

# Options for Water Reuse in Optical Fiber Production



**WPI**



*A Furukawa Company*

## **A Major Qualifying Project**

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## **Abstract**

The goal of this MQP report was to investigate the manufacturing process of optical fibers at the OFS plant in Sturbridge, MA. Our goal was to reduce water usage to encourage environmental benefits and cost-saving initiatives through our project work. First, we focused on constructing a lab-scale process to deionize the wastewater effluent stream so it could be repurposed. We then redesigned a seal water recirculation system that would eliminate a major source of single-pass water within the plant. The series of recommendations presented in this report and to the OFS team foresee large costs and water savings.

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## **Introduction**

OFS is an optical fiber production company located in Sturbridge, Massachusetts. They use water from the Town of Sturbridge in manufacturing, and once it has been used in the process it is sent to discharge to Sturbridge sewers. There is a cost to both this intake and release of town water. With the goal of saving money and improving sustainability in the plant, the employees of OFS presented our team with a two-fold problem. The first was to determine a way that the effluent stream of the wastewater could be recycled to a different part of the process. Treated water was sent immediately to the sewer, but ideally all or a portion of this water could be used again in a specific part of the manufacturing process. The second objective was to redesign a seal pump water recirculation system for the scrubber pumps located within the process. This is a source of single-pass water, but a recycle system can eliminate the plant's continuous water consumption.

## **Background**

### **About OFS**

OFS is a world-leader in design, manufacturing and production of optical fiber cables, connectivity, FTTX and photonics solutions, headquartered in Norcross, Georgia. The organization is part of the larger Furukawa Electric Company, a multi-billion-dollar company that has been in operation since 1884 and went into optical fiber manufacturing in 1974. OFS was the first to achieve commercial production of optical fibers, as well as the first to offer fiber optic products in the market that met the industry standards of today. These initiatives to develop cutting-edge and industry-leading technologies are supported through the best-in-class research delivered within OFS Laboratories (About OFS). The OFS Sturbridge facility only produces optical fibers.

### **Optical Fiber Production**

[Redacted] This portion is deemed confidential for the sponsor.

### **Wastewater Treatment**

The town of Sturbridge has specific regulations for water being sent to the sewer. In order to meet the proper wastewater standards, OFS has a wastewater treatment (WWT) process located downstream on the site. Process water is collected in a receiver tank. Water in this tank has both a low pH (2-5) and high levels fluoride (550-600 ppm), so both parameters need to be pretreated prior to discharge.

The WWT process contains four reactors in series. The first reactor adjusts the pH of water to between 6-9 standard units by adding a necessary amount of Sodium Hydroxide (NaOH). The second measures the amount of Fluorine located within the reactor and adds enough Calcium Chloride (CaCl<sub>2</sub>) to form Calcium Fluoride (CaF<sub>2</sub>) particles. The third reactor receives the water stream from reactor 2 and the water leaving the Ge recycling process. These two water streams are combined with a coagulant in order to encourage solid formation of CaF<sub>2</sub>. Finally in reactor four a flocculant is added to accelerate the rate of flocculation of CaF<sub>2</sub> solid particles in the water, i.e. encourages quicker separation of solid and liquid particles.

From reactor 4, the water is sent through a clarifier that allows for the removal of solid CaF<sub>2</sub> from the wastewater. The wet solid CaF<sub>2</sub> is then sent through a filter press, this dry solid CaF<sub>2</sub> can be deposited into a drum, which OFS then sends offsite, while the residual water from the filter press



is sent back through the clarifier. Before the wastewater can be completely sent to the sewer, it is collected from the clarifier into the effluent tank. The final effluent is tested through probes to ensure one final time the water meets the town of Sturbridge sewer regulations and standards.

### **Seal Water System**

During the fiber manufacturing process, glass rods are produced by passing a controlled mixture of Si, Ge and P chlorides through a high purity glass tube to create a glass rod. The gasses and emissions from MCVD to be treated before being released in the atmosphere. To clean the air, it is sent through a scrubber with a Series 1500 horizontal pump. The seals on the pump are continuously run with seal water. This is an absolute essential part in OFS manufacturing, as failed seals could shut down the entire manufacturing process. The pump has four mechanical seals that are flushed by 0.5 gallons of water per minute to stay lubricated to maximize their lifetime. In total, this is a rate of 2 gallons per minute, 2,880 gallons per day, 1,008,000 gallons per year (assuming around 350 days of operation per year), of town water being supplied to the four pumps every year. Town water currently sits at the cost of \$0.0693 / ft<sup>3</sup> and the cost of water sent back directly to sewer is another \$0.0982 / ft<sup>3</sup> cost. At OFS, this water is then sent directly to the facility's sewer system, but this was not always the case. A seal water recirculation system was put into operation at the OFS facility but has been out of commission for over 10 years due to issues with controls complexity and overheating.

To complete the team's objectives of conserving water and creating cost saving recommendations for OFS, the team planned to propose a redesign of the seal water recirculation system. The design would utilize the recirculation system still installed within OFS facility in addition to recommended changes to eliminate the use of single-pass water.

### **Process Controls**

Process controls are used in industry to maintain the desired conditions of an operation. To control conditions, indicators and transmitters are used to send current data to the control system. There are different indicators for each condition including temperature, flow rate, and level of product in a vessel. Thermocouples are temperature indicators widely used in industry due to their low cost and portability. Flow meters include rotameters or digital meters and are used to measure the flow rate of a product through a pipe. The flow rate can then be controlled by opening or closing a valve. Lastly, level indicators measure the amount of liquid in a vessel by using a sensor. Based on the level, automatic valves can allow more liquid into or out of the vessel.

Using the data from the transmitters listed above, different control systems can change the condition of the system. One type of controller is a programmable logic controller (PLC). A PLC is programmed with logic that specifies outputs based on certain inputs. A PLC takes an input from a transmitter and changes the output based on its programming (Seborg). For example, if a valve was supposed to open when a temperature transmitter reach 100°C, the PLC would be sent this transmission data, scan the program, and send a signal to open the valve if the temperature was 100°C.

## Ion Exchange Columns & Thomas Kinetic Equation

A popular and cost-efficient way to treat contaminated water is to use ion exchange columns packed with a specific resin material. Water whose contaminants are fully dissolved in ionic form can effectively be treated by running this contaminated water through a resin material. Resin material is microporous, entirely insoluble in organic materials alongside waste, and consists of a polymer backbone with a functional group attached to the backbone. Generally, these resins are sold in an ionic form in which an ion is pre-loaded, i.e., bonded to the functional group (Mazille, 2019).

The strength of the bond between the ion and the functional group is relatively weak. Therefore, when contaminated water passes through the resin, the ionized contaminants have a stronger affinity to kick the resin's pre-loaded ion out and bond with the resin at the functional group. This is how the term ion exchange was coined. The resin acts to exchange its initial loaded ion with the contaminated ion in the process fluid.

Resins generally come in 2 basic forms, cation and anion forms with few exceptions for special cases. Resin selection is based on the ion strength of the contaminant alongside the desired goal of either demineralization or softening. Demineralization fully removes any contaminant while softening removes most of the contaminants to a point where the water becomes potable (Basics). To accomplish either goal depends on the functional group. In demineralization applications, there are two ion exchange columns: one for the anion and one for the cation resin. Additionally, the column, or bed, can contain a mixture of both resins (mixed bed). The functional group is negatively charged in cation resins while the anion resin functional group is positively charged. In resins used for softening, the cation contains  $\text{Na}^+$  ions bonded to the functional group, while the anion resin contains  $\text{Cl}^-$  ions bonded to the functional group. Therefore, once the water is fully treated, its contaminants are replaced with either  $\text{H}_2\text{O}$  or  $\text{NaCl}$  depending on the application. After a time, the resin will become oversaturated with contaminants and can no longer filter out contaminants. When this occurs, the resin must be regenerated. This is done usually by backwashing the resin with a brine solution for softening purposes or an acidic and basic solution for demineralization purposes (Basics). Due to the inherent simplicity of the system, ion exchange has served to treat wastewater, industrial demineralization, ultrapure water production, etc. (Dupont, 2022).

Once a resin is selected, it must be tested under pilot scale conditions to determine the magnitude of scale up. For a fixed-bed column, performance is quantified by obtaining a breakthrough curve, which is the contaminant-effluent concentration versus throughput volume profile in the column. This is done by the Thomas Kinetic model shown in **equation 1**

$$\ln\left(\frac{C_0}{C} - 1\right) = -\frac{kC_0}{Q}V + \frac{kq_0M}{Q} \quad (1)$$

To use this equation to scale up, one must run water over time at a constant flow rate  $Q$ , where the cumulative volume that has passed as a function of time is  $V$  (i.e.,  $V = Q \cdot t$ ), through the column of known resin dry mass  $M$ , and measure its respective outlet concentration,  $C$ . When using the equation to determine scale up parameters,  $Q$  becomes the total volume ran through the scaled-up

column (in L/day). Additionally, when scaling up,  $V$ , becomes the total volume run through the column before regeneration must occur.

During pilot scale testing, all parameters are readily determined except for  $k$  and  $q_0$  which are the Thomas kinetic coefficients of the specific resin.  $k$  is the kinetic constant, while  $q_0$  is the maximum solid-phase concentration of the absorbed solute/contaminant ion (often written in terms of  $\frac{g \text{ solute}}{g \text{ resin}}$ ). Thus, finding  $q_0$  is relevant as it quantifies the ion sorption capacity of our resin. Looking at **equation 1**, one can see it follows the general slope form,  $y=mx+b$ . With:

$$\overbrace{\ln\left(\frac{C_0}{C} - 1\right)}^y = -\overbrace{\frac{kC_0}{Q}}^m \overbrace{V}^x + \overbrace{\frac{kq_0M}{Q}}^b \quad (2)$$

Therefore, one can run aliquots of volume through the column and measure the outlet concentration. Since the initial and effluent concentrations and throughput volumes can be directly measured, one can easily plot the *linearized* breakthrough curve,  $\ln[(C_0/C)-1]$  vs.  $V$ . One can then take the slope and the y-intercept of the curve to determine the relevant coefficients  $k$  and  $q_0$ , respectively.

With  $k$  and  $q_0$  known, the scale up now only involves determining the mass of the resin required. The engineer can specify the remaining variables by selecting a desired outlet concentration with a known inlet composition, regeneration period, and expected flow rate. Once the mass of the resin is solved for, the engineer can then select a column based off the resin mass needed.

## Methodology

### Preliminary Testing

To complete the objectives for this project, it was vital to first conduct preliminary tests on water samples taken from various sources in the process and identify potential areas for water recovery and reuse. During an initial visit to the OFS manufacturing site, seven water samples were taken from the following locations:

- 1) Wastewater treatment (WWT)
  - a) Receiver tank
  - b) Reactor tank 1 (RX-1)
  - c) Reactor tank 2 (RX-2)
  - d) Effluent tank
- 2) Chiller outflow
- 3) Ge recycle stream prior to WWT
- 4) Town water from the plant's faucet

While tests were conducted on each of these samples to analyze their components, we were primarily interested in the chemical make-up of the effluent stream and its variation from town water.

To confirm the absence of significant organic contaminants in the company's water sources, Fourier Transform Infrared (FTIR) Spectroscopy was conducted on each of the samples. FTIR is an effective analytical method that utilizes infrared radiation to detect functional groups, such as hydrocarbons, carbonyls, and amines, and characterize components in unknown samples. This form of spectroscopy delivers a spectrum that plots wavenumber ( $\text{cm}^{-1}$ ) against either absorption or percent transmittance. For these tests, percent transmittance was chosen as the more optimal option.

To further investigate potential sources for water reuse, we tested for the presence and concentration of calcium in the samples. As previously mentioned, copious amounts of calcium chloride are added during the WWT process to reduce the concentration of fluorine, which can result in a high concentration of calcium in the effluent. To seriously consider the effluent stream as a potential area for recycling, it was important to determine the calcium level in these samples and compare it to the water commonly used in their manufacturing operations, town water, as surplus calcium can lead to unwanted solid build-up in equipment and side reactions. Inductively coupled plasma (ICP) mass spectrometry was used to find the concentration of calcium in diluted samples of the town water and effluent streams.

In addition to calcium, chlorine contamination was taken into consideration. A Hach DR6000 UV-VIS Laboratory Spectrophotometer was used in tandem with colorimetric assay free and total chlorine test powder pillows to determine the free and combined chlorine concentrations in both effluent and town water samples. Based on our results from chlorine testing, which can be found in more detail in the preliminary testing portion of the results and discussion section, we decided chlorine contamination was not a major concern.

With the completion of these preliminary measurements, we determined that configuration of an ion exchange column with the appropriate resins to remove surplus calcium and fluorine was necessary to bring effluent water to a commensurate quality with town water.

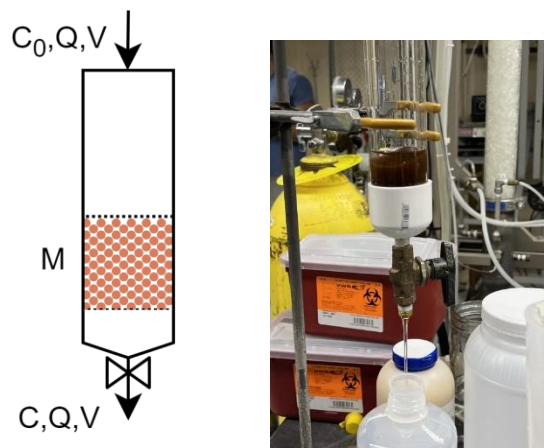
## **Ion Exchange**

### **Anion & Cation Resin Selection**

A strong acid cation resin (Purolite C100) and Type I strong base anion resin (Purolite A400) were selected as the packing materials for the ion exchange column experiments. Both resins are composed of a gel polystyrene crosslinked with divinylbenzene; however, the cation resin is functionalized with a sulfonic acid group, while the anion resin is functionalized with an ammonium group. Off the shelf, C100 and A400 are provided in hydrogen form and hydroxide form, respectively. These mobile hydrogen and hydroxide ions are what will be exchanged with our process ions of interest e.g., calcium, fluorine.

### **Operating the Bench-Scale Ion Exchange System**

The bench-scale ion exchange column was fabricated, as shown in Figure 1.



**Figure 1.** Left: Schematic of the packed bench-scale column. Right: Our group's Column.

Effluent water from OFS (which we will now refer to as “OFS Water” to prevent confusion with the effluent from the ion exchange columns) was collected to serve as the working fluid for our ion exchange experiments and stored in a refrigerator to reduce the gradual escape of any volatile ions into overhead gas volume. To obtain initial ion concentrations of the OFS water, a fluoride field test kit was used to measure fluoride concentration, a colorimetric assay was used to measure free and total chlorine concentrations, and (on a later day) one more sample was sent to ICP (Inductively Coupled Plasma) spectroscopic analysis for calcium ion concentration.

The column was prepared by first rinsing it with deionized (DI) water. Then, the dry cation resin was weighed out and packed into the column. For a given pre-determined throughput volume, the sample of OFS water was run through the column, and the effluent stream was completely collected in a storage bottle. A small-scale positive displacement piston pump was used and ran at 2.17 L/h through the column. When testing the respective resins, a cation resin mass of 274.81 grams and an anion resin mass of 248.45 grams were placed in the column. Outlet concentrations were taken at volume increments of 0.15, 0.3, 0.5, 0.8, 0.9, 1, 1.2, and 1.5 L. Volume was measured in terms of how much volume has passed through the column during the experimental period.

~10 mL aliquots of the effluent were also transferred to a vial for calcium testing using an Apera Instruments YD300 Portable Water Hardness Tester field tester. Due to the expected outlet concentrations being relatively low, TISAB was also used to emphasize the tester's capability to detect the calcium in the column effluent. These outlet samples were stored in ice or refrigerator. This was done for 8 different throughput volumes.

Following this, the column was emptied out such that the cation resin was removed and then rinsed with DI water. In a similar fashion, the column was now packed with anion resin. The 8 different column effluent waters collected from the cation resin experiments were then one at a time run through the column and collected into a beaker; a ~10 mL aliquot of the effluent was also transferred to a vial for destructive analyses of fluorine concentration. A Hanna Instruments High Range Fluoride Calorimeter fluoride field test kit was used to measure fluoride concentration in these samples.

## **Scale-Up Column Sizing**

### **Breakthrough Curve Analysis**

With each resin, there was at least one ion contaminant of interest we wanted to capture: cation resin for calcium ions and anion resins for fluoride ions. Therefore, two breakthrough curves were needed to determine the calcium ion sorption capacity of the cation resin and the fluoride ion sorption capacity of the anion resin. Each (linearized) breakthrough curve was obtained by plotting  $\ln[(C_o/C)-1]$  vs.  $V$ . The resulting slope and y-intercept were used to determine  $k$  and  $q_0$  for each resin-ion system. With these kinetic expressions we can then determine resin mass needed. With a predetermined outlet and inlet concentrations for each resin, we can determine the mass of resin required to reach the desired outlet concentration by solving for resin mass in the Thomas Kinetic Equation depicted in equation 1. With the resin mass known, the column can be sized in compliance with the resin mass required.

### **Heuristics & Equivalency**

Should the creation of a breakthrough curve not be successful general heuristics for resin mass and column sizing can be used. To determine resin mass, the resin total capacity can be used. Determining the mass of contaminant run through the column per year can then be used to determine resin mass required by dividing the mass of contaminant run through the column by the total capacity as referenced in appendix B. To determine heuristics for column sizing, Purolite was contacted. Purolite verbally provided heuristics for column sizing and operations. Generally speaking, the flow rate into the column should be 2-3 gpm/ft<sup>3</sup>, a linear velocity of 8-12 gpm/ft<sup>2</sup>, and a column diameter that can accommodate 36-60 inches of resin bed depth. Additional height should be considered if regeneration is occurring inside the column itself since backwash expansion must be considered.

### **Recirculation System**

A second source of water that could be utilized in both a sustainability and economic savings standpoint is the seal water used to lubricate the scrubber recirculation pumps. The town water supplied to the mechanical seals currently enters the pumping system through a 2" diameter pipe, and through a piping system with two manifolds. There are eight pumps with eight mechanical seals that are available in the scrubber system, but only four are used in operation at a time. The decommissioned recycled piping lines and seal water collection tank is still attached to the rest of the piping system and in theory could run if the proper valves were open. Though, because of the importance seal water has on the system, the issues of the old recirculation need to be addressed by redesigning the system.

To complete this redesign, the key concerns and issues that caused the shutdown of the recycle needed to be addressed. Through a conversation with an OFS Manager who has witnessed the installation and failure of the system, we learned one issue was the temperature rise due to the friction in the piping. This could also be witnessed by seeing the seal pump water tank, which was bulging likely due to its inability to support the temperature of the water it once held. Another concern was contamination, like unwanted organisms or bacteria growing in the tank or pipelines. There were pH probes testing the water to address these concerns, but flow meters would still occasionally be clogged by contaminants. To address these concerns and redesign a recycle system

we created a theoretical pumping system. The ideal system would maximize water reuse, while avoiding contamination and overheating concerns. Our system focuses on strategically inputting controls and the recommended design uses equipment from vendors recommended by OFS staff. We constructed P&IDs, layout diagrams and estimated a cost analysis for the seal water recirculation system design.

## Results and Discussion

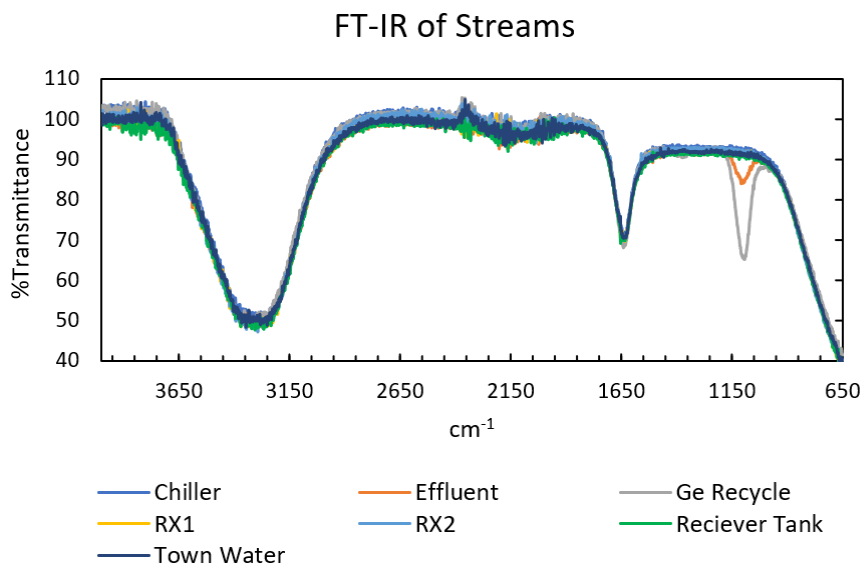
Using the methods previously described, we collected preliminary water sample data, collected data from a bench-scale ion exchange column, and outlined a recirculation system design. The results from our research and tests are discussed in the following sections.

### Preliminary Testing

We first tested water samples for various areas of the plant to determine concentrations of potential contaminants. Tests completed include Fourier transform infrared spectroscopy, inductively coupled plasma (ICP) mass spectroscopy, and UV-VIS spectrophotometry.

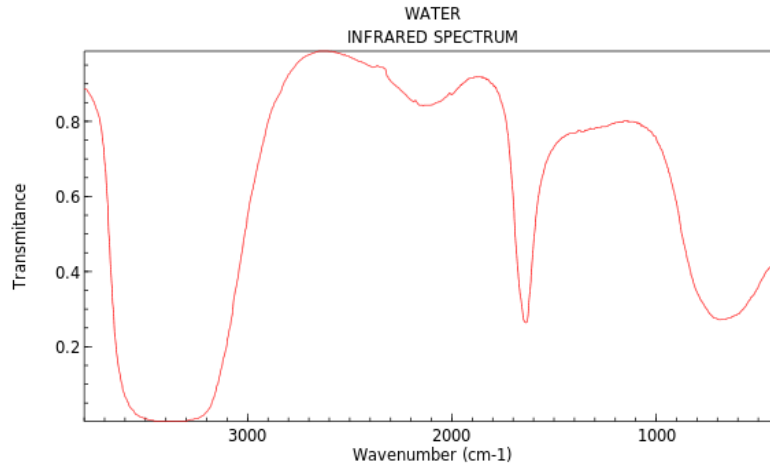
### Fourier Transform Infrared (FTIR) Spectroscopy

Using FTIR spectroscopy, we were able identify any potential organic contaminants in the water samples. Figures 2 and 3 display the spectra produced by the FTIR instrument for the water samples, in comparison to the expected FTIR spectrum found through research.



**Figure 2.** Experimental IR spectra for water samples





**Figure 3.** Expected IR spectrum for water.

As seen by the spectra above, the water samples do not appear to have any major contaminants as the experimental spectra is very comparable to that of the expected; the primary peaks observed around wave numbers  $3300\text{ cm}^{-1}$  and  $1600\text{ cm}^{-1}$  were reasonable and representative of the O-H stretch and H-O-H scissors of the water molecule, respectively. The similarity between these two plots verified that the water samples were, indeed, just water and did not contain any unforeseen compounds.

### Inductively Coupled Plasma (ICP) Mass Spectrometry

We knew there was buildup up of calcium particles within the wastewater treatment process, so we used Inductively coupled plasma (ICP) mass spectrometry to compare calcium concentrations in samples town water and effluent streams. From this testing, the following results were found:

**Table 1.** Concentration of calcium in town water and effluent streams, found through ICP testing.

Water Source	Unfactored Concentration (ppb)	Concentration (ppb)
Town Water	321.9	3,219.3
Effluent	1,127.8	11,278.1

While the effluent stream does appear to be significantly more concentrated with calcium than town water, it is still considered “soft” as it lies below the 60-ppm threshold, which is sufficient for OFS’ processes. However, it is still beneficial to implement a system for removing this excess calcium and lowering the level down to town water’s levels to avoid further accumulation and future issues from arising. From this information, we settled on a method for removing approximately 8,000 ppb (or 8 ppm) of calcium from the effluent to match town water’s standards.

### UV-VIS Spectrophotometer

To determine free and combined chlorine concentrations in the town water, and effluent water a Hach DR6000 UV-VIS Laboratory Spectrophotometer was used in tandem with colorimetric assay free and total chlorine test powder pillows. This analysis resulted in the following concentrations:

**Table 2.** Concentration of chlorine in town water and effluent streams, found through spectrophotometer testing.

Water Source	Free Chlorine Concentration (ppm)	Combined Chlorine Concentration (ppm)	Total Chlorine Concentration (ppm)
Town Water	0	0.02	0.02
Effluent	0.06	0.15	0.21

When conducting our analytical tests, OFS sent similar water samples to an external lab for additional testing to confirm the accuracy of our findings and equipment. Results from the Micobac lab were as follows:

**Table 3.** Concentration of chlorine in town water and effluent streams, found through external lab testing.

Water Source	Free Chlorine Concentration (ppm)	Combined Chlorine Concentration (ppm)	Total Chlorine Concentration (ppm)
Town Water	<0.05	<0.05	<0.05
Effluent	0.16	0.05	0.21

The tables of data above illustrate the difference between chlorine levels in town water and effluent. Since each source maintains a relatively low chlorine level, and the effluent stream does exhibit a higher but still relatively concentration the need to filter out chlorine for industrial use of the recycled water is not necessary. Overall, outcomes from both testing types were very comparable, and confirmed that we did not have to consider the filtration of chlorine ions due to the intended use of the recycled water being for industrial applications.

### Ion Exchange

#### Anion Resin for Fluoride Removal

The bench-scale ion exchange column was then packed with an anion resin to remove fluoride from the effluent water stream. Removing fluorides is necessary to reuse the effluent stream as we want to restore the ion concentrations to a level at or near town. Daily logs of the effluent steam are maintained by the OFS plant, and the concentration ranges from 5-30 ppm, where Sturbridge

town water is limited to a range of 0.6-1.1 ppm. The anion resin column was run twice, and in both scenarios, we had unforeseen results.

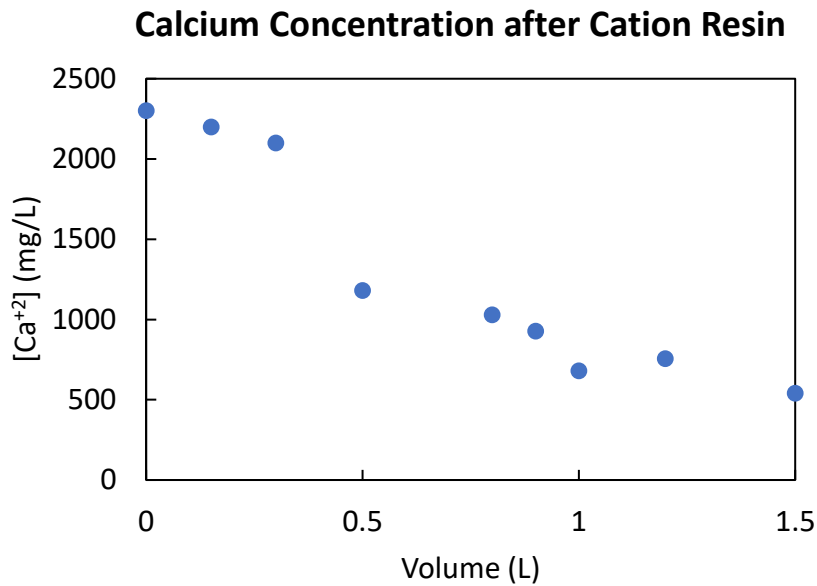
In both column runs the fluoride concentration did not get reduced, and in some cases even read more, than the initial concentration. The resin used to pack the column, sourced from Purolite, was a demineralizer resin, meaning in ideal operating conditions ions should be completely removed. Although we did not predict total removal in our bench-scale process, the idea that water passing through a packed column would result in minimal decrease or a rise in ion concentration was not possible. The measured fluoride concentrations and failed breakthrough curves can be seen in Appendix A.

As a team, we knew an increasing fluoride concentration after passing through a resin designed to remove fluoride ions was not feasible, so we weighed the potentials of what was going wrong in our process. In addition to the ion exchange column built, we performed a jar test, where the effluent water sat in a jar with a mass of resin for 30 minutes to see if the issue was that within the column equilibrium was not being reached. Even in this case, the fluoride concentration did not decrease.

We believe, knowing that in theory our constructed anion exchange columns and methods should work, that the fluoride meter or testing method was faulty. An in-series cation/anion column design was a suggested procedure for bench-scale testing when trying to deionize water samples (Geraud, 2016). Before taking our recommendation to construct an anion ion exchange column for fluorides, we recommend constructing further bench-scale ion exchange columns and testing the fluoride concentrations with a better, verified analytical tester. This can ensure that the methods and anion resin used was not the issue, but the inconsistent and unfeasible readings were in fact due to a faulty colorimeter checker.

### **Cation Resin for Calcium Removal**

In addition to the removal of fluorine ions, we performed a pilot-scale ion exchange with a strong acid cation resin (C100) packing to remove excess calcium ions from the effluent stream and deter the buildup of solid calcium residue during reuse. Based on the results found through preliminary ICP mass spectrometry testing, there was approximately three to four times as much calcium present in the effluent when compared to town water: 3.22 ppm in town water and 11.28 ppm in the effluent. This significant difference between calcium concentrations prompted us to run the cation resin column as a means of bringing the effluent's calcium to a level comparable to that of town water. Table 15 in Appendix B displays the calcium measurements taken for each of the predetermined volumes that flowed through the ion-exchange column. Using the raw data from this trial, we constructed the following figure, which plots sample volume against the concentration of calcium ions:



**Figure 4.** Plot of sample volume against calcium ion concentration, taken from raw data for cation resin ion-exchange trial.

As evident from figure 4 above, a distinct, roughly sigmoidal trend emerges that opposes the expected relationship between sample volume and calcium concentration: larger volumes resulted in smaller concentrations of calcium, whereas smaller volumes ran through the column yielded higher calcium levels. We also measured and determined an initial calcium concentration of 2300 mg/L for the effluent when using the field tester. Each of the samples contained a lower calcium concentration than the initial effluent stream, which demonstrated the cation resin’s ability to successfully remove amounts of the positively charged ions. However, the trend observed in the data and plot is not intuitive as we would anticipate an increasing concentration of calcium after sustained use of the resin due to increased exhaustion and growing contamination of the column.

The established relationship between volume and  $\text{Ca}^{2+}$  concentration also negatively impacted our ability to construct and utilize a breakthrough curve for the cation resin because the direction observed is the exact opposite of the expected trend; our attempted breakthrough curve and Thomas kinetic equation can be found in Figure 14 of Appendix B. As previously mentioned, the linearized breakthrough curve plots sample volume against the natural log of initial concentration divided by sample concentration minus one, which allows us to determine the necessary values,  $k$  and  $q_0$  for scaling our pilot-scale ion exchange column up to an industrial-scale. Since the slope of our “breakthrough curve” is positive, the rate constant and the maximum solid phase concentration of exchanged solute values yielded would not be feasible and would render our breakthrough analysis futile for scale-up purposes.

We believe the difficulty we experienced with extracting useable results from the cation ion-exchange column is attributed to the relatively short timeframe of our experimentation. It is very possible that the strong acid cation resin used requires a “priming” phase that is considered negligible under an industrial setting. However, due to the small scale of our experimentation, we

were not able to push past this priming phase and achieve a reasonable breakthrough curve. If the column were to run consistently for a longer time span, such as 3 or 4 four hours, we are confident a true breakthrough curve would be attained.

**Scale-Up Column Sizing**

As this report does not include a valid breakthrough curve, and  $q_0$  and  $k$  were not obtained experimentally. As future steps, we recommend obtaining a complete bench scale breakthrough curve to get these. These values can then be utilized to size the full-scale ion exchange columns more accurately via the Thomas Kinetic Equation, an example calculation can be seen in appendix C1.

Additionally, the total capacity of each resin can be used to determine the required scale up mass. Sample calculations shown in appendix C2 and C3.

**Operating Condition**

Although a required resin mass could not experimentally be determined from the Thomas Kinetic equation, general scale up heuristics can still be used. Provided in table 4 below by Purolite, are column specifications when using Purolite resins C100 and A400.

**Table 4.** Resin heuristics provided by Purolite.

<b>Flowrate (gpm)</b>	8.0
<b>Diameter (in)</b>	12.0
<b>Cross Sectional Area (in<sup>2</sup>)</b>	113
<b>Flow (BV/Hr)</b>	21.4
<b>Amount of Resin Needed (ft<sup>3</sup>)</b>	3.0
<b>Height of Bed (in)</b>	46
<b>Height of Vessel with 50% freeboard (in)</b>	68.8
<b>Linear Velocity (gpm/ft<sup>2</sup>)</b>	10.2
<b>gpm/ft<sup>3</sup></b>	2.7

The generated data comes from generic heuristics provided by Purolite which is shown below in table 5. The data listed below can appropriately be used to scale up even further if necessary.

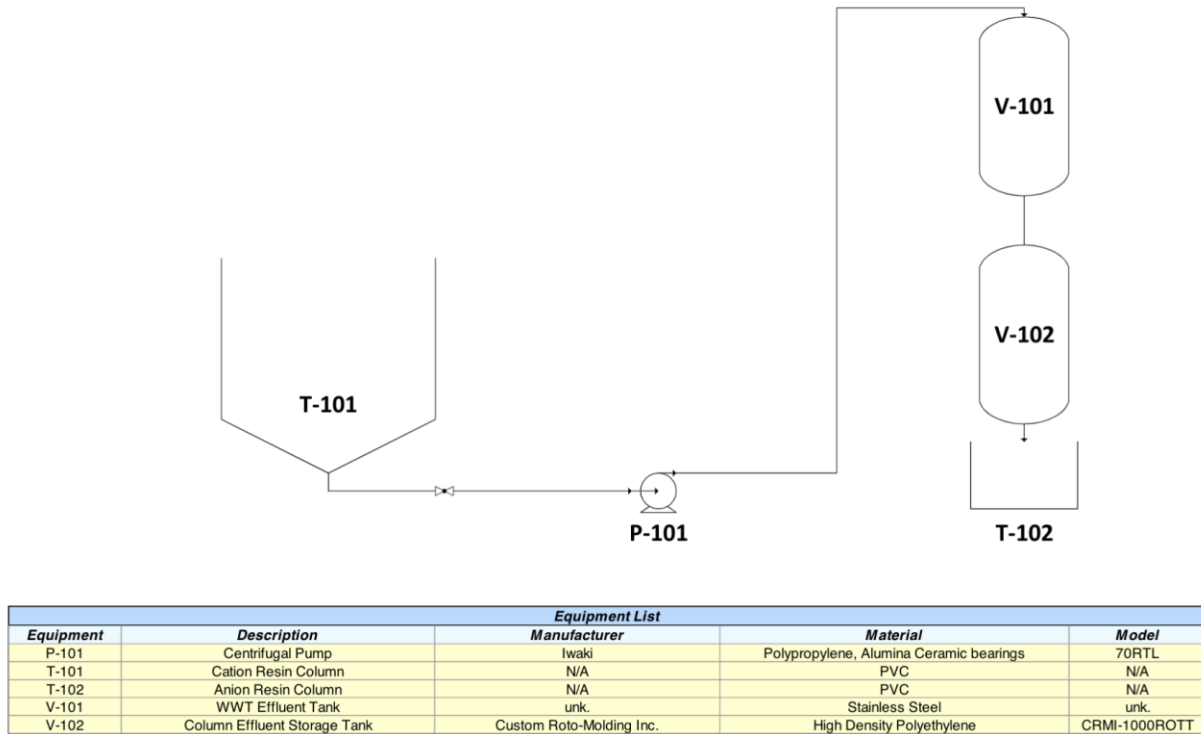
**Table 5.** Further heuristics are provided by Purolite.

<b>Flow rate based on Resin Volume (gpm/ft<sup>3</sup>)</b>	2-3
<b>Flow rate based on Bed Volume (BV/h) (equivalent to gpm/ft<sup>3</sup>)</b>	16-24
<b>Linear Velocity (gpm/ft<sup>2</sup>)</b>	8-12
<b>Resin Bed Depth (in)</b>	36-60

From the recommended flow rates in table 4, obtaining a pre-built column for the required resins is not feasible due to ion exchange traditionally being used in applications that call for flow rates more than 100 gpm. Additionally, due to the relatively small scale of this operation and conditions

being close to standard temperatures and pressure, the column can be constructed using low-cost materials such as PVC.

### Piping & Instrumentation Diagram



**Figure 5.** Piping & Instrumentation Diagram of Ion Exchange System

Figure 5 depicted above is the proposed P&ID. In this process, water would be fed from T-101, the WWT effluent tank, to P-101, a centrifugal pump, at roughly 8 gpm. The water would be fed to the ion exchange system which is composed of V-101, V-102, and T-102 alongside associated piping/coupling. The set-up is vertical due to the associated pressure loss that happens between columns. To eliminate the complexity of adding another pump in between columns, the columns are stacked in series. This allows gravity to run water through the second anion column. Once the water flows through the second column it will then settle below in T-102 which is a 1,000-gallon storage tank made of HDPE. There is a gate valve between T-101 and P-101. This valve can be opened when the ion exchange system needs to be operational and closed as needed. Under the assumption of wanting to treat 1,000 gallons the time for the valve to be open would be 2 hours and 5 minutes assuming an 8-gpm flow rate into the column. The time for this water to reach the storage tank would be slightly lower due to unsteady flow in the column from resin packing.

### Column design

From the recommended flow rates in table 4, obtaining a pre-built column for the required resins is not feasible due to ion exchange traditionally being used in applications that call for flow rates more than 100 gpm. Additionally, due to the relatively small scale of this operation and conditions

being close to standard temperatures and pressure, the column can be constructed using low-cost materials such as PVC.



**Figure 6.** A three-dimensional view of the custom-built ion exchange column.

Figure 6 depicts a three-dimensional view of the custom-built ion exchange column. The water will flow in through the top and into the first column which will be filled with the C100 cation resin. Following this, it will fall into the second A400 anion resin packed column secured by a rubber coupling. Once the water has made its way through the resin it will then settle in a 1,000-gallon storage tank not depicted in the figure.



**Figure 7.** Cross-sectional cut of the ion exchange column.

Figure 7 depicts the cross-sectional cut of the ion exchange column. A 1/8" PVC sheet with holes drilled is placed at the top of the column to act as a distributor for the inflowing water. A second

sheet is placed at the bottom of the first column. This second sheet acts as distributor for the second column alongside a method to hold the resin in place. A final sheet is placed at the bottom of the column to secure the resin.

Due to the various alternative options for reuse, a method for transporting water in T-102 is not depicted; however, the recommended tank can easily be modified to have a pipe connected from the bottom and be routed as desired. Another point of consideration is how the column will be supported. Currently figure 8 proposes a low-cost method in supporting the column. A frame around the column constructed of Unistrut beams can be used to support the column while in operation. This low-cost method of construction also allows for the column to slide in and out of the frame for service as the frame supports can be removed since the frame is not a fixed structure.

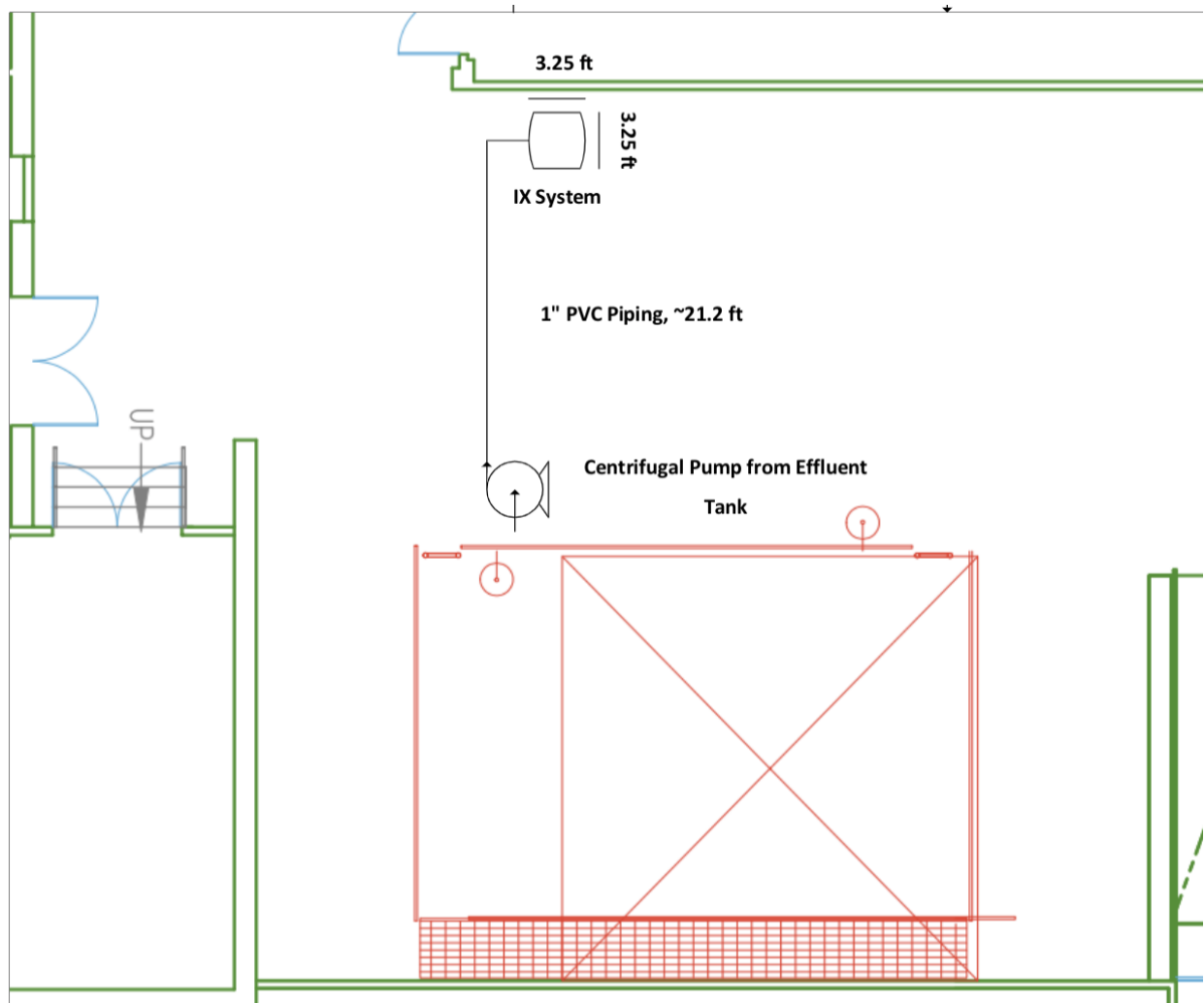


**Figure 8.** Three-dimensional view of the supporting frame for the ion exchange column.

As seen in figure 8 a three-dimensional view of the supporting frame is presented. The column is secured in the center of a rectangular shaped frame. At the bottom of the column a CNC cut metal sheet will be held up by brackets which will act as a support for the column to rest on. To provide lateral support a CNC cut metal sheet will be placed around the column which will also be secured by brackets. Please note that in this depiction the tank is fully secured underneath the frame. However, in actual operation it is recommended that part of the tank extends past the frame boundaries to save on the building material costs of the frame.



## Floor Layout



**Figure 9.** Floor layout for the proposed ion exchange column system.

Figure 9 provides a proposed location in which the ion exchange column can be placed. It is recommended that the system be placed approximately 21.2 ft away from the centrifugal pump and the pump be placed as close as physically possible to the effluent tank. Piping will run across the room and feed into the ion exchange column which should take up 3.25 ft x 3.25 ft of space against the northernmost wall. Placement of the ion exchange column is flexible, but any modifications made must require another friction loss calculation referenced in appendix D.

## Equipment List

Table 6 below outlines the equipment list that will be required to construct this column. The cost of accessory brackets and bolts required to construct the frame has not been included as the cost is minimal and the material is assumed to be readily available on site. The cost of PVC cement needed to secure the piping and column parts together is also not included as it is assumed that PVC

cement is readily available. Overall, the total cost of building materials should sum to be \$26,445. For column construction and regeneration instructions please reference appendix H.1 and H.2.

**Table 6.** Equipment list for the ion exchange column construction.

<b>Part</b>	<b>Part Description</b>	<b>Manufacturer</b>	<b>Part Number</b>	<b>Price</b>	<b>Quantity</b>
A	12" PVC Pipe -48 " Cut	Georg Fischer Harvel	H0401200PL1000	\$205.44 /ft \$9,861.12	1
B	12" PVC Pipe -46 " Cut	Georg Fischer Harvel	H0401200PL1000	\$205.44 /ft \$9,450.24	1
C	12" Socket Caps	Lasco	447-120	\$192.06 ea \$384.12	2
D	12" Rubber Coupling	Grainger Choice	1056-1212	\$56.78 ea	1
E	1" Pipe Fitting	United States Plastic Corp.	USP 27419	\$10.47 ea \$20.94	2
F	Adhesive for PVC sheet	B&K	16441	\$15.10	1
G	½" Thick PVC Sheet	United States Plastic Corp.	45856	\$11.26 ea \$33.78	3
H	1,000 Gallon Tank	Custom Roto-Molding Inc.	CRMI-1000ROTT	\$2,168.40	1
I	Centrifugal Pump	Iwaki	MD-70RLT-115	\$588.00	1
J	PVC Check Valve	PentAir	#wgv1	\$36.25	1
K	1" White PVC Plane End Pipe	IPEX Industrial Systems	022606	\$1.12 / ft \$40.32	36 feet
L	1" 90° PVC Pipe socket	United States Plastic Corp.	USP 28535	\$0.69 ea \$1.38	2
M	1 ½" P1100T	Gordon Electrical Supply	P1100T-20GR	\$900.00	100 feet
N	14 Gauge Sheet Metal	Generic – CNC Cut	60"x24"	\$900.00	2
O	10.5" Bracket	Global Industries	P1777EG	\$1,274.25	12
P	Post Base	Unistrut Service Company	P2072A-SQ- Powder Coat Green	\$119.32	4

Q	Cation Resin	Purolite	C100	\$170.00	3 ft <sup>3</sup>
R	Anion Resin	Purolite	A400	\$425.00	3 ft <sup>3</sup>

Calcium build-up is a visual issue seen in the WWT facility. Refence appendix H.3 to review a preliminary recommendation on how to resolve this issue.

### Savings Cost Analysis

**Table 7.** List of assumptions for calculating payback period.

<b>System Flowrate (gpm)</b>	8.0
<b>Cost of Regeneration</b>	\$482.25
<b>Frequency of Regeneration per Year</b>	4
<b>Frequency of Treat Water being Reintroduced per Day</b>	1
<b>Cost to Purchase Water (Per ft<sup>3</sup>)</b>	\$0.0693
<b>Cost to Send Water to Sewer (Per ft<sup>3</sup>)</b>	\$0.0982
<b>Project Lifetime (Years)</b>	15
<b>Discount Rate</b>	9.5%
<b>Annuity to Present Value Factor</b>	7.828

Table 7 depicts the assumptions used when calculating the payback period. It is assumed that 8 gpm are flowing into the column, the cost to send water to sewer from WWT is \$0.0982 per ft<sup>3</sup>, the ion exchange column will be in place for 15 years, a discount rate of 9.5%, and present dollar values were used when applicable. It is also important to note that it is assumed that the column treats 1000 gallons a day and that the plant uses those 1000 gallons each day. Equation 2 Below depicts how all annual costs, and savings were determined using an annuity to present value equation. Equation 2 was applied to the annual cost of removing water, annual cost of regenerating resin, and the annual savings values calculated.

$$Present Value = A \frac{(1+i)^n - 1}{i(1+i)^n} = (Annuity Value) \frac{(1.095)^{15} - 1}{0.095(1.095)^{15}} = 7.828(Annuity Value) \quad (2)$$

Typically, columns are ordered “off the shelf” but due to the scale of this column being much smaller than typical industrial uses, getting a quote for a custom-made column would make this project economically infeasible so a standard PVC column is designed at the cost of having to export resin for regeneration. The cost of resin regenerated is calculated to be \$482.25 based on the quote in appendix G. It is also assumed that regeneration is required 3 times per year Table 8 below highlights resin properties used in the calculations.

**Table 8. Resin Information**

<b>Mass of Cation Resin</b>	156 lb
<b>Mass of Anion Resin</b>	129 lb
<b>Density of Cation</b>	52 lb/ft <sup>3</sup>
<b>Density of Anion</b>	43 lb/ft <sup>3</sup>
<b>Cost of Anion</b>	\$425
<b>Cost of Cation</b>	\$170

The densities were obtained via product sheets and the cost of anion resin, A400, was priced at \$141.58 per ft<sup>3</sup> and the cost for the cation resin, C100, was priced at \$56.63 per ft<sup>3</sup>.

**Table 9. Cost analysis for the ion exchange columns**

<b>Total Cost (Considered Present Cost)</b>	\$41,545.55
<b>Annual Cost (P/A, i%, n)</b>	\$22,635.24
<b>Annual Savings with IX (P/A)</b>	\$37,506.39
<b>Total Savings</b>	\$14,871.15
<b>Payback Period (Years)</b>	2.8

Table 9 depicts the payback period when in present dollar amount. Values were calculated in appendix E1. The total cost encompasses the initial price of building the column and the cost of maintenance over the course of the project life. The annual cost takes the present value of sending the remaining water back to the sewer. The annual savings cost takes the difference in sending water back to the sewer with and without using ion exchange. This annual value is later factored to bring it to a present value.

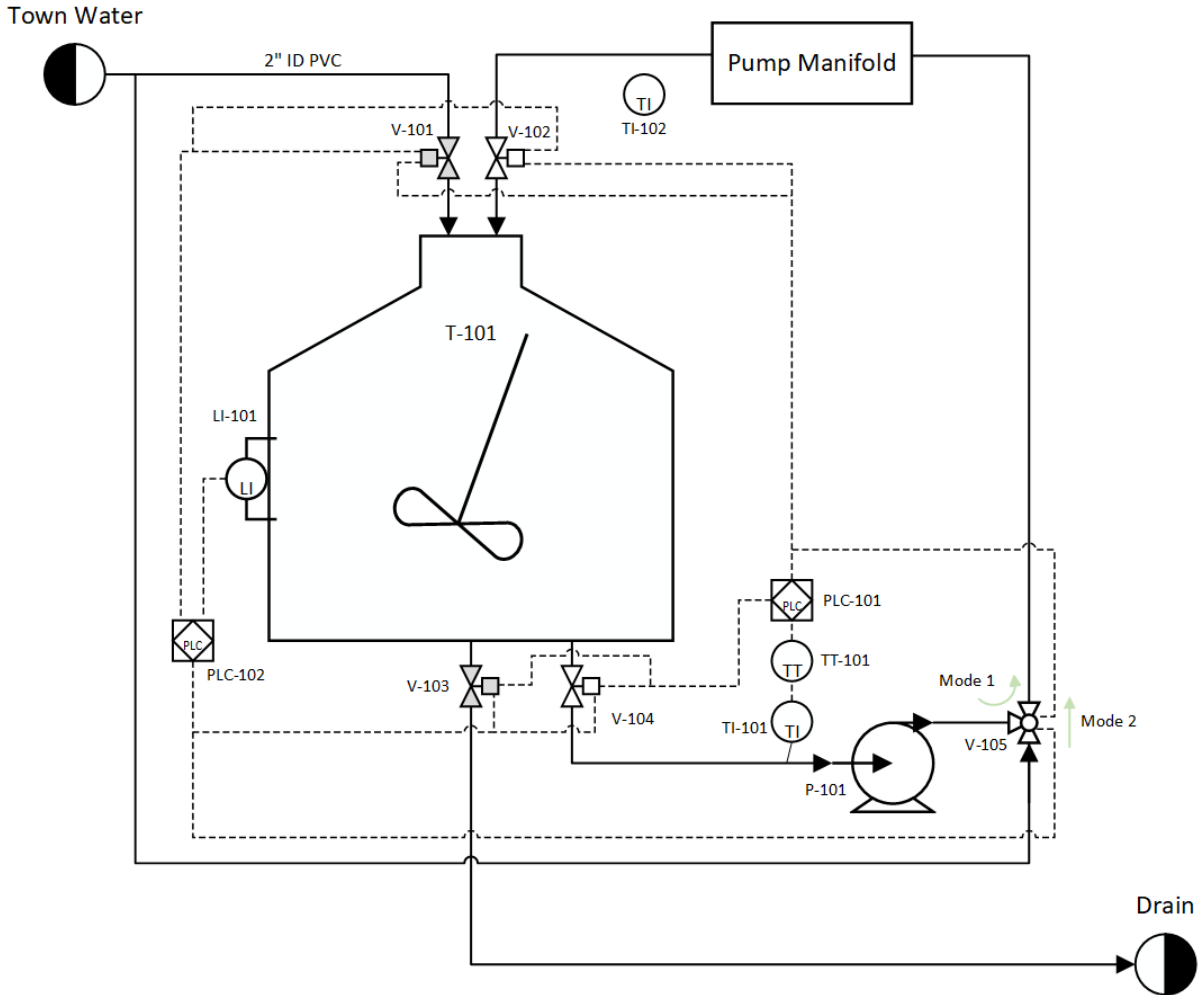
As one can see, the payback period is roughly 2.8 years using these assumptions. The cost savings over the course of the project life with a short payback period suggests that recycling the wastewater effluent water can prove to save roughly \$14,871 per year over a 15-year period with only an investment of \$41,545.55 when factoring maintenance costs to present value.

### **Seal Water Recirculation**

Our redesigned seal water system recirculates existing water through the scrubber pumps using an additional new tank and controls. These new controls will address concerns regarding the seal water heating up. It will combine with the already existing seal water system at OFS. By implementing this proposed design, OFS will use less water and therefore spend less on this operation.

### **Proposed P&ID**

The new seal water recirculation system will take existing infrastructure at OFS along with new items. The proposed piping and instrumentation diagram (P&ID) of the system is shown below.



**Figure 10.** The schematic diagram for the recommended new seal water recirculation system design.

The system will consist of a 500-gallon fiberglass tank. The tank will have two inlet electric control valves (V-101 and V-102) and two outlet electric control valves (V-103 and V-104). The system will operate in two ways. The first option is to have V-102 and V-104 open to create a closed recirculation loop. The second option will have V-101 and V-103 open, using fresh town water as seal water and discarding any water sent to the tank. More specifics on control parameters can be found in the following section.

### Controls

The tank will consist of two inlets, one source from town water controlled by V-101 and one from the seal water system pump manifold controlled by V-102. There will also be two outlets, with V-103 leading to the drain and V-104 going back to the pump manifold. When the temperature indicator, TI-101, is below 70 degrees Celsius, V-102 and V-104 will be open and V-105 will operate in mode 1. This will create a closed loop with the pump manifold. When TI-101 registers a water temperature over 70 degrees Celsius, V-104 will close and mode 2 on V-105 will open. This will circulate new town water through the pump manifold. Meanwhile, V-103 will open to drain the hot water from the tank. When the minimum level is reached on LI-101, V-103 will close

and V-101 will open to refill the tank with cold water. When the maximum level is met on LI-101, V-101 will close. V-104 will open and V-105 will switch back to mode 1 to create the recirculation loop. The table below provides a simplified summary of the control parameters. Additionally, appendix F is a flow chart describing this PLC system.

**Table 10.** Recirculation System control parameters.

Temperature	Water Level	V-101	V-102	V-103	V-104	V-105
< 70	Within Range	Closed	Open	Closed	Open	Mode 1
> 70	Within Range	Closed	Open	Open	Closed	Mode 2
Not Applicable	Minimum	Open	Open	Open	Closed	Mode 2
< 70	Maximum	Closed	Open	Closed	Open	Mode 1

### Equipment List

To successfully implement this design, new equipment will be needed. On the previous recirculation system, a polyethylene tank was used to store water. This resulted in the tank bulging under high temperatures. To combat this concern, we propose using a fiberglass reinforced plastic (FRP) tank. An impeller will be added to the tank to ensure the water does not stay stagnant. Temperature and level controls will be needed as well. Lastly, a pump will be needed to send the water exiting the tank to the pump manifold. Table 11 lists the equipment and a suggested vendor. The specifications and cost of the equipment are also included.

**Table 11.** Recirculation system equipment list.

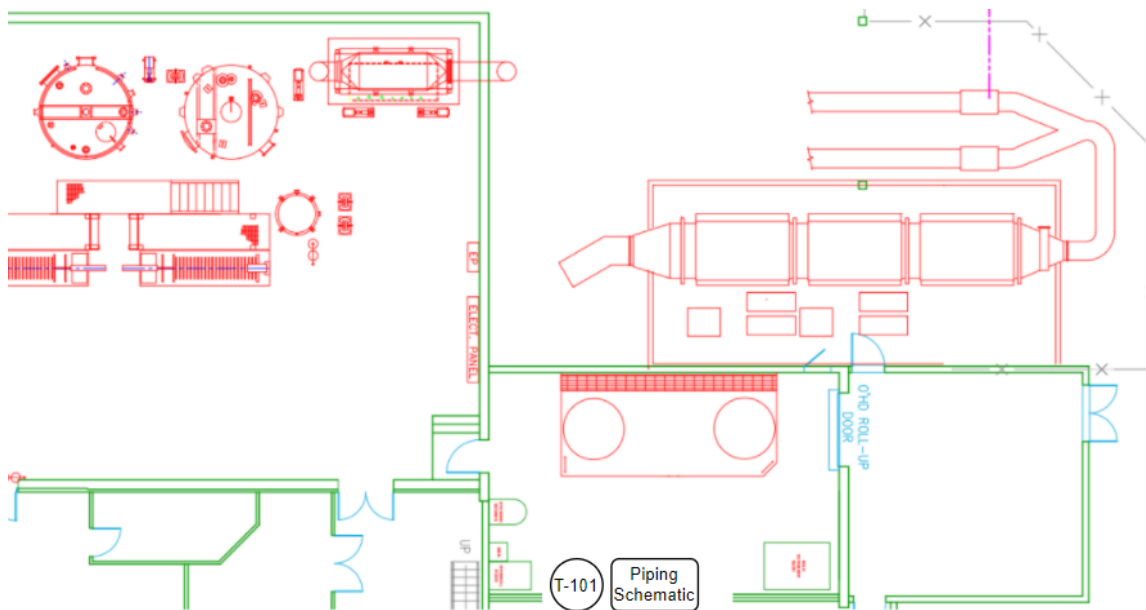
Part	Part Description	Manufacturer	Part Number	Price	Quantity
T-101	Fiberglass Tank	Found on Met-Chem	-	\$900.00	1
-	Impeller	INDCO	21A6004	\$1615.50	1
TI-101 TI-102	Temperature Indicator	Georg Fischer Signet	323501	\$417.00	2
TT-101 TT-102	Temperature Transponder	Novus	8803809706	\$101.65	2
LI-101	Level Indicator	Gems Sensor	138167	\$236.55	2
LT-101	Level Transmitter	Flowline	LG10-0003-00	\$974.18	2
V-101 V-102 V-103 V-104	Electronic Control Valve 2"	Plastomatic Valves	ABRA200VT-PP	\$709.87	4

V-105	3-Way Electronic Control Valve 2"	Baelz	342-B-DN50-PN16	\$1481.20	1
P-101	Pump	Iwaki	MD-20RZT-115NL	\$329.00	1

Since our proposed design will be combined with the existing seal water system, additional piping will be minimal, and we considered the costs negligible. Therefore, the total cost of all new equipment is \$10,624.

### Layout Diagram

In addition to the redesign of the seal water recirculation system, we also propose for an area in the plant for the new tank and equipment to reside, as well as relocation of the current pumping manifold. The current pumping manifold, that sends seal water to the scrubber pumps, resides on an impractical mezzanine that requires ascending a steep, vertical metal ladder to be reached. This area is cramped for any more than 2 people to reside on and is not suitable to host the tank and additional apparatuses we are recommending. Relocating the manifold to be adjacent to the manifold would be beneficial because it would reduce the additional length of piping. The area we recommend in the plant is currently empty, besides a few cleaning supplies, and the mezzanine the manifold currently resides is in the same room. The proposed layout diagram for the T-101 and recirculation system, in addition to the piping schematic already installed at the OFS site is below.



**Figure 11.** Layout diagram of the OFS plant, including the location of the recommended recirculation system and relocation of the scrubber pump manifold.

Piping will be reconfigured throughout the room, and because the pump manifold is being relocated to the same room, minimal additional piping will be needed. The exact placement of

the tank, pump and relocation of the manifold is flexible, but we recommend they stay close and in series to minimize piping and pumping costs.

### **Cost Savings Analysis**

OFS uses two gallons per minute to circulate seal water through the scrubber pumps. With inlet water rates of \$0.069/ft<sup>3</sup> and sewer discharge rates of \$0.0982/ft<sup>3</sup>, OFS spends \$23,504.52 per year on fresh seal water. Using temperature data provided by OFS, we estimate that the recycled water will reach 70°C after 8 days, calculated in appendix I. This means the tank would be emptied and refilled 46 times per year. This reduces the cost of seal water per year to \$517.96. Considering the initial cost of equipment, this provides a payback period of 0.48 years or 176 days. The annual savings for implementing this new design will be \$22,054. Detailed calculations for payback period and total savings can be found in Appendix E.2.

### **Potential Applications of the Post-Ion-Exchange Water**

Upstream there are several processes in which town water could be replaced by our treated recirculation water. It could be used as make up water in the liquid-vapor contact scrubbers, functioning to adsorb regulated contaminants from the exhaust MCVD gasses. The effluent from the scrubber is ultimately disposed of as hazardous waste (save for the recaptured germanium). It may also be used as reactor rinse to clear off depositing/fouling of the vessels during maintenance.

Additionally, the treated water can be used in conjunction with the proposed recirculation system. During the stage when the recirculation system is refilled with town water the treated ion exchange water can be used instead to refill the tank as the tank in the recirculation system is only 500 gallons while the storage tank in the ion exchange system is 1,000 gallons.

### **Additional Sources of Reuse**

Although we chose to focus on the effluent stream, other water sources around the plant have potential to be recycled. Another source of single-pass water within the OFS process is the discharged water created during reverse osmosis (RO) treatment. Reverse osmosis treatment is a way to remove contaminants from water by sending it through a semipermeable membrane while under pressure. The RO system is used to produce deionized (DI) water, water that has been completely stripped of all its ions, and this water is used where high-purity water is needed in fiber optics cable manufacturing, for example in the rinsing of cables after hydrofluoric acid etching.

RO discharge water is single pass, because for every 3 gallons of Sturbridge town water inputted into the system, only 1 gallon of DI water is produced. The other 2 gallons of water is immediately sent back to the town sewer system. This water is only slightly more elevated in ions than town water, and therefore could be utilized as an additional reuse source. To investigate this potential further, we used our fluoride tester and a chlorine spectrometer to compare the concentration levels in RO to town. That comparison is seen in Table 12 below.



**Table 12.** Ion concentration comparison between RO Discharge and Town Water.

<b>Water Source</b>	<b>Fluoride (ppm)</b>	<b>Total Chlorines (mg/L)</b>	<b>Free Chlorines (mg/L)</b>
<b>RO Discharge</b>	1.7	0.03	0.03
<b>Town Water</b>	0.6-1.1	0.02	0

Through the comparison of RO Discharge and Town Water, we recommend that the RO Discharge could be repurposed in other areas of the plant, rather than maintaining it as a single-pass water source. If the low ion concentrations are not a concern, the water could be immediately collected and used as any of the suggested sources, i.e., the scrubber make-up water, germanium reactor rinse or chiller water. If these slightly elevated concentrations could cause concern, the RO Discharge could also be sent through the ion exchange columns we recommended for the OFS site. The discharge combined with the treated effluent stream could be utilized as an even larger recycled water source.

## **Conclusions and Recommendations**

Lowering operating costs is a primary way for companies to increase profits margins. Water, a finite resource, costs money to both use and discharge back to the environment. At OFS, single-use water is heavily relied on for the waste treatment process. By reusing water, OFS will be able to reduce its total water consumption during manufacturing, in turn lowering manufacturing costs. After analyzing several water samples and researching the best methods for water treatment and reuse, we designed a way for OFS to reuse the wastewater effluent stream as well as a recirculation system for the seal water. With these designs we recommend the following list of actions be taken.

First, we recommend that OFS construct and implement an ion exchange column after the downstream wastewater treatment referencing the supplied equipment list, schematics, and construction instructions. This system, paired with a bag filter to catch solid calcium particles, will remove excess fluorine and calcium by using a dual-series column with anion and cation resins. Once treated, this water can be recycled to another process within the plant. Reusing this water in another operation in the plant will result in \$31,000 saved each year after a 2.8-year payback period.

Next, we recommend that OFS reconstructs the decommissioned seal water recirculation system for the scrubber pumps. Concerns for the previous system included the water temperature getting too high and potential for organic growth if the water remained stagnant in a tank for too long. The updated design will use temperature controls to keep the water under 70°C and an impeller in the tank with constantly mix the water. With this new design, OFS will save \$22,200 annually with a payback period of only half a year.

For future projects for OFS, we suggest focusing on reuse options for the RO discharge. The water's ion levels are only slightly more elevated than town water, meaning it could be easily reused in other areas of operation. RO discharge water can be used as scrubber make-up water, chiller water, reactor rinses, or water for the geranium rinse. Next steps would include researching each option and determining the feasibility of rerouting the discharge water to it.

By taking these steps, OFS can greatly decrease their annual water consumption. The ion exchange column and seal water recirculation system can save OFS over \$50,000 annually. In addition to the financial benefits of these changes, the manufacturing process also becomes more sustainable by reusing water. OFS will become less dependent on outside sources for water with the ability to reuse water within the plant. These improvements to the wastewater processes will allow OFS to become a more environmentally conscious company while also saving money.

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## Appendices

### A. Anion Data

To create a breakthrough curve, we ran the effluent water through an anion resin packed column and collected a series of output samples of 8 throughput volumes. We first measured the initial fluoride concentration using a fluoride meter and then also obtained the fluoride concentrations for the subsequent samples. A breakthrough curve was then attempted to be created for the runs, using the natural log of the fluoride concentration ratio and the sample volume.

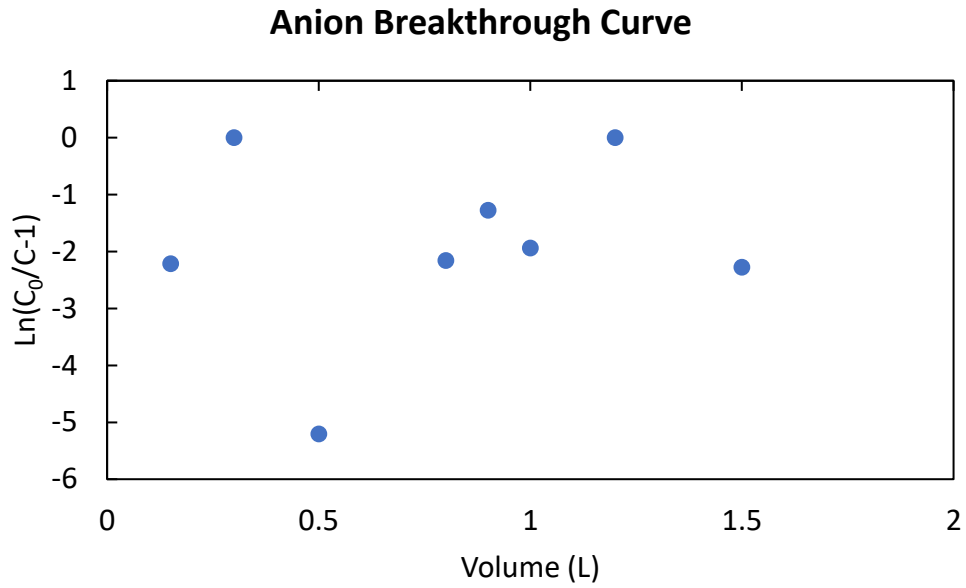
The natural log of fluoride concentration:

$$\ln\left(\left(\frac{C_o}{C_v}\right) - 1\right); C_o \text{ is initial concentration \& } C_v \text{ is concentration at specified volume}$$

Below is the 2 series of tables and attempted breakthrough curves for the measured fluoride concentrations for both series of column runs.

**Table 13.** First set of data for anion ion-exchange breakthrough curve to remove fluorine.

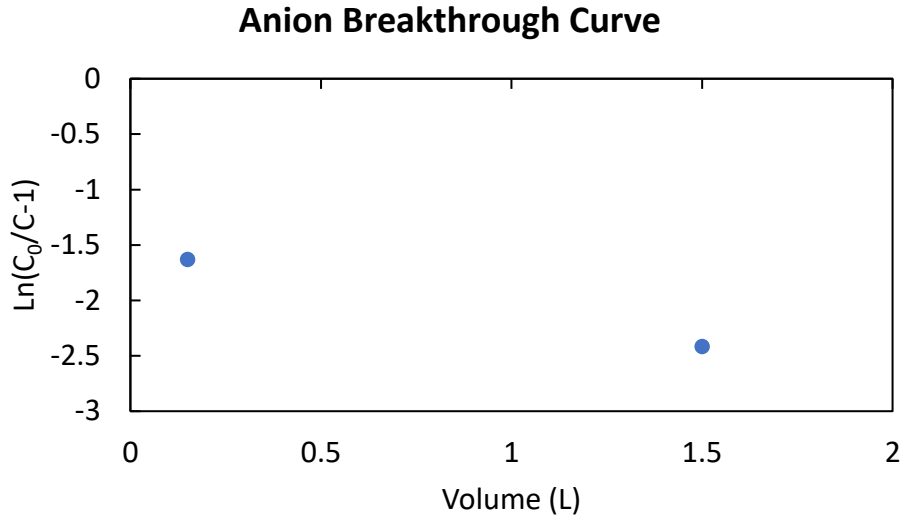
Measurement	Volume (L)	Fluoride Concentration (mg/L)	$\ln\left(\left(\frac{C_o}{C_v}\right) - 1\right)$
0	-	18.3	-
1	0.15	16.5	-2.215573716
2	0.3	18.6	#NUM!
3	0.5	18.2	-5.204006687
4	0.8	16.4	-2.155427449
5	0.9	14.3	-1.273965176
6	1	16	-1.939679599
7	1.2	18.5	#NUM!
8	1.5	16.6	-2.278774444



**Figure 12.** Anion breakthrough curve for first dataset.

**Table 14.** Second set of data for anion ion-exchange breakthrough curve to remove fluorine.

Measurement	Volume (L)	Fluoride Concentration (mg/L)	$\ln\left(\frac{C_0}{C_v} - 1\right)$
0	-	17.1	-
1	0.15	14.3	-1.63064012
2	0.3	17.6	#NUM!
3	0.5	19.1	#NUM!
4	0.8	19.4	#NUM!
5	0.9	19.6	#NUM!
6	1	19.4	#NUM!
7	1.2	18.4	#NUM!
8	1.5	15.7	-2.417188476



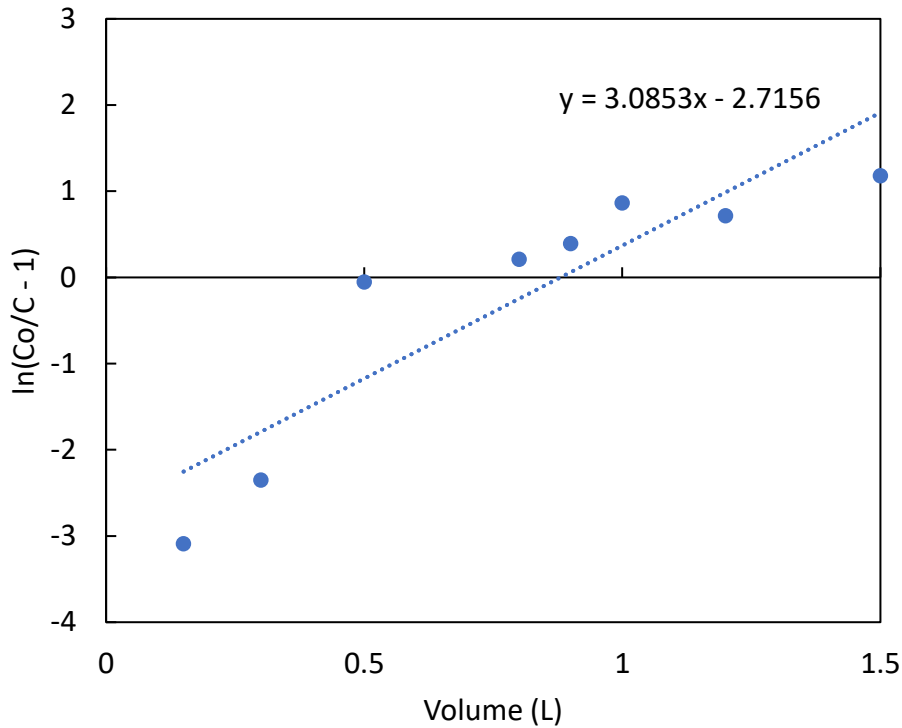
**Figure 13.** Anion breakthrough curve for second dataset.

Both Anion breakthrough curves did not give us expected results or a viable breakthrough curve. The natural log of the concentration ratio was an insolvable number in some cases because somehow the fluoride concentrations, after being passed through the anion resin, measured with a higher fluoride concentration than the initial concentrations. As mentioned in the above results and discussion, we are attributing this to faulty readings from the fluoride testing instrument.

### B. Cation Data

**Table 15.** Data set for cation ion-exchange breakthrough curve to remove calcium.

Measurement	Volume (L)	Calcium Concentration (mg/L)
0	-	2300
1	0.15	2200
2	0.3	2100
3	0.5	1180
4	0.8	1030
5	0.9	928
6	1	681
7	1.2	755
8	1.5	541



**Figure 14.** Cation Breakthrough Curve

### C. Scale-up Calculations

#### i. Using the Thomas Kinetic Equation

For sizing the cation exchange column, from breakthrough data,  $q_0$  and  $k$  would be obtained.

The Thomas Kinetic Equation is:

$$\ln\left(\frac{C_0}{C} - 1\right) = \frac{k_1 q_0 M}{Q} - \frac{k_1 C_0 V_t}{Q}$$

The *design* system will have a flow rate of 2 gpm and a minimum allowable throughput volume of 180,000 gallons (roughly half a year of usage, assuming a daily column flow of ~1000 gallons based on OFS's current operations). The water coming in will have the previously determined concentrations of 180 ppm of  $Ca^{+2}$ . The design allowable outlet concentration will be 30.5 ppm (to be like town water).

$$Q = 2 \frac{\text{gallons}}{\text{minute}}$$

$$V_t = 180,000 \text{ gallons}$$

$$C_0 = 180 \text{ ppm} = 0.68137 \frac{\text{g}}{\text{gallon}}$$

$$C = 30.5 \text{ ppm} = 0.11545 \frac{\text{g}}{\text{gallon}}$$

Then all parameters except the mass are known.

$$M = \frac{Q \ln\left(\frac{C_0}{C} - 1\right)}{k_1 q_0} + \frac{V_t C_0}{q_0} = \frac{2 \ln\left(\frac{0.68137}{0.11545} - 1\right)}{k_1 q_0} + \frac{(180000)0.68137}{q_0}$$

Given the nature of packed bed, the bulk density will be less than the particle density, so the true volume of the packed bed needed will be greater.

Then the bed volume is  $\frac{M}{\rho_{\text{bulk}}} = V_{\text{bed}}$

This volume would be used to determine the dimensions of our cylindrical column.

For the Anion Exchange column, the solution is carried out in the same manner as for the cation column, the only difference now is that the input  $C_0$  will be for the fluoride, so  $C_0 = 20 \text{ ppm}$  and  $C = 1 \text{ ppm}$

$C_0 = 0.0757 \text{ ppm}$

$C = 0.003785 \text{ ppm}$

$$M = \frac{Q \ln\left(\frac{C_0}{C} - 1\right)}{k_2 q_0} + \frac{V_t C_0}{q_0} = \frac{2 \ln\left(\frac{0.0757}{0.003785} - 1\right)}{k_1 q_0} + \frac{(180000)0.0757}{q_0}$$

The volume would be calculated similarly as above.

## ii. Using total capacity for the C100 Cation Resin

The absorption capacity for the C100 resin is  $43.7 \frac{\text{kg of Ca}^{2+}}{\text{ft}^3}$

The calcium concentration in the wastewater stream is approximated to be  $11 \frac{\text{mg}}{\text{L}}$  as recorded using ICP

The calcium concentration must be converted into kg that have been run through the column on a yearly basis

$$11 \frac{\text{mg}}{\text{L}} \frac{1 \text{ L}}{.264 \text{ gal}} \frac{\text{kg}}{1 \times 10^6} = 0.000041667 \frac{\text{kg of Ca}^{2+}}{\text{gal}}$$

There are 0.000041667 kg of  $\text{Ca}^{2+}$  for every gallon run through the column. Now we must consider the kg of  $\text{Ca}^{2+}$  run through the column each year

$$0.000041667 \frac{\text{kg of Ca}^{2+}}{\text{gal}} \frac{8 \text{ gal}}{\text{minute}} \frac{518,400 \text{ minutes}}{1 \text{ year}} = 172.8 \frac{\text{kg of Ca}^{2+}}{\text{year}}$$

The C100 resin absorbs 43.7 kg of  $\text{Ca}^{2+}$  ions for every  $\text{ft}^3$  of resin so now we must determine the  $\text{ft}^3$  required.

$$172.8 \frac{\text{kg of Ca}^{2+}}{\text{year}} \frac{1 \text{ ft}^3 \text{ resin}}{43.7 \text{ kg of Ca}^{2+} \text{ absorbed}} = 3.95 \text{ ft}^3$$

To treat the calcium through the column without needing to regenerate the resin we would need  $3.95 \text{ ft}^3$  of resin. Since our design is based off of  $3 \text{ ft}^3$  in the column we will need to regenerate the resin 2 times per year however, due to the A400 resin requiring greater regenerations the regeneration frequency will be based on the A400 resin requirements



### iii. Using total capacity for the A400 Anion Resin

The absorption capacity for the A400 resin is  $28.4 \frac{kg \text{ of } F^-}{ft^3}$

The fluorine concentration in the wastewater stream ranges from 7 to  $27 \frac{mg}{L}$  so a fluorine concentration of  $17 \frac{mg}{L}$  will be used for the calculations.

The fluorine concentration must be converted into kg that have been run through the column on a yearly basis

$$17 \frac{mg}{L} \frac{1 L}{.264 gal} \frac{kg}{1 \times 10^6} = 0.000064352 \frac{kg \text{ of } F^-}{gal}$$

There are 0.000064352 kg of  $F^-$  for every gallon run through the column. Now we must consider the kg of  $F^-$  run through the column each year

$$0.000064352 \frac{kg \text{ of } F^-}{gal} \frac{8 gal}{minute} \frac{518,400 minutes}{1 year} = 266.88 \frac{kg \text{ of } F^-}{year}$$

The A400 resin absorbs 28.4 kg of  $F^-$  ions for every  $ft^3$  of resin so now we must determine the  $ft^3$  required.

$$266.88 \frac{kg \text{ of } F^-}{year} \frac{1 ft^3 resin}{28.4 kg \text{ of } F^- \text{ absorbed}} = 9.4 ft^3$$

To treat the fluorine through the column without needing to regenerate the resin we would need  $9.4 ft^3$  of resin. Since our design is based off of  $3 ft^3$  in the column we will need to regenerate the resin 4 times per year.

### D. Ion Exchange Pump Calculation

To determine the total dynamic head, we will consider that the minimum level in the effluent tank is 30" or 2.5 ft and the vertical distance the fluid must travel is equivalent to 3" plus the IX system. The total height of the IX system is 174" or 14.5 ft.

This value includes both columns (94"), the coupling (6"), the end caps (6" each) pipe fittings (1" ea), tank height (48"), distance between the tank and the bottom fitting (6"), and the height of piping above the top pipe fitting (6").

Thus, the total head when neglecting friction is 144" or 12 feet. The total piping distance including horizontal, and vertical is estimated to be roughly 36 feet.

To determine frictional head loss the Hazen-Williams Friction Loss equation is used. This equation can predict frictional head losses with accuracy when the fluid is water through empirical correlations. Since we are only working with water the Hazen-William equation is appropriate for this purpose.

$$h_{100ft} = \frac{0.2083 \left( \frac{100}{c} \right)^{1.852} * q^{1.852}}{d_h^{4.8655}}$$

Whereas:

- q is equivalent to the flow rate in gpm (8 gpm)
- c is the Hazen-Williams roughness coefficient (150 for PVC)
- $d_h$  is the hydraulic diameter (1")

$$h_{100ft} = \frac{0.2083 \left(\frac{100}{150}\right)^{1.852} * 8^{1.852}}{1^{4.8655}} = \mathbf{4.62 \text{ ft per } 100 \text{ ft } H_2O}$$

The estimated pipe length is 36 feet so  $\frac{(4.62)}{100} * 36$  equals **1.66 ft H<sub>2</sub>O** frictional head loss.

There are 2 90° 1" PVC pipe elbows and 1 fully open gate valve which will contribute to minor losses that must be accounted for.

The loss coefficient used for the gate valve will be 0.15 (Engineering Toolbox)

The loss coefficient used for the 90° elbows will be 0.3 (Engineer Toolbox)

The linear velocity is 10.2 gpm/ft<sup>2</sup> which equates to 1.375 (ft<sup>3</sup>/min)/(ft<sup>2</sup>) which equals 1.36 ft/min or 0.41 m/min or 0.0069 m/s

$$\frac{10.2 \text{ gpm}}{\text{ft}^2} \frac{1 \text{ ft}^3}{7.418 \text{ Gallon}} = 1.375 \frac{\text{ft}^3}{\text{min}} \frac{1}{\text{ft}^2} = 1.375 \frac{\text{ft}^3}{\text{min ft}^2} = 1.375 \frac{\text{ft}}{\text{min}}$$

Continuing on to place our velocity into meters per second

$$1.375 \frac{\text{ft}}{\text{min}} \frac{0.3048 \text{ m}}{\text{ft}} \frac{\text{minute}}{60 \text{ second}} = 0.0069 \frac{\text{meters}}{\text{second}}$$

The equation for head loss is

$$h_{minor} = (\text{loss coefficient}) * \left(\frac{\text{velocity}^2}{2g}\right)$$

The total loss is:

$$h_{minor} = \left( (0.15) * \left(\frac{0.0069^2}{2 * 9.8}\right) \right) + \left( (0.3) * \left(\frac{0.0069^2}{2 * 9.8}\right) \right) = 0.45 \text{ m}$$

0.45 m lost which equates to **1.47 ft lost**.

Therefore, the total dynamic head required is **15.13 ft** and the specified pump's maximum head is 27.5 ft which means there is ample head available for the operation of the IX system.

## E. Cost Analysis Calculations

### i. Ion Exchange System

The total cost of the system from the equipment list is \$26,445.00.

The cost of sending water back to the town from the WWT effluent stream is \$0.0982 per ft<sup>3</sup> and OFS dumps approximately 1,590 gallons each day according to data logs provided.

$$1590 \text{ gallons} \frac{ft^3}{7.481 \text{ gallons}} 0.0982 \frac{\$}{ft^3} = \$21.05 \text{ per day}$$

With this information we know that without using ion exchange OFS spends \$21.05 per day on dumping water which equals \$7,682.71 per year.

Under the assumption that OFS implements an ion exchange system and treats 1,000 gallons each day for 365 days a year then OFS would spend the following on dumping water each year.

$$590 \text{ gallons} \frac{ft^3}{7.481 \text{ gallons}} 0.0982 \frac{\$}{ft^3} = \$7.92 \text{ per day}$$

This equates to \$2,891.51 per year spent on water. The cost savings each year is

$$\$7,682.71 - \$2,891.51 = \$4,791.2 \text{ saved each year}$$

From total capacity calculations in Appendix A, a regeneration frequency of 4 times a year is called for at a cost of \$482.25 per regeneration. Therefore, the total cost of regeneration.

$$\$482.25 \times 4 = \$1,929 \text{ per year}$$

The savings cost will be considered profit and the regeneration cost will be considered as part of the initial cost once brought to present value. These annuity values must be brought to present value. A discount rate of 9.5% is used with a project lifetime of 15 years. The Annuity to Present value equation is used.

$$Present \ Value = A \frac{(1+i)^n - 1}{i(1+i)^n} = (Annuity \ Value) \frac{(1.095)^{15} - 1}{0.095(1.095)^{15}} = 7.828(Annuity \ Value)$$

Therefore, the total cost of expenses

$$7.828(\$2,891.51) = \$22,635.24$$

The total savings is

$$7.828(\$7,682.71 - \$2,891.51) = \$37,506.39$$

The total up front cost includes the total cost of equipment plus the maintenance cost brought to present value. Therefore, the upfront cost is

$$\$25,250 + 7.828(\$1,929) = \$41,545.55$$

Since savings is considered profit the annual profit is depicted below.

$$\$37,506.39 - \$22,635.24 = \$14,871.15$$

Thus, the payback period becomes

$$\frac{\text{Total Cost of Investment}}{\text{Annual Profit}} = \frac{\$41,545.55}{\$14,871.15} = 2.8 \text{ years}$$

It will take 2.8 years for OFS to recoup their initial investment through the annual savings cost over the course of the project life.

### ii. Seal Water Recirculation

First, we determine how much OFS currently spends on water to seal the pumps on the scrubbers.

$$\left(2 \frac{\text{gal}}{\text{min}}\right) \left(0.0639 \frac{\$}{\text{ft}^3}\right) \left(\frac{1\text{ft}^3}{\text{gal}}\right) + \left(2 \frac{\text{gal}}{\text{min}}\right) \left(0.0982 \frac{\$}{\text{ft}^3}\right) \left(\frac{1\text{ft}^3}{\text{gal}}\right) = \$0.0448/\text{min}$$

Assuming 8400 operating hours per year,

$$\left(\frac{\$0.448}{\text{min}}\right) \left(\frac{60\text{min}}{\text{hour}}\right) (8400 \text{ hrs}) = \$22572.19/\text{year}$$

Next, we find the operating costs to drain and refill the 500-gallon tank in the proposed design.

$$\left(0.0639 \frac{\$}{\text{ft}^3}\right) \left(\frac{1\text{ft}^3}{\text{gal}}\right) (500 \text{ gal}) + \left(0.0982 \frac{\$}{\text{ft}^3}\right) \left(\frac{1\text{ft}^3}{\text{gal}}\right) (500 \text{ gal}) = \$11.12$$

Assuming we need to refill the tank every 7.89 days (see appendix I for calculations), we can now determine how much OFS will spend annually spend using the recirculation system.

$$\left(\frac{\$11.12}{7.89 \text{ days}}\right) \left(\frac{365}{1 \text{ year}}\right) = \$517.96/\text{year}$$

We can now find the payback period on this project using the estimated equipment costs and operating costs.

$$\frac{\$10623.94}{\$22572.19/\text{year} - \$517.96/\text{year}} = 0.48 \text{ years} = 176 \text{ days}$$

### F. P&ID PLC Controls

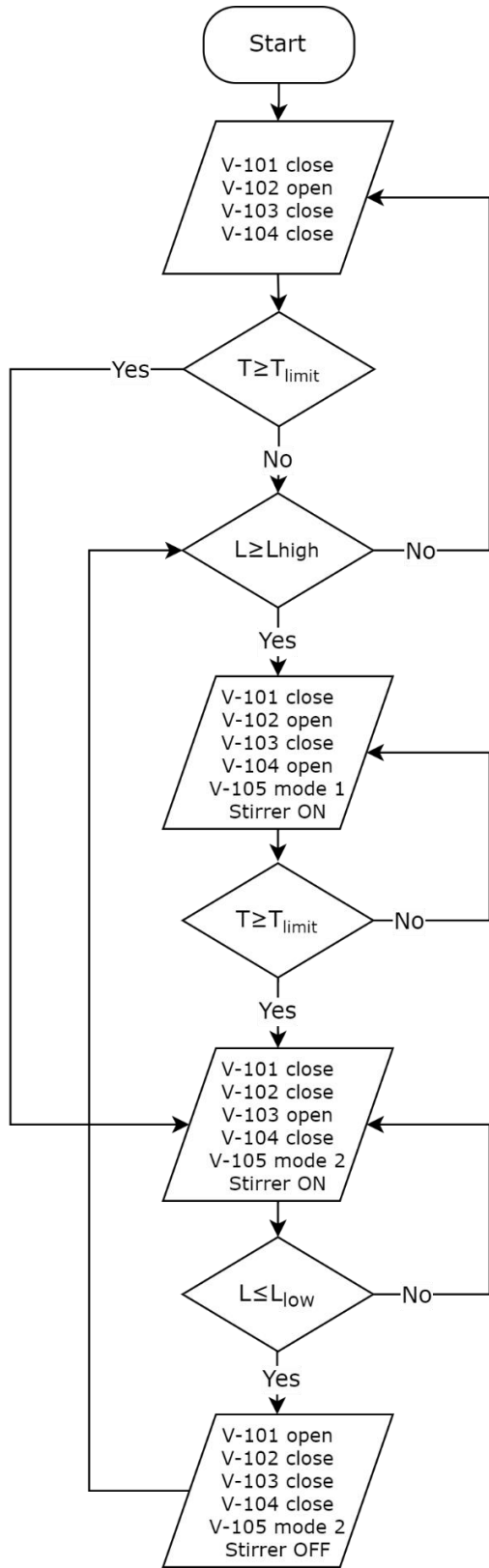


Figure 15. PLC Controls

## G. Quotes from Vendors



April 14, 2022

**Quoted To:** Sam Elaskalani  
Worcester Polytechnic Institute  
100 Institute Rd.  
Worcester, MA 01609

**Quote No.:** 41422JMC-02

RE: SAC and SBA Resin Regeneration

Sam:

Thanks for the opportunity to quote regenerating three (3) cu ft of each strong acid cation H+/H form resin and strong Base Anion H+/H form resin.

**Resin Regeneration:**

Qty	Part No	Description	Price
3 cu ft	SAC/REGEN	Strong Acid Cation Resin Regeneration (\$75.00 per cu ft)	\$225.00
3 cu ft	SBA/REGEN	Strong Base Anion Resin Regeneration (\$85.75 per cu ft)	\$257.25

**TOTAL ... \$482.25**

The above price is quoted in US Dollars. The price is FOB ACM (Forest Hill, MD). Freight, taxes, fees, are not included and will be invoiced separately.

**Validity:**

30 days from the date of this proposal

**Payment Terms**

Net 30 Days upon ACM Technologies approved credit

**Schedule**

Shipment: 4-7 days after receipt and acceptance of confirming purchase order, a signed copy of this proposal, and approval of credit and terms.

Please feel free to call me on my cell at 443-356-7009 or email me at [jmccormick@acmix.com](mailto:jmccormick@acmix.com) if you have any questions or need additional information.

**QUOTE IS GOOD FOR 30 DAYS**

Equipment will be ordered upon receipt of your purchase order and deposit. Installation is not included. All material is guaranteed to be as specified. Any alteration or deviation from stated specifications, involving extra costs, will be executed only upon written orders, and will become an extra Charge over and above the original quotation. All agreements are contingent upon strikes, accidents, or delays beyond our control.

REPRESENTATIVE

SIGNATURE: Jim McCormick INDUSTRIAL SALES  
(For seller) (Title)

**ACCEPTANCE OF PROPOSAL** – The above prices, specifications, and conditions are satisfactory and are hereby accepted. You are authorized to do the work as specified. When signed by both parties, this becomes a legal and binding contract.

SIGNATURE: \_\_\_\_\_  
(For buyer) (Title)

DATE: \_\_\_\_\_ PURCHASE ORDER # \_\_\_\_\_

## H. Preliminary Instruction and Suggestions for OFS

### i. Ion Exchange Column Installation Instructions

To construct the column, it must be noted that some parts must be secured with PVC cement while others will not be due to the need to remove resin periodically for off-site regeneration.

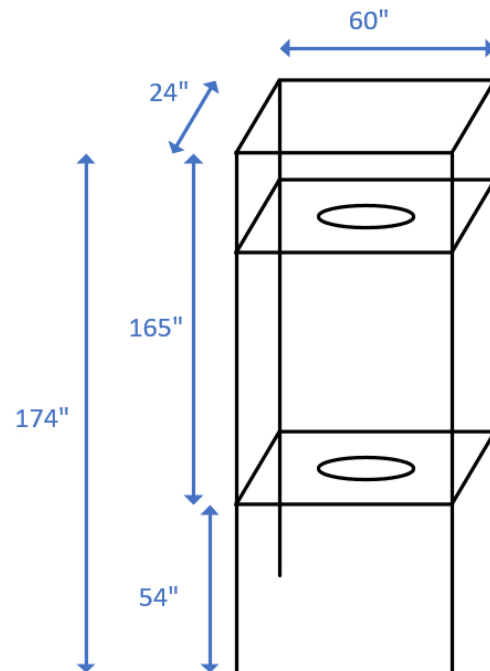
One part will need minor modification, the 1/8" thick PVC sheets (part G) must be cut into circular shapes to be equal to the outer diameter of the 12" PVC pipes (Parts A & B) which equals 12.75". All 3 sheets must be cut. Additionally, each sheet must have multiple small holes drilled through them to allow the water to flow through the column. Hole sizes between 1/16" to 1/8" should be appropriate.

V-101 is attached to V-102 by a 12" rubber coupling. This coupling will not require cement as it will be secured by the hose clamps. The modified 1/8" sheet will be cemented on with part F to the bottom of V-102, and the bottom of V-101. However, the sheet that rests on top of V-101 will not be adhered and will rest between the end cap (part C) and top of V-101. The end cap (part C)

which rests on the top of V-101 will also not be secured using cement. Since this cap is on the top of the column it should remain secure. This is so the resin can easily be removed during service in both columns. The 1" pipe fittings (Part E) will be fitted to both end caps (Part C) and will be connected to any required piping.

The following parts will not need to be glued to the column:

- End cap (Part C) on the top of the column
- 1/8" circular sheet on the top of the first and second column



**Figure 16.** Sketch of the ion exchange column frame design.

Construct the column frame according to the dimensions shown above. The column frame and column can both be constructed on its side or vertically. It is recommended to construct both vertically then begin constructing the column itself within the frame to minimize risk of injury.

Once the frame is set up, begin building the column from the bottom up ensuring each part is properly secured in place by the frame. Another point of consideration is that the CNC cut metal sheets that secure the column is provided in a CAD file. The CNC cut metal is also supported by P1100T rails and brackets (Parts M and O) and must be secured appropriately.

### **ii. Regeneration Instruction**

To remove resin from the column two operators will be required alongside two sturdy ladders. It is recommended to first disconnect the piping attached to the top end cap (part C) and remove the 1/8" clear PVC sheet. Once the top column resin is exposed, scoop out the cation resin until empty. Once it is empty, loosen the hose clamps around the 12" rubber coupling then proceed to wiggle V-101 out of the top end cap (part C). Once it is wiggled out enough. Have one operator hold the column in place while the other can scoop out the packed resin in V-102 into a bucket. Once all resin is removed, secure the 12" rubber coupling again and allow the resin to dry prior to shipping



the resin to an off-site facility. Information about off-site resin regeneration can be found in the regeneration quote attached in appendix G.

### iii. Implementation of a Bag Filter

Another concern brought to our attention during several visits to OFS's Sturbridge manufacturing site is the build-up of solid calcium compounds throughout the latter portion of their wastewater treatment's tanks and equipment. This issue can be attributed to the copious amounts of calcium chloride delivered to their reactors to reduce the concentration of free fluorine ions. Coagulants and flocculants are added to the wastewater stream and allow to easy removal of this white precipitate in the clarifier tank; however, the established system is not completely effective and results in excess calcium "caking" their effluent tank and clogging the pipes leading to sewer. This issue requires periodic cleanings and maintenance from operators, which can be time consuming and has the potential to bring the company's manufacturing operations to a halt.

To remediate this issue, we believe OFS would benefit from the addition of a bag filter within their wastewater treatment process. Filter bags are an effective, cost-efficient method for capturing contaminants and removing solid particles from a liquid stream. Calcium compounds, such as calcium carbonate, typically have particle sizes that range from 0.2 to 30 microns (Science Direct). Based on the severity of the issue, we recommend the use of a 0.2 micron filter bag, which would cost OFS about \$15.00 (Far West Fungi). A potential location for the bag filter includes within the preexisting metal strainer that cycles water through the effluent tank, similar to a reflux loop. This option would allow for the addition of a bag filter with a larger diameter opening, like 7 inches. Given the relatively low price and the negative impact of sold calcium build-up, we believe a bag filter would overall provide great benefit to the company.

### I. Estimation of Time Required to Reach Critical Temperature

From thermal data logs at OFS, the pumps that they use show a seal fluid temperature rise of about 1.1°C. To estimate the time that it will take for heat in the recirculation tank to accumulate past the temperature limit, we shall assume that this temperature rise is constant.

The amount of time that a "slug" of water travels through the tank, the recirculation piping, and the pump manifold can be simplified as simply the residence time  $\tau$  of the tank (this is where any "slug" of water will spend most of its time, so we are only considering this).

For a tank, the mean residence time is:

$$\tau = \frac{V_{tank}}{Q} = [hours]$$

$$\tau = \frac{500 \text{ gal}}{2 \text{ gpm}} = 250 \text{ minutes}$$

At the molecular scale, this represents the average amount of time a "slug" of water will spend in the reactor from inlet to outlet. Realistically, some will exit faster, and some will get stuck, but our calculated  $\tau$  is the mean value.

So, after 1000 minutes, the "slug" of water exits the tank and passes the pump where it is heated with a temperature rise of 1.1°C.

Based on the materials of construction (MOCs) of our recirculation system, we arbitrarily determined  $T_{limit} = 70^{\circ}C$ .

Assuming an initial temperature of  $20^{\circ}C$ , then the amount of cycles which a given slug of water can pass is

$$\frac{(70 - 20)^{\circ}C}{1.1 \frac{\circ C}{cycle}} = 45.45 \text{ cycles}$$

$$45.45 \text{ cycles} * 250 \text{ minutes} = 11362.5 \text{ minutes} = 7.89 \text{ days}$$