam.

8. Copy example problems (not solutions) from the text or your notes and set them aside. Later, try to see if you can solve the problem.

9. Carefully examine each chapter objective. Choose a problem or question that will test your understanding of the material related to that objective. Don't just go down the list and say "I know that, I know that," etc. Understanding it when I discuss it or work those problems in class is very different from having to generate your own thinking process.

10. The week before the exam begin reviewing your material. If you have kept up as described above, then this will not be too hard.

Problem solving:

a) Read the problem.
b) ***Write down*** what is known and what you are asked to find.
c) ***Write down*** the relationship among these quantities (an equation)
d) Solve the equation for the unknown quantity ***before*** plugging in numerical values.
e) Plug in the known quantities ***including units***.
f) Do math and check to see if the units are correct for the unknown quantity and that the answer is reasonable.

Human Sciences Department
MTSU P.O. Box 86
Murfreesboro, Tennessee 37132
Office: (615) 898-2884
FAX: (615) 898-5130

MIDDLE TENNESSEE STATE UNIVERSITY

13 October 2004
Protocol Title: Evaluation of Student Learning in Alternative Forms of Course Delivery: Intensive vs. Traditional or Online vs. Traditional Chemistry Courses
Protocol Number: 05-045
lwilson@mtsu.edu and hallm@clarkstate.edu

Dear Dr. Wilson and Ms. Hall,

The MTSU Institutional Review Board, or representative of the IRB, has reviewed your research proposal identified above. It has determined that the study poses minimal risk to subjects and qualifies for an expedited review under 45 CFR 46.110 and 21 CFR 56.110.

Please note that any unanticipated harms to subjects or adverse events must be reported to the Office of Sponsored Programs at (615) 898-5005.

Approval is granted for one (1) year from the date of this letter for 775 subjects in the form of educational records.

You will need to submit an end-of-project report to the Office of Research and Sponsored Programs upon completion of your research.

Please note that any change to the protocol must be submitted to the IRB before implementing this change.

Good luck with your research.

Sincerely,

Catherine Stogner

Catherine D. Stogner
Department of Human Sciences
5522

MIDDLE TENNESSEE STATE UNIVERSITY Office of Research and Sponsored Programs

Office of Compliance

Institutional Review Board ("IRB") Progress/Final Report

Instructions for completing this form:

1. Save this file to your computer in order for you to fill out. To avoid losing data, save as you go along as well as when you are finished.

2. Complete the form by either typing or pasting your answers in the appropriate right-hand box. The boxes will expand to fit your answers.

3. Finally, submit the completed form as an e-mail attachment to irb@mtsu.edu or fax it to (615) 898-5028.

What is the status of the project?

_______ Completed; this is the Final Report.

__X_____ Ongoing; this is a progress report and attached is my request for continuing review for IRB approval for an additional 12 months.

A. Reference

Protocol Number:	05-045
Primary Investigator:	Linda Wilson
Project Title:	Evaluation of Student Learning in Alternative Forms of Course Delivery: Intensive vs. Traditional or Online vs. Traditional Chemistry Courses

B. Timing & Completion

Project Start Date	September, 2004
Project Completion Date	August, 2007

C. Unexpected Events

Describe any unusual or unexpected events that occurred which caused deviations from the standard procedures approved in the application.	None
Was the event reported and if so, when?	

D. Distribution of Results

Who received the final research report on findings?	Still in progress
Were findings made available to participants?	_______ Yes ___X___ No
If yes, how were they made available?	
List any definite or tentative publications arising from this project.	The following poster sessions presented tentative finding from this project: *"Do Students Perform as Well in an Intensive Course Format? Linda A. Wilson and Mildred V. Hall, presented at the Annual Scholar's Day, Middle Tennessee State University, Murfreesboro TN Oct. 22, DO STUDENTS PERFORM AS WELL IN AN INTENSIVE COURSE FORMAT? Linda A. Wilson and Mildred V. Hall, presented at the Annual Meeting of the Tennessee Academy of Science, Columbia State Community College, Columbia TN November 19, 2004. Intensive courses in introductory chemistry: Does student performance suffer? Linda Arney Wilson and Mildred V. Hall, presented at the 229th ACS National Meeting, in San Diego, CA, March 13-17, 2005*

E. Storage & Disposition of Primary Data and Identifiers

Describe secure data storage and final disposition arrangements, i.e. disposal, for each type of data medium collected.	Student names were replaced by codes; all educational records stored electronically on password-secured computers.
Who has/had direct access to the participants or their primary data during	Access to data restricted to investigators.

the project? Provide their name, role in the project, and whether data was separated from identifiers at the time of access.	

F. Participant & Research Team Statistics

How many potential participants were contacted?	Educational records of 272 students have been analyzed thus far.
How many participants started the study?	N/A
How many participants completed the study?	N/A
If there is a deviation from the number of participants that started the study to the number of participants that finished the study, please explain why.	N/A
How many researchers, including the primary investigator and all research assistants, were involved in this study?	Two, the principle investigator and DA student, Mildred Hall

PLEASE TYPE

PROTOCOL NUMBER:

SUBMISSION DATE:

MIDDLE TENNESSEE STATE UNIVERSITY
INSTITUTIONAL REVIEW BOARD
HUMAN SUBJECTS RESEARCH REVIEW FORM

Documentation of IRB Training (attach appropriate documentation):
Internet training certificate ☒ Workshop certificate ☐ Registration for workshop ☐

Request for Expedited Review ☒ Full Review☐

Investigator(s) name(s) Dr. Linda Wilson, Mildred V. Hall

Investigator(s) e-mail: lwilson@mtsu.edu, hallm@clarkstate.edu

Investigator(s) address: Department of Chemistry, MTSU Box 409
Arts & Sciences Division, Clark State Community College, 570 E. Leffel Lane, Springfield, OH, 45501-0570

Project Title: Evaluation of Student Learning in Alternative Forms of Course Delivery: Intensive vs. Traditional or Online vs. Traditional Chemistry Courses

Campus telephone: 2489

Campus address: MTSU Box 409

Department or University Unit: Department of Chemistry

Investigator Status (For each investigator)
- ☒Faculty/Staff
- ☒ Graduate Student
- ☐ Undergraduate Student
- ☐ Other

If the principal investigator is a student, list name department and local telephone of faculty supervisor. Please note that THE FACULTY SUPERVISOR MUST INDICATE KNOWLEDGE AND APPROVAL OF THIS PROPOSAL BY SIGNING THIS FORM.

Faculty Supervisor Name Dr. Linda A. Wilson

Faculty Supervisor e-mail: Lwilson@mtsu.edu

Address & Telephone MTSU Box 409 615-898-2489

Social Security # 411-86-72-89

Source of funding for project departmental funds

Expected starting date for project October, 2004

Is this project expected to continue for more than one year? ☒ Yes ☐No
Anticipated Completion Date 8/07

Approval for projects is valid for one year only. Investigators must request a continuation of the approval yearly if the activity lasts more than one year. Only two continuations will be granted for a given project. After three years, the project must be resubmitted.

PROJECT DESCRIPTION

- The following information is required for all projects.
- Limit your answers to the space provided. (Further information may be attached to supplement this description, but not to replace it)
- Attach copies of all questionnaires, testing instruments or interview protocols; include any cover letters or instructions to subject.

DESCRIPTION
Provide a **BRIEF** description, in **LAYMAN'S TERMS**, of the proposed research:

Do students learn chemistry as well in accelerated or online courses as they do in traditional courses? The rise of popularity of these alternative format courses implies a need for this type of evaluation.

We propose to investigate this issue in two parts:

Part I will compare test scores of students in four Introductory General Chemistry 1010 (MTSU) 3-week semesters to test scores of students in four Introductory General Chemistry 1010 15-week semesters. All the courses were taught by Dr. Linda Wilson. ACT and placement test scores will be used to determine the similarity of the two test groups.

Part II will compare test scores of students in 12 traditional sections of Fundamentals of Chemistry 110 (Clark State Community College) with those of students in 12 online sections of Fundamentals of Chemistry 110 (Clark State Community College). All the courses will be taught by Mildred Hall. Placement test scores and grades in Introductory Algebra I (CPE 101) will be used to determine the similarity of the two test groups.

Appropriate statistical analyses of the data from both parts will be carried out to determine what differences in student learning, if any, occur between the traditional courses and the alternative format courses.

METHOD (check all that apply)
☐ QUESTIONNAIRE ☐OBSERVATION ☐ TEST
☐ INTERVIEW ☐FILES ☐TASK
☐ TREATMENT
☒ OTHER Educational Records

NUMBER OF SUBJECTS:

Part 1: 275
Part 2: 500

SUBJECT POPULATION (check all that apply)

☒ ADULT ☐ MINOR
☐ PRISONER ☐ MENTALLY RETARDED
☐ MENTALLY ILL ☐ PHYSICALLY ILL
☐ DISABLED ☐ OTHER
Specify:

SUBJECT SELECTION

Are subjects to be drawn from the Psychology subject pool? ☐ Yes ☒ No

- If yes, a completed sample sign-up sheet must be submitted.
- If no, describe how subjects will be selected for participation in this project and any payment to be received by the subject:

Subjects for Part I are those enrolled in the Introductory General Chemistry 1010 at MTSU taught by Dr. Linda Wilson in the following terms: Fall 00 through Fall 03 and Summer 01 through Summer 04. Subjects for Part II are those enrolled in Fundamentals of Chemistry 110 at Clark State Community College taught by Mildred Hall from Fall 03 through Spring 06. As the project involves analysis of educational records, no payment will be given to the subjects. Approximately 275 students will be subjects for part I. Part II will involve approximately 500 students.

NOTE: If the subjects are to be drawn from an institution or organization (e.g., hospital, social service agency, prison, school, etc.) which has the responsibility for the subjects, then documentation of permission from that institution must be submitted to the Board before final approval can be given.

CONFIDENTIALITY

Specify steps to be taken to guard the anonymity of subjects and/or the confidentiality of their responses. Indicate what personal identifying indicators will be kept on subjects. Specify procedures for storage and ultimate disposal of personal information. Federal guidelines require all study related documents (documentation of informed consent, surveys, study notes, data analysis, and all study-related correspondence) be kept for at least 3 years.

Numerical codes will be substituted for student names prior to the analysis of the data. Dr. Wilson will encode the class data prior to transferring the data to Ms. Hall.

CONSENT

Specify how subjects will be informed of the following: a) the nature of their participation in the project, b) that their participation is voluntary and that they may withdraw at any time without repercussions, and c) that their responses are confidential. (If a consent form is being used, attach a copy. If presented orally, a copy of presentation must be submitted.)

The data to be used are all educational records.

ADDITIONAL PROCEDURAL INFORMATION

INDICATE BELOW WHETHER YOUR PROJECT INVOLVES ANY OF THE FOLLOWING. FOR EACH ITEM CHECKED, PROVIDE THE REQUESTED INFORMATION IN THE ADDITIONAL PROCEDURAL INFORMATION SECTION BEGINNING ON PAGE 5

- ☐ A) Risk (p. 5)
- ☐ B) Minors as subjects (p. 5)
- ☐ C) Psychological intervention (p. 6)
- ☐ D) Deception (p. 6)
- ☐ E) Physiological intervention (p. 7)
- ☐ F) Biomedical procedures (p. 7)

SEE THE PAGE INDICATED FOR A MORE DETAILED DESCRIPTION OF THESE CATEGORIES

SIGNATURES

The **Principal Investigator** must sign this form.

I certify that 1) the information provided for this project is accurate, b) no other procedures will be used in this project, and c) any modifications in this project will be submitted for approval prior to use.

[signature] 9/8/04

Signature of Investigator   Date

If the P.I. is a student, his/her **Faculty Supervisor** must also sign this form.

I certify that this project is under my direct supervision and that I am responsible for insuring that all provisions of approval are complied with by the investigator.

Linda Wilson 9/24/04

Signature of Faculty Supervisor   Date

Committee Use Only

NOTE: APPROVAL OF THIS PROJECT BY THE IRB ONLY SIGNIFIES THAT THE PROCEDURES ADEQUATELY PROTECT THE RIGHTS AND WELFARE OF THE SUBJECTS AND SHOULD NOT BE TAKEN TO INDICATE UNIVERSITY APPROVAL TO CONDUCT THE RESEARCH.

Expedited Review

Approved:________________________________ ____________
College Representative Date

Committee Review

Approved:________________________________ ____________
Committee Chair Date

Application Checklist

Investigator(s): Please **read** and **initial** each item.

Checklist item	Initial
Is all information typed?	[initials]
Is documentation of IRB training attached?	[initials]
Is the investigator email address and other contact information included?	[initials]
If student research, is the faculty advisor email and other contact information incuded?	[initials]
Are surveys, questionnaires, tests, interview forms / scripts attached?	
Is the number of subjects indicated?	[initials]
Is the method of subject selection indicated?	[initials]
If using the Pyschology Department subject pool, is signup sheet attached?	
If a consent form is being used, is a copy of the consent form attached?	
For research involving minors, is an assent form attached?	
For research at outside institutions (e.g., schools), are permission letters on official letterhead attached?	[initials]
Has the application been signed by the investigator (and if student, but faculty advisor)?	[initials]

Incomplete applications may result in delay of research.

Human Participant Protections Education for Research T

Completion Certificate

This is to certify that

Mildred Hall

has completed the **Human Participants Protection Education for Research Teams** online course, sponsored by the National Institutes of Health (NIH), on 08/31/2004.

This course included the following:

- key historical events and current issues that impact guidelines and legislation on human participant protection in research.
- ethical principles and guidelines that should assist in resolving the ethical issues inherent in the conduct of research with human participants.
- the use of key ethical principles and federal regulations to protect human participants at various stages in the research process.
- a description of guidelines for the protection of special populations in research.
- a definition of informed consent and components necessary for a valid consent.
- a description of the role of the IRB in the research process.
- the roles, responsibilities, and interactions of federal agencies, institutions, and researchers in conducting research with human participants.

National Institutes of Health
http://www.nih.gov/

Human Participant Protections Education for Research Teams

Completion Certificate

This is to certify that

Linda Arney Wilson

has completed the **Human Participants Protection Education for Research Teams** online course, sponsored by the National Institutes of Health (NIH), on 11/11/2003.

This course included the following:

- key historical events and current issues that impact guidelines and legislation on human participant protection in research.
- ethical principles and guidelines that should assist in resolving the ethical issues inherent in the conduct of research with human participants.
- the use of key ethical principles and federal regulations to protect human participants at various stages in the research process.
- a description of guidelines for the protection of special populations in research.
- a definition of informed consent and components necessary for a valid consent.
- a description of the role of the IRB in the research process.
- the roles, responsibilities, and interactions of federal agencies, institutions, and researchers in conducting research with human participants.

National Institutes of Health
http://www.nih.gov

Clark State Community College

Office of the Vice President, Academic and Student Affairs

570 East Leffel Lane
Post Office Box 570
Springfield, Ohio
45501-0570

937/328-6025
Fax: 937/328-6142
E-mail: runyana@clarkstate.edu
http://www.clarkstate.edu

September 13, 2004

To: Middle Tennessee State University Institutional Review Board
Subject: Research Review Approval

Dear Sirs,

After reviewing the Human Subjects Research Review Form for the research project titled "Evaluation of Student Learning in Alternative Forms of Course Delivery: Intensive vs. Traditional and Online vs. Traditional Chemistry Courses" by Dr Linda Wilson and Mildred V. Hall, Clark State Community College would agree to allow Mildred V. Hall to conduct the investigation on our campus.

In order to ensure continued compliance with the Family Educational Rights and Privacy Act (FERPA), all data collected from our campus shall have all personally identifying information including but not limited to name, Social Security number, college-assigned ID number, address, and phone number removed from any records used in the study.

We look forward to seeing the results of the investigation.

Thank you.

Sincerely,

Andy Runyan

Andy Runyan, Ph.D.
Vice President
Academic and Student Affairs

cc: Dr. Karen Rafinski, President

From: "Bochmann Manfred Prof (CAP)" <M.Bochmann@uea.ac.uk> — Monday - July 30, 2007 4:03 AM

To: "Midge Hall" <mvh2a@mtsu.edu>

Subject: RE: request

Happy to give permission for you to use this figure.

Professor Manfred Bochmann
Head of School,
School of Chemical Sciences & Pharmacy,
University of East Anglia,
Norwich NR4 7TJ, UK
Tel./Fax (+44) 1603 592044 (direct)
Secretary: (+44) 1603 593143
http://www1.uea.ac.uk/cm/home/schools/sci/cap/people/faculty/mb

-----Original Message-----
From: Midge Hall [mailto:mvh2a@mtsu.edu]
Sent: Monday, July 30, 2007 1:57 AM
To: Bochmann Manfred Prof (CAP)
Cc: hallm@clarkstate.edu
Subject: request

Dear Professor Bochmann,

I am a doctoral student in chemistry at Middle Tennessee State University, working, in part, under the supervision of Professor Gary Wulfsberg. I am currently writing my dissertation, part of which includes a discussion of weakly coordinating anions.
Would you be willing to grant me permission to use a figure from one of your publications? Specifically, I would like permission to copy and use the structures of the diborate and hexahalocarboranyl anions found on p. 3692 of the article, "Isobutene Polymerization and Isobutene-Isoprene Copolymerization Catalyzed by Cationic Zirconocene Hydride Complexes," published in Macromolecules, 2003, 36, 4276-4287.
A positive reply to this email would be sufficient to provide the necessary certificate of permission.

Thank you for your consideration,

Midge Hall
Department of Chemistry
Middle Tennessee State University

From: Tobin Marks <t-marks@northwestern.edu> Monday - July 30, 2007 6:25 PM
To: Midge Hall <mvh2a@mtsu.edu>
Subject: Re: Request

OK with me

At 06:16 PM 7/29/2007 -0500, you wrote:
>Dear Professor Marks,
>
>I am a doctoral student in chemistry at Middle
>Tennessee State University, working, in part,
>under the supervision of Professor Gary
>Wulfsberg. I am currently writing my
>dissertation, part of which includes a
>discussion of weakly coordinating anions.
>
>Would you be willing to grant me permission to
>use a figure from one of your
>publications? Specifically, I would like
>permission to copy and use the structures of
>perfluorinated aryloxy metallate ions found on
>p. 3692 of the article, â€œWeakly Coordinating
>Al-, Nb-, Ta-, Y-, and La-Based
>Perfluoroaryloxymetalate Anions as Co-catalyst
>Components for Single-Site Olefin
>Polymerization,â€ published in Organnometallics, 2002, 21, 3691-3702.
>
>A positive reply to this email would be
>sufficient to provide the necessary certificate of permission.
>
>Thank you for your consideration,
>
>Midge Hall
>Department of Chemistry
>Middle Tennessee State University

From: "Herbert W. Roesky" <hroesky@gwdg.de> Monday - July 30, 2007 4:38 AM

To: Midge Hall <mvh2a@mtsu.edu>

Subject: Re: Request

You are welcome to use that figure.Good luck and best regards.
Herbert Roesky

Midge Hall schrieb:

> Dear Professor Roesky,
>
> I am a doctoral student in chemistry at Middle Tennessee State University, working, in part, under the supervision of Professor Gary Wulfsberg. I am currently writing my dissertation, part of which includes a discussion of weakly coordinating anions.
>
> Would you be willing to grant me permission to use a figure from one of your publications? Specifically, I would like permission to copy and use one of the carbaalanate structures found in in Scheme 1 on p. 5855 of the article, â€œAluminum Hydride Cations Stabilized by Weakly Coordinating Carbaalanates,â€ published in Inorganic Chemistry, 2005, 44, 5854-5857.
>
> A positive reply to this email would be sufficient to provide the necessary certificate of permission.
>
> Thank you for your consideration,
>
> Midge Hall
> Department of Chemistry
> Middle Tennessee State University

Jerome Lacour <Jerome.Lacour@chiorg.unige.ch> Tuesday - July 31, 2007 4:14 AM
To: Midge Hall <mvh2a@mtsu.edu>
Subject: Re:

Dear Mifge,
But of course. No problem whatsoever.
Here are the drawings of the article in ChemDraw format if it can of help.
Good luck with the PhD write-up.
Best
Jerome

Midge Hall wrote:
Dear Professor Lacour,

I am a doctoral student in chemistry at Middle Tennessee State University, working, in part, under the supervision of Professor Gary Wulfsberg. I am currently writing my dissertation, part of which includes a discussion of weakly coordinating anions.

Would you be willing to grant me permission to use a figure from one of your publications? Specifically, I would like permission to copy and use one of the trisphat ion structures found in figure 1 of the article, "Large-Scale Synthesis and Resolution of TRISPHAT Anion," published in J. Org. Chem., 2004, 69, 8521-8525.

A positive reply to this email would be sufficient to provide the necessary certificate of permission.

Thank you for your consideration,

Midge Hall
Department of Chemistry
Middle Tennessee State University