A comprehensive laboratory course is an important component for adequately preparing engineering students for future careers in industry. The University of Delaware (UD) Department of Chemical and Biomolecular Engineering (CBE) has recognized the importance of such a course and has committed significant resources and effort towards continuous improvement. Chemical Engineering Laboratory I, more commonly known as Junior lab or J-lab, is offered to students in the Spring semester of their junior year, and seeks to reinforce ChE fundamentals, technical writing, oral presentation, teamwork, leadership skills, and safety practices. Five experiments focused on chemical kinetics, fluid mechanics, thermodynamics, heat and mass transfer, and engineering instrumentation provide the connection with the theory taught in the core ChE courses. This article provides an overview of the Junior lab taught at UD and a detailed analysis of the recently introduced chemical kinetics experiment.

CHEMICAL ENGINEERING LABORATORY I

The Junior lab provides the first opportunity for undergraduate students to apply the knowledge and skills acquired from coursework to actual chemical engineering experiments. The undergraduate lab provides a comprehensive and well-developed program that can lead to significant academic growth for the students. The UD CBE department dedicates significant resources for the Junior lab. A team of four professors, four graduate teaching assistants (TAs), and two laboratory technicians operate, maintain, and work to continuously improve the laboratory. The laboratory consists of five different experiments: fundamentals of measurement (FOM), heat exchange (HEX), vapor-liquid equilibrium (VLE), fluid mechanics (FLO), and chemical kinetics (KIN). The FOM laboratory introduces students to the operation of basic temperature, pressure, and flow measurement devices. The lab also provides students with background on analog and digital data acquisition and control hardware and an introduction to LabView® computer programming. The FLO experiment features a series of pipes and instrumentation to

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study flow in the laminar, transition, and turbulent regimes. The VLE experiment consists of four ebulliometers for measuring infinite dilution activity coefficients to calculate binary phase behavior.\textsuperscript{[2]} The HEX experiment features tube and tube, shell and tube, and plate and frame heat exchangers for evaluating and comparing heat transfer correlations. The KIN experiment measures the reaction order and activation energy for the iodination or bromination of acetone reaction. A detailed description of the KIN experiment is provided in the next section.

All students start by running the FOM experiment, which is focused on understanding the principles and operation for a variety of temperature, pressure, and flow measuring devices. The instruments are mounted on portable racks that can be moved between the laboratory and classroom. The FOM experiment provides students with a thorough understanding of engineering instrumentation before they need to use it in the four additional experiments (FLO, VLE, HEX, KIN). Students select two out of the four remaining experiments to complete during the rest of the semester. The experiments are based on the core chemical engineering classes that serve as prerequisites for the lab: CHEG341 (Fluid Mechanics), CHEG231 and CHEG325 (Thermodynamics), CHEG342 (Heat and Mass Transfer), and CHEG332 (Chemical Kinetics).

To complete three experiments with approximately 80 to 100 students within a 13-week semester requires a rigorous schedule be followed. The lab is split into three cycles with each cycle lasting approximately four weeks (and the oral/video presentations lasting one week). At the beginning of a cycle, the laboratory groups, typically consisting of four students, each elect a group leader. The group leader is responsible for division of labor, organization of the group, assignment submission, and student-teacher correspondence. A new group leader is elected for each cycle so that all students have the opportunity to lead an experiment or an oral presentation.

Given such a demanding schedule, professors, TAs, and students utilize Sakai\textsuperscript{[3]} for exchanging course information. Sakai provides professors and TAs the ability to post resources, schedule meetings, send out messages, announce assignment deadlines, and provide grades, all of which are archived and easily accessible by the students. In addition, students are able to electronically submit draft and final reports via Sakai to eliminate the need for hard copy submissions. The electronically submitted reports provide users with a confirmation email, which includes the attached files and time of submission. TAs grade draft reports and the professors grade the final reports. Due to the short time frame between experiments, the professors and TAs aim for a two- to three-day turnaround on their comments and grades. This provides students with important feedback before beginning the next experiment. Significant improvement in report writing and lab performance is often seen from the first to third cycle.

During the first week of the semester, faculty and TAs provide lectures on topics such as instrumentation, statistics, error analysis, and how to write an effective engineering report. The first week of each cycle features a prelab lecture given by the assigned professor for the experiment and a prelab tour given by the TA so that students can become familiar with the laboratory equipment. Instructional videos of experiments are also available online for students to watch and see the equipment in operation.\textsuperscript{[4]} A prelab meeting is held with the assigned faculty member and TA for each experiment. The prelab meetings are one to two hours and include a 20-minute presentation by the students to the professor and TA as a demonstration of their preparedness for the upcoming lab. The prelab meetings provide the faculty member time to thoroughly cover the theory for the experiment and any safety concerns for the lab to ensure all students understand the hazards. The prelab meeting also provides the opportunity for the students to ask in-depth questions. The professors assigned to each experiment meet individually with all groups, which is a significant time commitment, but ensures that the students are well prepared for the lab.

During the second week, the students work in the lab as organized groups and each experiment can be completed within the allotted 4-hour time period. The third week provides time for analysis and report writing. The report is due two weeks after the groups’ completion of the in-lab experiments. The average report consists of approximately 20 pages with a supplemental appendix. Students are allowed to submit a rough draft of their report to the TA up to 72 hours before the report deadline so that the TA can provide comments on data analysis and report writing.

The first cycle is identical for all groups, with every group completing the FOM lab. Cycles two and three vary between groups, with each group completing two of the four possible remaining labs. The fourth cycle is a presentation on one of the completed experiments. Each group is given the option of either creating an instructional laboratory video or giving a professional oral presentation. The videos are presented during a video night at the end of the semester, with the best videos receiving awards. The best videos are also used to help instruct the next year’s students on the operation of the equipment and how to successfully complete each experiment.\textsuperscript{[4]} The oral presentation is given in a television studio at UD, in front of the Junior lab professors and TAs. The other student groups are also invited to attend. The presenters have access to an “Oral Communication Fellow,” usually a senior communications major, to assist them in the design and delivery of the presentation. Following a period of questions after the presentation, the students are provided a DVD copy of their presentation, and asked to write a critique of both their speaking skills and overall presentation quality.

In 2014, 92 students were enrolled in the Junior lab, and these students worked in groups to complete three of the five labs over the course of the semester. The students were given
CHEMICAL KINETICS EXPERIMENT

The growing number of students in the UD CBE department required another experiment to be added to the Junior lab course. In 2013, we added a KIN experiment in combination with the FLO, VLE, and HEX experiments. In the KIN lab, the undergraduate students run either the iodination or bromination of acetone reaction. During Spring 2014 the students studied the iodination of acetone. The reaction is shown in Eq. (1) and uses HCl as a catalyst. The following sections will describe in detail the critical elements for the KIN lab.

\[ \text{H}^+ + \text{Ac} + \text{I}_2 \rightarrow \text{Acl} + \text{HI} \]

\[ \text{H}_2\text{O} \]

Apparatus and safety

The kinetics laboratory is equipped with two identical reactors. An example of one of the reactor setups can be seen in Figure 1. The 1-liter glass reactor (fabricated...
The reaction mechanism for the iodination of acetone can be written in three elementary steps. The first step [Eq. (2)] is an equilibrium reaction between acetone (Ac) and the acid (HCl) to form the protonated acetone intermediate (AcH+). The second step [Eq. (3)] is the slow step to form the enol intermediate (Ac*). The fast step [Eq. (4)] between the Ac* and the iodine (or bromine) produces the iodoacetone (or bromoacetone). The rate-determining step is the slow step and the rate equations for each step can be written in order to determine the overall rate expression for the reaction, which is shown in Eq. (5). The reaction is first order in acetone and HCl and zero order in iodine (or bromine).

$$Ac + H_2O \rightleftharpoons_{k_1} AcH^+ + H_2O$$ (equilibrium step) (2)

$$AcH^+ + H_2O \rightarrow_{k_2} Ac^- + H_3O^+$$ (slow step) (3)

$$Ac^- + I_2 \rightarrow_{k_3} AcI + HI$$ (fast step) (4)

$$r = k(T) \cdot [Ac]^{\alpha} \cdot [I_2]^{\beta} \cdot [HCl]^{\gamma}$$ (5)

Students are taught how to use the method of initial rates [Eq. (6)] to solve for the rate constant and orders of reaction:

$$\frac{r_1}{r_2} = \frac{k_2(T) \cdot [Ac]^{\alpha} \cdot [I_2]^{\beta} \cdot [HCl]^{\gamma}}{k_1(T) \cdot [Ac]^{\alpha} \cdot [I_2]^{\beta} \cdot [HCl]^{\gamma}}$$ (6)

where \( r \) is the reaction rate, \( k \) is the rate constant, \([Ac]\), \([I_2]\), and \([HCl]\) are reactant concentrations, and \( \alpha, \beta, \) and \( \gamma \) are the orders of reaction, and the subscripts 1 and 2 denote two individual reactions.

The iodination (and bromination) of acetone reaction is a color change reaction. The water, acetone, and HCl solution are added in order and the mixture is colorless. Once the iodine (or bromine) is added the reaction mixture changes to a yellowish-brown color and the reaction begins. The final product iodoacetone (or bromoacetone) is colorless so a

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**Methodology and data analysis**

The chemicals (HCl, acetone, and iodine-water solution) are weighed on analytical balances inside a chemical fume hood. Several graduated cylinders (30, 50, 100, 1000 mL) are available for students to weigh acetone and water, and 10 mL plastic syringes (BD Luer-Lok™) with detachable needles (18 gauge, BD PrecisionGlide) are used for volumetric measurement of HCl and iodine water solution. The graduated cylinders and syringes are all weighed on mass balances (OHAUS Scout Pro SPE2001) to obtain the most accurate weights.

During the prelab meeting, the faculty members assigned to the experiment ask the students a series of safety questions to make sure they are aware of all the experimental hazards. In addition, all students are required to take a mandatory 1-hour safety training course before entering the lab. Students must also thoroughly cover all aspects of safety during their prelab presentation. Specifically for the kinetics experiment, the concentrations of the iodine (or bromine) and HCl used are 0.1 N and 1 N, respectively. The dilute nature of these chemicals is a designed safety feature for this laboratory. In addition to the mandatory safety training, all students must wear personal protective equipment (splash goggles, lab coat, nitrile and ChloroFlex™ gloves, closed-toe shoes, and long pants) and dispense hazardous chemicals in a ventilated chemical fume hood. Students are required to read and become thoroughly familiar with the chemical Safety Data Sheets (SDS) and calculate amounts that if spilled could exceed allowable exposure limits (AEL) and flammability limits such as lower and upper explosivity limits (LEL and UEL). The laboratory is also equipped with a safety shower, eyewash station, and spill kit. OSHA-certified waste disposal containers are provided for the reactor products, wash solutions, and solid waste such as paper towels. The waste jugs are located in secondary containers for spill protection. A sharps bin is available for disposal of used syringe needles. Plastic transfer trays are used to minimize safety risks when moving syringes around the laboratory and syringes have protective covers. Needles are removed using a special tool to prevent finger pricks. Glass funnels are used when pouring large volumes of reagents such as water and acetone into the reactor to reduce the possibility of spills. Finally, in lab long hair is tied back and loose clothing, such as ties and jewelry, are removed to prevent contact with the rotating agitator shaft. Process safety management is a constant theme throughout Chemical Engineering Lab I, with the other experiments having equally strong safety features in place. Finally, adjunct professors from industry often participate or teach experiments in Junior lab and reinforce the importance of maintaining a strong safety culture.
The spectrometer can be used to measure the change in color from yellow-brown to clear. The spectrometer measures the change in intensity of the iodine at a wavelength of 510 nm (450 nm for bromine). Concentration is obtained from absorbance data using the Beer-Lambert Law:

\[ A = \varepsilon b c \]  

(7)

where \( A \) is absorbance, \( \varepsilon \) is the molar absorption coefficient, \( b \) is the sample path length, and \( c \) is the molar concentration.\(^7\) Calibration standards are prepared by the TA and a calibration curve can be constructed to determine \( \varepsilon b \), which over the range of iodine concentrations measured remains essentially constant.

During the prelab meeting, students will provide a detailed analysis of their methodology and experimental plan for obtaining the kinetic parameters. Typically, students perform one baseline experiment and then vary the concentration of one component at a time while holding the others constant including temperature which results in seven to 10 experiments. These experiments are typically run by two of the four students in one of the reactor setups.

The activation energy for the reaction can be determined using the Arrhenius equation:

\[ k(T) = A e^{-\frac{E_a}{RT}} \]  

(8)

where \( k \) is the reaction constant, \( A \) is the Arrhenius parameter, \( E_a \) is the activation energy, \( R \) is the ideal gas constant, and \( T \) is the temperature.\(^8\) Typically, the other two students in the group will also run the baseline experiment in the second reactor and focus on changing the temperature of the reaction while keeping the concentrations constant in order to determine the activation energy. Students usually run three to five experiments at temperatures from about 298 K to 308 K. Changing temperature can take longer than changing concentrations so having a second reactor allows all four students to run experiments and finish within the 4-hour time period. The key to high-quality measurements is maintaining a constant concentration or temperature depending on which variables students plan to keep fixed and understanding the impact of any deviations in the detailed error analysis for the order and activation energy.

Conceptually, the ideas are simple; however, the biggest problem students encounter with the initial rates method involves the difficulties in keeping reactant concentrations constant when preparing their solutions, and keeping temperature constant when performing each of the reactions. Each group of students must determine a value and an uncertainty for the orders of reaction for iodine, acetone, and hydrochloric acid, as well the Arrhenius parameter and activation energy. Students who are well prepared with good lab skills and who analyze their data in lab typically obtain excellent results.

Iodine concentration data as a function of time is shown in Figure 2. The spectrometer is turned on before any iodine is added so the initial baseline concentration (or absorbance) is zero. When the iodine is added, the concentration immediately increases as shown and the reaction begins. As the reaction progresses the iodine concentration steadily decreases to produce iodoacetone. The change in iodine concentration as a function of time can be fit using linear regression to determine the rate of reaction. As shown in Figure 2, a typical reaction takes about 15 to 20 minutes (900 – 1200 seconds) so 12 to 16 experiments can be completed in a 4-hour lab period.

To determine the reaction order for HCl, the concentration of iodine and acetone along with the temperature must be kept constant with respect to the initial baseline experiment. This simplifies Eq. (6) so that the rate constant \( k \) and the acetone and iodine concentrations cancel out. Next, Eq. (6) can be linearized by taking the natural log of both sides. Figure 3 is a
plot of the ratio of the rates versus the ratio of the HCl concentrations where the slope is the reaction order of interest for HCl. The same procedure is repeated for determining the orders for acetone and iodine (or bromine). Again, the method appears simple, but in practice how close the students maintain the concentrations and temperature they plan to cancel out determines how well they can obtain the theoretical orders for the reaction while minimizing their uncertainty.

To determine the activation energy and the Arrhenius parameter for this reaction, the rate constant $k$ must be determined as a function of temperature assuming either the theoretical or the experimentally determined orders. Eq. (8) can be linearized and $\ln(k)$ can be plotted versus the inverse of the absolute temperature as shown in Figure 4. The activation energy can be calculated by multiplying the slope of the trend line by the gas constant, $R$. The Arrhenius parameter can then be determined by taking the exponential value of the vertical intercept.

Non-linear regression can also be applied to solve the reaction orders simultaneously using all the rate data. The data has to be good quality and an initial guess for the orders provided based on the theoretical orders for acetone, HCl, and iodine (or bromine) of 1.0, 1.0, and 0. In some cases if the temperature-dependent data is also high quality the activation energy and Arrhenius parameter can also be determined along with the orders. In 2013, the use of non-linear regression was not mandatory; however, many students were able to develop a model and make comparisons with the initial rates method. In 2014, students were provided background on non-linear modeling and required to include this analysis in their final report. All groups were able to successfully model their data using non-linear regression and make comparisons with the initial rates method. Students typically employed Microsoft Excel for the linear regressions, and either MATLAB or MiniTab for the non-linear regressions.\[9,10\] In most cases when the data were of high quality the results using both methods were similar to within the experimental uncertainty.

**ASPEN modeling**

Students were taught the basics of ASPEN Plus, so that they could model the iodoacetone reaction and perform a sensitivity analysis.$^{[11]}$ Video tutorials were prepared using Camtasia® software so students could watch and learn online how to create a reactor ASPEN model.$^{[14]}$ The videos can be run simultaneously while students create their own model in a separate ASPEN window on the computer. This method of teaching engineering software has been popular with students at UD because they can watch the videos on their own as many times as necessary to learn the fundamentals while performing the exercises. Most groups chose the CSTR model (RCSTR) to evaluate the effect of changing kinetic parameters such as the reaction orders for acetone ($\alpha$), $I_2$ ($\beta$) and HCl ($\gamma$), activation energy ($E_a$) and Arrhenius parameter ($A$) on the production of iodoacetone as shown in Figure 5.

As expected the reaction order for iodine has no effect on the production of iodoacetone (i.e., zero order), increasing the reaction order for acetone increases the production of iodoacetone (i.e., [acetone] > 1.0), and increasing the HCl reaction order decreases the production of iodoacetone (i.e., [HCl] < 1.0). The activation energy ($E_a$) and/or temperature have the largest effect on the production of iodoacetone, which is expected since both are within the exponential as shown in Eq. (8).
A few groups also modeled the reactor using the batch ASPEN model (RBATCH), which provided the change in iodine concentration as a function of time. A good fit between the experimental and model data was obtained as shown in Figure 6. This provides students with a model comparison of their time-dependent concentration data.

Students use their ASPEN models to estimate the reactor size and operating conditions necessary for commercial-scale production of iodoacetone (or bromoacetone). This is the first opportunity students get to use ASPEN to model an experiment and although they only have time to go to lab once to run this experiment, they can use their models to expand upon the effect of changing operating conditions such as reactor temperature and volume or operating a continuous flow reactor versus a batch reactor. In other words, the students can run additional experiments using their ASPEN models without having to return to the laboratory.

Assessment and experiences

The average kinetic parameters obtained for the KIN experiment in 2013 and 2014 are provided in Table 2. The bromination of acetone in 2013 was run by 13 groups (52 students) and the iodination of acetone in 2014 was run by 12 groups (48 students). In both years, students came close to obtaining the theoretical orders for the halogen (I$_2$ or Br$_2$) of 0, the acetone of 1.0, and the catalyst (HCl) of 1.0. The activation energy (E$_a$) for the bromination of acetone was about 84.1 ± 2.0 kJ/mol, which is in good agreement with reported literature values (86.7 ± 0.5 kJ/mol).$^{12}$ The activation energy and Arrhenius factor were expected to be similar for both reactions as shown because the rate limiting step is not a function of the halogen [see Eq. (3)].

Students are encouraged to be creative in Junior lab. During their prelab meetings with the faculty member and TAs other experiments and modeling are discussed that the group might try if time allows. A few examples in 2014 include one group performing a mixing experiment to evaluate the speed of mixing on the rate of reaction and another group performing a basic heat transfer analysis on the temperature difference between inside the reactor versus inside the spectrometer cuvette cell. In some cases these additional experiments have improved the design of the experiment. For example, a group brought to our attention the fact that the cooling/heating jacket around the reactor did not completely envelop the 1 liter fill volume recommended for each experiment. The group determined if the reactor were filled to approximately 0.6 liters the temperature was easier to maintain and control; however, the reduced volume did lead to larger experimental errors when preparing reactant concentrations because smaller masses for each reactant must be weighed.

CONCLUSIONS

The undergraduate Junior laboratory at the University of Delaware has evolved over several decades and each year continues to implement improvements based on student feedback. Students have responded very positively to the Junior laboratory when surveyed at the end of the course. As part of the survey, students were asked to rate whether or not the following three ABET objectives were achieved:

1. Plan an optimum set of experiments that meet well-defined objectives;
2. Recognize and properly use laboratory safety procedures. Identify major hazards in an experiment. Collect data, analyze and interpret experimental measurements, and compare to existing theories, and;
3. Learn to communicate results and conclusions effectively through both written reports and oral presentations.

During the Spring 2014 semester, 70 out of 92 students...
responded to our survey. On a scale of 1 to 5, with 1 being very dissatisfied and 5 being very satisfied, these questions received average scores of 4.53, 4.75, and 4.59, respectively.

Many students also take a special interest in Junior lab after completing the course and want to know what they can do to improve the lab for the future. Students have assisted instructors during the summer and winter sessions with many upgrades including the design, construction, and documentation for the KIN experiment and modifications and calibrations to the VLE, FLO, HEX, and FOM experiments. Currently a group of students is studying the iodination of 2-butanol and another student is evaluating other strong acids that can catalyze the iodination reaction as future kinetic experiments.

Chemical Engineering Laboratory I features an informative instrumentation experiment and four unique labs each directly relating to a core course in the ChE curriculum. The KIN experiment has been successfully demonstrated for both the iodination and bromination of acetone. ASPEN modeling provides the opportunity for students to run additional experiments on the computer outside of laboratory time with results that are comparable to their laboratory data.

Many students describe the Junior lab as one of the most memorable courses they take during their undergraduate education at UD. The course prepares students to not only connect the theory from core courses with actual experiments, but also to work together efficiently in teams, to have the opportunity to lead a project, to learn how to effectively write and present engineering reports, and to operate equipment safely. The Junior lab provides a comprehensive learning environment to help prepare students for future careers as chemical engineers.

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