Automatic Titration Proposal

Team 59

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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:

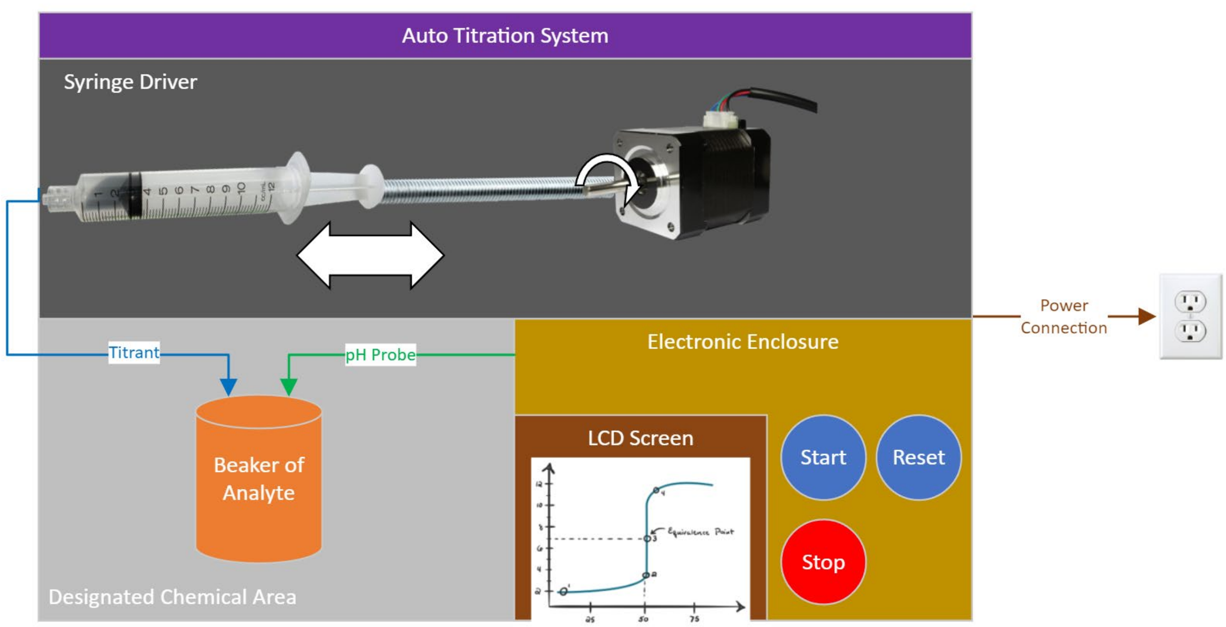

Figure 1: High Level Visual Overview of Titration System, highlighting chemical locations and User I/O

3D/CAD Initial Design Concept:

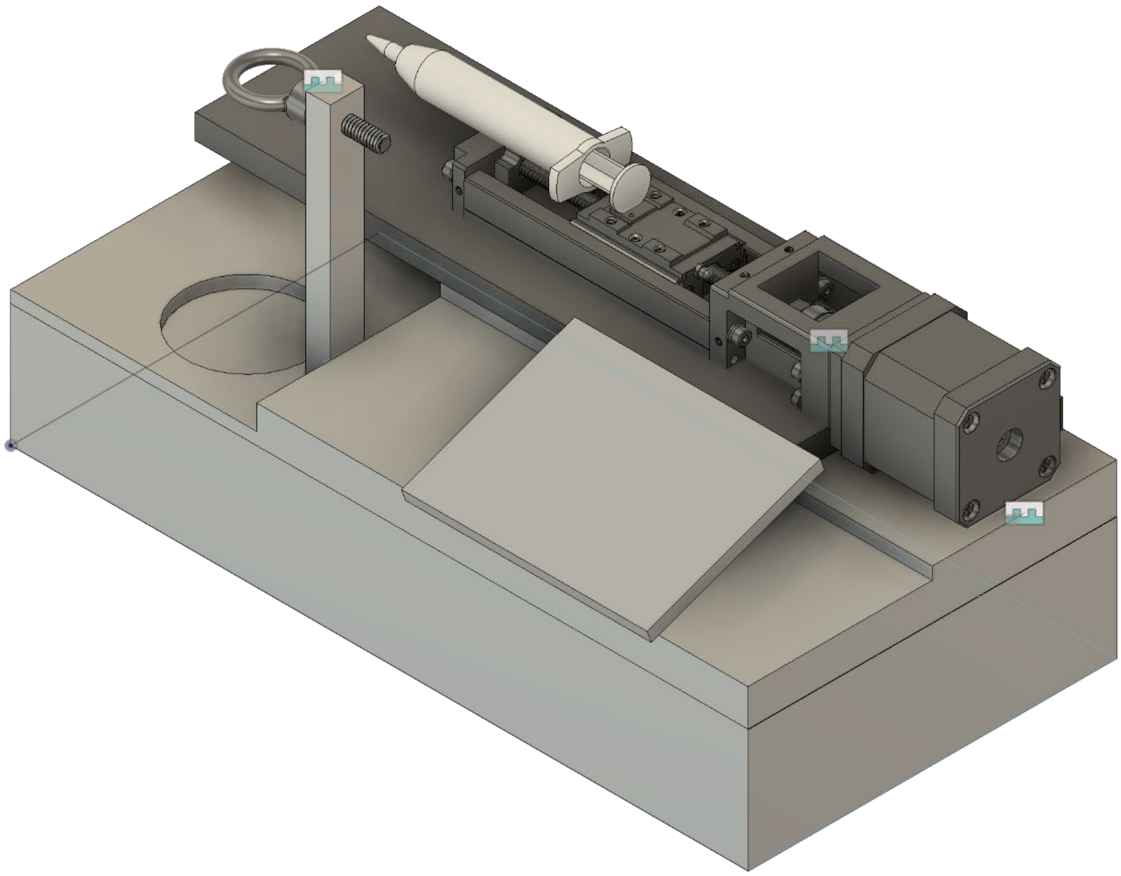

Figure 2: 3D/CAD Initial Design Concept

-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:

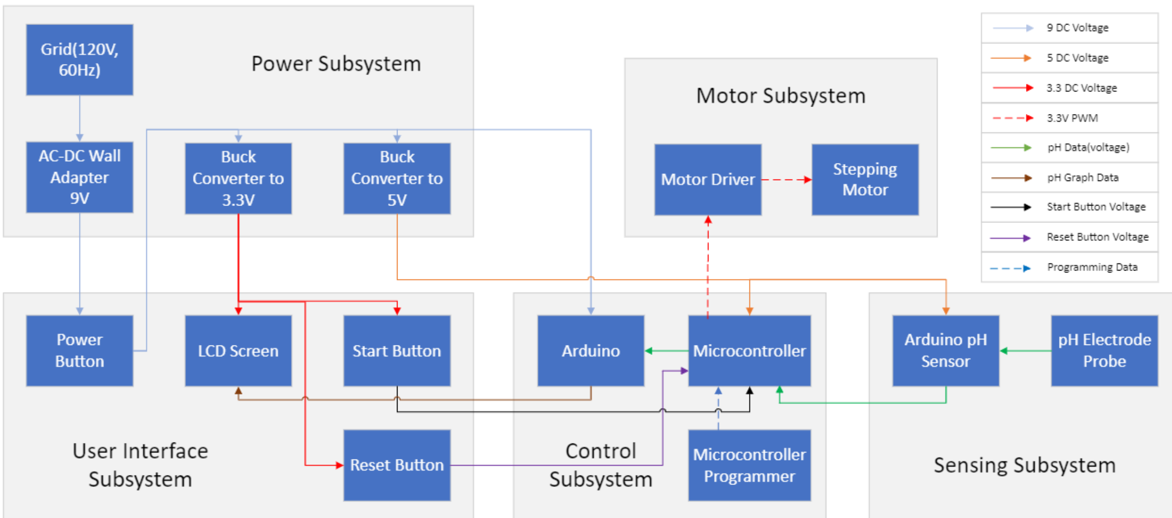

Figure 3: Diagram of Titration System Consisting of The Five Subsystems: Power, Motor, Sensing, Control and User Interface

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 multiple buck converters to shift-down the voltage to different values.

Control Subsystem

- This subsystem contains the microcontroller and Arduino. The microcontroller is used to collect data from the sensing subsystem and to control the speed of the motor system through a PWM signal. The Arduino is used to display the pH and volume data in a graph format on the LCD screen from the User Interface subsystem. It will also take the data derivative 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 reset button, and a power button (Emergency stop). The reset button is used to fill the syringe with the titrate by retracting the plunger. 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. A difference in hydrogen-ions against a ground voltage results in an electrical charge from -415 mV to 415 mV. This charge 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.

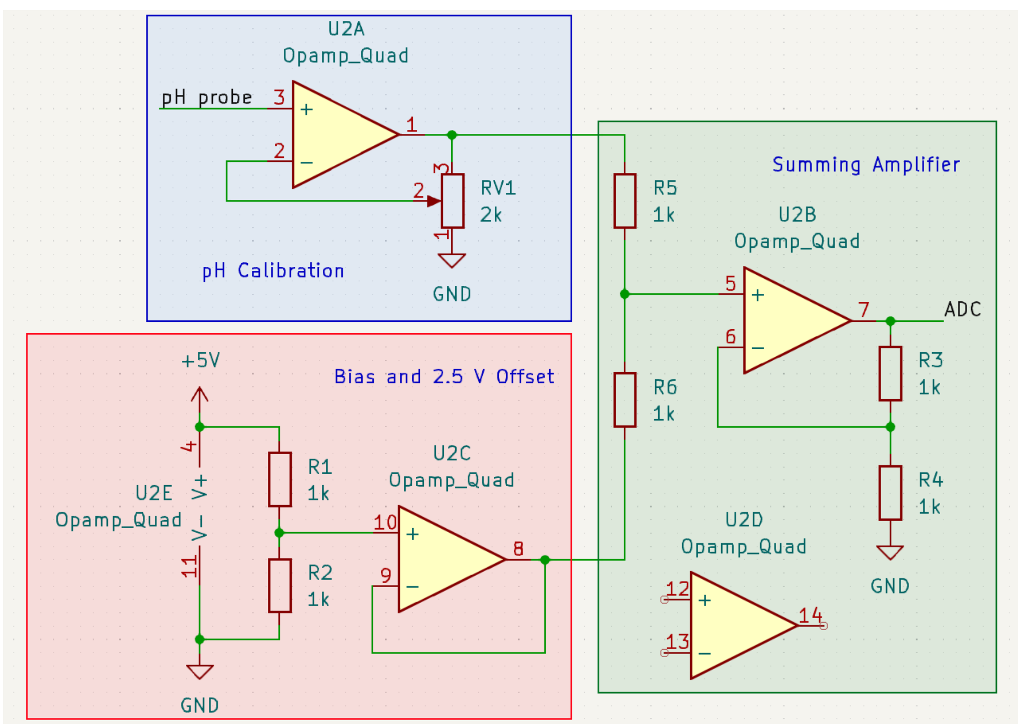

Figure 4: Schematic for pH probe output amplification and scaling to match ADC input pin voltage requirements

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

Subsystem 2: Power System

We will be using an AC (120V, 60Hz) wall to 9V DC adapter. Additionally, we will need to use dc-to-dc adapters for the varying dc voltages needed for the varying subsystem devices including the microcontroller and pH sensor at 5V, and the LCD screen, start and stop buttons, and stepping motor at 3.3V. The 9V 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 9V signal will also be applied to the Arduino in the Control System.

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
• Subsystem produces a 3.3 +/- 0.1 V output to power the LCD screen	• Validate the 3.3 V regulator can accept the voltage produced by the wall adapter. • Ensure the regulator is outputting a voltage at five volts and within tolerance

Subsystem 3: Control

The microcontroller will be taking the live output voltage from the sensing subsystem and process 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 a graph of the current pH level (a titration curve) on the LCD screen in the User Interface subsystem. The x-axis of the graph will be the amount of titrant dispensed into the beaker. This volume amount will be determined based on the step size of the motor. This will be a live graph. The endpoint of the titration curve, also known as the steepest part of the curve, 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 • Adjust the code's constraints to determine how far the plunger is pulled out of the barrel • Click the reset button to determine how far the plunger is pulled • 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 Pulsed signal to control the motor dependent on step size. For a design specific time interval, the syringe will need to produce one drop of titrate.	• Adjust the duty ratio of the pwm depending on your system until one drop comes out per cycle • 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

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.

Requirements	Verification
• The motor driver will convert the microcontrollers pwm signal, into one 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

Subsystem 5: User Interface

The user interface consists of four components: an LCD screen, a start button, a reset button, and a power button. The LCD screen will be displaying the graph(s) created by the Arduino. The reset button is used to return the syringe to its extended state. It will initially remove any excess titrate of the previous test and then pull back, allowing the syringe to fill up. 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 reset button performs the opposite action of the start button, but resetting the plunger to its original position in the barrel of the syringe	• The motor will be programmed to move the plunger the same distance, but opposite direction, as when the start button was pressed • When pressed, the plunger should return to the original closed position

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: 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 have to keep in mind the possibilities of spills in the lab area, although we will be using non-toxic substances in the lab to showcase and test our project, we need to understand the risks and the ability to clean up any issues. According to the Division of Research Safety [3] outlines to keep chemically resistant gloves, googles, acid & base neutralizers, and chemical spill absorption powder. 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.

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.

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