

LSC Use Only

Number: _____

Action: _____

Date: _____

UWUCC Use Only

Number: 91-41

Action: _____

Date: _____

CURRICULUM PROPOSAL COVER SHEET
University-Wide Undergraduate Curriculum Committee

I. Title/Author of Change

CH 412-Advanced Structural and Synthetic Methods
Course/Program Title: _____ in Inorganic Chemistry

Suggested 20 Character Course Title: Adv Strct Inorg Chem

Department: Chemistry

Contact Person: Dr. John Woolcock

II. If a course, is it being Proposed for:

X _____ Course Revision/Approval Only
_____ Course Revision/Approval and Liberal Studies Approval
_____ Liberal Studies Approval Only (course previously has been
approved by the University Senate)

III. Approvals

John C Woolcock _____
Department Curriculum Committee Department Chairperson

Almonesty _____
College Curriculum Committee College Dean *

Director of Liberal Studies
(where applicable)

Provost (where applicable)

*College Dean must consult with Provost before approving curriculum changes. Approval by College Dean indicates that the proposed change is consistent with long range planning documents, that all requests for resources made as part of the proposal can be met, and that the proposal has the support of the university administration.

IV. Timetable

Date Submitted
to LSC: _____

Semester to be
implemented:
Fall 1992

to UWUCC: 9/91

Date to be
published
in Catalog:
Fall 1992

I. CATALOG DESCRIPTION

CH 412 Advanced Structural and Synthetic Methods in Inorganic
Chemistry

3 credits
2 lecture hours
2 lab hours
(2c-2l-3sh)

Prerequisites: CH 410 or CH 322 and CH 411

Advanced techniques used in the synthesis and characterization of inorganic compounds will be explored. In lecture, emphasis will be placed on the theory and application of structural and spectroscopic methods of characterization. In lab, emphasis will be placed on advanced methods of synthesis and structural characterization using representative examples of important classes of inorganic compounds.

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2. SUMMARY OF PROPOSED REVISIONS

We wish to revise CH 412 for two reasons. We want to create a course that better complements CH 410, our new inorganic lab course. We also wish to provide an inorganic laboratory course for graduate students. In the first part of the semester, 2 hours of lecture per week on the theory and interpretation of inorganic structural data will replace the current lectures on descriptive chemistry of the elements. These will be complemented by two hours of laboratory each week in the first half of the semester, focusing on spectroscopic instrument operation and the collection and analysis of structural data as well as advanced synthetic techniques. In the second half of the semester each student will complete an inorganic laboratory project that will combine the structural tools learned in the previous part of the course with advanced synthetic and separation techniques.

3. COURSE SYLLABUS

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II. Course Objectives

- A. Introduce the student to advanced methods used in the preparation and separation of inorganic compounds.
- B. Examine in detail the theory and application of spectroscopic and other structural techniques to the characterization of inorganic compounds.
- C. Require each student to complete an inorganic laboratory project that will combine the structural tools learned in the previous part of the course with advanced synthetic and separation techniques.

III. Course Outline

A. Lecture (25 hours)

1. Introduction to Synthetic and Structural Methods (2 hours)
2. Nuclear Magnetic Resonance Spectroscopy (5 hours)
3. Infrared Spectroscopy (4 hours)
4. Ultraviolet and Visible (Electronic) Spectroscopy (4 hours)
5. Mass Spectroscopy (3 hours)
6. X-ray Methods (2 hours)
7. Inert Atmosphere Synthetic Techniques (5 hours)

B. Laboratory Work (18 hours)

1. Multinuclear (^1H , ^{13}C , ^{31}P , ^{195}Pt) NMR Spectroscopy of Platinum Phosphine Complexes (3 hours)
2. Inert Atmosphere Synthesis and IR Spectroscopy of Organoboron Compounds or Borane Adducts (4 hours)
3. Inert Atmosphere Synthesis and Mass Spectroscopy of Organotin (4 hours)
4. Interpretation of Single Crystal X-ray Data for the Determination of Molecular Structure (2 hours)
5. Electronic Transitions as a Probe of the Structure in Coordination Complexes (3 hours)

C. Inorganic Project (15 hours)

This project may be chosen by the student with the approval of the instructor, from experiments found in the bibliographic references. This project must include both a synthetic and structural component and use as many of the characterization techniques as possible that were described in the first part of the course. Once the laboratory work is completed the student will prepare either a final report on the project or make a poster presentation of the results.

IV. Evaluation Methods

The final grade for the course will be determined as follows:

- 10%-Homework Problem Sets (taken from the text)
- 20%-Final Exam (lecture material only)
- 30%-5 Laboratory Reports (based on the experiments found in part B of the course outline)
- 40%-Laboratory Project Report or Presentation (based on part C of the course outline)

V. Required Texts

1. Ebsworth, E.A.V.; Rankin, D.W.H., Cradock, S. Structural Methods in Inorganic Chemistry, Blackwell Scientific Publications: Boston, 1987.
2. Shriver, D.F.; Drezden, M.A. The Manipulation of Air-Sensitive Compounds, 2nd edition, John Wiley and Sons: New York, 1986.

VI. Special Resources Requirements

1. Each student is expected to purchase a pair of safety goggles for use in the laboratory.
2. Bound "composition" book to be used as a lab notebook.

VII. Bibliography

1. Angelici, R.J. Synthesis and Technique in Inorganic Chemistry, University Science Books: Mill Valley, CA, 1986.
2. Jolly, W.M. The Synthesis and Characterization of Inorganic Compounds, Prentice-Hall: Englewood Cliffs, NJ, 1970.
3. Szafran, Z.; Pike, P.M.; Singh, S. Microscale Inorganic Laboratory, John Wiley and Sons: New York, 1990.
4. Inorganic Syntheses, Volumes 1-25, John Wiley and Sons: New York, 1965-1990.
5. Journal of Chemical Education, American Chemical Society: Washington, D.C.

4. RATIONALE/JUSTIFICATION FOR THE CHANGE

We are currently in the process of revising all our undergraduate inorganic chemistry courses to retain American Chemical Society certification of the B.S. degree. To do this we have created a new, laboratory only inorganic course, CH 410, that will be required of all chemistry majors. It has as its focus the synthesis and characterization of a wide variety of inorganic compounds such that a different compound or group of compounds will be prepared each week. To clearly distinguish CH 410 from CH 412/512 we are revising Inorganic Preparations so that at the undergraduate level it builds on the work done in CH 410 using formal lectures on spectroscopic and synthetic techniques which are not covered in CH 410. We are also providing students with the opportunity to do an independent project. This will require them to bring together all of their knowledge and skills in the area of inorganic chemistry to solve a synthetic/structural problem.

As mentioned above CH 412 is a follow-up course for those undergraduate students that have already taken the Advanced Inorganic Laboratory, CH 410. As CH 512 it will also act as an introduction to inorganic chemistry lab for graduate students who have never had such a course. Since the amount of inorganic laboratory work in the undergraduate curriculum varies to such a large extent both in the US and abroad, it is important for graduate students that a detailed background in structural and synthetic techniques be presented. Since the students who have taken CH 410 also have not had this formalized background, the first part of the course (part III.A. and III.B. of the course outline) will be virtually identical for both graduate and undergraduate students. The major difference between CH 412 and

CH 512 will be in the type of project a student selects in the second half of the semester. We would expect the graduate students to select projects that are more challenging in terms of synthesis, purification and structure of the compounds they wished to study. For this reason we would encourage them to use the more advanced projects found in the bibliographic references above or select a project based on their own research or even one recently published in the chemical literature.

Appendix

- I. *Previous Syllabus for CH 412.*

CH 412 - Inorganic Preparations
 Dr. John Woolcock
 Weyandt 239C, X4828
 Spring 1988

LECTURE/LAB - Tuesday 8:00-12:00

OBJECTIVES: Through a series of lectures and laboratory experiments, we will introduce the techniques used in the synthesis and characterization of a wide variety of inorganic compounds.

REQUIRED TEXTS: Angelici, R.J. Synthesis and Technique in Inorganic Chemistry; University Science Books: Mill Valley, CA; 1986.

Ebsworth, E.A.V.; Rankin, D.W.H.; Craddock, S. Structural Methods in Inorganic Chemistry; Blackwell Scientific Publications; Palo Alto, CA; 1987.

COURSE REQUIREMENTS:

Lab Reports	75%
Homework Sets	20%
Lab Notebook	5%

GRADES: Grades will be based on your percentage out of the total number of points.

90-100	- A
80-89	- B
70-79	- C
60-69	- D
0-59	- F

LAB REPORTS: These are required at the conclusion of each experiment and must include the following:

- Title Page
- Comments on procedure, summary of data
- Characterization and spectroscopic analysis
- Questions
- Appendices (containing all spectra)

REQUIRED EXPERIMENTS

- Synthesis and Characterization of $[\text{Co}(\text{NH}_3)_4\text{CO}_3]\text{NO}_3$ and $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$ (Ref. 1, p. 13)
- Aquation of $[\text{Co}(\text{NH}_3)_5\text{Cl}]^{2+}$ (Ref. 1, p. 26)
- Synthesis of Chromium Acetate (Ref. 2, p. 422 and handout)
- Magnetic Susceptibility (Ref. 1, p. 46)
- Chromatography of Ferrocene Derivatives (Ref. 1, p. 157)
- Synthesis of $[\text{PCl}_4][\text{SbCl}_6]$ and $[\text{PCCl}_5]_3\text{Cl}][\text{SbCl}_6]$ and an Introduction to Inert Atmosphere Techniques (Ref. 1, p. 81)
- Organotin Compounds (Ref. 1, p. 169; Ref. 2 p. 475-480)
- Two-Dimensional NMR Spectroscopy of Palladium and Platinum Diolefin Complexes (Handout)
- "Open Experiment"

There will also be one required experiment that can be chosen by the student, an "Open Experiment", from Reference 1 or other sources. This must be chosen and approved by the instructor by March 29.

NOTE: You must be "checked out" on NR80 AF NMR and Nicolet 20 DXB IR during the first week of classes.

LECTURES: Topics will include introductions to various synthetic techniques, methods of spectroscopic characterization, and the theory and background associated with various types of inorganic compounds. Although no exams or quizzes will be given, the material presented in lecture will directly apply to the required and optional lab experiments. Homework sets from reference 4 will be collected and graded after completion of each chapter or section.

LAB NOTEBOOKS: Each student must keep a lab notebook in which all observations and discussions of compound characterization, etc. are entered. See reference #1 for more details. Lab notebooks will be examined periodically and must be turned in at the end of the semester for grading.

REFERENCES

1. Angelici, R.J. Synthesis and Technique in Inorganic Chemistry; University Science Books: Mill Valley, CA; 1986.
2. Jolly, W.J. The Synthesis and Characterization of Inorganic Compounds; Prentice-Hall: Englewood Cliffs, NJ; 1970.
3. Mayo, D.W.; Pike, R.M.; Butcher, S.S. Microscale Organic Laboratory; Wiley: New York; 1986.
4. Edsworth, E.A.V.; Rankin, D.W.H.; Craddock, S. Structural Methods in Inorganic Chemistry; Blackwell Scientific Publications: Palo Alto, CA; 1967.
5. Drago, R.S. Physical Methods in Chemistry, Saunders: New York; 1977.

GENERAL GRADING SCHEME FOR LAB REPORTSTotal 100 pts.Pts.15 Title Page and Organization

- a) Does each section start on separate page?
- b) Are all tables labeled?
- c) Are references in correct form? ACS Style Guide.
- d) Cluster spectra as an appendix. Organize by compound.
- e) General neatness and readability of discussions.

20 Comments on Procedure

- a) Are literature preps referenced?
- b) Table of reagents present?
- c) Notes on experiment? Are they appropriate?
- d) Table of product weights and % yield, simple phys. measurements (color, density, m.p., etc.)

40 Characterization

- a) Tables of spectral data-appropriate choice of most important features? Literature values (if any)? Properly referenced?
- b) Thorough interpretation of spectra.
(In some experiments data other than IR, NMR, UV-Vis are collected. This section should be used to analyze this data also).
- c) Are spectra of good quality?
- d) All peaks must be explained, including solvent and impurities.

25 Questions

(At times there will not be any or only a few questions. In this case the points for this section will be merged with the characterization section (d).)