Advanced Laboratory III
The University of Toledo
College of Natural Sciences and Mathematics
Department of Chemistry and Biochemistry

CHEM 4880 - 001 (CRN 42029)

Instructor: Slavica Isailovic
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Lab Location: Bowman-Oddy 2089
Lab Day/Time: T, R 8:30 am - 11:20 am
Credit Hours: 2.000
Term: Fall 2018

COURSE/CATALOG DESCRIPTION Laboratory experiments and techniques relating to subjects developed in CHEM 4300. Six hours of laboratory per week. Approved chemical safety goggles meeting the American National Standard Z87.1-1968 must be worn by every student during every laboratory class meeting.

COURSE OVERVIEW Advanced Laboratory III (CHEM 4880), is a class that is the final course in the Advanced Laboratory series. This course is a continuation of the previous two laboratory experiences that will introduce students to new laboratory techniques and chemical instrumentation.

STUDENT LEARNING OUTCOMES Upon completion of this course, the student will be able to use methods and chemical instrumentation present in modern analytical laboratories. Students will learn how to separate, identify and quantify various samples using: gas and liquid chromatography (GC and HPLC), gel electrophoresis (PAGE), size exclusion chromatography (SEC), mass spectrometry (MS), atomic absorption (AA), fluorescence (FL), ultraviolet-visible (UV-VIS) and infra-red (IR) spectroscopy as well as electrochemistry methods (potentiometry, cyclic voltammetry).

PREREQUISITES AND COREQUISITES Prerequisite: CHEM 3860 for level UG with min. grade of D- and CHEM 4300 for level UG with min. grade of D- (may be taken concurrently)

REQUIRED TEXTS AND ANCILLARY MATERIALS
- standard laboratory notebook, duplicate page style
- colored pencils, pens or markers
- approved safety goggles

TECHNOLOGY REQUIREMENTS
- flash data storage drive for exporting instrument data
- access to utoledo.edu BlackBoard
UNIVERSITY POLICIES
Policy Statement on Non-Discrimination on the basis of Disability (ADA)
The University is an equal opportunity educational institution. Please read The University’s Policy Statement on Nondiscrimination on the Basis of Disability Americans with Disability Act Compliance

Academic Accommodations
The University of Toledo is committed to providing equal access to education for all students. If you have a documented disability or you believe you have a disability and would like information regarding academic accommodations/adjustments in this course please contact the Student Disability Services Office.

ACADEMIC POLICIES
There is no make up work for missed class. Excused absences will be handled according to University’s policy (https://www.utoledo.edu/facsenate/missed_class_policy.html) If you have a valid excused absence you will be allowed to participate in the remaining portion of the lab (the techniques are important). Excused absences have 24 hours to contact the instructor or Chemistry Department Main Office by email or phone. Documentation must be provided within seven (7) days of the missed class.

University regulations on academic honesty will be strictly enforced throughout this course (http://www.utoledo.edu/dl/students/dishonesty.html). Violation of these policies can result in a grade of F for the subject laboratory report or even for the entire course. Please note that academic dishonesty in this course includes (but is not limited to) plagiarism of another’s work (website, text, any part of a peer’s lab report, etc), falsification of data, etc.

It is the responsibility of the students to ensure that they are familiar with the university regulations at the websites provided.

Laboratory Safety: Everyone working in the laboratory is responsible for laboratory safety. Common sense will take care of most situations, but if you have a concern or question, please ask the instructor. Anyone who endangers the safety of themselves or others in the laboratory will only be given one warning. A second warning will result in the removal of the student from the laboratory and a grade of zero for the laboratory experiment. In addition, anyone who seriously damages any instrument will also be removed from the lab and receive a grade of zero for that experiment. Each student must wear approved safety goggles in the laboratory. Only one warning will be given; each additional warning will result in a five point deduction from the laboratory grade. No shorts, sandals or open toed shoes will be allowed. These rules are for your protection and no exceptions will be permitted.

COURSE EXPECTATIONS
Lab work: Students will usually work in pairs with each individual recording results and observations independently.

Laboratory Notebooks: Each student is required to keep and maintain a well-organized laboratory notebook. The laboratory notebook is the record of all pre-experimental preparation (prelabs), solution documentation, experimental observations, data calculations, and conclusions. The laboratory notebook should be maintained in such a manner that anyone familiar with scientific methodology can read and understand (i) how the experiment was performed, (ii) what equipment and materials were used, (iii) what results were obtained, and (iv) what was learned from the experiment. Completeness is more important than neatness; however, everything should be legible. The first page should be a running Table of Contents for the entire notebook. Each page should be numbered. All notebook work must be
written in ink. Duplicate sheet style notebooks are required: copy of lab notebook pages is submitted for grading with the lab reports.

**Prelabs:** Prelabs must be written in lab notebook prior to starting the laboratory period. If the prelab material is deemed not adequate the student will not be allowed to start work.

**Lab Reports:** Due at the beginning of the laboratory period **one week** from the date of lab completion unless it is the first or last report of the term or special permission is given by the instructor. Upon satisfactory completion of experiment and clean up of work space **the instructor will initial first page of lab in notebook and mark the date it is due.** Student is checked out for that lab. Reports without instructor’s initials/date due will not be graded. Lab report due but not handed on time because of absence will be accepted for both, excused and unexcused absences. Unexcused will have a 20-point per workday deduction (that is NO points if report is turned one week after due date!) unless turned in by someone else (classmate, roommate, etc) at the time due.

**Laboratory Technique:** In addition to learning instrumental methods and applications, Advanced Laboratory III will continue student’s development and practice of good laboratory techniques. These include awareness and compliance with safety rules, proper handling of reagents, glassware and instrumentation, safe disposal of waste material, and proper lab maintenance, i.e., cleanliness. Every student is required to sign in log book of every instrument used and save all recorded/generated files in individual student folders on instrument’s computer.

**GRADING**
The grade for the course will be determined from percent average of all labs. Every lab is worth 100 points: 50 pts for Lab Notebook and 50 pts for Lab Report.

Typical Grading Scale

(+ grades upper 1/3 of range and – grades lower 1/3 of range)

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<td>D</td>
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LAB NOTEBOOK

Prelabs (30/50 points):

The prelabs will be written in lab notebooks prior to the start of the experiment. If prelabs are not completed, student will not be allowed to start experiment. Prelabs will contain the following information in student’s own words:

I. Title:

II. Lab Partner(s):

III. a) Purpose (5 pts): (no more than 3 sentences)
   
   b) Answers to PreLab Questions (10pts)

IV. Materials (5pts): Include chemical reactivity, spill control, disposal procedures, toxicity, routes of exposure, LD50, any other safety issues.

V. Experimental Procedures (10pts):

   a. Summarize what equipment you will be using (include diagrams of equipment set-ups, make and model of instrument).

   b. Write all calculations for the preparation of solutions, reagents or standards.

   c. Summarize what you will be doing for this experiment. EXAMPLE: Solutions of 0.1875M, 0.375M, 0.75M, 1.5M, and 3M of NaCl will be made up in DI water using volumetric flasks. The weight of the pycnometer will be determined using a Mettler Analytical balance at RT. The pycnometer will be filled with either DI water or one of the NaCl solutions above and then placed in a waterbath at 30°C for 15 minutes. After incubation, the flask will be wiped off and placed at RT for 15 minutes, weighed, rinsed out, and dried.

Labs (20/50 points):

VI. Results (15pts): Should include the following:

   a. Any procedural changes that have been made or any important information given during the laboratory that was not included in the prelab write up.

   b. Important experimental parameters and instrumental conditions when measurements are made.

   c. The actual weights or volumes used.

   d. The actual concentrations for any solution.

   e. Tabulation of all measurements (raw data and complete calculated values).

   f. Presentation of correlations, concentration determinations, or unknown identification.

   g. Preparation of graphs (computer generated graphs are preferred). Figures should be organized, labeled and taped into laboratory notebook.
VII. Conclusion (5 pts):

a. Summarize the data and draw any conclusions that you feel you can make. (Do not make statements like I went blind in one eye and could not see out of the other when reading the thermometer”). Statements like these can invalidate and experiment if this notebook is used to help defend a patent or other legal document).

b. How does your result(s) compare to what was expected?

c. DID YOU SATISFY THE PURPOSE OF THE EXPERIMENT?

LABORATORY REPORTS

Lab Report (50 points):

Student prepares Laboratory reports on experiments. They must be neatly organized and presented. Numerous computers are available on campus for word processing. When lab reports are handed in, they must have the copy pages from the laboratory notebook attached to them. There is no page number requirement for the reports, however all essential elements of the report must be present. In general, these elements should include the following items:

Cover Page: The cover page should include the title of the experiment, your name, the date the experiment was performed, the date the experiment is due, the name of your laboratory partner(s) and grading table/template. Grading table is to have 2 columns, one for the sections of notebook and report with maximal points listed in parentheses and an empty column for the actual earned points to be entered by instructor/TA during grading. A report will not be graded (will receive a score of 0 points) if no cover page.

Abstract (5 pts): This should be a paragraph that summarizes the purpose of the experiment, the specifics of the system studied, and the final results and conclusions drawn from the work. Size should be restricted to 750 words or less.

Introduction (10pts): This section will describe the purpose of the experiment, the chemistry involved, the instrumentation involved (keep in mind: this is instrumental analysis lab where the use of a particular instrument and technique is demonstrated - always addressed it in intro), and the scientific principles upon which the experiment is based. Use of references is expected here.

Materials and Methods (10 pts):

1) A list of reagents used with any necessary safety precautions and chemical disposal procedures

2) A summary of the experimental protocol including any procedural changes that have been made or any important information given during the laboratory that was not included in the original handout.

3) The instrument(s) names, diagrams, schemes and equipment used. Make and model of the instrument is required. Important experimental parameters and instrumental conditions. (Up to 5-point deduction if omitted!)
4) Statistical analysis protocols, equations and formulas, if used. All equations and formulas must be numbered.

Results (10 pts):

1) All data has to be presented in an organized fashion using tables (for numerical data) and instrument generated graphs (for spectra, chromatograms, etc)

Every table and graph has to be numbered, titled and properly labeled (measured values, units, axes, data symbols). There will be a 2-point deduction (up to max of 10 points) for every occurrence of table or graph not numbered, or titled, or labeled.

2) All student generated graphs must have a table associated with it showing the values used to make a graph.

3) Do not discuss what the data means or how it may differ from what is expected. This is for the Discussion section.

Discussion/Conclusions (10pts):

Describe the results and refer to tables and graphs here. EXAMPLE: Resolution of the protein on the column resulted in four peaks being observed when measuring absorbance at 280 nm (Fig. 2, Table 2). Referring to data tables and graphs by their numbers (as presented in Results section) is required. Generally, every data set (table, graph) should get its space in Discussion/Conclusions section of lab report. Not including it will result in points being deduced up to maximal 10 points. The Discussion and Conclusions section should discuss the results, draw conclusions from the data, and discuss any errors that may have affected your results. For example, you must decide whether the intended correlations are correct, whether your spectra make sense, and what the results tell you. When possible you should compare your data with the literature or available data (references). The reader must be able to judge whether you have accomplished what was intended (or understand what you actually ended up with) and asses the quality of your results.

References (5 pts):

All literature references used to write the laboratory report must be listed in this section (in order of appearance) and properly cited in the text. Use American Chemical Society standard reference format. Points will be taken from any section using a particular reference if it is not correctly cited.

Experiments will be usually conducted in pairs and each student will have the same Results. However, all pre-lab preparation and post-measurements/data collection work should be completed independently and represent student’s individual laboratory work and experience. Any duplicated/copied parts of lab reports (except in Results) will result in zero points for all students involved.
COURSE SCHEDULE (tentative, subject to change)

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Laboratory Experiment Abbreviations

PIP - Pipette lab (handout)
BL – Beer’s Law (exp. 6-1)
PH – Potentiometric Methods (exp. 2–1)
BCA – Protein Assay (handout)
FL – Fluorescence Spectroscopy (exp. 10–1)
IR – Infrared Spectroscopy (handout)
ABS – UV–VIS spectroscopy (exp. 6–4)
SEC – Size Exclusion Chromatography (exp. 14–1)
PAGE – Polyacrylamide gel electrophoresis (handout)
GC – Gas Chromatography (exp. 12–3)
MS – Mass Spectrometry (exp. 12–3, handouts)
LC – HPLC (exp. 13–1)
EC – Electrochemistry (exp. 4–1)
AA – Atomic Absorption Spectroscopy (handout)