Automated Titration System

ECE 445 Design Document – Spring 2024

Project #59

Jason Flanagan, Jack Viebrock, Matthew Weyrich

Professor: Jonathon Schuh

TA: Selva Subramaniam

Table of Contents

Part 1: Introduction

Problem:

Titration is a time-consuming process that can introduce large amounts of error from the manual procedure, such as improper burette reading, accidental extra analyte added, and guessing on the endpoint with a color indicator. Automatic titration systems can help reduce this error but cost over $3,000, restricting their application to wealthy labs. Someone starting a small wine business might want to perform a manual titration to determine the acid content of their products, but repeated manual titrations take up lots of time and can be inaccurate.

Solution:

We will create a lower-cost automatic titration system to bridge this gap in the market to make it affordable to have high-quality titration data accuracy over manual methods. This will be achieved using a pH-sensor and microcontroller to control the speed and accuracy of titration. By compiling the data collected from the pH-sensor, the data will be simultaneously displayed of the current pH, along with the derivative of the graph, representing the endpoint of the titrate and solution. With all the necessary components and the help of the machine shop, the system cost will be around $180.

Visual Aid:

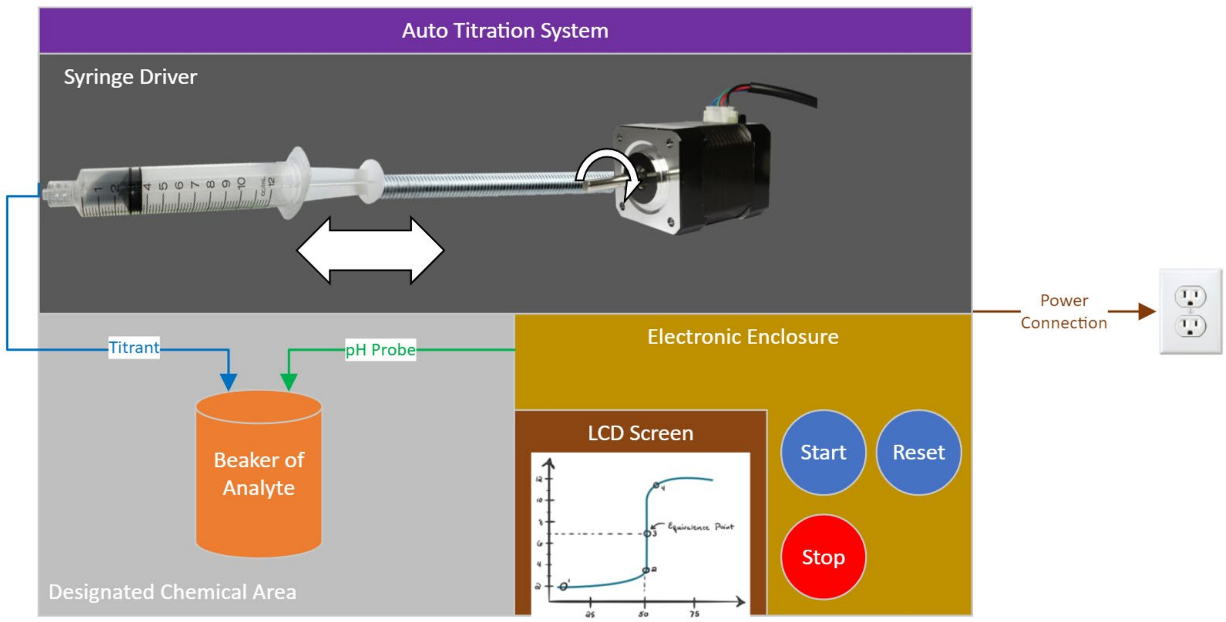

Figure 1: High Level Visual Overview of Titration System, highlighting chemical locations and User I/O

3D/CAD Initial Design Concept:

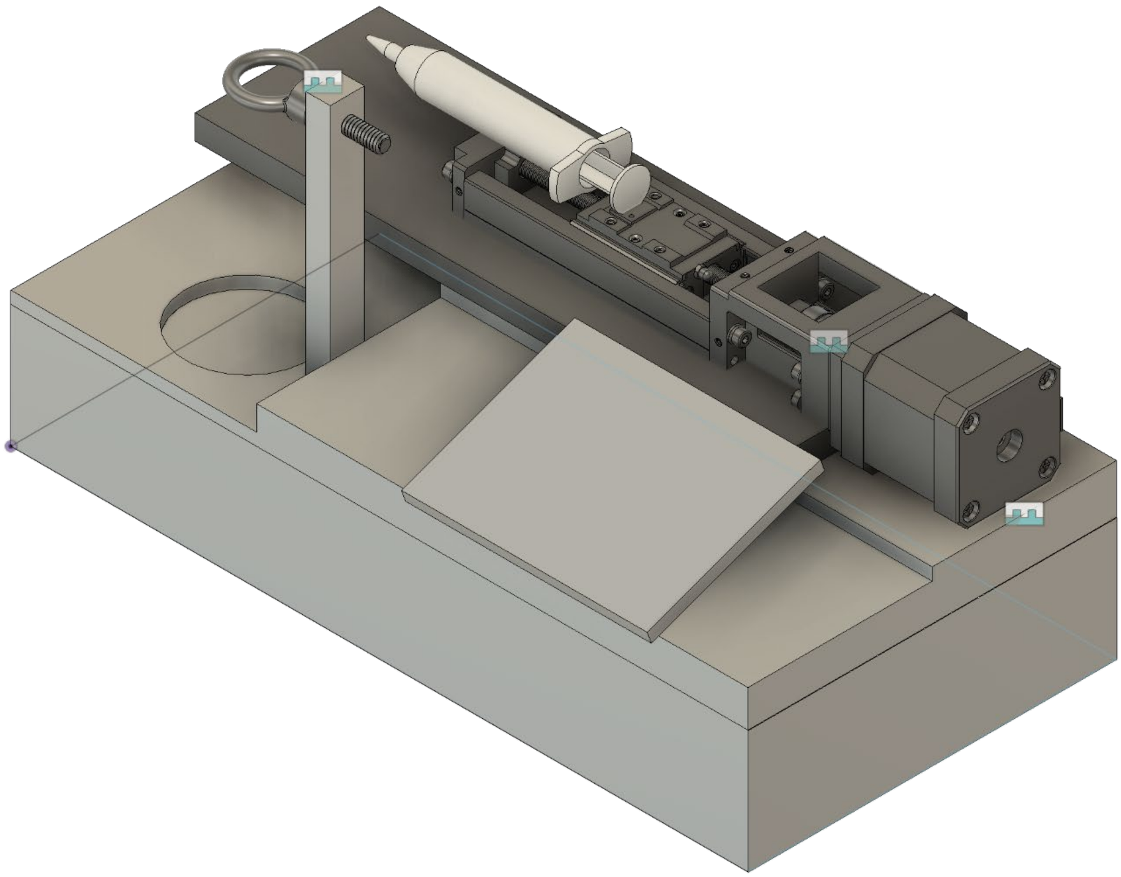

Figure 2: 3D/CAD Initial Design Concept

-Parts in the CAD drawing do not resemble final components but proof of concept of the layout. Some changes may be made based on what the machine shop fabricates, the components and functionality under test conditions.

-The back section resembles the syringe driver; the front left section resembles the beaker's place with a spot for the ph probe and syringe tubing to route through; and the front left section resembles the interface where the LCD Screen and Buttons will be placed with the other electronic components placed underneath the base.

High-level requirements list:

- 1st Requirement (Precision): Repeat titration with only +/-0.5% deviation between measured titration endpoints

- 2nd Requirement (Speed): Perform a titration as fast or faster than five minutes
- 3rd Requirement (Accuracy): Measure pH with the pH probe within +/-0.056 (+/- 0.02V of the correlated voltage; 0 V +/- 0.02V for 0 pH, and 5 V +/- 0.02V for 14 pH)

Part 2: Design

Block Diagram:

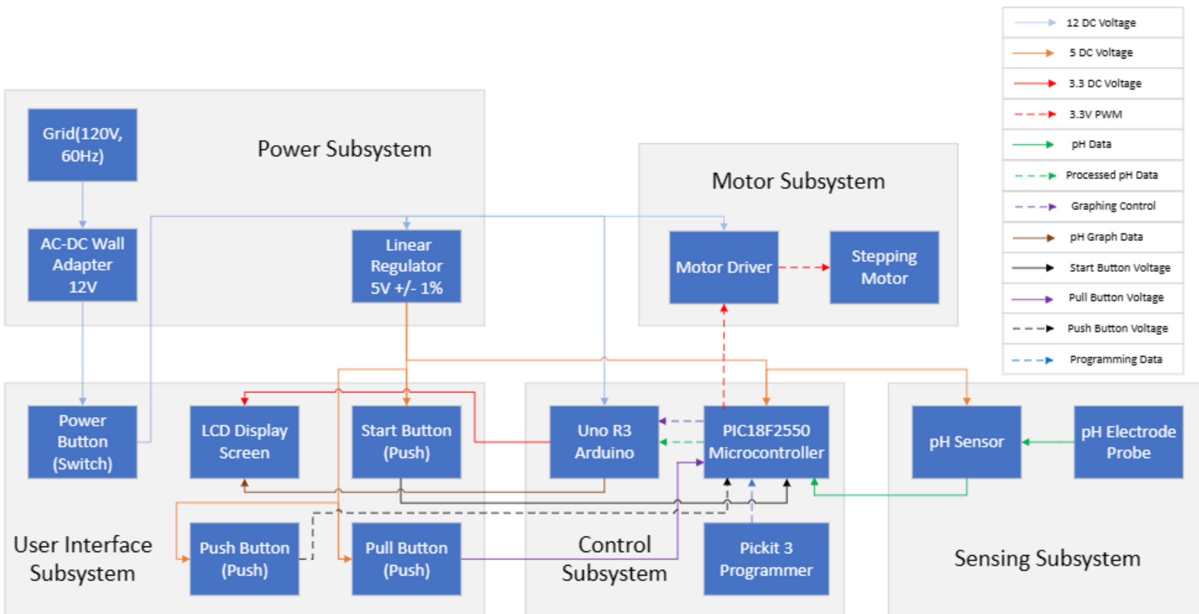

Figure 3: Diagram of Titration System Consisting of The Five Subsystems: Power, Motor, Sensing, Control and User Interface

Subsystem Overview:

Sensing Subsystem

- The sensing system's main purpose is to read the pH of the current solution. This will be achieved by using an electrode probe to determine the pH of the solution. Since the electrode outputs miniscule amounts of voltage, a pH sensor is needed to amplify the signal and add a DC offset. The pH sensor will be powered by the power subsystem and will send the data from the pH sensor to the microcontroller in the control subsystem.

Power Subsystem

- This system converts the grid's voltage to a DC voltage. The system contains a linear regulator to step-down the DC voltage to 5V, allowing the power subsystem to power the microcontroller and pH sensing circuitry.

Control Subsystem

- This subsystem contains the microcontroller and Arduino. The microcontroller is used to collect data from the sensing subsystem and to control the motor through four control signals. The Arduino is used to display the pH and volume data on the LCD screen from the User Interface subsystem. It will also take the derivative of the data to determine the solution's endpoint.

Motor Subsystem

- Contains the stepping motor that will be used to control the speed of release from the syringe containing the titrate.

User Interface Subsystem

- This module contains an LCD screen, a start button, a pull button, a push button, and a power button (Emergency stop). The pull button is used to fill the syringe with the titrate by retracting the plunger. The push button is used to press the plunger further into the barrel. The start button is used to initiate the titration. The LCD screen is used to display the data being produced from the Arduino. The power button is used as an emergency stop switch in case anything goes wrong or to cut the power when the system is not in use.

Subsystem Requirements:

Subsystem 1: Sensors

The titration system will not need an indicator to evaluate the endpoint. Instead, a pH electrode and accompanying circuitry, together called the pH sensor, will be used. The pH electrode works by detecting the amount of hydrogen ions in a solution. The demo will feature vinegar, a dilute form of acetic acid, in a beaker to start. The pH probe will be responsible for measuring the pH of this solution as we continuously add a strong base, NaOH. This will change the pH from acidic to neutral and then finally end up as a basic solution, and this circuitry will determine those changes. More care on the chemicals used is given in Part 4: Ethics and Safety. The pH, a measure of hydrogen ions in solution, will generate a potential against a ground voltage resulting in an electrical potential from -415 mV to 415 mV from the probe itself. This voltage is quite small, so an amplifying circuit is required before being connected to the microcontroller for analog to digital conversion (ADC). To boost the signal, the amplifying circuit will be powered by a 5 +/- 0.1 voltage being produced by the power subsystem. The voltage can then indicate the current acidity of the solution on a scale of 0-14, where 7 is the neutral value. This corresponds to a voltage in the range from 0 to 5 V which can be read by the ADC on the microprocessor. The total current drawn based on the chosen potentiometer and resistor values will maximize at 8.75 mA.

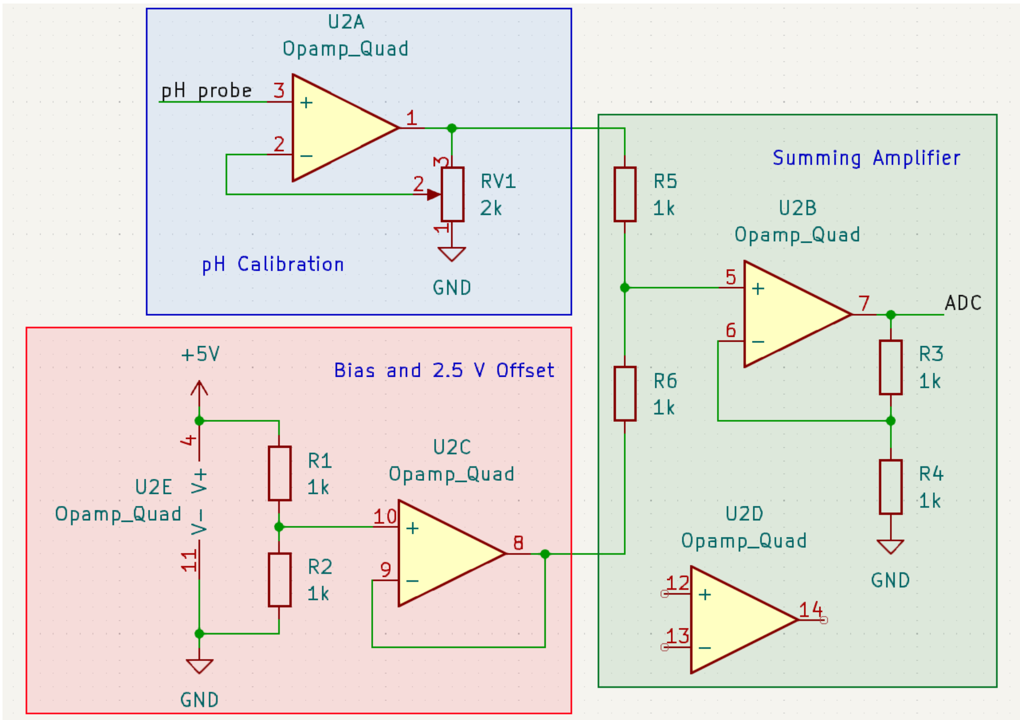

Figure 4: Schematic for pH probe output amplification and scaling to match ADC input pin voltage requirements

Requirements	Verification
• The pH sensor will utilize a 5 +/- 0.1 V input	• Place multimeter probes on the Vdd and GND pins on the pH sensor PCB to determine the voltage
• The pH sensor will output voltages between 0 and 5 +/- 0.1V with a current up to 24 +/- 1 mA	• Place multimeter probes on the output to ADC and GND pins on the pH sensor circuitry to determine the voltage and current • If the current or voltage is too large, the microcontroller will not be able to reliably read the data. Adjust the output current by altering the resistance at the output.
• Measure pH with the pH probe within +/-0.056 (+/- 0.02V of the correlated voltage; 0 V +/- 0.02V for 0 pH, and 5 V +/- 0.02V for 14 pH)	• Obtain demineralized water, pH4 and pH7 solutions • Place the electrode in the pH7 solution

	• After the measurement becomes stable (~ one minute), adjust the pH sensor to output 2.5 volts • Rinse the electrode well with demineralized water • Repeat the previous three steps with pH4 solution (achieve a reading of 1.429V), until the pH sensors output voltage is within +/- 0.02V for both solutions [6]
• Reduce the amount of electrical interference around the instrument	• Remove any unnecessary machinery in the immediate area to reduce electrical noise

Table 1: All requirements and verification needed for the Sensing Subsystem

Subsystem 2: Power System

We will be using an AC (120V, 60Hz) wall to 12V DC adapter. Additionally, we will need to use a 12-to-5 linear voltage regulator to power the microcontroller, pH sensor circuitry, and to route through the start and stop buttons in the user interface subsystem. We will be using a 5V 250 mA linear regulator that will output 5V +/- 1% with an average current draw of 0.6 mA. The 12V DC signal from the wall adapter will be routed through a power button in the user interface subsystem to allow a quick way of cutting power. This 12V signal will also be applied to the Arduino in the Control System and the stepper motor driver in the motor subsystem.

Requirements	Verification
• System Protection	• When deciding what wall adapter to use, make sure it can protect the system against power surges, inserting the plug in backwards, and other safety features
• AC-DC wall adapter that produces a DC voltage between 7-12 volts to power the Arduino	• Use a multimeter to ensure the wall adapter is producing a voltage between the Arduinos specified values
• Route the wall adaptors DC voltage through an emergency shutoff button	• Connect the wall adapter to the power button in the user interface subsystem • Ensure the button is operating correctly by using a multimeter to ensure no

	voltage is being supplied to the system when the switch is open
• Subsystem produces a 5 +/- 0.1 V output to power the pH sensor and microcontroller	• Validate the 5 V regulator can accept the voltage produced by the wall adapter. • Ensure the regulator is outputting a voltage at five volts and within tolerance

Table 2: All requirements and verification needed for the Power Subsystem

Subsystem 3: Control

The microcontroller will be taking the live output voltage from the sensing subsystem and processing it. At the same time, the microcontroller will determine the speed of the motor depending on the amount of titrate left in the syringe and the collected data. The microcontroller will also be in-charge of starting and resetting the pump when the start and reset button are pressed, respectively. The processed data will be sent from the microcontroller to the Arduino. The Arduino will display the current pH level on the LCD screen in the User Interface subsystem. The current volume in the beaker will be determined based on the step size of the motor. The endpoint of the titration curve, also known as the steepest part of the curve (based on the data points), will also be determined and displayed. The Arduino and microcontroller will be powered from the power subsystem.

Requirements	Verification
• The microcontroller will operate the motor to avoid pushing the plunger too far into the barrel, or by pulling the plunger out of the barrel.	• Attach a closed syringe to the stepper motor's rod • Hold the pull button to determine how long it takes to fill the plunger • Adjust the code's constraints to determine how far the plunger is pulled out of the barrel • Reset the plunger to the initial closed state by pressing the start button • Repeat the previous three steps to the desired plunger distance
• The microcontroller will create a four wired signal to control the motor dependent on step size. For a design	• Adjust the wire outputs based on the driving mode of the motor [8] until one drop comes out per cycle

specific time interval, the syringe will need to produce one drop of titrate.	• Achieve at least 30 drops for 30 duty cycles to confirm that no more or less drops dispended out of the syringe
• The Arduino will be used to solely graph the processed data and to determine the endpoint. The graph will be of pH vs. milliliter of titrate	• Data will be retrieved and connected to the LCD screen in the User Interface • Check that the data being graphed is correct by using the pH solutions that were used to calibrate the electrode

Table 3: All requirements and verification needed for the Control Subsystem

Subsystem 4: Motor

Our implementation of an automatic titration system will imitate a burette by using a syringe driver, which is a stepper motor to precisely administer titrant with a syringe. The motor will need to be connected to a motor driver so it can be controlled through the microcontroller. Without the motor driver, the microcontroller will not be able to produce enough voltage and current to operate the stepper motor. We will be using the *17N19S1684MB-200RS Nema 17* stepper motor, which has a 0.04 mm lead/step to allowing us to compress the syringe exactly to our specifications. The syringe will then be attached to a plastic tube with a pointed end to minimize drop size, thus further increasing precision on titrant dispense.

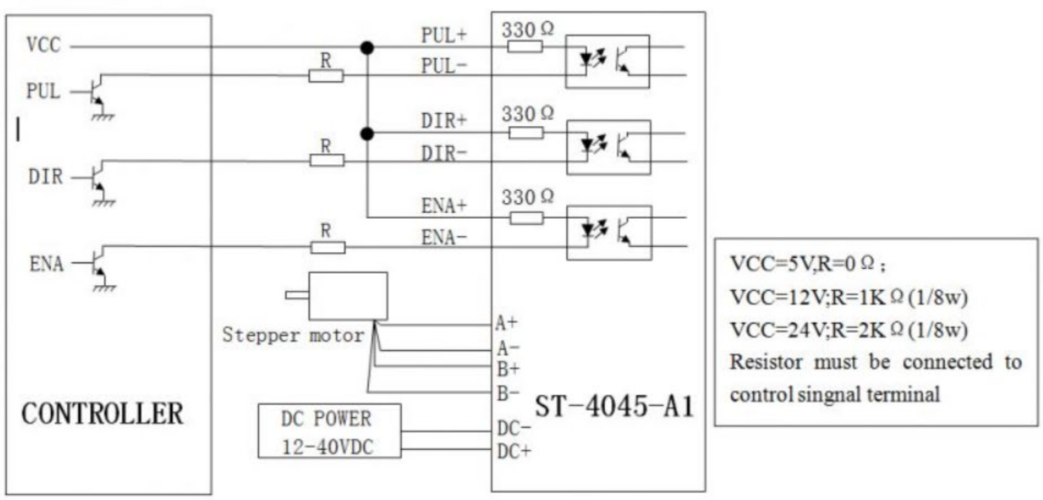

Figure 5: Schematic for TB6600 Stepper Motor Driver

Requirements	Verification
• The motor driver will amplify the microcontrollers signals, into ones that the motor can operate at by boosting current and voltage	• Observe the motors movement of the motor when the start or reset button is pressed in the user interface subsystem
• The motor must produce at least 33 Newtons of force [7] to pull and push the plunger out of the barrel	• When deciding what motor to use, guarantee it can produce enough force to operate the syringe • Since the torque is being converted from angular to linear, a part of the force will be used in this conversion, resulting in the necessity for a motor that can produce more than 33 newtons of force

Table 4: All requirements and verification needed for the Motor Subsystem

Subsystem 5: User Interface

The user interface consists of four components: an LCD screen, a start button, a pull button, a push button, and a power button. The LCD screen will be displaying the data created by the Arduino. The pull button is used to return the syringe to its extended state. The push button is used to further insert the plunger. The start button will be used to start the act of titration, indicating that the titrate and system has been properly setup. The power button is used to avoid sudden surges of power when plugging the system into the outlet, while also offering an emergency stop button.

Requirements	Verification
• The DC power from the wall adapter is fed through an emergency/power button	• Connect the wall adapter to the power button • Ensure the button is operating correctly by using a multimeter to ensure no voltage is being supplied to the system when the switch is open
• The start button initiates the motor to start pressing the plunger into the syringe	• When the start button is pressed, the syringe will start to close • The syringe will stop moving when it has reached its approximate endpoint that was determined in the microcontroller's code

• The push and pull buttons will be used to load and unload any titrate within the syringe	• When the pull button is pressed, the plunger is pulled further out of the barrel of the syringe • When the push button is pressed, the plunger is pushed further into the barrel of the syringe

Table 5: All requirements and verification needed for the User Interface Subsystem

Tolerance Analysis:

One of the project's key requirements is to make it more precise than manual titration. To do this, the syringe driver will have to be able to produce a drop of titrant smaller than that of a standard burette. A standard burette produces a drop size of 0.0357 ml according to [5]. To determine the precision of the syringe driver, the syringe and the stepper motor needed to be further researched.

First, the size of the syringe barrel needs to be calculated. The syringe barrel has a diameter of 22.1996 mm. This means that the barrels cross-section or surface area can then be computed by $SA = \pi r^2$. This results in the surface area being $SA = \pi \left(\frac{22.1996}{2}\right)^2 = 387.0617\, mm^2$.

According to the design document of the selected stepper motor, the thread on the lead will rotate 0.04 mm per step with a step accuracy of +/- 5.00%. By multiplying the cross-section of the barrel by the distance per step traveled of the stepper motor, the volume per step is 15.4825 +/- 0.7741 $\frac{mm^3}{step}$.

Since 1 milliliter is equivalent to $\frac{1000\, mm^3}{1\, ml}$, the amount of liquid dispensed per step of the motor would result in 15.4825 +/- 0.7741 micro-liters, which is 2.2 times more precise than a standard burette.

Part 3: Cost and Schedule

Cost Analysis:

The average starting salary for a UIUC Electrical Engineer Graduate is $42.20/hour. With an overhead factor of 2.5 and working and estimated 90 hours on this project throughout the semester, the cost comes to $9,495 per group member. Since there are three group members, the cost of our labor comes to $28,485. For the machine shop, the average salary of a machine shop worker is around $20/hour. With our design taking an estimated 20 hours to complete, the total cost of the machine shop would be $400.

Description	Manufacturer	Part #	Quantity	Cost
Gen 2 Mini Lab Grade pH Probe	Atlas Scientific	#ENV-20-pH	1	$60.99
10 mL Plastic Syringe	Betevie	BETUS-LTL	10	$8.48
12V DC 500MA POWER SUPPLY Wall Adapter	Lab	N/A	1	$0.00
+5 Voltage Regulator	STMicroelectronics	L4931CZ50-AP	1	$0.93
LCD 12x2	Lab	N/A	1	$0.00
Nema 17 Non-captive Linear Stepper Motor Actuator 48mm Stack 1.8 Deg 1.68A Lead 8mm/0.31496" Lead Screw 200mm	OYSTEPPER	*17N19S1684MB-200RS Nema 17*	1	$37.90
Stepper Motor Driver	Yisheng Technology	B08SG7L54W	1	$9.69
PCB.SMAFRA.HT	Taoglas Limited	931-1361-ND	1	$3.54
Quad Op-Amp	Microchip Technology	MCP6499T-E/SL	1	$0.88
External potentiometer	Bourns Inc.	3310Y-001-103L-ND	1	$3.82
PIC® 18F Microcontroller IC 8-Bit 48MHz 32KB (16K x 16) FLASH 28-SOIC	Microchip	PIC18F2550	1	$0 (Electronic Services Shop)

Push Button	N/A	N/A	2	$0 (Electronic Services Shop)
Toggle Switch	N/A	N/A	1	$0 (Electronic Services Shop)
Misc. PCB Components	N/A	N/A	X	$0 (Electronic Services Shop)

Table 6: Cost layout for all necessary components for the project

The total cost of the parts ends up being $127. Along with the cost of the group's labor and the machine shop, the total cost for the project will be $29,017.90.

Schedule:

Week	Expected Sequence of Events	Required Deliverables
1/15	• Initial Team Contact and Formation – Team • Independent Brainstorming and submission of post - Individual	• Initial Web Board Post
1/22	• Evaluate post feedback and process through brainstorming ideal to filter out project to propose – Team • Complete Assignments Individually - Individual	• Laboratory Safety Training • Early Project Approval • CAD Assignment
1/29	• Complete RFA – Team • Start on Project Proposal once RFA approved – Team • Start Initial Whole Concept Design – Team • Soldering Assignment work and finish - Individual	• Project Approval
2/5	• Design more finalized system overview and mechanical layout – Team • Get signoff from machine shop on viability – Team • Initial selection of architecture/components – Team	• Soldering Assignment • Proposal • Team Contract • Initial Contact with Machine Shop
2/12	• Design each subsystem in additional depth with component decisions for controls, power, motor, sensors, interface – Team • Jason – Sensor subcircuit	• Design Review Sign-Ups Open

	• Matthew – Power, Interface, Control • Jack – Power, Motor	
2/19	• Complete Design Document and Presentation – Team • Complete Regrade of Project Proposal – Team • Design of PCB Initialized – Team	• Design Review Sign-Ups Close • Design Document • Proposal Regrade
2/26	• Design of PCB Ready for Inspection – Team • Design of PCB Reviewed by TAs • Finalized Mechanical Dimensions to Machine Shop for build to begin – modules by individuals and put together by team. • Buy First Round of Components (Everything Machine Shop Needs) – Team	• Design Review • PCB Review
3/4	• Final week that component orders (external vendors) should be made – Team • Final information to Machine Shop for build – Team • Start Working on Final Paper – Team	• First Wave PCB Orders • Teamwork Evaluation • Friday-Last day to get revisions to machine shop
3/11	SPRING BREAK	N/A
3/18	• PCB Test, Assemble and Debug - Jason o Start revisions if necessary. • Test Power Converters – Jack and Matthew • Assemble Electrical Components: Arduino, Screen, Probe, PCB, Etc. - Team • Final paper work - Team	• Second Wave PCB Orders

3/25	• Resubmit PCB revisions if necessary
	• Check Progress with Machine Shop for Assembly - Jack
	• Test Modules/Subsystems
	  o Assemble, Code and Test Graph System - Matthew
	  o Test pH system - Jason
	  o Ensure Proper Power Input - Jack
	  o Test User Interface responsiveness/functionality - Matthew
		• Third Wave PCB Orders
		• Individual Progress Reports

4/1	• If Machine Shop Complete, Assemble modules on setup - Team • Test Motor Program for functionality, debug - Jack	• Fourth Wave PCB Orders

	• Test System Integration with submodules and start tests of functionality – Matthew and Jason • Final Paper Work - Team	
4/8	• Debug system integration and perform test on functionality • Final Paper Work and start presentation work	• Fifth and Final Wave PCB Orders
4/15	• Refine prototype further seeing how mock demo performs and is evaluated • Final Paper and Presentation Work	• Mock Demo • Team Contract Fulfillment
4/22	• Give Final Demonstration • Finalize Presentation, checking over: • Technical Information – Jack • Formatting - Jason • Time Constraints and Flow - Matthew	• Final Demo • Mock Presentation
4/29	• Give Presentation and submit final documentation - Team	• Final Presentation • Final Paper • Lab Notebook Submission

Table 7: The complete timeline for the project and the teammate specific work

Part 4: Ethics and Safety

There are many ethics and safety concerns when dealing with our project of titration, especially with the use of varying chemicals. We acknowledge that the IEEE ethics code [1] I.1 to "hold paramount the safety, health, and welfare of the public" will be chief in ensuring that our product will provide safe manipulation of chemicals in multiple applications and environments. This will be kept in mind for all design aspects of the project.

We also believe that the ACM's code of ethics 1.3 "Be honest and trustworthy" [4] is one that plays into all factors of our project from the design, build and functionality. It should provide a reminder to always cite sources, check patents and be honest about the origins of ideas. Additionally, when showcasing our project, it is important to be honest about the limitations and features. We must also be trustworthy between teammates to ensure we keep a good working relationship and continue working efficiently and effectively.

Safety is of utmost importance when considering the design and execution of this product. We stand behind the procedures set by the Division of Research Safety. This includes standards for chemical waste management making sure that the chemical compounds used in titration are not directly poured down the drain unless authorized which includes rinses that do not include heavy metals or acutely toxic waste with others on a case-by-case basis [2]. Additionally, all other waste should be placed in a proper container and use DRS waste tags for identification.

We also must keep in mind the possibilities of spills in the lab area, and we need to understand the risks and the ability to clean up any issues. The 445 lab already has a bright yellow spill kit that we intend to have present during the titration demo. The Division of Research Safety [3] outlines chemically resistant gloves, googles, acid & base neutralizers, and chemical spill absorption powder as essential when working with chemicals. You must use the PPE to protect yourself from the effects of these chemicals and properly use the absorption powder and understanding the classifications of chemicals to do so properly. For the demo we have planned, the chemicals used will be acetic acid (vinegar) and sodium hydroxide. The vinegar used will be food grade and can be used without PPE, but we will still have a safety data sheet on hand for acetic acid in case of emergencies. Sodium hydroxide will be used at 0.1 M concentration, which is dilute but still can cause damage to the user so nitrile gloves, googles and a lab coat will be worn for the demo. The volume of NaOH used will be 20 mL or less because we will use a 20 mL syringe, and the volume of acetic acid that can be neutralized with 20 mL of NaOH is around 2.4 mL, so 3 mL or less of acetic acid will be needed for the demo. For chemical storage, the base will need to be in its own container explicitly labeling the presence of the base, and the acid can be stored in the red box of flammable materials already in the lab. The safety data sheets for both chemicals will be added to the yellow safety folder located at the entrance of the lab. Lastly, because of the nature of the demo, any observers for the demo will need to be seated or standing at least 6 feet away from the demo to prevent any splashes or spills from reaching people not wearing PPE.

Additionally, the risk of fire is possible with the testing of electronics and in real world applications when flammable chemicals are in use. It is important to understand the difference in fire extinguishers that ABC extinguishers can be used for electrical fires and many chemical fires. Although some chemical fires from combustible metals would require a class D extinguisher. Making note of all emergency exits and fire extinguishers near the demo location will be done before demoing.

The use of moving pieces includes a motor and moving syringe provide a need to have rules set in place to keep hands, cords, long clothes, and hair out of reach of these moving components and chemicals/liquids. Additionally, you want to avoid these types of objects as to not pull the lab setup over which would result in spilled chemicals and possibility for fire.

Sources

[1] "IEEE code of Ethics," IEEE, https://www.ieee.org/about/corporate/governance/p7-8.html

[2] "Division of Research Safety," Illinois, https://drs.illinois.edu/Page/Waste/ChemicalWasteProcedures

[3] "Division of Research Safety," Illinois https://drs.illinois.edu/Page/IncidentResponse/EmergencyPreparedness

[4] "The code affirms an obligation of computing professions to use their skills for the benefit of society.," Code of Ethics, https://www.acm.org/code-of-ethics

[5] "Acid-Base Titration" Vernier.com https://www.vernier.com/files/sample_labs/CHEM-A-07-acid_base_titration.pdf (accessed Feb. 2, 2024)

[6] "How to calibrate a ph meter?," Royal Brinkman International - Global Specialist in Horticulture, https://royalbrinkman.com/knowledge-center/mechanical-equipment/ph-meter-calibration.

[7] M. S. Read, "Pressure effects of syringes," *Anaesthesia*, vol. 48, no. 11, pp. 1017–1017, Nov. 1993. doi:10.1111/j.1365-2044.1993.tb07504.x

[8] Sourav Gupta, "Interfacing Stepper Motor with PIC Microcontroller", Circuit Digest, https://circuitdigest.com/microcontroller-projects/interfacing-stepper-motor-with-pic16f877a.