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Leading an Effective Unit Operations Lab Course

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Leading an Effective Unit Operations Lab Course

Abstract
This paper is focused on the logistics and unique learning opportunities present in supervising a Unit Operations laboratory course. Specifically, the paper outlines some best practices that have been used to effectively manage the course. It is not a broad assessment or review of how different institutions run the lab, but rather highlights the unique features of the Unit Operations Laboratory at the author's institution. The goal of this paper is to provide chemical engineering faculty who will manage laboratory courses with proven best practices that are used in support of the Unit Operations Laboratory.

This paper is geared to faculty who will lead or assist with chemical engineering laboratory sessions. The expected outcome for faculty includes an understanding of the how these learning objectives are realized in the course through a variety of student assessments. Faculty who manage similar laboratories will be able to identify tools and techniques that have proven effective in the Unit Operations Laboratory. This is particularly important as the dual pressures of increasing enrollment and tight budget pressures combine to increase the challenge to running an effective undergraduate laboratory.

Overview of the Laboratory

Unit Operations Laboratory courses are ubiquitous in chemical engineering departments and often represent a rite of passage for upperclassmen. Key goals of the laboratory courses include application of chemical engineering principles in experiments, technical report writing, and pilot scale equipment operation. Ancillary goals include enhancing soft skills with respect to group dynamics and opportunities to improve techniques related to technical presentations. Typically, the unit operations laboratory is taught in the junior and senior years, when most of the core courses have been completed. The theoretical framework for analyzing relevant chemical engineering problems has been established, and students should be able to analyze data obtained in a laboratory setting relative to the theory. Some of the most important aspects of the laboratory experience involve determination of the reasons why the data from an experiment does not match the theory.

The Unit Operations Laboratory offers an opportunity to reduce theory to practice. Students perform experiments ranging from batch distillation to liquid-liquid extraction to fuel cells to fluidization. Most of the equipment in the lab is pilot scale, enabling students the opportunity to learn practical equipment and instrumentation handling skills and laboratory safety best practices. Historically, the laboratory was offered in an intensive summer session, but recently, the laboratory was moved into the traditional school year. Students take the laboratory course for two semesters, for a total of four (4) semester hours over the two semesters (2 x 2). There were several reasons behind the move, including avoidance of conflicts with summer internships, a move from quarters to semesters, and an expansion of undergraduate enrollment. A secondary benefit of conducting the lab during the semesters is that short experimental summaries can be conducted to support other courses. For example, when the Separations course is reviewing the theory of liquid-liquid extraction (LLE), students can witness batch and continuous LLE in the lab.

Specific content in the paper is divided into three parts. First, the paper provides an overview of the Unit Operations Laboratory, including details on how the course is integrated into the curriculum and learning objectives from the laboratory. Second, the session will summarize management of the lab, including course setup and details relevant to a professor running a chemical engineering laboratory course. Third, the session will focus on a single experiment and will give an example of the experimental measurements and linkage to theory.

High level learning objectives from the Unit Operations course include the following:

- Plan efficient laboratory experiments to collect relevant data while minimizing error
- Design and conduct experiments in the laboratory
- Compare experimentally measured results with literature data and quantify the sources of error that contribute to differences between measured data and literature data
- Prepare high quality written reports and oral presentations to summarize a project in a professional and informative manner.
- Practice effective group dynamics to work as a member of a team
- Apply safe laboratory practices important in the chemical industry, including laboratory safety protocols, interpretation of material safety data sheets (MSDS), and proper handling, storage, and disposal of chemicals.

The core component of the unit operations laboratory is the experiments and lab reports drafted by the students. However, there are other aspects of the course that complement the laboratory sessions. There is a once per week lecture that includes a combination of technical and soft skills topics. Technical topics covered in the lecture include overviews of instrumentation (fluid flow measurement, pumps and compressors, temperature and pressure measurement), emergency vents, error analysis, and problem solving with case studies from chemical plants. Lectures on soft skills include overviews of project management terminology and tools, presentation skills, teamwork, and six sigma analysis. The remainder of the lectures are focused on safety, including an overview of the following topics: laboratory safety, occupational exposure to hazardous chemicals, flammable liquids, Material Safety Data Sheets (MSDS), static electricity, compressed gases, cryogenic liquids, electrical safety, and radiation safety.

Experimental Focus and Groups

Since the unit operations laboratory is mandatory for undergraduate students, enrollment in each semester is quite large. To manage the volume of students in the lab at a given time, the class is divided into four sessions, with each session meeting in the lab every four weeks. Groups of four or five are randomly assigned, with several factors used to assign groups. The experiments in the lab are sub-divided into three (3) focus areas: Classical, Environmental, and Biological. For example, the Classical track includes distillation, gas-solid fluidization, shell and tube heat exchanger, plug flow reactor (PFR), and liquid-liquid extraction (LLE). The Environmental track includes absorption, reverse osmosis, hydrogen fuel cell, circulating fluidized bed, and continuous stirred tank reactor (CSTR). The Biological track includes plate heat exchanger, adsorption, mixing dynamics, and gas-solid-liquid fluidization.

A common challenge for any Unit Operations Laboratory is the selection of appropriate chemicals and chemistry for the experiments. The instructor must balance experimental effectiveness and realism with student safety, waste disposal costs, and raw material costs. Several of the experiments utilize acetic acid, either as a starting material (LLE, adsorption) or as a product of a reaction (CSTR and PFR, where acetic anhydride is hydrolyzed to acetic acid). Acetic acid is relatively inexpensive and benign, and storage and disposal costs are negligible. Other experiments utilize compressed air, glass beads, water, and poppy seeds (the three fluidization experiments). The two heat exchanger experiments utilize hot and cold water, with the hot water generated via direct steam injection in the plate heat exchanger and via an electrical heater in the shell and tube heat exchanger.

In the first week of the class, students are given a tour of the lab, and a short summary of each experiment is provided. Students fill out a selection form, with their first and second choices specified. They also note any potential conflicts, either with other students and/or with dates. Based on the focus selection forms, groups are formed for the remainder of the semester. Each group is exposed to six different experiments in the course of a semester. Two of the six experiments are performed in depth, with laboratory sessions from 8am-1pm on Fridays (5 hours in the laboratory for each experiment). The other four experiments are summarized in short overviews, where the groups rotate around the lab, with the teaching assistant (TA) running the equipment and describing the key features and data analysis for the given experiment. For the overview sessions, students spend one (1) hour at each of the four (4) experiments.

Preliminary Assignment

In the week in which a group performs their in-depth experiment, the group meets with the TA several days before the lab session. A group leader is assigned for each laboratory rotation, and the group leader coordinates team members' schedules and the TA availability to schedule the meeting. The TA gives a short summary of relevant theory and outlines the operation of the equipment. Specific safety considerations for the experiment in question are also reviewed. Students are given a pre-lab quiz to assess their knowledge of the experimental theory and the experimental procedure. Students also prepare and submit a preliminary assignment, with the following topics addressed:

- Details on the safety hazards and precautions to protect against them;
- Safety data sheets (SDS) for all materials in the lab;
- Detailed flowsheet of the experiment;
- Completed experimental procedure and data sheets;
- Outline of the Introduction section of the report, detailing relevant theory;
- Identification of the sources of error and plans to minimize and quantify error;
- Experimental plan for conducting the experiment, including the sequence of operations, assignment of responsibilities for group members, and a material and supplies checklist.

Ideally, the Preliminary Assignment should make the lab session more productive and enhance the learning experience for the students during the data collection portion of the lab.

Laboratory Sessions

Lab sessions start on Friday mornings at 8am. Students are assigned hard hats in the first week of the semester, and appropriate Personal Protective Equipment (PPE) is required for entrance to the lab. PPE includes a hard hat, safety glasses, long sleeve shirts, long pants, and closed toed shoes. Each student executes their responsibilities, as outlined in the Preliminary Assignment. Data collection has migrated from written records in laboratory books to automated data collection imported to laptops. A TA is assigned to each experiment and monitors their experiment, answering student questions during the lab, questioning students on relevant theory, and ensuring all safety procedures are followed. At the end of the laboratory session, the TA reviews the data that was collected during the lab and releases the group after the experimental station has been cleaned up and returned to its pre-lab state.

Lab Report

After the lab session, students have ten days to complete a comprehensive laboratory report. Lab reports include the following sections:

- Transmittal letter;
- Abstract;
- Table of Contents;
- Background and Theory;
- Experimental Description;
- Results and Discussion;
- Error Analysis;
- Conclusions;
- Recommendations;
- Design Extension.

The design extension is a supplemental problem given to the students after the lab. The design extension can include scale-up calculations using data from the laboratory session as a basis, process simulations, economic calculations, or derivative problems related to the experimental theory.

With the lab on Fridays, this gives the groups two weekends on which to work on the report. Efficient groups will tackle many of the calculations on the first weekend and will come armed with questions and areas of clarification for the professor and TA early the following week. More commonly, the bulk of the work occurs in Days 7-10, with many late night weekend sessions before the report gets turned in. The lab report is submitted on the second Monday following the lab session. Hard copy, bound reports are required for the submission. After submitting the report, each group member takes a post-lab quiz with questions tailored to the individual role.

The TA is provided with a grading rubric for evaluation of the lab reports. Maintaining consistency from experiment to experiment is a challenge in the lab, but the rubric assists in providing some framework in support of this goal. TA are also provided with reports from previous years so that they can benchmark their grades. The students are provided a general points allocation for the mandatory sections of the report, and the TA are provided with the next level of detail in the points breakdown. Teaching Assistants grade the reports on a scale from 0-100.

Course Grade Components

The overall grade for the unit operations course is a combination of individual and group points. Sixty-four percent of the total points for the course are for an individual's contribution, while the remainder is for the group contribution. The points allocation has been varied over the past decade in an attempt to balance the individual to group ratio, enabling a motivated student to achieve his or her objective grade without undue influence from other group members. The lab experimental grade and final exam grade are both worth 31% of the overall grade for the course. Three quizzes on lecture topics account for 23% of the grade. Other contributions to the overall grade include points for an individual's safety performance, a group safety audit, the EH&S training module completion, and a group presentation.

Example Experimental Detail

There are currently fourteen (14) experiments in the unit operations lab. The scope of this paper does not permit a detailed description of each experiment, but one experiment (gas-solid fluidization) is selected an example of the resources provided the TA and students. This experiment was selected as an example for several reasons. First, the apparatus and instrumental required for this experiment are simple and inexpensive. Departments interested in expanding their experimental repertoire for their laboratory could construct the experiment for less than

$5,000. Second, this experiment covers technology that is directly relevant to many chemical engineering applications, but is often only covered at a high-level, if at all, in undergraduate curriculums. Fluidization technology is commonly used in the engineering field, with examples ranging from pneumatic conveying of solids to freeze drying of fruits to heterogeneous catalytic reactions. Figure 1 shows the various applications of fluidization, with mapping to different topical areas. Third, this experiment is readily scalable. In its current manifestation, it is completed in a five-hour laboratory session, but the experiment could be compressed to accommodate shorter laboratory time frames.

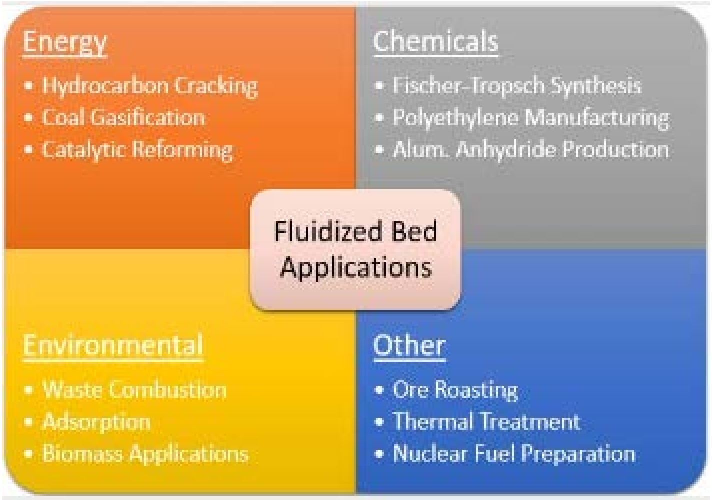

Figure 1. Fluidized Bed Industrial Applications

The basic properties of gas-solid fluidization (GSF) are investigated as part of this experiment. Fluidization occurs when solid particles are fully suspended in a fluid, with the suspension behaving like a dense fluid. If the bed is tilted, the top surface shifts so that it is parallel to the gravity vector. Large objects will sink or float, depending on the ratio of their density to that of the suspension. Fluidized solids can be drained from a bed, similar to a liquid. One of the critical parameters determining the state of the bed is the superficial velocity, which is defined as the volumetric flow rate divided by the cross-sectional area. At low superficial velocities, the drag force on the particle is less than the force of gravity, and the particles act as a packed solid. As the superficial velocity is increased, the drag force balances the gravitational force, resulting in fluidization of the bed. By the end of the experiment, students should develop an understanding of the principles underlying gas-solid fluidization (GSF). This includes, but is not limited to, the following:

- Bed height expansion after minimum fluidization is reached;
- Vapor holdup in the column and how it changes throughout the experiment;
- Pressure increases prior to fluidization;
- Pressure overshoot near minimum fluidization;

- Hysteresis in bed height and pressure readings after fluidization;
- Porosity values as a function of particle diameter and particle shape.

Several different types of solid particles (soda glass beads with average diameters of 300, 600, and 800 microns; mixed beads with diameters ranging from 300-800 microns; and table salt) are tested in the GSF column (Figure 2). It is a simple apparatus that could be built for an investment of several hundred dollars, with acrylic tubes and a silicone gasket near the bottom of the column that joins two separate sections. The volumetric flow rate of air is varied, and the bed height and pressure drop are recorded. Three (3) Dwyer Magnehelic pressure gauges are used to measure the differential pressure at various portions of the column. Multiple rotameters (2x100 SCFH and 1x1000 SCFH) are used to control volumetric flow of air into the column. Figure 3 shows the front of the GSF column.

Figure 2. Gas-Solid (GS) Fluidization Column

Figure 3. Fluidized Flow Control and Differential Pressure Meters

Prior to the experimental session, students are provided several different resources to obtain the required background to plan their experiment and to understand what critical measurements need to be made. This includes technical papers related to fluidization and an Operating Procedure (OP) for the experiment. Students plan the division of labor between group members based on the OP and prepare data log sheets to facilitate data capture during the laboratory session. Prior to the lab, students are expected to recognize all parameters in the Ergun equation, which relates the friction factor to the particle Reynolds number. The relevant theory for fluidization includes expected behavior for two key dependent variables, the pressure drop across the bed and the bed height. Figure 4 shows the theoretical predictions for these two variables. Key transitions for both variables occur at the minimum fluidization velocity.

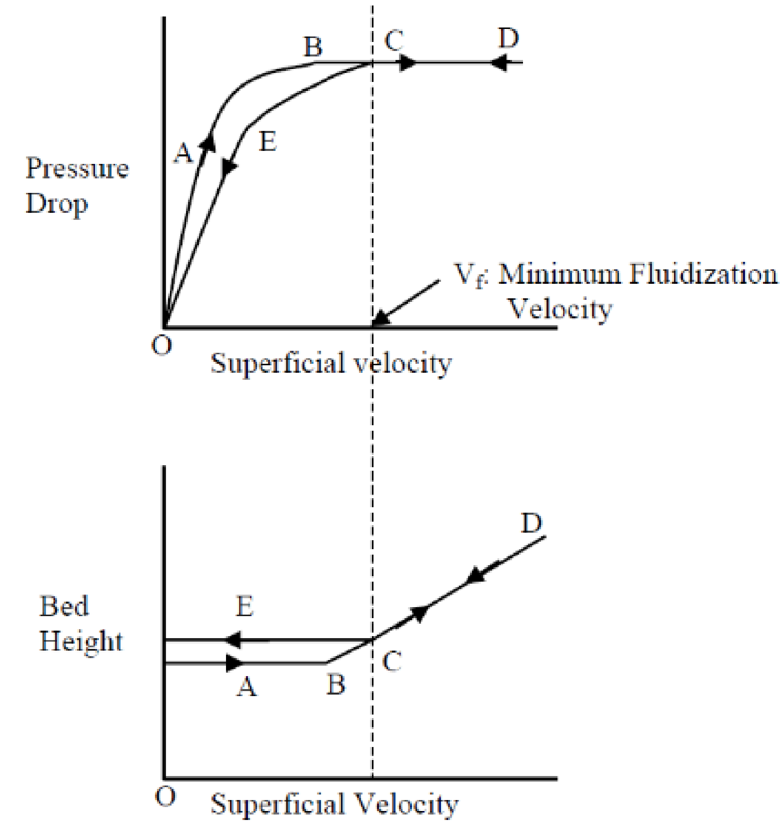

Figure 4. Theoretical Predictions for Pressure Drop and Bed Height in a Fluidized Bed

The actual experiments for the GS experiment are quite simple. Students alter the volumetric flow rate of gas through the column, monitoring the pressure drop at different locations and the bed height. The volumetric flow rate is increased in prescribed increments until the bed is completely fluidized. The flow rates are then decreased and the bed height and pressure drop are monitored as a function of superficial velocity.

In practice, the experimental data is similar to the theoretical predictions, but deviates due to systematic and random error. Comparing theoretical predictions with actual data and clearly explaining the differences is one of the key learning opportunities for students in the Unit Operations Lab. Figures 5 and 6 show the pressure drop and bed height from actual experimental data. These students were able to quantify the error in pressure drop and bed height, using error propagation techniques discussed in the lecture component of the course. Error bars on superficial velocity, pressure drop, and bed height are clearly shown in the figures and are expanded in the discussion of results.

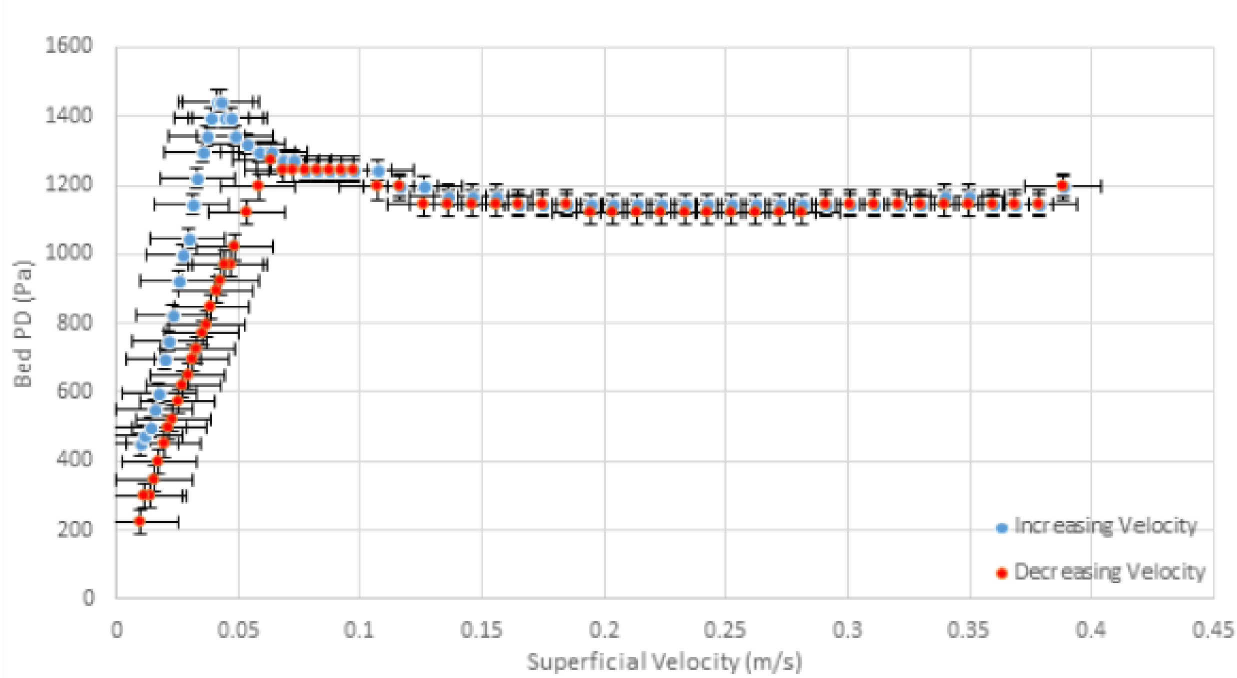

Figure 5. Pressure Drop Across the Fluidized Bed as a Function of Superficial Velocity

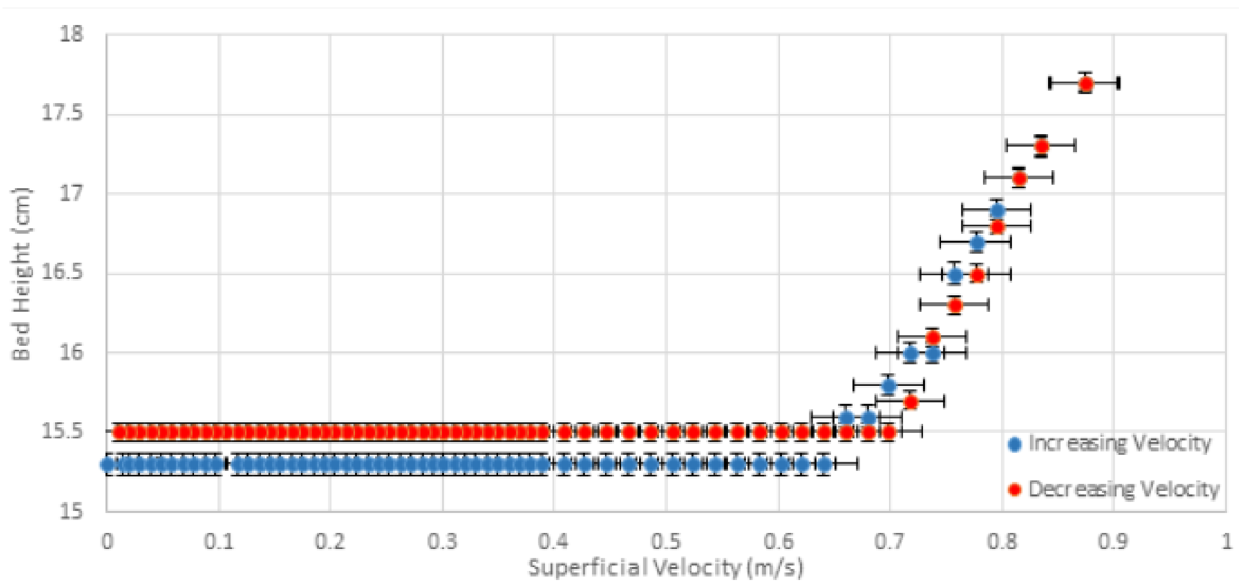

Figure 6. Fluidized Bed Height as a Function of Superficial Velocity

In parallel to the fluidization experiments, porosity of the solid particles is measured in both dry and wet tests. For the dry porosity experiment, a fixed volume of beads is added to a graduated cylinder. With a known bead density, calculation of the porosity is trivial. The experiment is repeated using a wet measurement technique, in which hot water is poured over the beads in a graduated cylinder until the water meniscus is at the top of the beads. The porosity values are measured for the monodisperse beads and for the mixed beads, and the students comment on any differences. As expected, the porosity for the mixed bead samples is lower than for the monodisperse beads. The measured porosities are used to calculate the bed density in the column.

Summary

The unit operations laboratory is the fundamental laboratory course in the undergraduate curriculum. It provides a vital link between theory and practice, allowing students to collect and analyze data from pilot scale equipment. Students learn how to design and conduct experiments in a laboratory, work effectively as a member of a team, and prepare high quality written reports and oral presentations that summarize a project in a professional and informative manner. The course places an emphasis on safety, and students understand how plant safety is integrated in data collection and laboratory experiments. The lab will continue to evolve to better match the evolving needs of industry, incorporating new experiments and expanding and enhancing existing experiments to enrich the undergraduate learning experience.

APPENDIX

UNIT OPERATIONS LABORATORY GRADING RUBRIC

WRITTEN REPORT GRADING FORM

Date Received.:________________________

Group No.:____

Group Members: ____, ______________
____,

Experiment Number: ____Title:_________________________________
____

Lab Session No.: ______________________

1. Title Page, Contents, Notation, References, and Appendices	/4
2. Abstract	/4
3. Purpose	/4
3. Introduction (Theory)	/15
4. Experimental Description	/10
5. Discussion of Results	/20
6. Error Analysis	/15
7. Conclusions	/4
8. Recommendations and Future Work	/4
9. Design Extension	/10
10. Experimental Summary Report	/5
11. Overall Report Quality	/5
CONTENT SUBTOTAL	/100
FORMAT DEDUCTIONS (See page 2)	-
TOTAL	/100

TA's Name: ________________________________

WRITTEN REPORT GRADING FORM, Continued

Format Deductions

Group No.:____ Experiment No.:___________________
Lab Session No.:__________________________

Grading Aspect	*Deduction value*	Deduction
Consistency: All figure labels on bottom; all table labels on top	.5/mistake	
Consistency: All report sections are similarly aligned (left or justified)	1	
Consistency: All section headings are in same format	1	
Consistency: All sections begin in the same location	1	
Consistency: All cited material is referred to similarly and correctly	1	
Page numbering is correct (Purpose is page #1)	1	
Figure and table labels are completely descriptive	1	
No grammar or spelling mistakes in body of report (exclude appendix)	.5/mistake	
Topics discussed in proper sections	1/mistake	
Page limits followed	2/mistake	
Use of professional language and appearance	.5/mistake	
Equations are numbered	1	
All figures and tables are discussed in the text	.5/mistake	
Report is bound in correct manner, in order and complete	2	
TOTAL DEDUCTIONS	14 max	

COMMENTS:

Fall 2016

GROUP LEADER'S REPORT (Confidential)

Today's Date:____

This report is to be turned in to the TA personally the day the report for the experiment is due. All the information in this report must be completed as accurately as possible, failure to do so will reflect on the Group Leader's performance and appraisal.

Group Number:______________________________________

Experiment Number: _________ Title: ____________________________ Lab Session No.:____________

Group Leader's Name: _______________ Teaching Associate's Name:______________________________

____

I. Time Report

Group Members	Time in Laboratory	Time in Calculations	If absent from lab or Computation, tell why
1.			
2.			
3.			

II. Report on Performance in Laboratory, Computations and Report Preparation

Give grades 0 through 4 (0 the lowest, 2 is average, 3 is above average, 4 is only for going above and beyond expectations; excellent).

Member	Team work	Responsibilities performed	Initiative	On-timeliness	Supervision required	Sum: (max=20)	Calculation: (= Sum/10)
1							
2							
3							

Fall 2016

T. A. EVALUATION FORM (Confidential)

Today's Date:____

Group Number:_______________________

Experiment Number: ____________________ Title: _____________________________________
Lab Session No.:_______________________

Group Leader's Name: __________________Teaching Associate's Name:________________

Give Point Scores (See range of score values):

Name of Member	Lab Performance (0-4) @	Quiz (0-4)	Preparation Assignment (0-4) +	Teamwork Assessment (0-4) +	Total (T) (0-16)	Calc: (=T/2)	Deduct for Safety/time violations *

@ A score of 3 is for a student who is fully prepared and always on-task. A score of 4 is for a fully prepared, always on-task person who goes above and beyond their duties to take charge or solve problems: overall exceptional. A score of 2 is for an average student's participation on an experiment: on-task but needs direction a lot of the time and not quite prepared enough. Scores below 2 are for below average performances

* If any, negative points. -1 for each safety violation; -0.3 for each time late < 5 mins. -1 for each time > 5 mins. late

+ Group score- same score for every member