

An Educational Laboratory Experimental System for Teaching Chemical Reaction Process Dynamics and Control

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Abstract

Chemical processes are governed by reaction rates that convert one set of chemical species into others. While these reactions are at the heart of all chemical engineering industrial processes, it is surprising that there are few laboratory experiments that introduce students to the time-domain evolution of chemical reactions. The vast majority of commercially available process dynamics and control educational equipment encountered by undergraduate chemical engineering students considers the dynamics of heat and mass transport. Students are able to study the time rate of change of temperature in a process, or the rate of flow in a process, but not the rate of reaction.

This paper presents the concept and the design for an inexpensive experimental apparatus that makes the dynamic study of chemical reactions accessible to any undergraduate chemical engineer. The proposed laboratory system allows for an easy connection between the theoretical differential equations used to model such a system and the actual behavior observed experimentally. The experiment uses opacity of a liquid solution as a surrogate for chemical concentration of a reactant thereby allowing a change in concentration to be relatively easily transduced with a light source and photodetector.

The experimental hardware consists of a stand in which a transparent beaker with two reactants is placed. A light source, such as a laser pointer, is on one side and a photodetector is on the other. The stand is mounted on a Peltier device allowing for thermal regulation of the reactants. In this paper we use a crystal violet bleaching reaction as the example but others can be substituted. The crystal violet bleaching reaction consists of combining a purple crystal violet solution and a clear sodium hydroxide solution together to form an initially purple solution which turns clear as the crystal violet concentration decreases to zero. The photodetector measures the percent transmittance of light through the solution which is converted to the concentration of crystal violet. The top of the beaker is unobstructed, which allows more crystal violet solution to be added in order to test the dynamic system response. This open setup provides increased flexibility in that liquid can be added at multiple times, something unavailable with a spectrophotometer. Additionally, the Peltier device allows both cooling and heating, demonstrating the direct influence of temperature on reaction rate.

In addition to the experimental hardware, this paper describes a simulation software tool developed in Matlab Simulink that is designed to simulate these types of reactive systems. The environment is a graphical programming one, with easy drag-and-drop icons already created so that relatively little programming is required. The simulations provide an intermediate link between the mathematical representation and the physical experiment thereby increasing student understanding. Overall, the simulation and experimental package provide an excellent foundation for understanding reaction rates and chemical processes with first order dynamics. Examples will be given of laboratory exercises that can be performed with the setup. Additionally, there is a discussion of process feedback control opportunities that could be available for advanced undergraduate courses.

1. Introduction

In a standard chemical engineering education, a gap exists between the differential equations governing chemical reaction dynamics learned in class and how chemical plants operate in industry. Improved student understanding of the time-domain evolution of chemical reactions can help fill this gap. Thus, there is a need for a safe and affordable experimental laboratory system and simulation tool that investigates the dynamic properties of chemical reactions, a core concept of chemical engineering.

For a hands on laboratory experience, it is difficult to find experiments that directly investigate chemical reaction rates often due to safety concerns and prohibitive costs. Many reactions that might be otherwise suitable are exothermic or produce gases which can be dangerous or require special facilities, such as a fume hood. Process control laboratory exercises often include heat transport or mass transport instead of reaction dynamics. Specifically, a common mass transport lab is maintaining the liquid level in a tank and is conducted at many universities including the University of Utah and the New York University Tandon School of Engineering^{1,2}. Feedback Instruments produces process control laboratory modules for temperature control and mass flow rate control as shown in Figure 1. These modules use heat and mass as surrogates for chemical reaction process control.



Figure 1. Temperature and mass flow rate process control training systems³.

In the absence of laboratory exercises that directly investigate process control of reactions, a common approach in undergraduate chemical engineering courses is to focus on textbook problems. Many undergraduate textbooks abstract away reactant and product names and instead use generic reagents⁴⁻⁹. For example, a usual problem formulation is $A + B \rightarrow C$, which means adding reactant A and reactant B to form product C with no physical explanation of what A, B, and C represent. Therefore, students often solve differential equations for how the concentration of reactant A changes with time without any connection to a realistic reaction or situation. For many students, simply solving these differential equations is inadequate for a thorough understanding.

To support student learning, simulation tools can be useful to connect theory and real experimental reactions. Many simulation tools currently exist including Matlab Simulink which is commonly used, as well as AMESim and Modelica¹⁰⁻¹². However, these more established graphical simulation toolboxes focus primarily on mechanical and electrical systems rather than chemical process systems. Alternatively, there are chemical process simulations developed specifically for educational purposes such as Reactor Lab and PISim^{13,14}. Reactor Lab, developed at the University of California, San Diego, is a free downloadable desktop app that provides the user with options to simulate different chemical reactors, such as a dynamic continuous stirred

tank reactor (CSTR), steady state CSTR, or packed bed reactor¹³. A shortcoming of Reactor Lab is that most reactions do not have the option of using reagents with physical meaning.

Additionally, Reactor Lab has limited options for changing parameters which can make it challenging to represent a physical situation. For instance, for the dynamic CSTR, a user enters a single value for UA, where U represents the jacket heat transfer coefficient and A represents the reactor surface area. By only allowing a user to enter a single value for UA, the exact physical size of the reactor remains unknown. Another simulation toolkit is PISim, developed at the University of Strathclyde, which aims to bridge the gap for students between pipeline and instrumentation diagrams (P&ID) and what the instrumentation and control system looks like in industry¹⁵. PISim allows for more complex systems to be constructed than Reactor Lab, but it also has limited physical meaning.

This paper presents an experimental apparatus and modeling toolkit, which can improve the undergraduate chemical engineering student learning experience. A safe, inexpensive, and repeatable experimental system with example laboratory exercises and sample data is presented. Additionally, a complementary simulation package developed in Matlab Simulink is shown. The simulation toolkit is in a graphical drag-and-drop environment and grounded in physical meaning. Lastly, an explanation of how feedback control can apply to the experimental system and a demonstration of control applied in simulation is included. The connection between classroom theory and real physical systems is bridged by the cohesiveness of a physically meaningful simulation package, hands on experimental system, and standard industry process control application.

2. Hands On Laboratory Exercise

Common challenges for laboratory experiments include safety and cost. As previously mentioned, many chemical reactions are exothermic which can quickly become dangerous; additionally many other reactions require special and thus expensive facilities. Another challenge specific to chemical dynamics is reliability. For example, Michaelis-Menten kinetics experiments are often conducted at universities^{16,17} and ubiquitously taught in biochemistry and enzyme kinetics textbooks¹⁸. However, the rate of reactions is usually calculated from a change in concentration with time and this process of differentiating is necessarily inexact¹⁹.

With these challenges in mind, the crystal violet bleaching reaction was carefully selected as a suitable experiment. The reaction does not produce heat or gas, which eliminates many safety concerns. Additionally, it is low cost and requires no fume hood or specialized protective equipment. The simplicity of a first order rate law contributes to reproducible results. The reaction combines a crystal violet dye solution and a sodium hydroxide solution (NaOH). The hydroxide molecules react with the crystal violet as shown in Figure 2 and the chemical reaction is represented by Equation 1



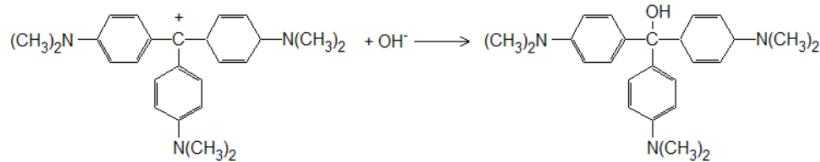


Figure 2. Crystal violet and sodium hydroxide reaction structure²⁰.

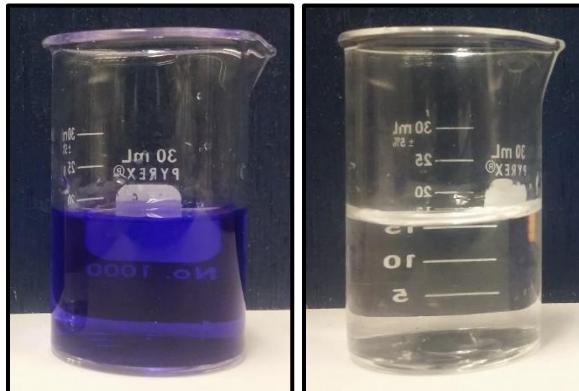


Figure 3. Crystal violet reaction at 1 minute (purple color) and 9 minutes (colorless).

When the reaction begins, the crystal violet dye causes the solution mixture to be a deep purple color. However, as the reaction progresses and the crystal violet ions disappear, the solution gradually transforms from a purple solution to a clear solution as shown in Figure 3. The visualization of how the concentration of crystal violet ions decreases is important for student interest and for easy detection by a photoresistor, rather than a more expensive and time intensive mass spectrometer. The crystal violet reaction follows a first order rate law, which means the unforced dynamic equation which governs the concentration of crystal violet is given by

$$\frac{dC_{CV}}{dt} = -kC_{CV}; C_{CV}(t=0) = C_{CV}(0), \quad (2)$$

where C_{CV} is the concentration of crystal violet and k is the reaction rate given by

$$k = k(T) = Ae^{\frac{E}{RT}} C_{OH}. \quad (3)$$

This is an Arrhenius equation with E , R , T , C_{OH} , and A . Here A is the pre-exponential factor, E is the activation energy, R is the gas constant, T is the temperature, and C_{OH} is the concentration of hydroxide ions. An important part of the reaction rate, Equation 3, is that the rate depends on temperature. The temperature dependence provides an easily adjustable process parameter to change the system time constant. To control the temperature, the experiment is conducted on a Peltier cooler as shown in Figure 4. The Peltier cooler is a thermoelectric device that can heat or cool the reagents. The experimental setup relies on measuring the optical transmissibility as a function of time by shining a light through the beaker with the reagents onto a photoresistor. The photoresistor is connected to a National Instruments myRIO, an embedded hardware device, which allows for simple data collection using LabVIEW. The experience using LabVIEW is a useful skill for undergraduates to obtain²¹. Since the reaction evolves on the time scale of seconds, the data acquisition device does not need to have as fast a sampling rate as a myRIO. Therefore, cheaper options like a SparkFun RedBoard Arduino or a BeagleBone Black would suffice. The custom stand for this laboratory exercise was 3D printed using High Impact

Polystyrene (HIPS) filament. The beaker has no cover so that additional reactants can be added throughout the reaction, unlike a spectrophotometer. The light source hardware choice is flexible, with the simplest option likely being a laser pointer. The total cost of the experimental apparatus is less than \$750, with the tabulated costs shown in Appendix A, Table 4.

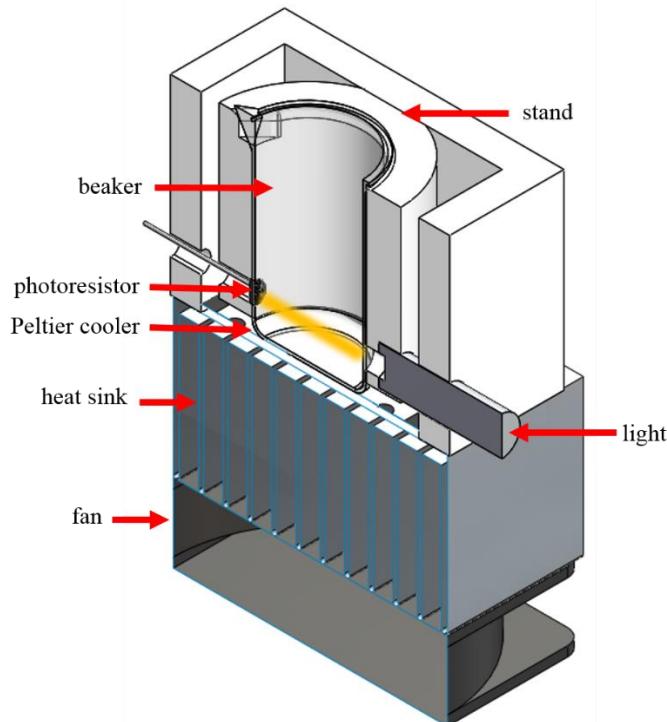


Figure 4. Experimental apparatus CAD model showing key elements, such as how the optical transmissibility of reactants in the beaker is detected with a light and photoresistor.

For this experimental apparatus and reaction, there are two laboratory exercises which both involve collecting analog output data from the photoresistor as a function of time. The first exercise is to conduct the reaction at various temperatures and observe how the reaction rate and time constant change as a function of temperature. The second exercise is to conduct the reaction at room temperature and, as the reaction progresses, to add a discrete amount of crystal violet solution to observe the step response of the system. For both exercises, the instructor provides 2.7×10^{-5} M crystal violet solution and 0.1 M sodium hydroxide solution. Standard chemical safety procedures should be followed with students wearing gloves and safety glasses. Outlines of both exercise procedures are provided in Appendix B and sample data are provided in Figure 8 and Figure 9. These exercises provide an interactive and highly visual experience to build intuition for the effect of a temperature dependent reaction rate, as shown in Equation 3, and how a first order system responds to step inputs.

3. Simulation Toolkit

A system modeling toolkit created in Matlab Simulink¹⁰, a block diagram environment, contains a library of chemical process component blocks. For example, the library of blocks includes, but is

not limited to, a continuous stirred tank reactor (CSTR), pipe, mass source, pump, split, and mass sink as shown in Figure 5.

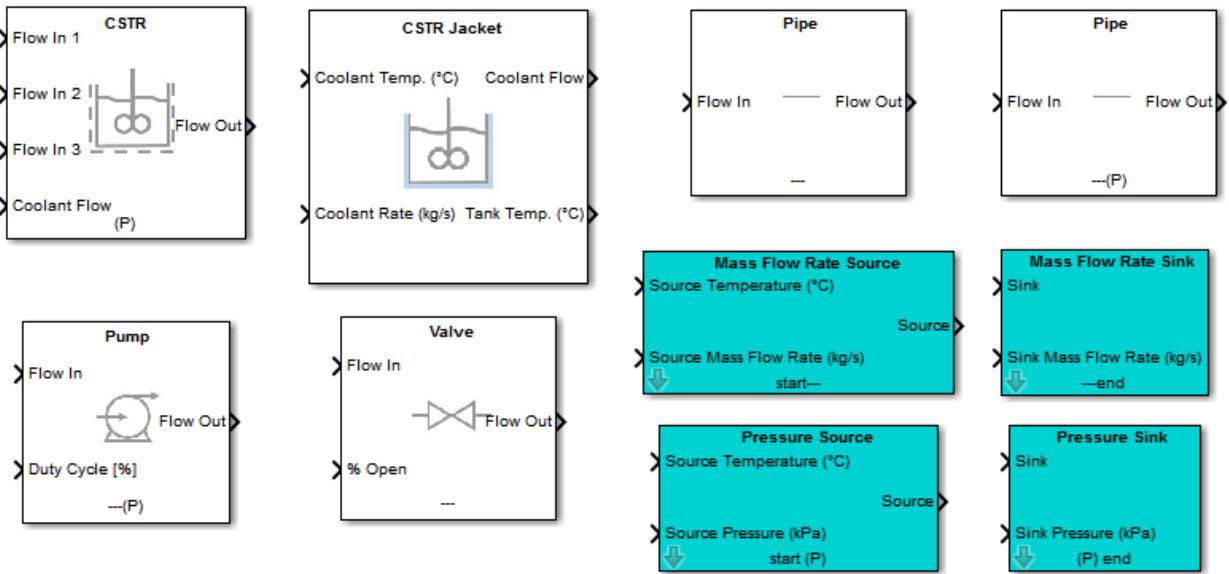


Figure 5. Example of Simulink library blocks that can be connected to create a model of a chemical process.

The blocks can be dragged and dropped into a Simulink model and connected to one another. The connection between blocks allows mass to flow between the different components to model the dynamics of a chemical process as shown in Figure 6. The flows into the CSTR contain the fluid temperature ($^{\circ}\text{C}$), mass flow rate (kg/s), reactant concentration (kg/m^3), density (kg/m^3), specific heat capacity ($\text{J/kg}^{\circ}\text{C}$), and heat of formation (J/kg).

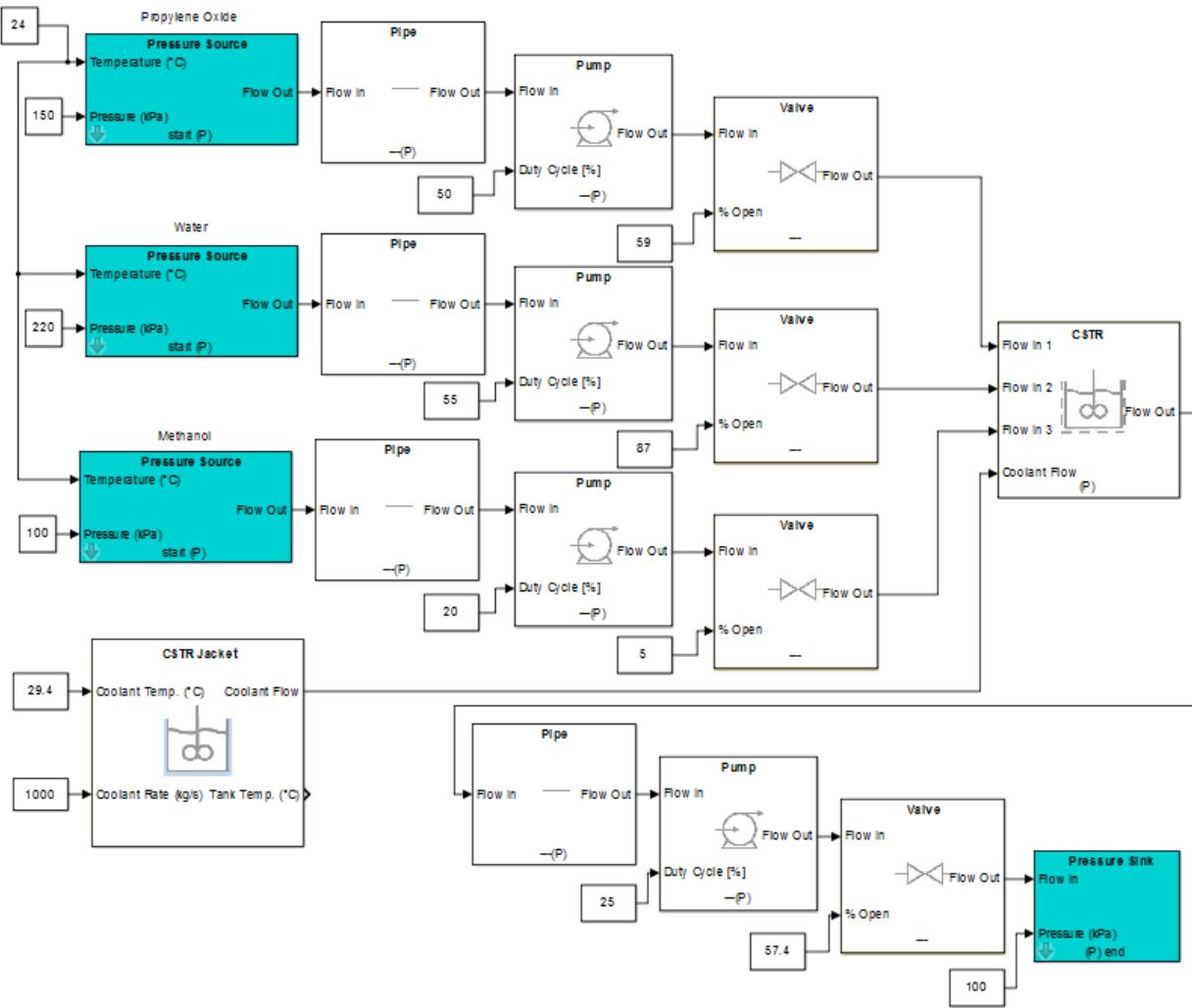


Figure 6. Example model configuration for the formation of propylene glycol specifically and in general an $A + B + C \rightarrow D$ example CSTR process.

Within each block, the dynamic differential equations for how values change, such as the temperature or concentration, are embedded. For example, within the CSTR block, the energy balance

$$\frac{dT}{dt} = \frac{UA_r(T_a - T) - \dot{W} - F_{A0}C_{ps}(T - T_0) + \Delta H_{RX}(-kC_A V)}{C_{A0}VC_{ps}}, \quad (4)$$

mass balance

$$\frac{dC_A}{dt} = \frac{C_{A0}v_0}{V} - \frac{C_Av_0}{V} - kC_A, \quad (5)$$

and rate law

$$k = Ae^{\frac{E}{RT}}, \quad (6)$$

are included exactly as students learn in class with the parameters defined in Table 1.

Table 1. CSTR energy and mass balance parameter and state definitions.

A	pre-exponential factor	F_{A0}	inlet mass flow rate of reactant A	T_0	inlet temperature
A_r	reactor surface area	ΔH_{RX}	heat of reaction	U	jacket heat transfer coefficient
C_A	reactant A concentration	k	rate constant	V	reactor volume
C_{A0}	inlet concentration of reactant A	R	gas constant	v_0	inlet total volumetric flow rate
C_{ps}	solution specific heat capacity	T	temperature	\dot{W}	shaft work
E	activation energy	T_a	jacket temperature		

Embedding energy and mass balances within the blocks reduces the amount of detailed programming and allows students to instead focus on the main ideas and explore the effects of changing key parameters. This is an important feature, because students often spend copious amounts of time entering the balance equations (Equation 4 and Equation 5) into Mathematica or Polymath, an ODE solver recommended for use within Fogler's textbook⁴. With this toolkit, the interface students work with instead is shown in Figure 7. Here, the geometric parameters tab is shown, which allows students to enter physically meaningful parameters such as tank diameter and height.

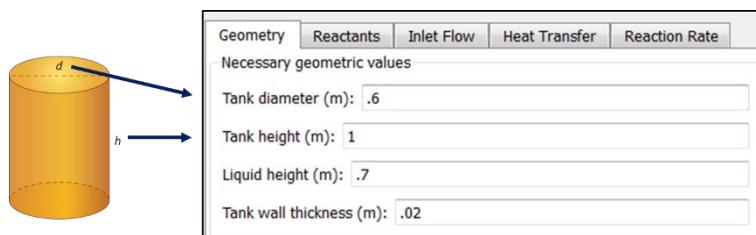


Figure 7. CSTR block user interface geometry tab with physically meaningful parameters.

Advantages of this simulation toolkit include physically meaningful reactants and parameter choices. Additionally, if desired, library blocks can be customized and new blocks added. For the purpose of this article, the modeling toolkit can be coupled with the crystal violet bleaching experiment previously described to supplement the theory and equations learned in class.

4. Comprehensive Results and Time Constant Analysis

After completing both the physical experimental laboratory exercises and developing a model in Simulink, students can compare experimental and simulation results. First, for the trials run at different temperatures, a comparison of experimental data and model results reveals good agreement as shown in Figure 8. Additionally, the model aligns well with the experimental results for the step input response of adding more crystal violet solution at 400 and 800 seconds

as displayed in Figure 9. The clean agreement allows students to investigate different scenarios with other temperatures or initial concentrations in the modeling toolkit and have confidence in the results.

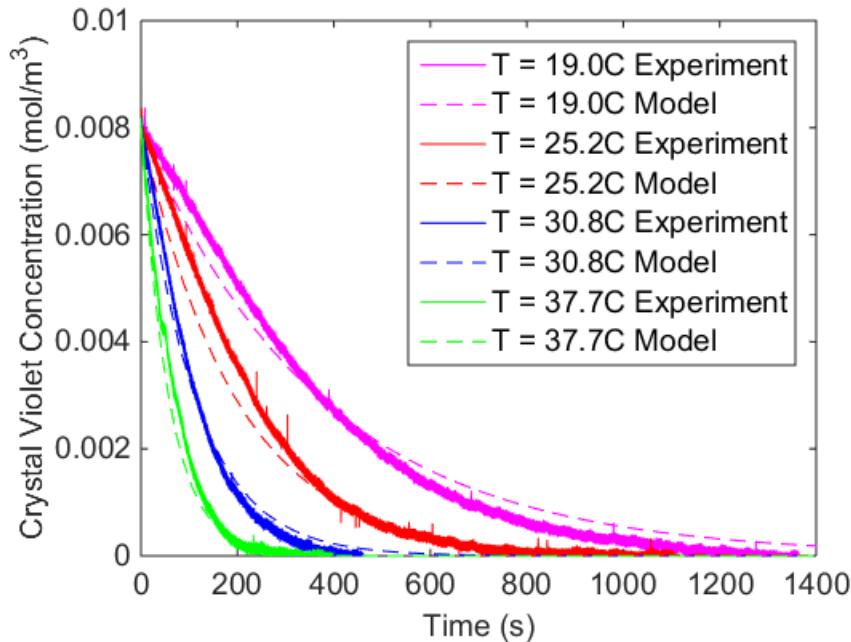


Figure 8. Crystal violet concentration versus time for experimental data and model results for different reaction temperatures.

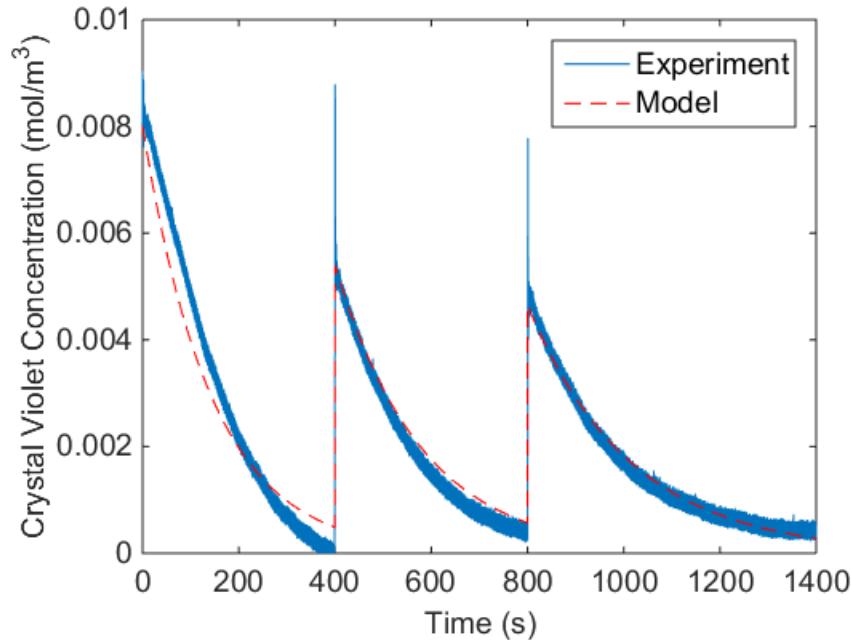


Figure 9. Crystal violet concentration versus time for experimental data and model results with added step inputs of additional crystal violet at 400 seconds and 800 seconds.

In addition to exploring different simulation scenarios, students can further analyze the results of a real first order chemical system. The first order differential equation for how the concentration of crystal violet, C_{CV} , evolves is given by

$$\frac{dC_{CV}}{dt} = -\underbrace{Ae^{\frac{E}{RT}} C_{OH}}_k C_{CV}, \quad (7)$$

with the solution

$$C_{CV}(t) = ae^{-kt}, \quad (8)$$

where A is the pre-exponential factor, E is the activation energy, R is the gas constant, T is the temperature, C_{OH} is the concentration of hydroxide ions, and t is time. Since this is a standard first order system, the following holds

$$\tau = \frac{1}{k} = \frac{1}{Ae^{\frac{E}{RT}} C_{OH}}, \quad (9)$$

where τ is the time constant. For this system, students can calculate the theoretical time constant, which will agree exactly with the model (dashed lines) in Figure 8. Then, students can estimate the time constant from the experimental data by determining the time when the experimentally measured concentration is $C_{CV} = \frac{1}{e} * C_{CV}(t=0) = \frac{1}{e} * 0.008 = 0.0029 \text{ mol/m}^3$.

5. Process Feedback Control Opportunities

After students gain familiarity with the crystal violet reaction and how the crystal violet concentration changes with time, the opportunity arises to introduce simple process feedback control. A pedagogical approach can be applied to a crystal violet reaction in a CSTR. A CSTR scenario means there is continuous mass flow into the reactor and out of the reactor to maintain a constant volume, which is different than the batch reactor scenario in the laboratory experiment with no mass flow out. Considering a crystal violet reaction in a CSTR allows students to write transfer functions for real system, understand system type, and design proportional and proportional-integral controllers.

First, the differential equation that governs how the concentration of crystal violet changes with time given as

$$\frac{dC_{CV}}{dt} = -\left(\frac{kV + v_0}{V}\right)C_{CV} + \underbrace{\frac{C_{CV0}v_{CV0}}{V}}_{\text{input}}, \quad (10)$$

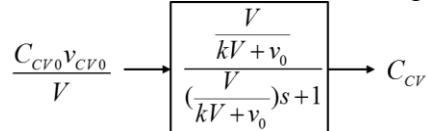
where C_{CV} is the crystal violet concentration in the reactor, k is the rate constant, V is the liquid volume, v_0 is the total mass flow rate, C_{CV0} is the inlet crystal violet concentration, and v_{CV0} is the inlet crystal violet mass flow rate. The inlet mass flow rate of the crystal violet solution, v_{CV0} , is the input term that will be modified by the controller to achieve a desired output of crystal violet concentration, C_{CV} . The other input term, inlet mass flow rate of sodium hydroxide will be held constant.

To write as a transfer function, Equation 10 must be converted to the frequency domain

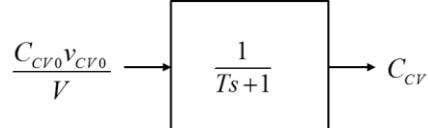
$$sC_{CV} = -\left(\frac{kV + v_0}{V}\right)C_{CV} + \frac{C_{CV0}v_{CV0}}{V} \quad (11)$$

$$\frac{C_{CV}}{\frac{C_{CV0}v_{CV0}}{V}} = \frac{1}{s + \frac{kV + v_0}{V}}, \quad (12)$$

where Equation 11 is rearranged into Equation 12 to be in a more standard form of output divided by input. Equation 12 can be converted into a block diagram as follows



and simplified to



which captures the dynamic behavior. The general form of an open-loop transfer function is

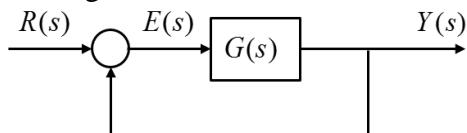
$$G(s) = \frac{K(T_a s + 1)(T_b s + 1) \dots (T_m s + 1)}{s^N (T_1 s + 1)(T_2 s + 1) \dots (T_p s + 1)} \quad (13)$$

where the system order is the order of the denominator ($p+N$) and the system type is the number of free integrators (N). For the crystal violet CSTR system, the open-loop transfer function is

$$G(s) = \frac{1}{Ts + 1}, \quad (14)$$

which confirms this is a first order system. Additionally, Equation 14 identifies that this reaction is a type 0 system, which will be important when considering how system types respond to different inputs.

In general, the closed-loop block diagram form is



with the closed-loop transfer function for error as

$$\frac{E(s)}{R(s)} = \frac{1}{1 + G(s)}, \quad (15)$$

where $R(s)$ is the reference signal, $E(s)$ is the error signal, $G(s)$ is the open-loop transfer function of the plant, and $Y(s)$ is the output signal. Equation 15 can be rearranged as

$$E(s) = \frac{R(s)}{1 + G(s)} \quad (16)$$

to solve for $E(s)$ to analyze the steady state error. With the final value theorem²² applied to Equation 16, the connection between the time domain and frequency domain is made with

$$e_{ss} = \lim_{t \rightarrow \infty} e(t) = \lim_{s \rightarrow 0} sE(s) = \lim_{s \rightarrow 0} \frac{sR(s)}{1 + G(s)}, \quad (17)$$

where e_{ss} is the steady state error between the reference signal and the output.

For this exercise, students can analyze the response to a unit step input, $R(s) = \frac{1}{s}$, because step inputs are characteristic of many industrial processes⁵. If a unit step input reference signal is substituted into Equation 17, then

$$e_{ss} = \lim_{t \rightarrow \infty} e(t) = \lim_{s \rightarrow 0} sE(s) = \lim_{s \rightarrow 0} \frac{sR(s)}{1 + G(s)} = \lim_{s \rightarrow 0} \frac{s(\frac{1}{s})}{1 + G(s)} = \frac{1}{1 + G(0)}, \quad (18)$$

governs the steady state error. Table 2 shows how there is a constant non-zero steady state error for type 0 systems, but for type 1 or higher systems there is zero steady state error.

Table 2. Steady state error for different system types.

System Type	$G(0)$	e_{ss}
0	$G(0) = \lim_{s \rightarrow 0} G(s) = K$	$e_{ss} = \frac{1}{1 + K}$
≥ 1	$G(0) = \lim_{s \rightarrow 0} G(s) = \infty$	$e_{ss} = 0$

Next, students could examine how proportional (P) and proportional-integral (PI) control affect the steady state error of a reference step input signal. The closed-loop block diagrams for P and PI control are shown in Figure 10.

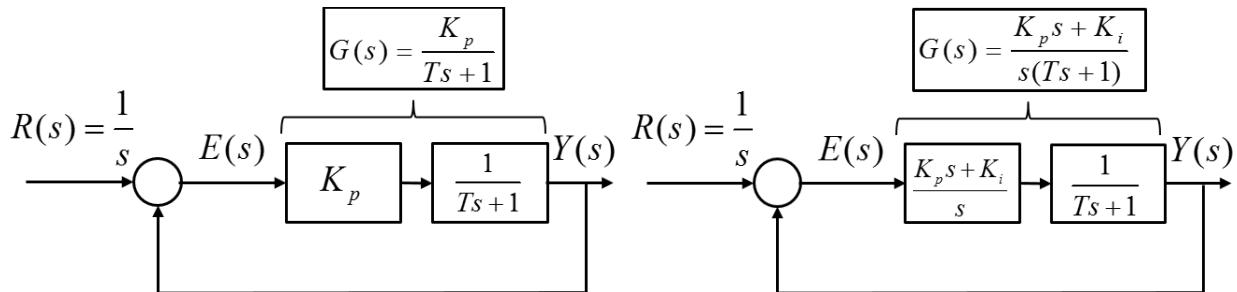


Figure 10. Proportional control (left) and proportional-integral control (right) for a unit step input and open-loop transfer function representative of a crystal violet reaction in a CSTR.

With P control applied, Figure 10 shows how the transfer function, $G(s) = \frac{K_p}{Ts+1}$, still takes the form of a type 0 system when compared with Equation 13. Therefore, P control does not eliminate the non-zero steady state error inherent to the plant. For this reason, the steady state errors present in Figure 11 are expected. However, for PI control, Figure 10 shows that the

transfer function, $G(s) = \frac{K_p s + K_i}{s(Ts+1)}$, has a free integrator, making it a type 1 system. Thus, with

PI control, zero steady state error is expected, which means that the concentration of crystal violet should be tracked exactly in Figure 12. Next, students can consider whether the inlet mass flow rate of crystal violet, the controller output, is reasonable as shown in Figure 13.

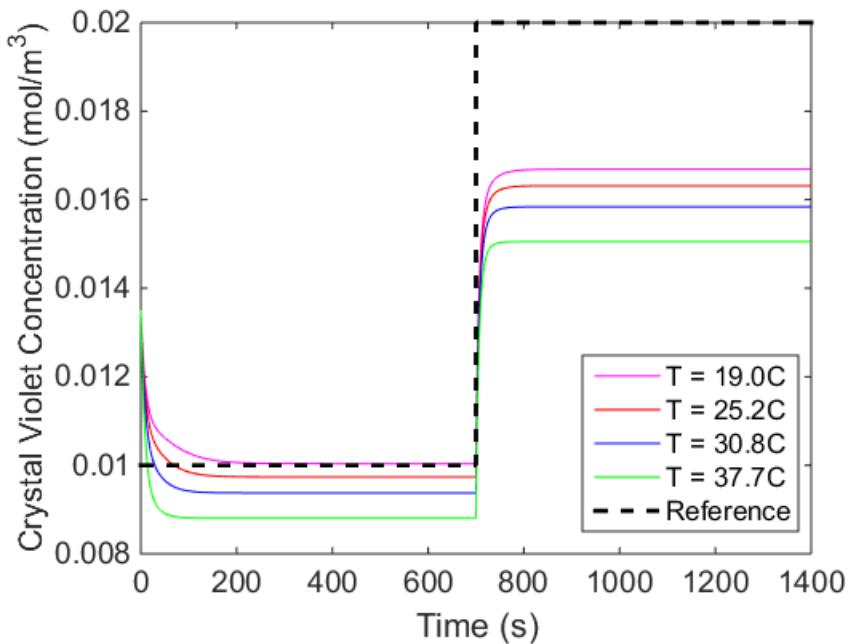


Figure 11. Crystal violet concentration versus time with proportional control of a step input at four temperatures showing non-zero steady state tracking errors to the dashed reference concentration.

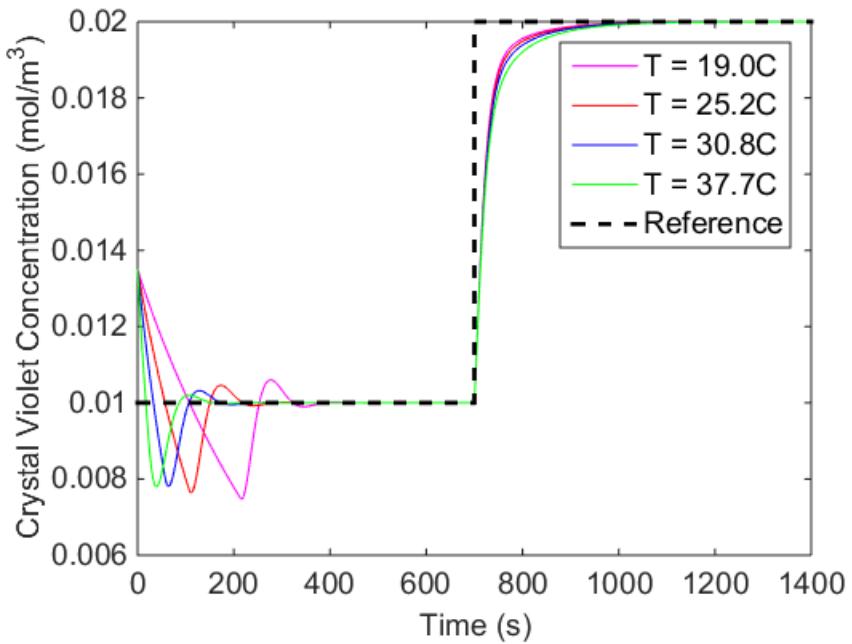


Figure 12. Crystal violet concentration versus time with proportional-integral control of a step input at four temperatures showing zero steady state errors to the dashed reference concentration.

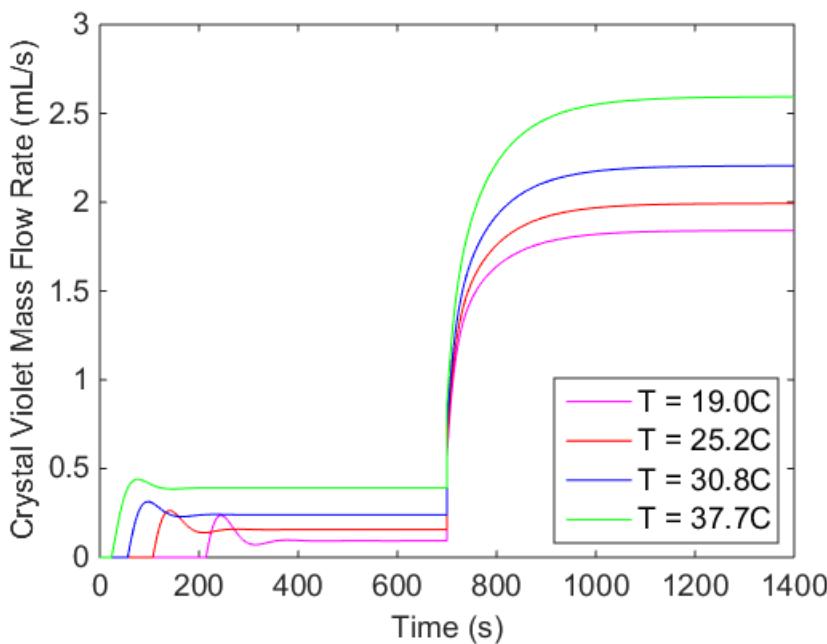


Figure 13. Crystal violet solution mass flow rate versus time determined by proportional-integral control at four temperatures to track the reference crystal violet solution shown in Figure 12.

Process feedback control topics covered:

- Translating time-domain differential equations into block diagrams
- Writing open-loop and closed-loop transfer functions
- Identifying system type
- Applying the final value theorem
- Designing P and PI controllers
- Identifying steady state error (can extend to overshoot, rise time, and settling time)
- Studying whether control outputs are reasonable

The direct application of these introductory feedback control concepts to a process that students have already explored through experiments and simulations aids in cementing the knowledge and building intuition for process control.

6. Conclusion

A hands on, safe, and reliable experimental laboratory exercise and complementary simulation toolkit are presented with suggested analysis and process feedback opportunities. The recent development of this learning setup has not allowed for student assessment yet. Therefore, the next step is to test this entire learning module with undergraduate chemical engineering students.

There are many ways to classify a “good experiment”, with initial criteria specifically for controls laboratory exercises in 1981 and as recently as 2016^{21,23}. However, good aspects of laboratory exercises transcend different disciplines. This laboratory exercise pairing a visual experiment and simulation toolkit with exploration of control objectives satisfies many of the criteria outlined in Roberts’ paper²¹ shown in Table 3.

Table 3. Criteria for a good experiment²¹ and analysis of whether this laboratory module satisfies the criteria (Y - yes or N - no).

“A good experiment should...”	Y/N – Reaction Dynamics Lab Module
Demonstrate important theoretical ideas	Y – temperature dependent rate laws, first order dynamic system response to step inputs
Reflect problems and situations students may encounter in real life	N – realistic to apply PI control to a chemical process ^{24,25} , but entire lab does not revolve around a real life situation
Be enjoyable, interactive, and promote active learning	Y – hands on visual lab exercises and control design in simulation both provide interactive experiences
Appeal to different types of learning styles	Y – visual, kinesthetic, and read/write styles
Provide a reasonable return on investment (ROI)	Y – main lab components reasonably priced and chemicals are affordable to restock
Be open and open-ended	N – straightforward procedure with space for independent student exploration only for the simulated control tasks
Be physically and logically robust	Y – experimental apparatus is durable and simple to provide easy troubleshooting
Safety	Y – requires minimal personal protective equipment and supervision
Exposure	Y – introduces students to proprietary software like LabVIEW and MATLAB, important tools for industry ²¹

This experiment can be improved by more closely reflecting real life problems and being more open-ended. Nevertheless, the experiment has the potential to serve as a uniquely safe, affordable, and visual experiment with a physically meaningful simulation tool to provide a cohesive learning experience about chemical reaction dynamics. Since this laboratory exercise investigates a first order chemical system, it is analogous to first order mechanical system laboratory exercises, like a DC motor velocity, or first order electrical system laboratory exercises, like an RC circuit voltage. These first order mechanical and electrical laboratory exercises are ubiquitous in undergraduate courses and this paper provides an equivalent pedagogical first order system laboratory module for chemical dynamics.

7. Acknowledgements

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References

1. University of Utah, D. of C. E. Liquid Level Control. (2017). at <https://www.che.utah.edu/undergraduate/projects_lab/equipment/liquid_level_control/>
2. NYU, M. C. L. Two Tank Experiment. (2017). at <<http://engineering.nyu.edu/mechatronics/Mpcrl/>>
3. Feedback. Feedback Products : Process Control. (2016). at <http://www.feedback-instruments.com/products/education/process_control>
4. Fogler, H. S. *Elements of Chemical Reaction Engineering*. (Prentice Hall PTR, 1992).
5. Seborg, D. E., Edgar, T. A. & Mellichamp, D. A. *Process Dynamics and Control*. (John Wiley & Sons, Inc., 2004).
6. Marlin, T. E. in (McGraw-Hill, Inc., 1995).
7. Levenspiel, O. *Chemical Reaction Engineering*. (John Wiley & Sons, Inc., 1999).
8. Davis, M. E. & Davis, R. J. *Fundamentals of Chemical Reaction Engineering*. (McGraw-Hill, Inc., 2003).
9. Bequette, W. *Process Dynamics Modeling, Analysis, and Simulation*. (Prentice Hall PTR, 1998).
10. MathWorks. Simulink. (2016). at <<https://www.mathworks.com/products/simulink.html>>
11. Siemens. LMS Imagine.Lab Amesim. (2016). at <https://www.plm.automation.siemens.com/en_us/products/lms/imagine-lab/amesim/>
12. Modelica. Modelica and the Modelica Association. (2016). at <<https://www.modelica.org/>>
13. Herz, R. K. Reactor Lab. (2016). at <reactorlab.net>
14. Postlethwaite, B. PISim Simulation - Exothermic CSTR. (2016).
15. Postlethwaite, B. The use of simulation in chemical process control learning and the development of PISim. in *2016 American Controls Conference (ACC)* 7328–7333 (2016).
16. Kentucky, U. of. EXPERIMENT 9 - Kinetic Methods of Analysis : Enzymatic Determination of Glucose. at <http://www.chem.uky.edu/courses/che226/Labs/090-Glucose_Kinetics.pdf>
17. Thompson, J. P. (York C. of P. Lab 3: Enzyme Kinetics. (2010). at <<http://faculty.ycp.edu/~jthompson/Biochemistry F2009/labs/enzyme kinetics 2009.pdf>>
18. Schnell, S. & Maini, P. K. A Century of Enzyme Kinetics: Reliability of the Km and Vmax Estimates. *Comments Theor. Biol.* **8**, 169–187 (2003).
19. Schnell, S. & Mendoza, C. Closed Form Solution for Time-dependent Enzyme Kinetics. *J. Theor. Biol.* **187**, 207–212 (1997).
20. Pasco. Order of Reaction Laboratory Handout. 1–18 (2016).
21. Roberts, D. & Borowski, A. P. A Revised Undergraduate Controls Lab Featuring Exposure-Based Experiences A Revised Undergraduate Controls Lab Featuring Exposure-based Experiences. *2016 ASEE Annu. Conf. Expo.* (2016).
22. Franklin, G. F., Powell, J. D. & Emami-Naeini, A. *Feedback Control of Dynamic Systems*. (Pearson, 2015).
23. Balchen, J. G., Handlykken, M. & Tysso, A. The Need for Better Laboratory Experiments In Control Engineering Education. in *8th IFAC World Congress* 3363–3368 (1981).
24. Kocijan, J., Žunić, G., Strmčnik, S. & Vrančić, D. Fuzzy gain-scheduling control of a gas-liquid separation plant implemented on a PLC. *Int. J. Control* **75**, 1082–1091 (2002).
25. White, A., Zhu, G. & Choi, J. Hardware-in-the-loop simulation of robust gain-scheduling

- control of port-fuel-injection processes. *IEEE Trans. Control Syst. Technol.* **19**, 1433–1443 (2011).
26. Scholar Chemistry. Sodium Hydroxide Solution, 0.1M MSDS. 1–2 (2009). at <http://www.onboces.org/safety/msds/S/Scholar_Chemical/Sodium_Hydroxide_Solution_0.1M_669.00.pdf>

Appendix A: Experimental Apparatus Cost

Table 4. Experimental apparatus cost breakdown.

Part	Cost (\$)
3D printed stand	4
Beaker, 30 mL	3
Crystal violet solution, 0.024M, 100 mL	8
Sodium hydroxide solution, 1.0M, 500 mL	7
Light source	10
Peltier cooler module	35
Power supply (for Peltier cooler)	215
Power supply (for photoresistor)	215
myRIO	249
Total	746

Appendix B: Experimental Laboratory Procedures

Temperature dependence procedure:

1. Measure 5 mL of 2.7×10^{-5} M crystal violet solution in a 10 mL graduated cylinder and pour into first 30 mL beaker.
2. Measure 5 mL of 0.1 M sodium hydroxide solution in a 10 mL graduated cylinder and pour into second 30 mL beaker.
3. Place both beakers on the Peltier cooler to heat or cool solutions to the desired temperature. Adjust temperature by changing the applied voltage of the power supply attached to the Peltier cooler.
4. Once both solutions have reached desired temperature, place stand, photoresistor, light source, and first beaker in place as shown in Figure 4. Keep the applied voltage to the Peltier cooler constant to maintain the same temperature throughout the reaction.
5. Begin acquiring data from the photoresistor with a sampling rate of one sample per second or faster.
6. Pour the second beaker with the sodium hydroxide solution into the first beaker to begin the chemical reaction.
7. Trial is complete when solution is clear.
8. Repeat steps 1-7 for desired variety of temperatures.

Step input procedure:

1. Set up stand, photoresistor, light source, and beaker as shown in Figure 4.
2. Measure 10 mL of 2.7×10^{-5} M crystal violet solution in a 10 mL graduated cylinder and pour into 30 mL beaker on the Peltier cooler.
3. Measure 10 mL of 0.1 M sodium hydroxide solution in a 10 mL graduated cylinder.
4. Begin acquiring data from the photoresistor with a sampling rate of one sample per second or faster.
5. Pour sodium hydroxide solution into beaker with crystal violet solution and begin timer.
6. Measure 5 mL of 2.7×10^{-5} M crystal violet solution in a 10 mL graduated cylinder.
7. At 400 seconds, pour additional 5 mL of crystal violet solution into the beaker.
8. Measure 5 mL of 2.7×10^{-5} M crystal violet solution in a 10 mL graduated cylinder.
9. At 800 seconds, pour additional 5 mL of crystal violet solution into the beaker.
10. At 1400 seconds, trial is complete.

Safety:

A key advantage of this experiment is the low level of safety concerns. However, it is important to note that general laboratory safety practices (chemical splash goggles and chemical resistant clothing) should be used when handling the sodium hydroxide²⁶.