Northeastern University College of Engineering

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# Mechanical Design of Sequential Injection Analysis System for Nitrate Sensing

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## Introduction and Motivation

### General Overview

- Nitrate pollution from fertilizer run-off is degrading our oceans and contributing to 'dead zones'.
- Low-cost sensors allow for large areas of the ocean to be better understood across space and time which will increase our awareness and ability to solve this problem.
- Ion-selective electrode (ISE) arrays are a promising low-cost alternative to more expensive optical sensors but require large amounts of training data to make them effective for field work and sea-water analysis.
- Designing a robotic system that will automate fluid analysis and decrease manual labor required in the lab.

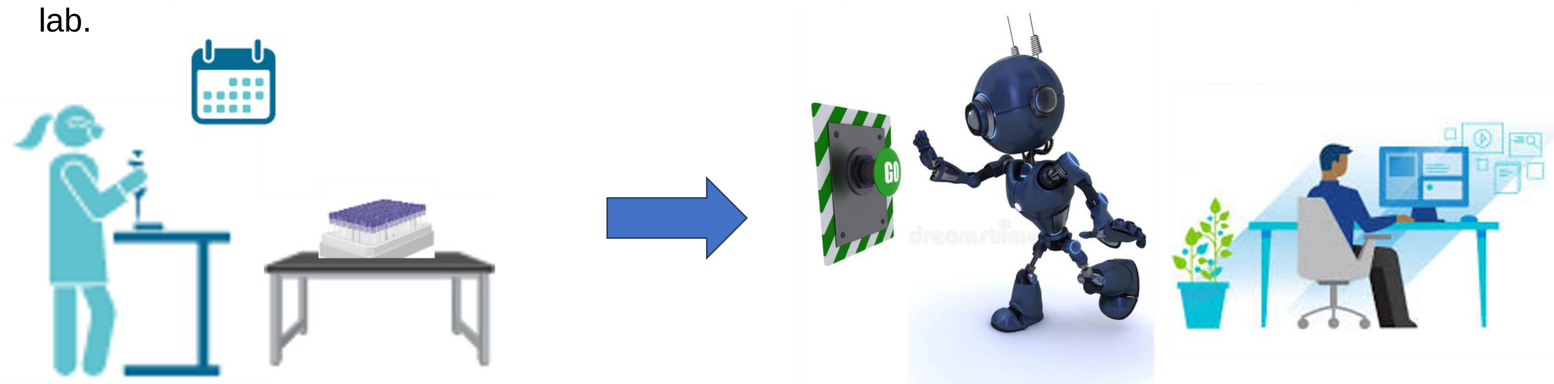


***Figure 1.*** *Animation depicting benefit of overall system design. Automated lab work for improved efficiency, accuracy, and bulk data collection.*

## Approach

Construct a multi-component (sequential injection analysis) system capable of moving fluids through various outports using a multi-position valve head to create an array of varying solution concentrations. Then test those solutions using a flow cell for fluid analysis.

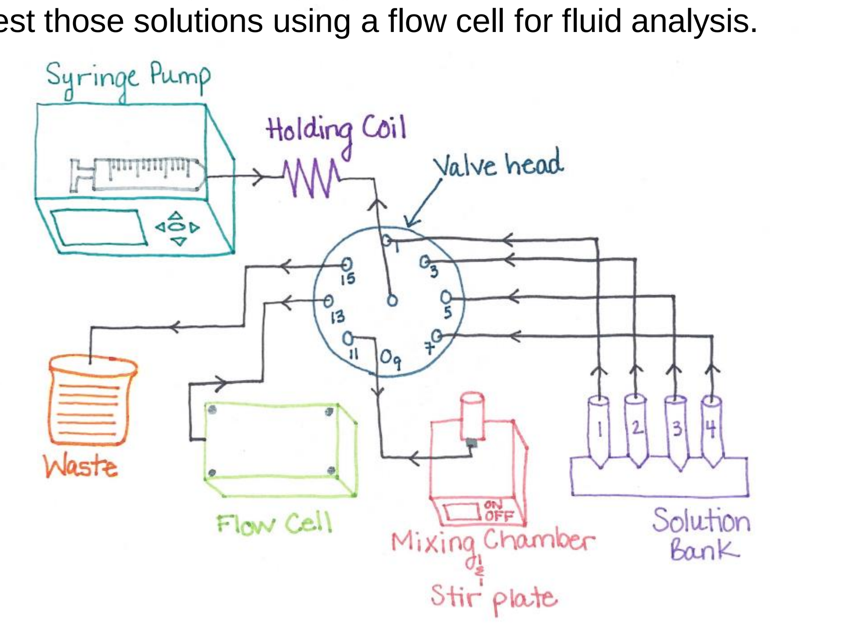



***Figure 2.*** *Sequential injection analysis conceptual diagram depicting various key components (color coded).*

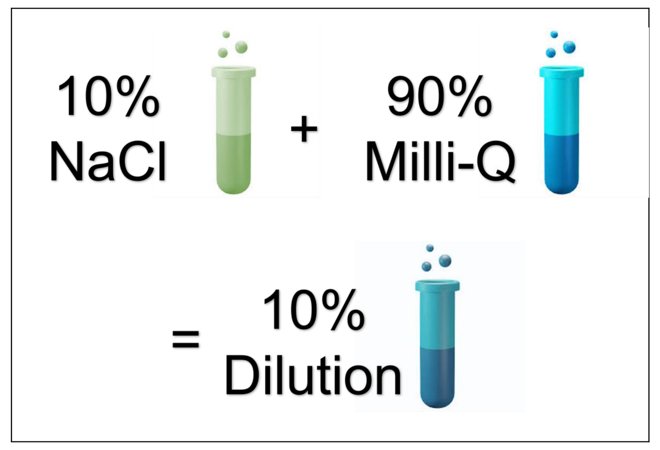



***Figure 3.*** *Concept animation for fluid mixing and solution analysis using SIA.*

### Main System Components

- Syringe Pump
- Multi-position Valve Head
- Mixing Chamber
- Flow Cell
- Solution Bank
- Waste

## Hardware System

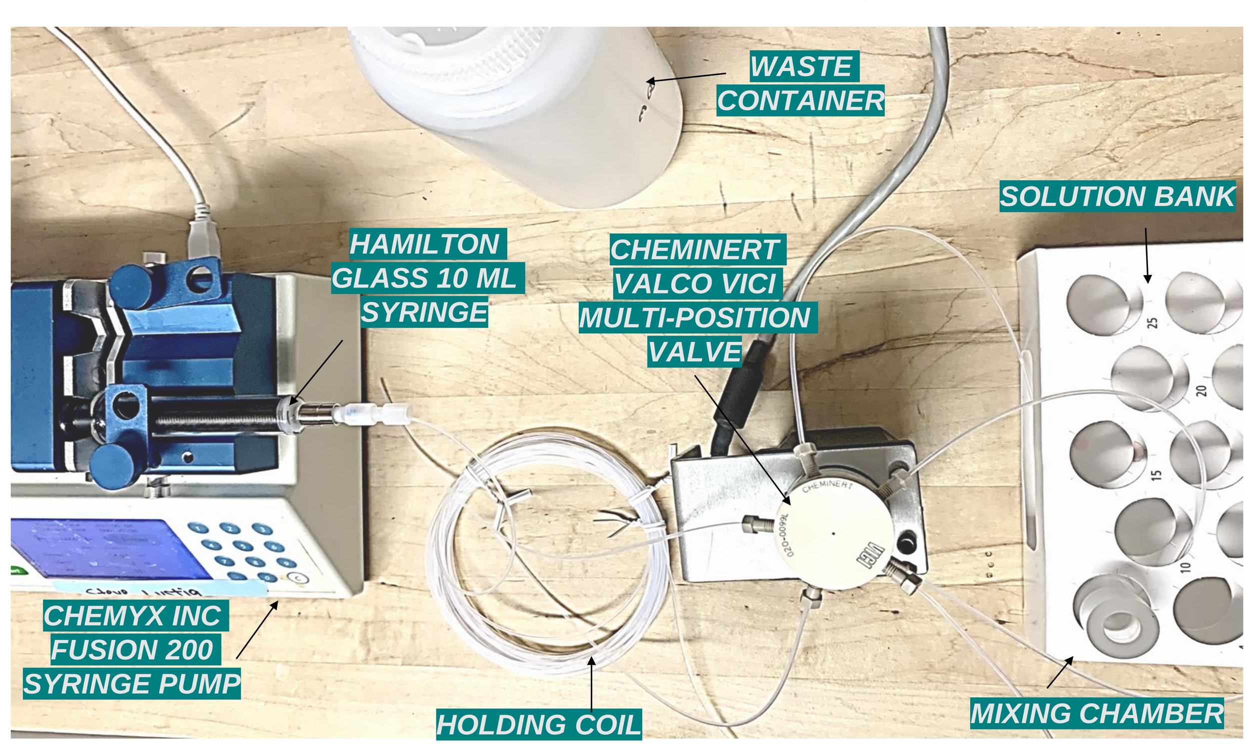
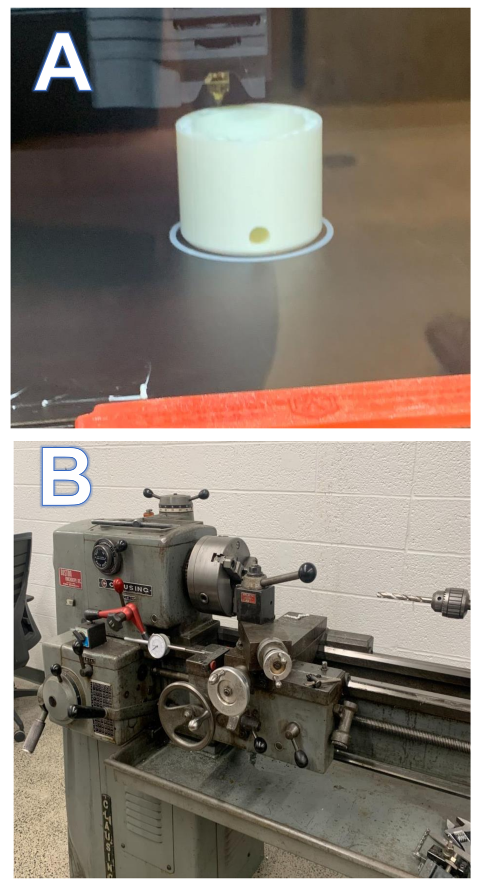
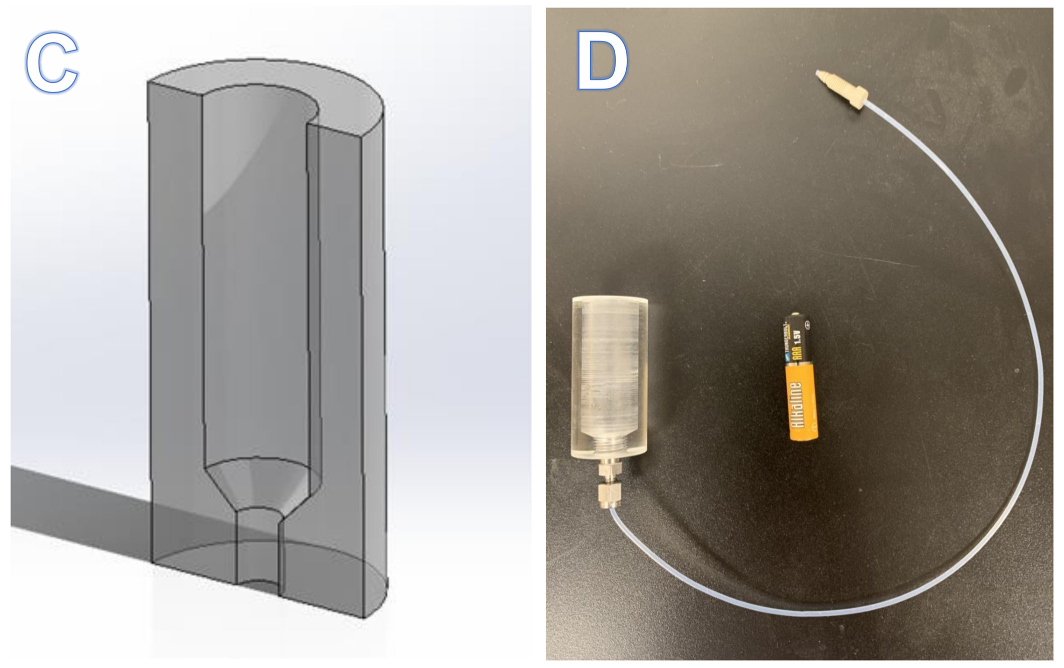



***Figure 4.*** *Image depicting some of the key system components. Syringe pump, multi-position valve head and actuator, mixing chamber, holding coil, solution bank and waste container. NOTE: flow cell and stir plate not depicted in this set up.*

| COMPONENT | DESIGN INTENT |
|---|---|
| Syringe Pump | Bi-directional pump, capacity for 10 ml glass, salt resistant syringe, LabVIEW compatibility preferred |
| Multi-Position Valve | Multi-position valve head capable of bi-directional movement, manual and automated control, RS-232 compatibility for computer connection, LabVIEW compatibility preferred |
| Mixing Chamber | Cast-acrylic mixing chamber with inner chamfer and 10 ml capacity, inlet/outport at base for proper drainage. |
| Fittings | Zero-dead volume fittings for improved accuracy, avoid fittings being part of the fluid flow path. |
| Materials | Teflon, stainless steel, and cast acrylic all used to minimize chemical interferences and increase corrosion resistance. |
| Communication | USB serial port connections for computer control, LabVIEW compatibility preferred but not required. |

## Mixing Chamber Design




***Figures 5.*** *A) 3D Printing prototype of mixing chamber. B) Lathe used to machine the cast acrylic rod into mixing chamber. C) Modified SolidWorks design of mixing chamber depicting inner chamfer and lower thread location. D) Mixing chamber designed to hold up to 10 ml of fluid and have compatibility with magnetic stirrer. Depicted with 1/16" teflon tubing and Swagelok 1/16" stainless steel compression to NPT fitting for zero-dead-volume fluid path.*

### PROGRAMMING AND DESIGN CONSIDERATIONS

**Prototyping:**
- SolidWorks
- Idea Maker
- 3D Printing

**Machining:**

Cast acrylic rod 1 ½" diameter
¼" wall thickness for threading
2" height with 1" inner diameter

## Flow Control Software Design

### FRONT PANEL

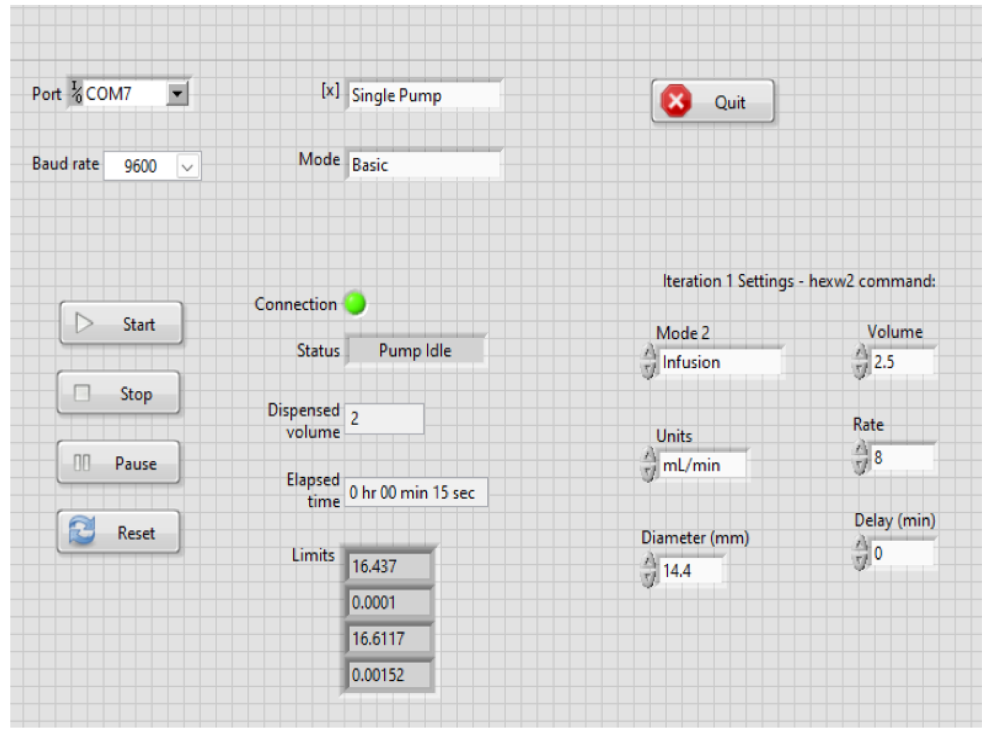



***Figure 6.*** *LabVIEW front panel. User interface for instrument control of syringe pump.*

### LabVIEW

- Allows for instrument control of multiple devices
- Part of the broader software module, this program allows configurations to be applied from front panel and sent to the pump.
- Controls the pump over USB serial port.
- Input settings include, diameter, volume, rate, delay, units and mode.
- Ability to adapt programming or expand into larger, more complex code.

| INSTRUCTIONS | MODE | VOLUME | PORT POSITION |
|---|---|---|---|
| Solution A to holding coil | Withdraw | 4.5 ml | 1 |
| Switch Valve to port 3 | | | |
| Solution B to holding coil | Withdraw | 0.5 ml | 3 |
| Switch valve to port 5 | | | |
| Holding coil to mixing chamber | Infuse | 5.0 ml | 5 |
| Delay 10 seconds to mix | | | |
| Mixing chamber to holding coil | Withdraw | 5.0 ml | 5 |
| Switch valve to port 7 | | | |
| Holding coil to flow cell | Infuse | 5.0 ml | 7 |
| Flow cell to holding coil | Withdraw | 5.0 ml | 7 |
| Switch valve to port 9 for waste container | | | |
| Holding coil to waste | Infuse | 5.0 ml | 9 |

***Table 1.*** *Example instruction for making 10% dilution*

## Proof of Concept Testing

### Phase 1: Hardware Testing

| COMPONENT | TESTING FOR | STATUS/OBSERVATIONS |
|---|---|---|
| Syringe Pump | Mode | Both working |
| | Force/rate | Must remain within syringe limitations according to chart |
| | Air bubbles | Small air bubble observed on withdraw |
| | Syringe fit | Adjusted to size manually |
| Valve | Port position | 10 positions, 20 ports |
| | Inlet/vent | Inlet = odd, vent=even |
| | Fluid flow path | Reverses same path |
| | Leak down test | Vent does not release fluid pressure after switching port |
| Mixing Chamber | Volume assessment | Approximately 10 ml |
| | Fluid flow | Fluid reverses initial path as desired |
| | Magnetic stirrer fit | Fits with room to move |
| | Air on withdraw | Not if withdrawn slowly |
| Holding coil | Volume assessment | Approximately 8.0 ml |
| Magnetic Stir Plate | Stand for mixing chamber | Works even with stand above it |

***Table 2.*** *Initial testing observations from individual components and overall system integration and functionality.*

- Testing of all system components for functionality and system integration.
- Fluid volumes and flow paths were also observed and tested.
- Programs such as LabVIEW and Hyperterminal used to verify automated control of valve and syringe pump individually

### Phase 2: Conductivity Testing for Fluid Concentration Verification

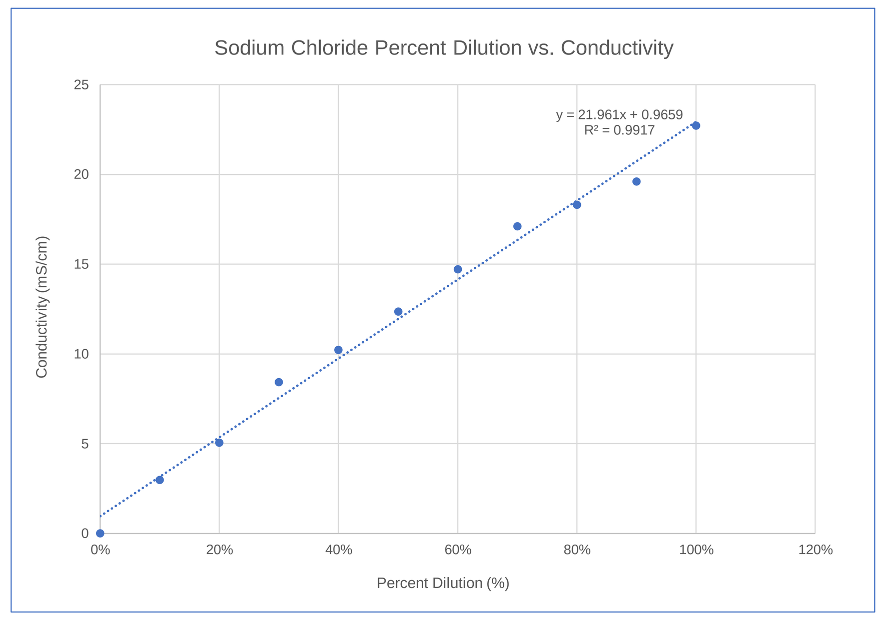



***Figure 7.*** *Sodium chloride conductivity testing results depicting linear fit.*

- Conductivity testing demonstrated that the current mixing procedure for solutions is making expected concentration levels accurately

| Milli-Q® (ml) | 0.25 M Sodium Chloride (ml) | Concentration (M) | Conductivity (mS/cm) | Expected Conductivity (mS/cm) | Percent Error (%) |
|---|---|---|---|---|---|
| 8.0 | 0.0 | 0.000 | 0.00134 | 0.000049 | 96.343 |
| 7.2 | 0.8 | 0.025 | 2.98 | 2.561 | 14.072 |
| 6.4 | 1.6 | 0.050 | 5.04 | 4.970 | 1.379 |
| 5.6 | 2.4 | 0.075 | 8.42 | 7.304 | 13.256 |
| 4.8 | 3.2 | 0.100 | 10.22 | 9.584 | 6.227 |
| 4.0 | 4.0 | 0.125 | 12.36 | 11.822 | 4.356 |
| 3.2 | 4.8 | 0.150 | 14.70 | 14.025 | 4.590 |
| 2.4 | 5.6 | 0.175 | 17.09 | 16.200 | 5.210 |
| 1.6 | 6.4 | 0.200 | 18.30 | 18.348 | -0.264 |
| 0.8 | 7.2 | 0.225 | 19.60 | 20.474 | -4.460 |
| 0.0 | 8.0 | 0.250 | 22.70 | 22.579 | 0.532 |

***Table 3.*** *Conductivity results for varying concentrations of sodium chloride.*

## Conclusions and Future Work

**Conclusions:**
- Preliminary tests showed promising results from overall functionality of the system and all integrated components.
- Conductivity testing followed a linear fit as expected showing mixing procedures are accurate and working as we hoped.

**Future Work:**
- Develop a priming procedure for the pump and all fluid lines to decrease possibility of air in the syringe or tubing.
- Further development of flow cell component and integration within system
- Continue programming LabVIEW for simultaneous instrument control of valve and pump
- Increase compound mixtures and solution bank

## Funding

SCHMIDT MARINE TECHNOLOGY PARTNERS

REU-PATHWAYS

MIT SEA GRANT COLLEGE PROGRAM

NSF National Science Foundation