

---

# The “Spin-Grower”: A Machine for Rapid Layer-by-Layer Assembly of Nanostructured Materials

## Final Report

---

Team #17

Matthew Bachner, Yeh Chuin Poh,  
Thomas Serbowicz, and Steve Vozar

Sponsors: Prof. Nicholas A. Kotov,  
Prof. John Hart, and Paul Podsiadlo

Section Instructors: Prof. Jyoti Mazumder  
Dr. Guru Dinda

ME450 Section 04  
Winter 2008  
Department of Mechanical Engineering  
University of Michigan  
Ann Arbor, MI 48109-2125

15 April 2008



## EXECUTIVE SUMMARY

Layer-by-layer assembly is a method of producing multi-layered thin films. The conventional method of production consists of repeatedly dipping a substrate into a series of electrolyte solutions and solvents. This process is very time consuming, as it takes up to eight minutes for each layer to be deposited. The dipping method does not give a good surface finish and the film thickness of each layer is also not uniform [1]. The machines currently used for the dip method of production in Professor Kotov's lab are not capable of producing large samples in a reasonable amount of time. They are also unreliable and often break down.

The purpose of this project is to overcome the problems with the current method of production by developing a spin-assisted LBL assembly device. In the spin-assisted assembly method, oppositely charged solutions are sequentially delivered to a rapidly spinning substrate. The centrifugal force from the spinning substrate combined with the material viscosity and solution concentration determines the film thickness. This method significantly reduces the production time and produces highly ordered films with a much better surface finish [1].

The engineering specifications and requirements for the device were determined through discussions with the project sponsors and background research. This spin-assisted layer-by-layer assembly machine will need to have a spinning platform that holds a 10 cm (4 inch) diameter silicon wafer and can rotate at speeds ranging from 1,000 to 8,000 rpm. The machine will need integrated pumps that can inject solutions onto the wafer at any orientation without mixing them prior to deposition on a substrate. This spin-grower machine needs to be able to control the solution flow rates at approximately 0.5 mL/s for a duration of about 2 seconds. The controller must be able to control the sequence at which the different solutions are deposited for up to 3000 bilayers. The machine will also need to store four liters of five different solutions. The tubes and body of the machine must withstand chemical solutions with pH ranging from 1-10 [2]. Another optional but highly desirable feature is to include a device to measure the thickness of the film in real time with precision on the order of nanometers.

The major components of the final design for our system are as follows: one SCS G3P-8 spin coater, five Watson-Marlow 314 VDL/D peristaltic pumps with brushless DC motors, 1/16" ID bioprene tubing, three National Instruments USB-6008 DAQ boards, and fully articulating Loc-line hosing. We have also designed several custom components for the integration of the different subsystems, including a sheet metal pump rack, a modified spin coater top, and an electronics box. After product demonstration, we determined that an affordable laser measurement system would not be accurate enough to justify integration into the system.

Once constructed, the design was validated through a series of visual checks, system tests, and calibrations of the prototype. With proper calibration, the pumps can deliver a desired amount of liquid to within 0.1mL, without dripping after deposition. We were able to produce a 10-bilayer film of a clay-polymer nanocomposite with thickness and uniformity similar to the manually created spin-coated films created in Prof. Kotov's lab.

Improvements that could be made to this system include adding communications between the pumps and the spin coater, so that different layers could be applied at different spin rates. Additionally, a nitrogen purge on the inside of the spin coater might reduce the amount of liquid that condenses on the spin coater top.

This prototype is a tool for the Kotov lab to quickly produce nanostructured LBL materials. In order to determine the parameters of the system that produce the optimal films, we recommend a fractional factorial design of experiments approach for each combination of solutions.

## TABLE OF CONTENTS

1 ABSTRACT.....	5
2 INTRODUCTION .....	5
3 PROJECT REQUIREMENTS .....	6
3.1 Customer Requirements .....	6
3.2 Engineering Requirements .....	9
4 CONCEPT GENERATIONS AND SELECTION .....	9
4.1 Brainstorming.....	10
4.2 Functional Decomposition .....	10
4.3 Concept Generation Tree .....	11
4.4 Product and Literature Search.....	11
4.5 Concept Selection .....	11
4.6 Subsystem Concept Generation and Selection.....	11
4.6.1 Spin Coater.....	11
4.6.1.1 Spin Coater Concept Generation .....	11
4.6.1.2 Spin Coater Product Selection .....	13
4.6.2 Flow Regulation.....	13
4.6.2.1 Flow Regulation Concept Generation .....	13
4.6.2.2 Flow Regulation Product Selection .....	15
4.6.3 Fluid Delivery .....	16
4.6.3.1 Fluid Delivery Concept Generation.....	16
4.6.3.2 Fluid Delivery Product Selection.....	17
4.6.4 Real-time Thickness Measurement (RTTM) .....	18
4.6.4.1 RTTM Concept Generation .....	18
4.6.4.2 RTTM Product Selection.....	19
4.6.5 Data Acquisition and Analog Output (DAQ).....	19
4.6.5.1 DAQ Concept Generation.....	19
4.6.5.2 DAQ Product Selection .....	20
5 SELECTED CONCEPT DESCRIPTION .....	21
5.1 Final Design Description .....	21
5.1.1 Team-Designed Components .....	23
5.1.1.1 Modified spin coater cover.....	23
5.1.1.2 Pump rack.....	24
5.1.1.3 Electronics rack .....	25
5.1.1.4 Chuck adaptor.....	25
5.2 Prototype Description .....	25
5.3 Parameter Analysis .....	25
5.3.1 Electronics.....	26
5.3.2 Fluid Delivery Analysis .....	27
5.3.3 Thickness Measurement.....	27
5.3.4 Team-Designed Components .....	28
5.3.5 Design Analysis Assignment .....	28
5.4 Fabrication Plan .....	29
5.5 Validation Results.....	30
5.5.1 Electronics.....	30
5.5.2 Fluid Dispense System.....	31
5.5.3 Film Thickness with Ellipsometer.....	32
5.5.4 Film Thickness with Keyence Laser .....	32

5.5.5 Substrate Spin Speed.....	33
5.5.6 Complete System Validation.....	33
6 DISCUSSION.....	33
6.1 Customer Requirements.....	33
6.2 Peristaltic Pumps.....	33
6.3 Fluid Condensation.....	34
6.4 Variation of Spin Speed.....	34
6.5 Centralized Control.....	34
6.6 Thickness Measurement System.....	34
7 RECOMMENDATIONS.....	35
8 INFORMATION SOURCES.....	35
8.1 Spin-assisted LBL Assembly.....	35
8.2 Thickness Measurement System.....	36
9 CONCLUSIONS.....	36
10 ACKNOWLEDGEMENTS.....	37
11 REFERENCES.....	38
APPENDIX A - GANTT CHART.....	41
APPENDIX B – BRAINSTORMING AND CONCEPT GENERATION.....	42
APPENDIX C - SPRAY TESTING RESULTS.....	44
APPENDIX D – COMPONENT SPECIFICATIONS.....	45
APPENDIX E – PROJECT BILL OF MATERIALS.....	50
APPENDIX F – FLOW REGULATION FULL PUGH CHART.....	51
APPENDIX G – FLUID DELIVERY SYSTEM FULL PUGH CHART.....	52
APPENDIX H – DIMENSIONED DRAWINGS.....	53
APPENDIX I – DESIGN CHANGES SINCE DR3.....	66
APPENDIX J – DESIGN ANALYSIS ASSIGNMENTS.....	67
APPENDIX K – ELECTRICAL CONNECTIONS.....	76
APPENDIX L – INSTRUCTIONS FOR USE.....	77

## 1 ABSTRACT

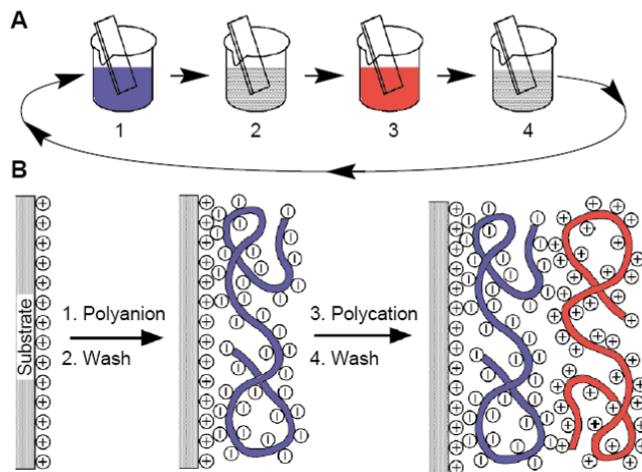
Layer-by-layer (LBL) assembly is a well-established method of producing multilayered nanostructured materials. In Professor Nicholas A. Kotov's lab at the University of Michigan, LBL assembly is often accomplished via a dip-coating process, which is time consuming and often performed on unreliable equipment. Spin-assisted LBL assembly has the potential to reduce the fabrication time of nanostructured materials by an order of magnitude and increase the quality of the films. The purpose of this project is to design and produce a spin-assisted LBL assembly prototype using a spin-coater and an automated fluid delivery system for the production of a variety of different nanocomposites.

## 2 INTRODUCTION

Layer-by-layer (LBL) assembly is a well-established method for the production of multi-layered thin films. In LBL assembly, oppositely charged compounds are sequentially stacked on a surface, which makes it ideal for the creation of nanostructured materials including sensing devices, optical devices, micromechanical devices, biological devices [3], antibacterial coatings, membranes, solar cells, batteries, and composite materials [4].

LBL assembly is usually performed by repeatedly dipping a substrate into a series of solutions (Fig. 1). However, this procedure is very time-consuming because the sample must be immersed in the electrolyte solution a long time for the substrate to be fully coated. Using the dipping technique, a single layer can take up to eight minutes to apply. Thus, producing a 300-layer sample takes almost 2 days of continuous dipping! In addition, the robots used to automatically perform this dipping process are unreliable and break often [2].

**Figure 1. LBL dipping method for adsorption of positive and negative solutions [5]**



LBL assembly can also be accomplished using a spin-assisted technique, in which the substrate spins rapidly while alternating compounds are delivered one layer at a time. The rapid spinning of the sample produces thin layers of a consistent thickness. Because of the centrifugal and viscous forces acting on the liquid compounds, molecules adhere to the substrate much faster than in the dipping process [1]. Layers can be created in a matter of seconds, thus reducing production times by an order of magnitude and creating the potential for thicker multilayer composites with new morphologies and properties [4].

The purpose of this project is to build a desktop-scale prototype system capable of implementing LBL assembly via a spin-coating process. The prototype will be able to automatically inject a pre-programmed amount of liquid per layer onto a substrate in a spin coater with controllable speeds. Auxiliary systems that could be implemented into this project if time allows include an optical device for the real-time measurement of sample thickness as well as an atmosphere (pressure and temperature) control system. This machine should be able to make an array of different functional nanomaterials for the ME450 design expo.

The sponsors of this project are Prof. John Hart in the mechanical engineering department as well as Prof. Nicholas A. Kotov and Paul Podsiadlo in the chemical engineering department at the University of Michigan.

### **3 PROJECT REQUIREMENTS**

We developed our qualitative project requirements by consulting with our project sponsors and observing the current LBL assembly setup. We then used a literature search combined with the sponsors' requests to develop engineering specifications for the prototype.

#### **3.1 Customer Requirements**

According to our sponsors, the spin-grower must be able to automatically deposit multiple solutions onto a substrate at a rapid pace. This is accomplished using the following mandatory requirements: The spin-grower must be capable of spinning at speeds high enough to create thin, uniform layers of liquid solution. The spin-grower should have the capability of holding substrates of silicon wafers or glass slides. It must also store five solutions that are to be kept separated prior to contact on the substrate, and the injection system should have the capability to prevent drips. The injection of these solutions must be precisely controlled. One of the solutions should be a solvent to allow rinsing between layer depositions. All components of the spin-grower that may potentially be in contact with the electrolyte solutions and solvent must be chemically compatible with each. The machine should be able to run continuously and autonomously to produce films with as many as 3000 bilayers. Due to the high speed spinning components, an adequate safety enclosure should also be incorporated. These key requirements are summarized in Table 1, p.7.

### **Table 1. Key Customer Requirements**

Automated injection  
Can hold silicon wafer or glass slides  
Chemically compatible  
Controllable delivery flow rate  
Deposit many bilayers (3000)  
Fast cycle time  
Fully flexible injection of fluid  
Multiple solution capability  
No mixing of solutions  
Precise volume delivery  
Prevents drips  
Safety enclosure  
Spinning platform

Other features exist that are desirable, but not as important as the requirements listed above. All operating functions should be centrally controlled and fully programmable. For increased automation, the system should have a large fluid capacity and allow fluid containers to be refilled easily. It would also be desirable to control the spin-coater speed from layer to layer, coordinating spin speed with individual fluid injections. If time allows, we would like to develop a real time measurement device that can report the growth of the thin film throughout the LBL process, as well as a means to control the atmospheric conditions in which the film is constructed.

Figure 2, p. 8 shows the Quality Function Deployment (QFD) chart or “house of quality” listing the customer requirements outlined above and their relation to engineering specification created to further quantify the customer requirements. The mandatory and highly important requirements are given customer weight values of 4 and 3, respectively. The less important and optional components were given weights of 2 and 1, respectively.





### 3.2 Engineering Requirements

We used the customer needs to determine the quantitative engineering specifications, towards which design efforts can be focused. The engineering specification categories were determined by brainstorming what aspects of the design would influence each customer requirement and weighted based on importance using values of 1,3, and 9 as indicated in the correlation legend in the QFD (Fig. 2 p. 8). Each customer requirement is related to at least two engineering specifications. Target values and acceptable ranges for the engineering specifications were developed through discussion with the customer and research of current spin coating processes. We have also determined the positive and negative interactions between the technical requirements and the magnitude of the interactions, indicated in the upper portion of the QFD. Table 2, below, contains the target engineering specifications determined for each of the technical requirements the range of acceptable values can be found in the full QFD on p. 8. The specification for nozzle diameter was changed to exit velocity as a more relevant engineering specification to avoid backslash.

**Table 2. Key engineering specifications**

<b>Engineering Specification</b>	<b>Target</b>
Minimum platform spin speed	1000 rpm
Maximum platform spin speed	8000 rpm
Platform acceleration	500 rpm/s
Substrate diameter	100 mm
Maximum fluid dispense rate	15 mL/min
Minimum fluid dispense rate	3 mL/min
Fluid dispense precision	100 µL
Minimum pH compatibility	1 pH
Maximum pH compatibility	10 pH
Nozzle quantity	5 qty
Power requirement	110 W
Reservoir capacity	4 L
Enclosure diameter	30.5 cm
Container quantity	5 qty
Exit Velocity	25.3 cm/s

## 4 CONCEPT GENERATION AND SELECTION

Once we established the customer requirements and engineering specifications for the project, we started generating ideas about how to solve our engineering problem. We used standard concept generation techniques such as brainstorming, functional decomposition, concept generation trees, and product and literature searches.

Our project is unique because it is heavily focused on the integration of different subsystems, rather than on the manufacturing of new parts. Each subsystem is almost fully independent of the other systems, allowing the design for each sub-function to be modular. Each subsystem design could be swapped with another design without significantly affecting the other components. For example, if we changed from

one fluid dispensing system to another, it would only affect how the dispensing system interfaces at the top of the spin coater. By combining all of the different ideas for subsystems, we could come up with dozens of different final designs!

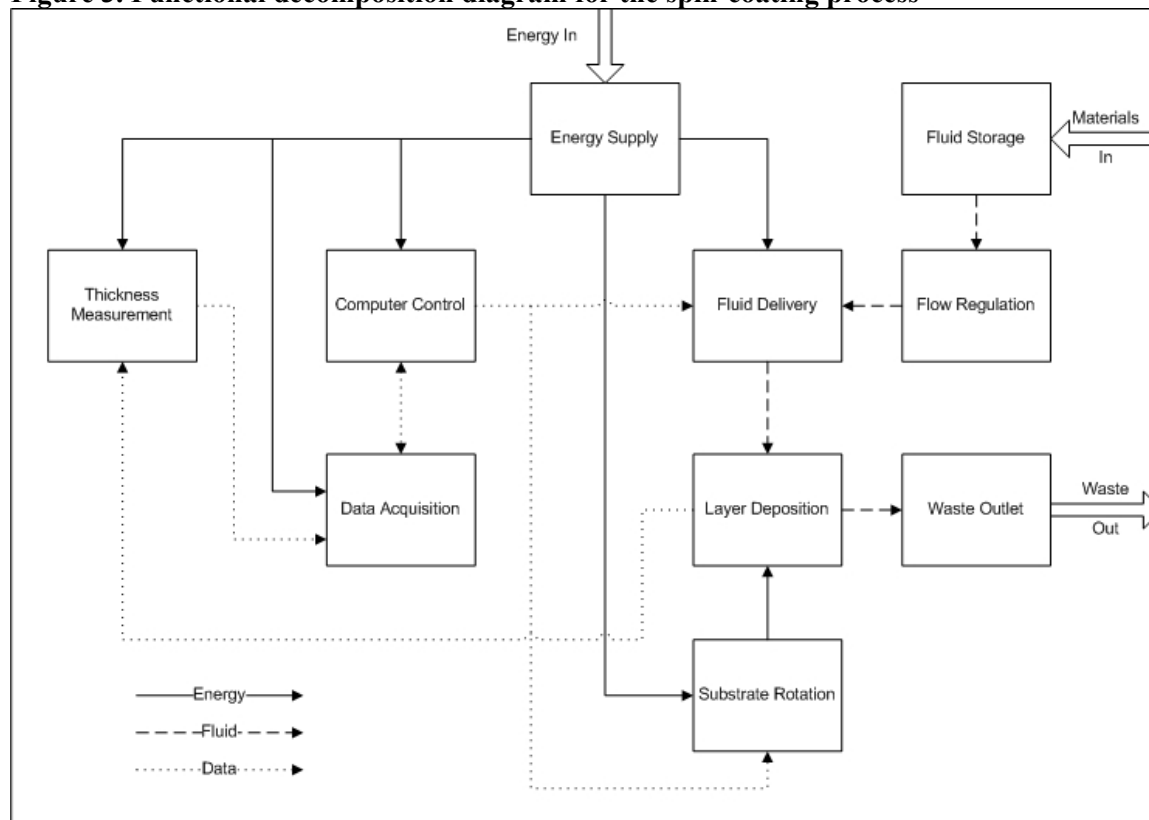
#### 4.1 Brainstorming

Brainstorming was done at the very beginning of the ideation process to develop many ideas very quickly. Ideas ranged from highly practical (standardize all power inputs, use LabView software to build a control program, use pipette tips as nozzles), to infeasible (build our own data acquisition hardware, store solutions in huge vats so they never need to be refilled). A full list of our brainstorming ideas can be found in Appendix B.1.

#### 4.2 Functional Decomposition

A functional decomposition for the process is shown in Fig. 3, below. The figure shows the flow of energy, data, and fluid. Producing and analyzing this decomposition helped us isolate different aspects of the system, and locate the difficult spots for the integration of the subsystems.

**Figure 3. Functional decomposition diagram for the spin-coating process**



### 4.3 Concept Generation Tree

A concept generation (branching) tree was developed for both the fluid delivery and flow regulation subsystems for easier organization of ideas. This way, we can further develop the branches with the most potential. The branching trees are shown in Appendix B.2.

### 4.4 Product and Literature Search

We spent a lot of time researching the best products to use in each of our subsystems. Because of the scope of the project, we are purchasing all of our major components, so our feasible concept selection is limited to the options available on the market today.

### 4.5 Concept Selection

We then narrowed down our ideas using a feasibility analysis taking into consideration our limited time and resources. The best ideas were then compared using a Pugh chart developed using the customer requirements of our QFD and weighting our selection criteria by order of importance. This took into account the function and engineering specifications associated with each of the options as well as the cost and availability. The highest ranking components were chosen for integration into the alpha design. These decisions were validated through discussion with our sponsors.

### 4.6 Subsystem Concept Generation and Selection

#### 4.6.1 Spin Coater

##### 4.6.1.1 Spin Coater Concept Generation

The spin coater is the central subcomponent of the system. All other components will interact with the spin coater in some way. The quality of the films produced will depend heavily on the spin coater itself, so the choice of this instrument is critical. The engineering requirements for the spin coater are shown in Table 3, below.

**Table 3. Engineering requirements for spin coater**

Spin Speed	1,000-10,000 RPM
Spin Acceleration	500 RPM/s
Substrate Size	4" (10.16 cm)
pH range	1-10

Since we are trying to create a centrally-controlled system, we would like to be able to control the speed of the spin coater remotely with a computer. Ideally, an "open-source" spin coater could be purchased that would allow us to input a voltage signal to regulate the spin speed. However, such a spin coater could not be found. Spin coaters are governed with analog, digital, and/or computer controls. It might be possible to hack into an analog control board and override the potentiometer inside, but this might be difficult or dangerous. It would be very difficult to hack into a digital control board without knowing the details of the control signals. A computer control system could run in the background along with a LabView program that we have written to communicate between the spin coater and flow regulation system. We briefly thought about building our own spin coater for this system, but quickly determined that the amount of time and effort required to build a spin coater would considerably reduce the scope of the project.

Other important aspects we considered when researching and choosing a spin coater were price, availability, number of necessary accessories (such as a vacuum chuck, a vacuum pump, or a lid for the coater), and safety and reliability of the system.

We performed a comprehensive product search and found five spin coaters that could possibly be integrated into our system.

First, we found the Laurell Medusa, which came as a complete package with a liquid delivery system and control software (Fig. 4a, p.13). However, at \$34,775, this system is very cost-prohibitive. Additionally, it might be difficult to later implement a thickness measurement system with the spin coater having a domed lid. Other products from Laurell were expensive (although not as expensive as the Medusa), and had to be controlled with either a digital keypad or proprietary computer software. The chemical compatibility, chuck size, spin speed, and acceleration for many Laurell systems met our engineering specifications [6].

Similarly, SCS produces spin coaters that can be controlled via a digital keypad or proprietary software (Fig 4b). However, the SCS coaters are approximately \$5,000, depending on the model, which is much more affordable than the Laurell machines. Additionally, the other engineering specifications are met by the G3-series spin coaters from SCS [7].

Both CHEMAT and Alfa Aesar sell very similar spin coaters controlled using analog knobs (Fig 4c). The price of both of these systems is about \$4000 (plus accessories). The spin speed is not quite as high as the other systems, and there is no integrated lid. These seem to be mid-quality spin coaters, but they are cheaper than the previous coaters, and the analog controllers would be easier to hack into than a digital control board [8],[9].

Finally, MTI produces a very cheap spin coater, which uses double-stick tape to hold the substrate to the spin coater (all the other systems use a vacuum) (Fig 4d). The maximum spin speed is only 5100 RPM [10], and the quality and reliability of this product is highly suspect. However, this spin coater is very affordable, and the analog controller might be easy to modify.

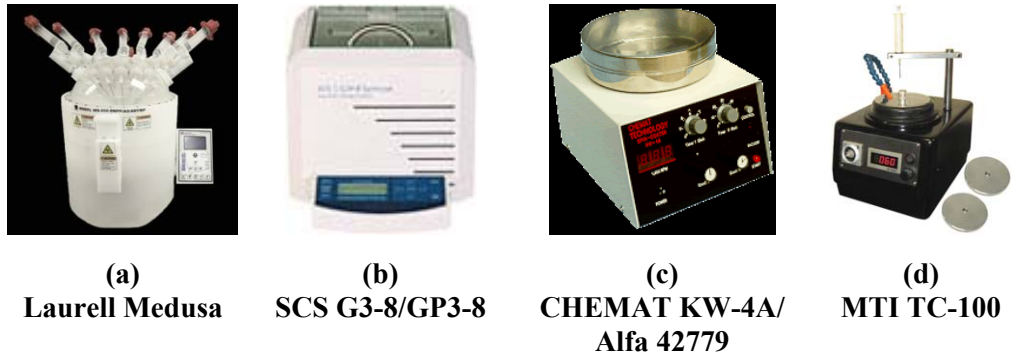
A summary of these products is given in Table 4, below.

**Table 4. Specifications of various spin coaters [6],[7],[8],[9],[10]**

	<b>Laurell Medusa</b>	<b>SCS G3P-8</b>	<b>SCS G3-8</b>	<b>CHEMAT KW-4A</b>	<b>Alfa 42779</b>	<b>MTI TC-100</b>
Base Price	\$34,775.00	\$4,797.00	\$3,250.00	\$4,050.00	\$4,056.00	\$1,495.00
Accessory Price	\$0.00	\$1,634.50	\$1,235.50	\$1,047.00	\$1,047.00	\$0.00
<i>Total Price</i>	<i>\$34,775.00</i>	<i>\$6,431.50</i>	<i>\$4,485.50</i>	<i>\$5,097.00</i>	<i>\$5,103.00</i>	<i>\$1,495.00</i>
Control Type	Computer	Computer	Digital	Analog	Analog	Analog
Max Spin Speed	10,000 RPM	10,000 RPM	10,000 RPM	8,000 RPM	8,000 RPM	5,100 RPM
Max Acceleration	~1,500 RPM/sec <sup>1</sup>	1,500 RPM/sec	1,500 RPM/sec	Unknown	Unknown	Unknown

<sup>1</sup>Experimentally determined

Figure 4. Photographs of compared spin coaters [6],[7],[8],[10]



#### 4.6.1.2 Spin Coater Product Selection

Using the information provided from each manufacturer, we were able to compile a Pugh chart to evaluate and rank each spin coater. Table 5 shows that the highest-ranking spin coater is the SCS model G3P-8. Since the spin coater is such a vital part of our design, we discussed this decision in depth with our project sponsor, who also agreed that that G3P-8 spin coater would be the best instrument for our project.

We also discussed the product in great detail with the manufacturer to ensure that we were getting everything that we needed. For example, the G3P-8 comes with a nitrogen purge that must be active in order for the instrument to turn on. This is to prevent hazardous gases from exiting the spin coater. However, we are not using anything that will produce hazardous gases, so we were able to get SCS to disable this requirement in the spin coater circuitry.

Table 5. Spin coater selection Pugh chart

Products (Spin Coater)													
Selection Criteria	Weight	Laurell Medusa		SCS G3P-8		SCS G3-8		CHEMAT KW-4A		Alfa 42779		MTI TC-100	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
Externally controllable	10	5	0.5	5	0.5	2	0.2	3	0.3	3	0.3	3	0.3
Ease of intergration to central control	5	5	0.25	4	0.2	1	0.05	3	0.15	3	0.15	3	0.15
Chemically compatible	10	5	0.5	5	0.5	5	0.5	4	0.4	4	0.4	2	0.2
Spin Rate	10	5	0.5	5	0.5	5	0.5	3	0.3	3	0.3	2	0.2
Spin Acceleration	5	5	0.25	5	0.25	5	0.25	3	0.15	3	0.15	3	0.15
Reliability	10	5	0.5	4	0.4	4	0.4	3	0.3	3	0.3	2	0.2
Chuck Size	5	5	0.25	5	0.25	5	0.25	5	0.25	5	0.25	4	0.2
Safety	10	4	0.4	4	0.4	4	0.4	3	0.3	3	0.3	2	0.2
Additional Accessories Needed	5	5	0.25	3	0.15	3	0.15	3	0.15	3	0.15	4	0.2
Availability	10	2	0.2	5	0.5	5	0.5	4	0.4	4	0.4	4	0.4
Price	20	1	0.2	3	0.6	4	0.8	4	0.8	4	0.8	5	1
Total Score	100	3.8		4.25		4		3.5		3.5		3.2	
Rank		3		1		2		4		4		6	
Purchase?		No		PURCHASE		No		No		No		No	

#### 4.6.2 Flow Regulation

##### 4.6.2.1 Flow Regulation Concept Generation

The flow regulation is the subcomponent of the spin-grower that will control the amount of fluid that will be delivered to the substrate. The engineering requirements for flow regulation are detailed in the QFD diagram on p.8, as well as summarized in Table 6, p.14.

**Table 6. Engineering requirements for flow regulation**

Volume flow	1 mL ( $\pm 0.2$ mL)
Flow rate	0.5 mL/sec
Volume capacity	min 3 Liters
pH range	1-10

The flow regulation component must prevent the solutions from mixing prior to deposition. Other qualities include low cost, external controllability, and safety. The main concepts considered include syringe pumps, flow meters, pressure vessels, and peristaltic pumps.

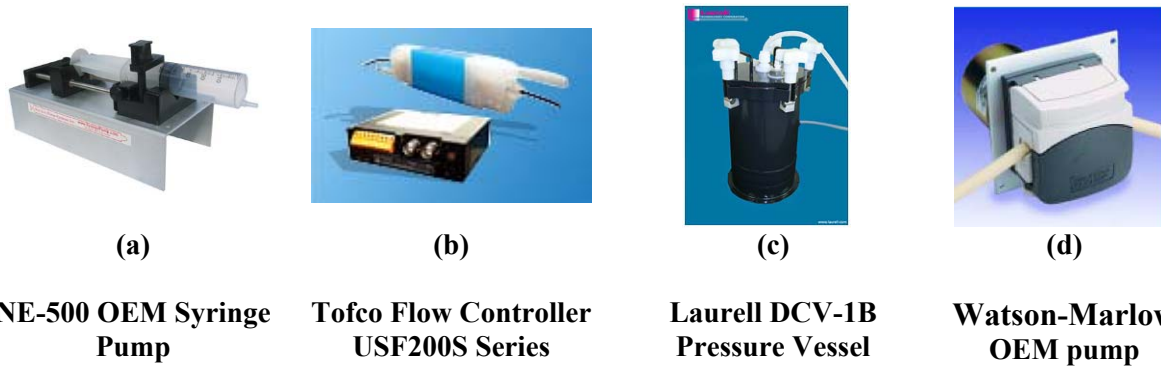
Syringe pumps could be used in our system as a flow regulator that would allow for high accuracy of volume flow and flow rate control (Fig. 5a, p.15). The pressure on the syringe would be voltage-controlled to allow for integration of a centrally controllable device. With the liquid only inside the cylinder and dispensing tubes, this concept could be made chemically compatible for our pH range. The low price and ease of integration to the system are advantages for this concept. Drawbacks with this concept include the low volume capacity and possibility of drips.

Flow controllers were another option for flow regulation (Fig. 5b). Flow controllers would have the benefit of drawing from a large capacity container using gravity to force the flow. Simplified calculations showed our flow rate would only require a few centimeters of head to reach desired flow rates. In order to meet our requirements, the fluid flow must also be able to completely stop. Flow controllers that met these requirements as well as chemical compatibility requirements were thousands of dollars each. Another drawback to this design is the possibility of drips.

Pressure vessels were considered while looking into the Laurell spin-coater system. Laurell had the option of syringe pumps or a pressure vessel for flow regulation (Fig. 5c). The pressure vessel allowed for a larger volume capacity. The pressure vessel would be centrally controllable with the spin-coater through Laurell software. Drawbacks of this concept are that it would require a pump to charge the vessel, and is integrated into the expensive Laurell system. A pressure vessel separate from the Laurell system would be difficult to integrate into a centrally controllable system.

Peristaltic pumps are another potential flow regulation system (Fig. 5d). In this solution, fluid only travels through a chemically compatible dispensing tube, which can draw from a large capacity reservoir. Multiple rollers on the pump allow for precise volume flow, and the tube diameter can be selected for the correct flow rate. The pump is controlled by a motor which is voltage controlled, and can be integrated into our system. Specifically, OEM peristaltic pumps are intended for integration into original designs, and are moderately expensive.

**Figure 5. Flow regulation concepts including syringe pumps (a), flow meters (b), pressure vessels (c), and peristaltic pumps (d) [11],[12],[6],[13]**



**4.6.2.2 Flow Regulation Concept Selection**

We did research to find suitable products for each concept, and used these products to compare among concepts. Table 7, below shows the comparison among concepts of OEM peristaltic pumps, OEM syringe pump, flow meters, and pressure vessels. From this Pugh chart we determined that peristaltic pumps are the optimal design. All four concepts met most of the demands, but the syringe pump did not have large capacity for fluids, and the flow meters were very expensive to be chemical compatible. The pressure vessels would work well within the Laurell system, but outside of that would be hard to control and integrate with the rest of the system. Table 8, p.16 shows the comparison of some peristaltic pumps we researched. The full list of flow regulation products compared is included in Appendix F.

**Table 7. Comparison of flow regulation concepts for engineering requirements**

Selection Criteria	weight	Concepts							
		OEM peristaltic pump		OEM syringe pump		Flow meter		Pressure vessel	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
External control	10	5	0.5	4	0.4	4	0.4	2	0.2
Ease of integration to central control	5	4	0.2	4	0.2	4	0.2	2	0.1
Chemically compatible	15	5	0.75	5	0.75	2	0.3	3	0.45
Controllable delivery flow rate	10	3	0.3	5	0.5	3	0.3	5	0.5
Deposit many bilayers (3000)	5	3	0.15	1	0.05	3	0.15	5	0.25
Easy to refill fluids	5	5	0.25	1	0.05	5	0.25	1	0.05
Fast cycle time	2	4	0.08	3	0.06	2	0.04	5	0.1
Large fluid capacity	13	5	0.65	1	0.13	5	0.65	4	0.52
Multiple solution capability	5	2	0.1	2	0.1	1	0.05	4	0.2
No mixing of solutions	5	4	0.2	4	0.2	5	0.25	4	0.2
Precise volume delivery	10	2	0.2	5	0.5	5	0.5	3	0.3
Prevents drips	5	5	0.25	1	0.05	1	0.05	3	0.15
Safety	10	4	0.4	5	0.5	5	0.5	2	0.2
Total Score	100	4.03		3.49		3.64		3.22	
Rank		1		3		2		4	
Continue?		Develop		No		No		No	

**Table 8. Comparison of peristaltic pumps for flow regulation**

Products (Flow Regulation: Peristaltic Pumps)									
Selection Criteria	weight	Watson-Marlow 300S OEM with Brushless DC motor		Barnant Multichannel Pump		Watson-Marlow 100S OEM		Watson-Marlow multichannel	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
External control	15	5	0.75	2	0.3	5	0.75	2	0.3
Ease of intergration to central control	15	4	0.6	2	0.3	4	0.6	2	0.3
Chemically compatible	5	5	0.25	5	0.25	5	0.25	5	0.25
Controllable delivery flow rate	5	3	0.15	5	0.25	2	0.1	5	0.25
Easy to refill fluids	5	5	0.25	5	0.25	4	0.2	5	0.25
Multiple solution capability	5	2	0.1	2	0.1	2	0.1	2	0.1
Precise volume delivery	5	4	0.2	5	0.25	3	0.15	5	0.25
Prevents drips	5	5	0.25	2	0.1	4	0.2	2	0.1
Safety	10	4	0.4	5	0.5	4	0.4	5	0.5
Availability	10	5	0.5	4	0.4	4	0.4	5	0.5
Price	20	4	0.8	2	0.4	5	1	2	0.4
Total Score	100	4.25		3.1		4.15		3.2	
Rank		1		7		2		6	
Purchase?		PURCHASE		No		No		No	

By researching various products and manufactures, we identified several peristaltic pumps that we could use for our application. We then used a Pugh chart to select the specific pump that we decided to purchase. Table 8 shows that we selected the Watson-Marlow 314VDL/D variable speed pump with a brushless DC motor for our design.

The peristaltic pumps work on the principle of positive displacement. The spinning rotor continuously squeezes the fluid through the tubing. This is similar to the way blood is pumped through the body. The motors of the pumps are controlled by a 0-4V input signal and a direction control signal. The specifications for pump speed and flow rates are given in Fig. D2 and D3 in Appendix D.

### 4.6.3 Fluid Delivery

#### 4.6.3.1 Fluid Delivery Concept Generation

The fluid delivery subsystem must be able to repeatedly deliver fluid onto the spinning substrate through the spin coater lid. It should also prevent mixing of the polyelectrolyte solution prior to contact on the substrate surface. The outlet velocity of the fluid is important because backsplash is undesirable. Four main concepts were developed for fluid delivery and they are summarized below.

Fixed center injection was the first concept developed (Fig. 6a, p.17). It would involve having five nozzles set to a fixed location and angle surrounding the center of the substrate. While this concept guarantees a repeatable single injection location, it lacks flexibility in injection location and angle.

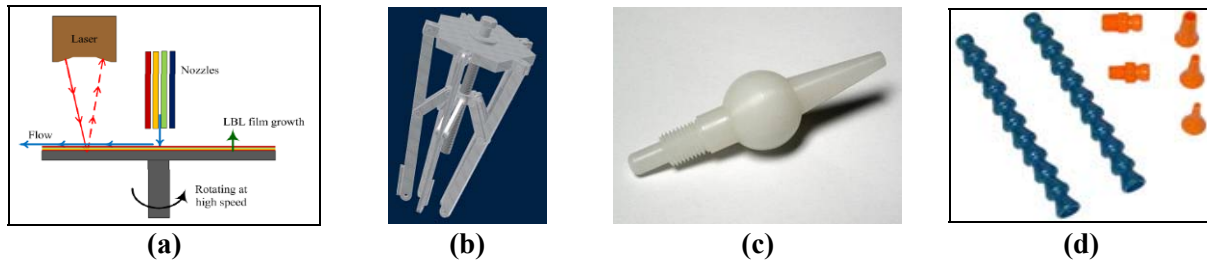
The uniform focus concept is another concept that was generated which involves a linkage mechanism with a central screw-drive that could adjust the injection location of all the nozzles equal distance from the center (Fig. 6b). The goal with this concept was to introduce some flexibility in injection location and allow the fluid to be injected from different heights while still aiming at the center of the substrate. Some drawbacks to this concept are the complexity of construction, material cost, and the dependence of the injection angle on nozzle location and height.

The ball joint concept is one that was developed through benchmark research on the Laurell spin-processor systems (Fig. 6c). A ball joint system would allow for good flexibility in injection location for each fluid independently; however the injection angle would still be dependent on injection location and height. This concept would be difficult to manufacture and be relatively high in cost.



Fully articulating tubing allows absolute freedom of location and injection angle for each fluid independently (Fig. 6d). This type of tubing is commonly used for coolant fluid injection in many machining processes and therefore has a low cost and high availability. The hose will be easily integrated with the spin coater lid with the aid of incorporated National Pipe Thread (NPT) connectors. This system will remain chemically compatible because it allows for the peristaltic pump tubing to be run through the inside, preventing fluid from ever coming in contact with the inside surfaces.

**Figure 6. Concepts for fluid delivery including vertical nozzles (a), focusing mechanism (b), ball joint (c), and fully articulating tubing (d) [14],[15]**



#### 4.6.3.2 Fluid Delivery Concept Selection

The concept selection process for the fluid delivery subsystem involved a review of the project customer requirements to determine which requirements directly related to fluid delivery. These requirements were then listed as selection criteria and given a weight based on their relative importance. A matrix was then compiled in the form of a Pugh chart (Table 9, below). Based on the results of the Pugh chart, the fully articulated tubing concept was chosen to develop. A Pugh Chart showing all fluid delivery concepts can be found in Appendix G.

We have chosen to purchase 1/4" ID fully articulated tubing from Loc-line. We will insert our 3/16" OD bioprene pump tubing from the peristaltic pumps through the Loc-line to ensure chemical compatibility. The inside diameter of the bioprene tubing is 1/16", which produces an outlet velocity that will not cause splash back (see Section 8.2, p.27).

**Table 9. Pugh chart comparison of fluid delivery systems**

Selection Criteria	Concepts								
	weight	Fixed Vertical Injection		Uniform Focus		Ball Joint		Fully Articulating Tube	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
Repeatable delivery position	15	5	0.75	4	0.6	4	0.6	3	0.45
No mixing of solutions	15	3	0.45	4	0.6	4	0.6	4	0.6
Ability to vary outlet velocity	7	3	0.21	3	0.21	2	0.14	2	0.14
Ease of integration into lid	6	4	0.24	2	0.12	1	0.06	5	0.3
Complexity	6	5	0.3	1	0.06	3	0.18	5	0.3
Low Cost	9	5	0.45	2	0.18	1	0.09	4	0.36
Chemical Compatibility	15	5	0.75	4	0.6	4	0.6	4	0.6
Freedom of deposition location	11	1	0.11	3	0.33	5	0.55	5	0.55
Freedom of deposition angle	11	1	0.11	1	0.11	3	0.33	5	0.55
Ease of integration with flow regulation system	5	4	0.2	2	0.1	2	0.1	4	0.2
Total Score	100		3.57		2.91		3.25		4.05
Rank			2		4		3		1
Continue?			No		No		No		Develop

## 4.6.4 Real-time Thickness Measurement (RTTM)

### 4.6.4.1 RTTM Concept Generation

Real-time thickness measurement is not a key customer requirement. It is an optional but highly desirable feature to include in the LBL assembly process. Every polyelectrolyte layer deposited is approximately 5 nanometers thick, and it is desired for the optical measurement system to measure the thickness of every layer deposition in real time. The spin coater will be spinning at approximately 3000 RPM and thickness measurement should be done while the substrate is still spinning. Stopping the spin coater to measure the thickness is not desired as it would drastically slow the pace of film growth.

Because we want a real-time, noninvasive, highly precise measurement, optical techniques are the only practical solutions. We considered three different optical measurement systems: an interferometer, an ellipsometer, and a laser displacement sensor.

Figure 7a, below, shows an interferometer from SIOS. An interferometer is a laser device that makes highly accurate and traceable measurements. It is able to measure thickness layers with a resolution of  $\pm 0.5\text{nm}$  and has a high sampling rate. The substrate that is being measured has to be static. Any motion will cause a change in the beam angle and the beam reflectors will need to be readjusted. The cost of this system is over \$90,000 [16].

Figure 7b shows an example of an ellipsometer from J.A. Woollam. An ellipsometer utilizes the reflection angle of a beam to measure the thickness of a substrate. It can measure thickness to less than one nanometer. The ellipsometer uses a CCD detection system that provides a real time contrast image of the sample, which provides information about film thickness and reflective index [17]. The substrate that is being measured has to be static. Any motion will cause the deflection of the beam to change. The cost of this system is over \$70,000.

Figure 7c is an example of a Keyence laser displacement sensor. A displacement sensor is a device which continuously measures distance as an object moves. It can also be used to measure dimensions such as height, width, or thickness of an object. Due to our experimental conditions, we are focusing on a Keyence laser displacement sensor that has the world's fastest sampling rate of 50 kHz. It has a resolution of up to 10 nm and linearity of  $\pm 0.03\%$  of full scale (full scale =  $\pm 1\text{ mm}$ ). The influence of target surface conditions is minimal. The cost of this system is \$7,000 plus the cost of the mounting equipment [18].

**Figure 7. Concepts for RTTM including an interferometer (a), ellipsometer (b), and laser displacement sensor (c) [16],[17],[18]**



(a)



(b)



(c)

#### 4.6.4.2 RTTM Concept Selection

**Table 10. Comparison of optical measuring devices for engineering requirements**

Products (Thickness measurement: Optical Measuring Device)							
Selection Criteria	Weight	Interferometer		Ellipsometer		Laser Displacement Sensor	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
Resolution	5	5	0.25	4	0.2	3	0.15
Safety	5	3	0.15	3	0.15	3	0.15
Sampling Rate	10	3	0.3	2	0.2	5	0.5
Vibration Isolation	10	5	0.5	5	0.5	5	0.5
Ease of Integration	15	3	0.45	3	0.45	5	0.75
Minimum Wobbling Effect	10	2	0.2	3	0.3	4	0.4
Availability	15	2	0.3	2	0.3	5	0.75
Price	30	1	0.3	2	0.6	5	1.5
Total Score	100	2.45		2.7		4.7	
Rank		3		2		1	
Continue?		No		No		<b>Develop</b>	

The feasibility of integrating the different measuring devices was considered during the concept selection process. There will be some splashing of fluids during the LBL assembly through spin coating. This requires the substrate to be contained and the laser sensor needs to be outside of the spin coater. An interferometer requires a beam splitter and mirrors. Splashing of fluids on the beam splitter and mirrors will disrupt the readings. It becomes an additional challenge to mount the beam splitter and mirrors externally.

Cost was also consideration in the selection process. Due to the incredibly high cost of the interferometer and the ellipsometer, they are not practical for our application.

The most feasible, affordable and reasonable solution for thickness measurement is the Keyence laser displacement sensor. This is confirmed by our Pugh chart for measurement systems (Table 10, above). The challenge will be isolating the sensor head from the mechanical vibration of the apparatus. Even though the laser displacement sensor has a resolution of 10 nm, it has a high sampling rate of 50 kHz. The thickness measurement may not be as accurate as an ellipsometer or interferometer, but the high sampling rate will give an average thickness reading and it will also show the rate of thickness development in real time. The Keyence laser displacement sensor also has another advantage: It is programmable and gives control over the thickness development. It will allow the user to set the thickness limit upon which the spin coating system will stop.

A Keyence representative came with a sample laser displacement sensor to test whether it fulfils our engineering requirements and whether it is feasible to integrate. Some tests were run on the laser displacement sensor and the results were not favorable. We have decided not to purchase the laser sensor as it is expensive and is not currently suitable for our application. It might be possible to implement a real time thickness measuring system if more time is available to perform further testing. Detailed results of the tests are given in Section 5.5.4 Validation Plan of film thickness (p.32).

#### 4.6.5 Data Acquisition and Analog Output (DAQ)

##### 4.6.5.1 DAQ Concept Generation

We need data acquisition and analog output (DAQ) hardware to interface the control aspects of our system. The spin coater has an output trigger of 24VDC that will allow us to coordinate different spin

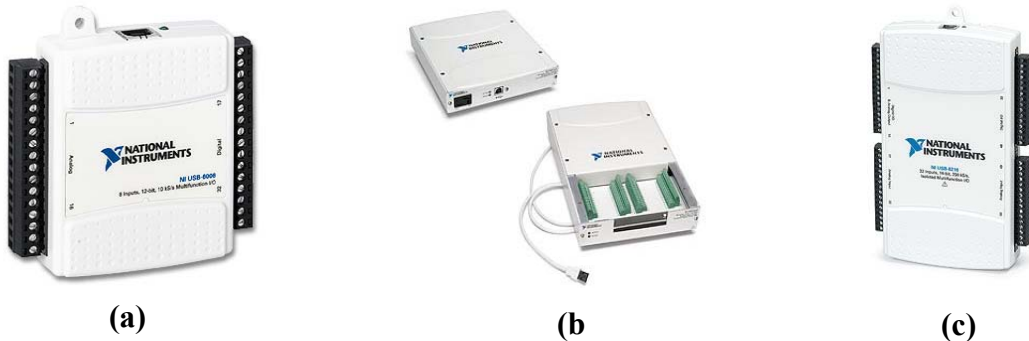
cycles with different solution injections [19]. The thickness measurement system also has one analog output that needs to be fed into the computer. Additionally, each pump has a speed input, ranging from 0-4V, and a direction input (clockwise/counterclockwise), ranging from 0-5V. This means that we need 10 analog outputs [20]. However, since only one pump will be running at a time during the spin coating process, we could control all of the direction inputs with one signal, which would reduce the number of outputs to six. When purging the air out of the pumps, they would all be running the same direction, so reducing the number of outputs would not affect the purge process.

Since we are planning to use LabView to write a central control program for the system, we decided to look only at National Instruments DAQ products. There are third party systems available, but in order to keep our search reasonable, we restricted our search to NI products. We spoke with Becky Linton, the NI representative for the University of Michigan, who suggested two products, the NI USB-6251 and NI USB-6218, both of which are higher-end DAQ boards. We also spoke with ME undergraduate lab supervisor Tom Bress, who recommended the more economical NI USB-6008 and NI USB-6009 boards. Table 11, below, compares the four products. We are only considering USB connections for the DAQ boards because they don't require any additional hardware, making them easy to implement and switch between computers.

**Table 11. Comparison of DAQ hardware [21]**

	NI USB-6009	NI USB-6008	NI USB-6251	NI USB-6218
Bus	USB	USB	USB	USB
Analog Inputs	4 differential	4 differential	8 differential	16 differential
Analog Outputs	2	2	2	2
Input Resolution	14 bits	12 bits	16 bits	16 bits
Output Resolution	12 bits	12 bits	16 bits	16 bits
Max Input Rate	48 kS/s	10 kS/s	1.25 MS/s	250 kS/s
Output Rate	150 Hz	150 Hz	2.86 MS/s	250 kS/s
Input Range	$\pm 1$ to $\pm 20$ V	$\pm 1$ to $\pm 20$ V	0 to $\pm 10$ V	0 to $\pm 10$ V
Output Range	0 to 5 V	0 to 5 V	0 to $\pm 10$ V	0 to $\pm 10$ V
Price	\$242.10	\$143.10	\$1,214.10	\$1,079.10
Ships within	1-2 days	1-2 days	2-5 days	1-3 days

**Figure 8. Photographs of the NI USB-6008/6009 (a), NI USB-6251 (b), and NI USB-6218 (c) [21]**



#### 4.6.5.2 DAQ Product Selection

The most important aspect we considered for our DAQ boards was price. Since none of the options we looked at had more than 2 outputs, we would need to purchase three boards to get six outputs. Additionally, the input resolution is less important, because we plan to use a different DAQ board with a

resolution of higher than 16 bits to collect the data from our chosen laser system, but this will be purchased later if we decide to implement the thickness measurement system. This also means that the input range is not important because the only input signal is now a 0-24VDC binary signal coming from the spin coater. The actual magnitude of this voltage could be brought down to 5 or 10 V using a voltage splitter circuit.

The outputs of the DAQ board are a more important consideration than the inputs. The engineering analysis section (see Section 8.1, p.26) shows that the 150 Hz, 12 bit output should be sufficient for our system.

While the more expensive DAQ boards are faster and more powerful, the low-end systems would be fine for our needs. Thus, the decision basically came down to cost, which meant that that choosing three NI USB-6008 DAQ boards for our six outputs and one input was the obvious choice. The Pugh chart for DAQ boards (Table 12, below) confirms this choice.

**Table 12. DAQ selection Pugh chart**

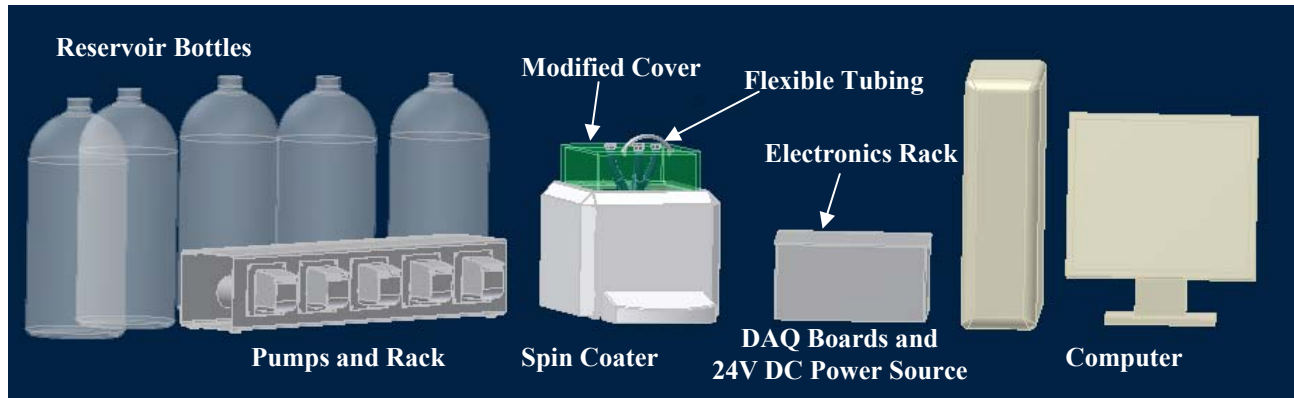
Products (DAQ)									
		NI USB-6009		NI USB-6008		NI USB-6251		NI USB-6218	
Selection Criteria	Weight	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
Input Resolution	5	4	0.2	3	0.15	5	0.25	5	0.25
Output Resolution	10	3	0.3	3	0.3	5	0.5	5	0.5
Sampling Rate	10	3	0.3	2	0.2	5	0.5	4	0.4
USB	10	5	0.5	5	0.5	5	0.5	5	0.5
Ease of Integration	10	5	0.5	5	0.5	4	0.4	4	0.4
Availability	15	5	0.75	5	0.75	4	0.6	5	0.75
Price	40	4	1.6	5	2	2	0.8	2	0.8
Total Score	100	4.15		4.4		3.55		3.6	
Rank		2		1		4		3	
Purchase?		No		<b>PURCHASE</b>		No		No	

## 5 SELECTED CONCEPT DESCRIPTION

### 5.1 Final Design Description

The major components chosen through the concept generation and selection process integrate with the team-designed components to form our final design. This design keeps all of the subsystems modular, facilitating flexible setup layouts and isolating electrical components from the potentially damaging aqueous solutions. Figure 9, p.22 is a rendering of how all of the components integrate into the final desktop system, and Fig. 10 is an image of the final setup. Figure 11, p.23 shows the flow of power, fluid, and data through the system. A summary of the selected purchased components and team designed components is shown in Table 13, p.22. The final bill of materials for the project is listed in Appendix E.

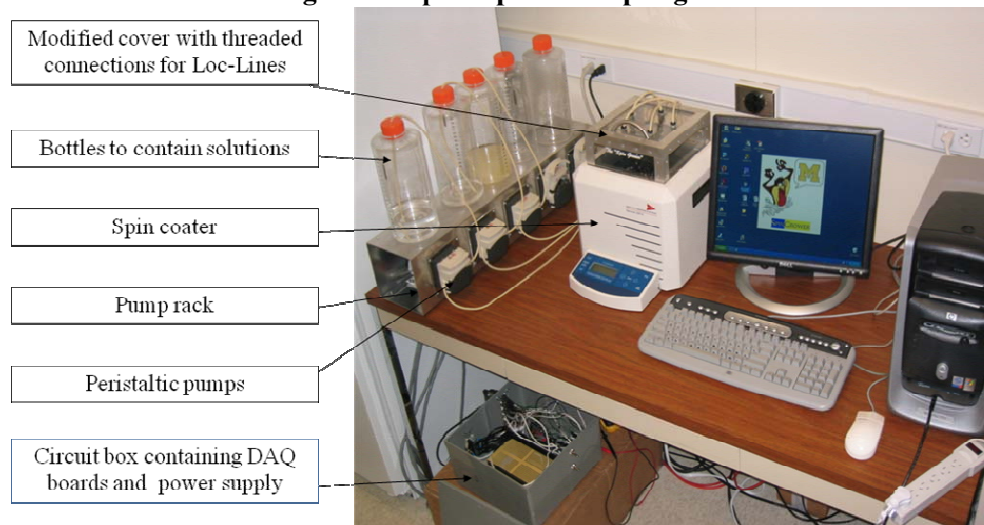
**Figure 9. The final design desktop setup for the spin-grower**



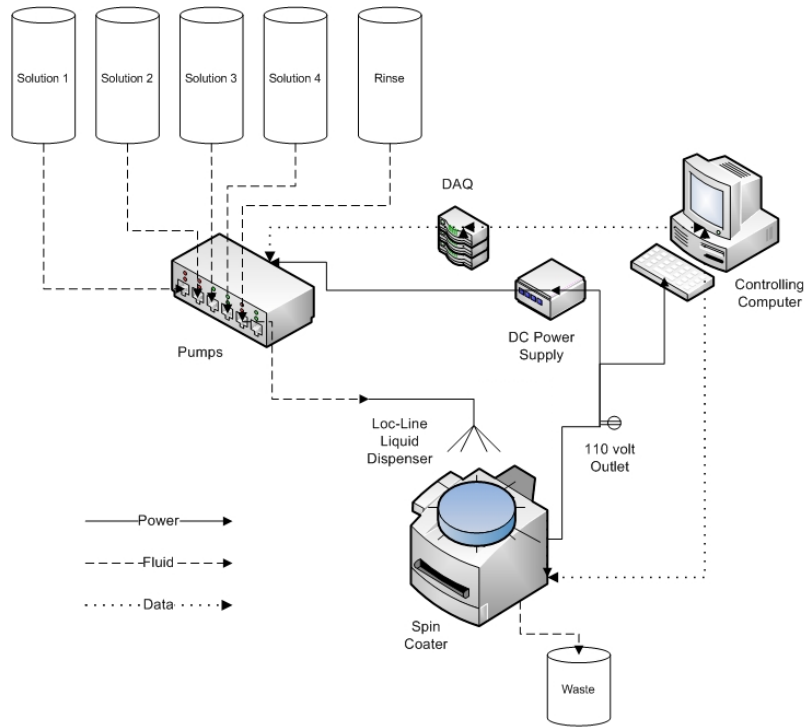
**Table 13. Description of final design system components.**

Component	Description
Manufacturer Purchased Components	
Computer	Dell Optiplex (OS: Windows Vista)
Spin Coater	SCS G3P-8
Peristaltic Pumps	Watson-Marlow 300 series high-precision OEM pumps
Pump Tubing	Watson-Marlow Bioprene Tubing, ID 1/16", Wall Thickness 1/16"
Data Acquisition Boards	National Instruments USB-6008
24V DC Power Source	Acopian Gold Box Unregulated DV Power Source
Fluid Delivery	Loc-Line Modular Hose System
Reservoir Bottles	(Available in sponsor's laboratory)
Team Designed Components	
Pump Rack	Simple aluminum rack bent in a break with holes for mounting pumps
Electronics Rack	PVC box with port or wires in back
Modified Coater Cover	Boxed up cover to allow sufficient length of tube to achieve all angles

**Figure 10. Picture of the final design desktop setup for the spin-grower**



**Figure 11. Diagram of alpha design system flows and connections**



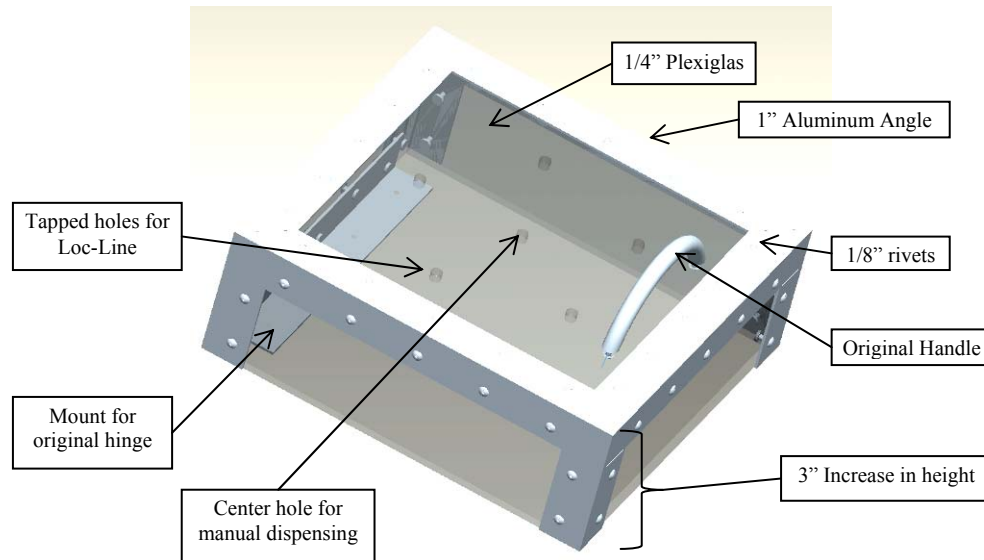
### 5.1.1 Team-Designed Components

Each of the individual subsystems and their key purchased components are discussed in detail in the Concept Generation section. The team designed components will now be discussed in more detail. The team-designed components are essential in the integration of all subsystems.

#### 5.1.1.1 Modified spin coater cover

The modified spin coater cover is a “bolt-on” addition to the coater, fully integrating the flow regulation, fluid delivery, and spin coater subsystems. The original flat cover left insufficient space to mount the Loc-Line tubing for fluid delivery. The modified cover adds three inches to the height and allows adequate space for the fully articulating tubes. The Plexiglas cover provides sufficient impact resistance against flying debris while still allowing the coating process to be viewed. The articulating tubes mount in the center of the underside of the spin coater cover via 1/4 inch NPT connectors incorporated into the tubing. The tubes are mounted regularly around a 5 inch diameter circle centered above the substrate. Care was taken to ensure that the surface area of the connection to the cover was large enough to prevent cracking due to substantial torque generated when adjusting the hose. A model of the new cover is shown in Fig. 12, p.24, detailing how the three subsystems will integrate. Engineering drawings of the modified spin coater cover are included in Appendix H.

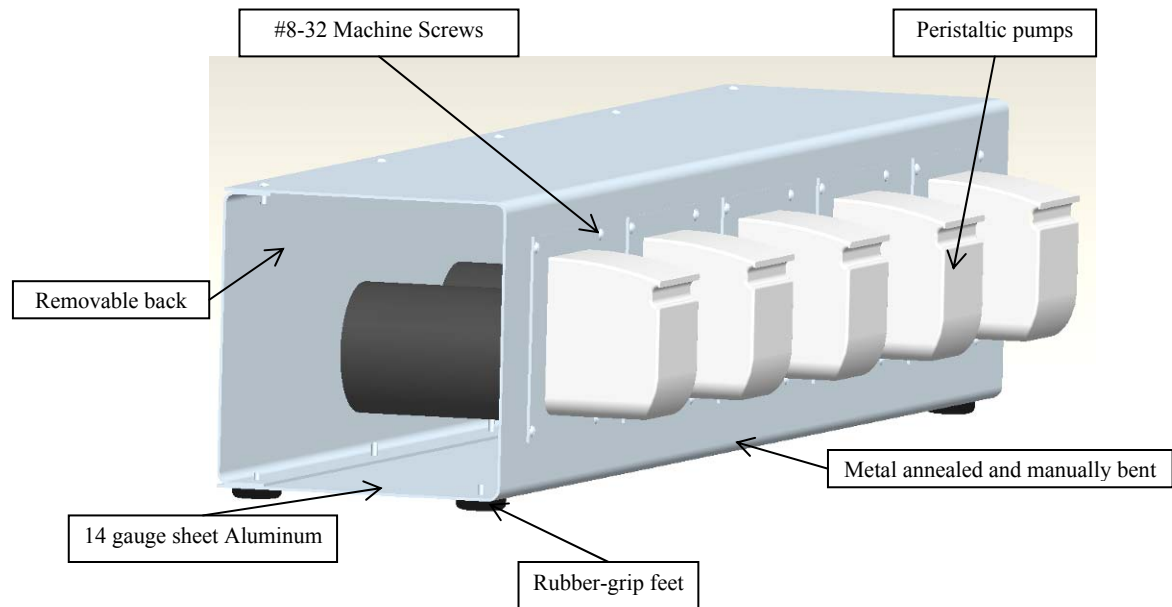
**Figure 12. View of modified spin coater lid**



### 5.1.1.2 Pump rack

The pump rack shown in Fig. 13, below, holds all five peristaltic pumps. The pump rack is made out of 14 gauge sheet aluminum and is manufactured to the dimensions specified in Appendix H. To shape the aluminum, we annealed it with an oxyacetylene torch and then bent it in a manual brake. The bending returns the aluminum close to its initial strength. Rubber feet on the bottom level the rack and ensure no slipping while the pumps are running. A removable back to mount wire connectors also allows for easier access to remove the pumps. Aluminum bolts, #8-32 x 3/8", mount the pumps and the removable back.

**Figure 13. Model of pump rack holding all five peristaltic pumps**





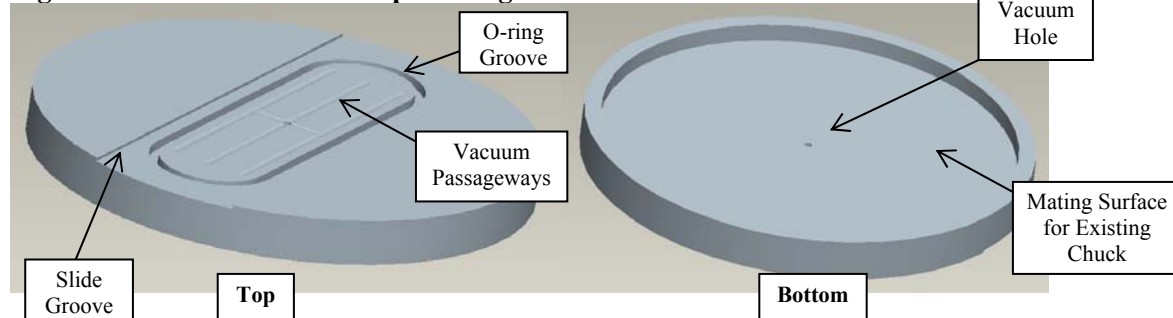
### 5.1.1.3 Electronics rack

The electronics rack houses all three DAQ boards as well as the 24V DC power source and a USB hub. It is a fully enclosed box with a port on the back for power input/outputs and communication cables. It is constructed out of a PVC junction box. There is sufficient space around the power source so that adequate convection can occur and prevent the power source from overheating. A detailed drawing of the modified junction box used for the rack is shown in Fig. H11, in Appendix H.

### 5.1.1.4 Chuck Adaptor

Due to the relatively high price of silicon substrates and the desire to use substrates that are smaller and of different geometry than the 10cm diameter round silicon wafers, an adaptor was created to hold 1" x 3" glass microscope slides. Originally we planned on creating an entirely new spin chuck that would be interchangeable with the one used to hold the silicon wafers, however due to foreseeable manufacturing difficulties, the new concept of creating an adaptor for the existing vacuum chuck was proposed. The vacuum chuck adaptor utilizes the vacuum passageways on the existing chuck as well as a precisely sized round pocket on the underside to hold itself in place. The chuck also has a one inch wide slot on the top side with an array of vacuum passageways for holding the microscope slide firmly in place. When the vacuum is engaged by the spin coater, the adaptor and microscope slide simultaneously seal to the existing chuck and adaptor respectively. A Viton O-ring in a groove prevents liquid from being able to enter the vacuum passageways. Assuming no free air flow when the vacuum is engaged, based on the design of our vacuum passageways and the capabilities of the vacuum pump a microscope slide is held in place with approximately 19.25 N. A model for the adaptor is shown in Fig. 14, below.

**Figure 14. Model of chuck adaptor for glass slides**



## 5.2 Prototype Description

For this project, we are creating a fully-functional laboratory instrument that will be used to produce thin-film nanostructured materials, thus our prototype fully demonstrates the complete final system. If desired, more instruments could be made using the documentation provided in this report. However, there are several design changes that could be implemented if this were to become a mass-produced commercial product.

First, we would have to be more concerned with the assembly of the device. For example, the modified spin coater top that we designed contains many parts that need to be put together. We also designed the top so that it could be machined using the tools and materials available to us. However, if we were going to make hundreds of these instruments, we might consider using a vacuum-formed plastic top instead of the riveted Plexiglas structure we have detailed. This would reduce the number of assembly parts and would thus reduce assembly time. Similarly, a plastic housing could be designed to hold the peristaltic pumps, instead of the metal pump rack, thus reducing component weight and cost.

If we were making a commercial product, we might also think about breaking away from the LabVIEW

control software and creating proprietary software that includes both the spin-coating software and the pump control algorithm. This might require us to work with the manufacturer of the spin coater to develop such software, or we could try to develop our own spin coater and write new software ourselves.

### 5.3 Parameter Analysis

For this project we used many of the engineering fundamentals we have learned in prior classes. Our knowledge of fluid mechanics allowed us to design an effective liquid injection system. The solutions used will have similar viscosity to water, but range from 1-10 in pH values. To accommodate for these types of solutions, the material selection for all components is crucial. We have also had to use our knowledge of electronics and controls to interface the system with a central computer controller. The laser measurement system draws on our knowledge of optics as well as vibration isolation.

The analyses presented in this section validate the purchases we have made and the designs of our custom components. Because they are the most expensive and most important aspects of our project, we performed a more detailed analysis on the purchased components. We had to make sure that we were purchasing quality components that would meet our engineering specifications and integrate well together. We used a less detailed analysis for the team-designed components. For easy integration, they are mostly based on the geometries and designs of the purchased components.

#### 5.3.1 Electronics

The power supply we purchased to power the five peristaltic pumps is rated to supply 24VDC and 10A, giving a maximum power output to 240W. Each pump runs on 24VDC, and consumes 35W at maximum power, thus drawing 1.46A. Therefore, even if all five pumps are running at full capacity, the power supply will be able to output more than enough power and amperage.

We used a cable with 18 AWG wire to transfer power and data to the pumps. This wire is rated at 5 amps [40], which is less than the maximum current that will be flowing through the wire. This wire was relatively thick, and to be able to be soldered into our D-connectors, we had to remove four out of the seven strands in the wire at the solder connection. However, we believe that the extra solder will make up for the reduced capacity of the trimmed wire at the connection. Additionally, the trimmed wire ends were approximately the same size as the wires that came with the pumps. Finally, the power rating for the wire is given for continuous power supply, but the pumps will not be running all the time. The rinse pump will be running the most, and with a 10-second dry time, and 2 second rinse and fluid deposition time, current will only be supplied for 2 out of every 24 seconds, for a duty cycle of 0.083. We had originally planned on using standard serial cables to transmit data and power, but they were 26 AWG or higher (rated at less than 0.8 amps [40]), which might be safe given the previous duty cycle, but we wanted to make sure the wires did not overheat.

The minimum output rate of the chosen DAQ hardware is 150 Hz, meaning that the signal will change in  $1/150^{\text{th}}$  of a second. Using 1/16" ID tubing, the max flow rate is 1.25 mL/sec [20]. Thus, at 150 Hz output rate, we would be able to provide a signal to control flow to within less than 0.01 mL of the desired amount, which is within our engineering specification of 0.1 mL.

Additionally, a 12 bit output is sufficient for our system. The motor input is 0-4V, which can be separated into  $2^{12}=4096$  discrete amounts, giving a motor input resolution of  $4V/2^{12}=1\text{mV}$ . Since the motor runs at 1000 RPM/volt, this gives us a resolution of 1 motor RPM. The pump is geared down from the motor such that the pump runs at 350 RPM when the motor is running at 4000 RPM [20], giving us 0.0875 pump revolution per motor revolution. Since the flow is 0.25mL/pump revolution [20], the flow is  $0.0875*0.25=0.0219$  mL/motor revolution. Multiplying this by our motor resolution of 1 RPM, we get a flow resolution of  $0.022$  mL/min= $0.0004$  mL/sec, which is well beyond our desired precision. This

resolution error will be insignificant compared to the other errors introduced into the system by stretching of the tube, electrical fluctuations, and pump inertial forces. Thus, a 12 bit output resolution is adequate.

### 5.3.2 Fluid Delivery Analysis

A high fluid outlet velocity may cause unwanted splash back and may tear the film. To determine an acceptable range of outlet velocities which prevented splashing, testing was conducted with a syringe and varying outlet diameters (see Appendix C). The testing resulted in a maximum acceptable outlet velocity of 60 cm/s for a substrate spin rate of 3000 rpm. The current nozzle diameter is 1/16 inch, equivalent to the inside diameter of the tubing being used in the flow control subsystem. At the target flow rate of 0.5 ml/s the resulting outlet velocity is calculated to be 25.3 cm/s with Eq. 1, below.

$$\dot{V} = vA \quad \text{Equation (1)}$$

Where  $\dot{V}$  the volumetric flow rate,  $v$  is the outlet velocity, and  $A$  is the outlet area.

To assure system chemical compatibility, the tubing used in the flow control subsystem was analyzed with a chemical compatibility chart. This is the main component that will be in direct contact with the pure solutions. The exceptions are minor splashes and mist contacting the external surface of the articulating tube, and excess solution in the spin coater pan.

### 5.3.3 Thickness Measurement

Dynamic measurement of the layer thickness is much more complicated than static measurement. The rotation of the substrate will likely cause wobbling of the surface. Also, the center of the substrate has to be positioned on the chuck very accurately because any form of eccentricity will cause vibrations.

Isolating the optical measuring device from the rest of the apparatus is a challenge. Fundamental engineering knowledge on dynamics and vibration is needed to address this issue. Simple analysis was done using idealized systems of a mass-spring-damper. The mass,  $m$  (kg) was assumed to be infinitely rigid while the spring is weightless with stiffness,  $K$  (lbs/in) and the damper is weightless and its damping coefficient is  $C$  (lbs/in./sec). [38]

The natural frequency,  $f_n$  (Hz cycles/sec) and critical damping,  $C_c$  is calculated using the simplified equations below. [38]

$$f_n = 3.13\sqrt{K/mg} \quad \text{Equation (2)}$$

$$C_c = 78396mf_n^2 \quad \text{Equation (3)}$$

A harmonic motion with sinusoidal motion of frequency  $f$  will have a transmissibility  $T$ , which is the ratio of the vibrational amplitude  $x$ , and mass response amplitude  $y$ . The equation for transmissibility is shown below. [38]

$$T = \frac{y}{x} = \left\{ \frac{1 + \left[ 2 \left( \frac{f}{f_n} \right) \left( \frac{C}{C_c} \right) \right]^2}{\left[ 1 - \left( \frac{f}{f_n} \right)^2 \right]^2 + \left[ 2 \left( \frac{f}{f_n} \right) \left( \frac{C}{C_c} \right) \right]^2} \right\}^{\frac{1}{2}} \quad \text{Equation (4)}$$

If the transmission of vibrations from the spin coater to the laser measurement system becomes an issue, we could use the analysis above to design dampers to isolate the laser system from mechanical vibrations.

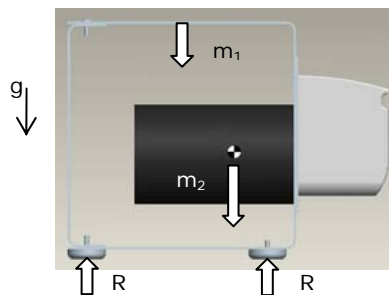
Testing with a demo laser measurement system showed significant drift of the reading between measurements. This is a result of more than just vibrations of the spin coater and table. A full discussion is given in Section 6.6, p.34.

### 5.3.4 Team Designed Components

The team-designed components were designed as simply as possible with basic engineering fundamentals in mind. We also considered the reliability, safety, and manufacturability of the components when determining their designs. However, intense engineering analysis for these components was not necessary. The most important factor we used to design the components was the geometries of the other components that they are integrated into.

For example, the pump rack was designed by measuring the dimensions of the pumps and spacing them far enough apart along the rack such that the tubing would not get tangled or kinked. We also assumed that there would be no significant forces on the pumps from the tension of the tubing. This can be ensured by leaving some slack in the tube lines. By simply picking up the pumps, we could tell that the center of mass was located in the pump motor. Thus, we designed the pump rack so that the center of mass of the pumps was between the two rubber supports (see Fig. 15, below). The other team-designed components were designed similarly, using basic engineering judgment.

**Figure 15. Center of mass of pump and pump rack is clearly located between the two reaction forces from the rubber supports, resulting in a stable system.**



The chuck adaptor features vacuum passageways to increase the surface area of the vacuum-glass slide interface. The grooves provide a  $2.187 \times 10^{-4} \text{ m}^2$  area, and the purchased pump provides a vacuum of about 88kPa [19]. Thus, the additional normal force on the slide is 19.25 N. This should be enough to prevent the slide from slipping off of the spin chuck if it is reasonably centered over the spinning axis.

### 5.3.5 Design Analysis Assignment

The additional assignments for this project were useful in developing and critiquing our design. Although they did not have a large impact on how our prototype was created, they would be useful if this were to be redesigned as a final product

Using the CES software we found that the materials we originally selected (Plexiglas and Aluminum 6061) were also recommended by the software. The details for material selection are in Appendix J1. The software was also useful at determining suitable manufacturing processes if our product were to be mass produced and details are shown in Appendix J2.

The SimaPro software allowed us to get a larger vision of the environmental impact from the aluminum and Plexiglas chosen. The aluminum had a much larger impact than the Plexiglas, but it is still an appropriate material for the amount used in the project. Details of design for environmental sustainability are in Appendix J3.

With fast rotating motors in our machine, and acidic chemicals, safety was always a concern. The designSafe software provided supporting evidence that with proper use our machine does not have any high risk associated with it. Details of design for safety are in Appendix J4.

The design for assembly assignment focused more towards how a redesign of the prototype would make the final product easier and faster to assemble. We estimate that the redesign proposed would reduce assembly time from 840 seconds to about 458 seconds. Details of design for assembly are in Appendix J5.

### 5.4 Fabrication Plan

The fabrication plan for the team designed components was developed with several important considerations in mind. First, the components must be designed for use within a laboratory. We therefore want a quality of construction supporting prolonged and reliable equipment use. Second, they must be made using the available machine shops. Finally, they must be designed for fabrication with readily available materials in a reasonable amount of time. Table 14, p.29-30 shows the process plan for the team-designed components.

**Table 14. Process plan for team-designed components (Continued on next page)**

Ops No.	Machine Process	Part Name	Step Instructions and Parameters	Tool used
<b>1. Obtain Materials</b>				
1.1	Purchasing	Buy Plexiglas	24x36x0.2"	
1.2	Purchasing	Buy Angle Aluminum	120x1x0.0625"	
1.3	Purchasing	Buy Sheet Aluminum	36x48", 14 gauge	
1.4	Purchasing	Buy Cable	50ft long, 7 wire, 18ga.	
1.5	Purchasing	Buy Electronics Box		
1.6	Purchasing	Buy Misc. Assembly Materials	Pop rivets, silicone caulk, backing washers, Molex connectors, USB hub	
<b>2. Manufacture of modified spin coater cover top panels</b>				
2.1	Etching	Plexiglas - Delivery hole locations	Etch locations of 5 fluid delivery holes	Laser Cutter
2.2	Laser Cutting	Plexiglas - Spin Cover Panels	Cut geometry of modified spin coater top, including rivet holes	Laser Cutter
2.3	Drilling	Plexiglas – Delivery Holes	Drill fluid delivery holes	7/16" Drill
2.4	Tapping	Plexiglas – Delivery Holes	Tap fluid delivery holes	1/2" NPT Tap
2.5	Filing	Plexiglas	File all sharp edges	File
<b>3. Manufacture of modified spin coater side brackets</b>				
3.1	Cutting	Angle Aluminum Bracket	Cut Aluminum angle to size and shape	Bandsaw
3.2	Drilling	Angle Aluminum Bracket	Drill rivet holes in angle bracket	
<b>4. Assembly of Modified Spin Coater Top</b>				
4.1	Assembly	Spin Coater Top	Assemble spin coater top with pop rivets and silicone caulk	Pop Rivet Gun, Caulk Gun
<b>5. Manufacture of Pump Rack</b>				
5.1	Shearing	Sheet aluminum	Shear sheet aluminum to proper shape	Shear
5.2	Drilling	Sheet aluminum	Drill bolt holes in sheet aluminum	#29 (0.136")
5.3	Tapping	Sheet aluminum	Tap appropriate bolt holes in sheet aluminum	8-32 Tap
5.4	Annealing	Sheet aluminum	Anneal aluminum sheet in locations to be bent	Oxy-Acet. Torch
5.5	Bending	Sheet aluminum	Manually bend aluminum to pump rack geometry	Brake
5.6	Cutting	Pump Rack	Cut out holes for pumps and connectors	Hacksaw
5.7	Filing	Pump Rack	File all sharp edges	File

**Table 14. Continued**

Ops No.	Machine Process	Part Name	Step Instructions and Parameters	Tool used
<b>6. Assembly of Pump Rack</b>				
6.1	Assembly	Pump Rack	Assemble Pump Rack with bolts, nuts, washers, and pumps	
<b>7. Manufacture and Assembly of Data/Power Cables</b>				
7.1	Cutting	Cable	Cut multi-wire cable to 5 10' lengths	Wire Cutter
7.2	Stripping	Cable	Strip end of each wire 0.25"	Wire Stripper
7.3	Soldering Connector	Cable/Connectors	Solder wire into D-connectors, insulate with electrical tape and heat shrink	Soldering Iron, Heat Gun
7.4	Install Hoods	Cable/Hoods	Install hoods over connectors	Screwdriver
7.5	Connect Cable	Cable	Connect Cables to pumps, power, and DAQ	Screwdriver
<b>8. Manufacture and Assembly Electronics Box</b>				
8.1	Drilling	Electronics Box	Drill wire and mounting holes in electronics box	Hand Drill
8.2	Soldering Connector	Wires/Connectors	Solder individual wires into D-connectors to be mounted on box, insulate with heat shrink	Soldering Iron, Heat Gun
8.3	Mounting	Electronics Box	Mount DAQ Boards and Power supply in Electronics box	Screwdriver
8.4	Connect Cable	Cables, Connectors, Termination Strip	Connect power to termination strip, and DAQ USB Cables to computer	Screwdriver
<b>9. Manufacture of Chuck Adaptor</b>				
9.1	Cutting	Delrin	Cut Delrin 3.75" Delrin stock to size	Bandsaw
9.2	Turning	Delrin	Turn down Delrin to 3.5", Face bottom cavity, Drill Vacuum Hole	Lathe, #55 Drill bit
9.3	Milling	Delrin	Face top of chuck adaptor, mill slide cutout, mill vacuum passageways	Mill, 1/2" End Mill
9.4	Soldering	Viton O-ring kit	Cut and solder Viton O-ring to size	Soldering Iron
9.5	Assembly	O-ring, Chuck adaptor	Install O-ring into chuck adaptor with O-ring grease	

Tolerances on all parts of the modified spin coater top are important due to the fact that the final product involves the assembly of many parts with rivets, and we want to ensure good aesthetic quality. The hole locations on the pump rack are also critical to ensure proper fit between the front and back panel, however the size of the pump cutouts can be held to a lower tolerance due to the pump backing plates being slightly over-sized. The finish of the mating surfaces of the chuck adaptor must be very fine, to keep the vacuum sealed.

Figures 12 and 13, p. 24 detail the assembly of the team-designed components. For the anticipated small-scale production of these instruments, the fabrication would be very similar to the way we have constructed the prototype. On a large scale, further analysis would need to be conducted to determine the most efficient methods of production.

## 5.5 Validation Results

Quantitative engineering specifications were determined by evaluating customer needs. The targeted engineering specifications for each of the technical requirements are listed in Table 2, p. 9. Relations between customer requirements and engineering specifications may be found in the full QFD diagram on page 8. This section describes a systematic means of how the engineering specifications were met.

### 5.5.1 Electronics

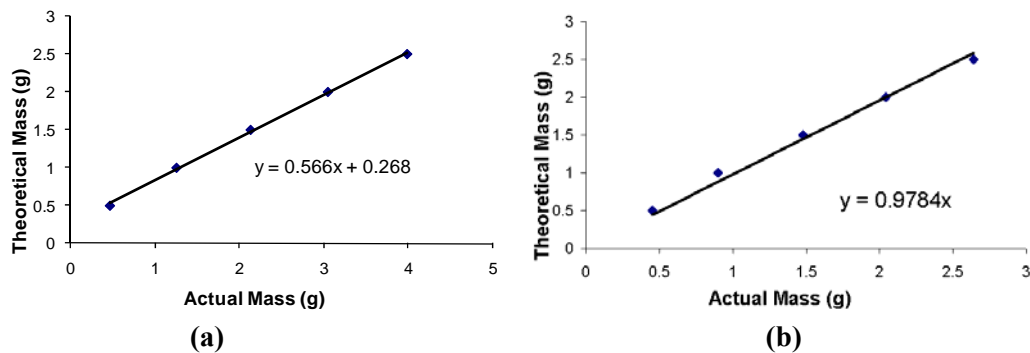
The necessary electrical power to run all five pumps has to be supplied by the DC power supply. A digital multi-meter that is readily available in our sponsor's lab will be used to determine that each pump receives the appropriate electrical input. We ran the pumps for several minutes and did not observe any

overheating of components or electrical shorts. However, *care should be taken not to connect or disconnect the cables between the pumps and control box when the DC power supply is on, because doing so can cause a voltage arc.*

### 5.5.2 Fluid Dispense System

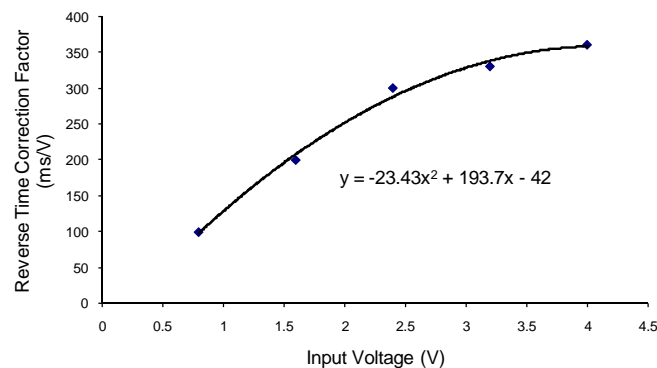
One of the most important engineering specifications is the fluid dispense rate and volume. Sufficient fluid needs to be dispensed on the substrate at the desired velocity. The validation was done using distilled water, and the dispensed fluid was massed to get an accurate volume measurement. It is a valid assumption that all solutions that will be used in this system have similar viscosity to water and will flow at the same rate. To calibrate volume, the dispensed volume was taken at rates of 15, 30, 45, 60, and 75 ml/min. A linear fit was used to adjust the LabVIEW voltage output. Figure 16a shows the calibration curve for volume, and Fig. 16b shows the expected vs. experimental volumes after calibration. Because of the variation in pumps, each pump has its own calibration curve. The volume flow rate is validated from the volume and time of flow; however this value is not as precise because of the acceleration and deceleration of the motor. Specifications for the motor rotation are included in Appendix D. Also, validation test has been done to ensure back splash does not occur (see Appendix C).

**Figure 16. Volume calibration for a pump (a), and results after calibration (b)**



To ensure no dripping of solutions, the motor reverse at the end of fluid delivery is also calibrated for each pump. Figure 17 shows the Labview input voltage empirically follows a quadratic relation to reverse time correction factor. Using this calibration, it is visually evident that the pump reverses correctly for all input voltages. This calibration must be done for each pump. Calibrations can be re-done if necessary due to wear on the system or changing system parameters.

**Figure 17. Reverse calibration curve for one pump in the system**



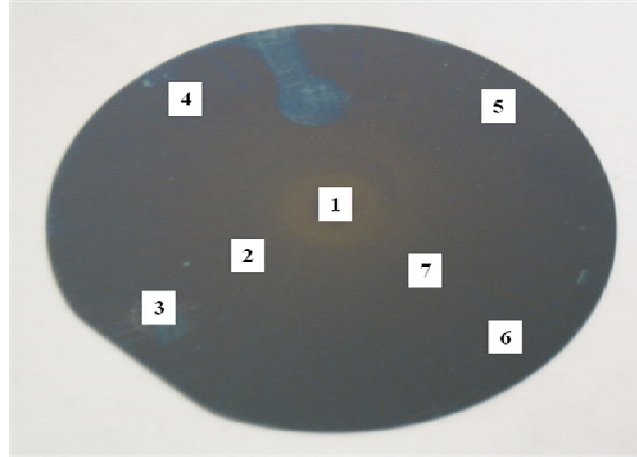
### 5.5.3 Film Thickness with Ellipsometer

Test films have been created with the spin-grower. To validate film thickness an ellipsometer was used to accurately measure the thickness across the silicon wafer. The measurement of a 10 bi-layer nanofilm is shown in Table 15, using points shown from Fig 18. The film shows more growth toward the edges, as previously experienced spinning at 2000 rpm [41].

**Table 15. Thickness measurements of a 10 bi-layer film**

Point	1	2	3	4	5	6	7
Thickness $\pm 0.50$ (nm)	83.38	86.99	90.10	90.61	90.40	94.35	87.80

**Figure 18. Location of measurements for a 10 bi-layers film**



### 5.5.4 Film Thickness with Keyence Laser

Validation on the laser displacement sensor concept was done with a demo unit provided by Keyence. Tests were run on the LK-G 15 which is the laser sensor with the highest resolution. The sensor has a linearity of 0.03% of Full Scale (FS = 1mm) and a resolution of 10 nm. Fluid was deposited manually with the spin coater lid open. Measurements were taken over 10 layers and the averages of 20000 readings were taken. The results of the tests are shown below in Table 16.

**Table 16. Thickness measurements of a 10 bi-layer film**

Number of Layers	0	4	8
Thickness (mm)	-1.28735 (baseline)	-1.27344	-1.27658
Change in Thickness from previous reading (nm)	0	13910	-3140

It is not necessary to see the growth over 10 layers but our major concern is the drift of 10 microns from one reading to the other. This drift may be due to thermal expansion of the spin coater, small movement of the spin coater across the table, or any number of other uncertainties on the micro-scale. Just touching the table could affect the measurements. The sensor did not give any good readings after 10 layers. Unless the thickness of the film builds up to more than 30 microns, the laser sensor will not show any conclusive results, thus we did not purchase it. It will be easier to measure the film thickness using the existing ellipsometer to determine the growth kinetics of the film. The ellipsometer also has the advantage of



being able to measure different points on the substrate. The laser displacement sensor would have been fixed at one radius.

### 5.5.5 Substrate Spin Speed

The spin speed and platform acceleration of the spin coater does not require further validation. The spin coater has a built in system that measures the spin speed and platform acceleration in real time. It allows the user to control the speed and acceleration of the spin coater from the front panel. The calibration and validation of this system was already done by the manufacturer of the spin coater.

### 5.5.6 Complete System Validation

The entire system has been tested by creating several sample clay-polymer films. The optimal parameters for running the system will be determined by the users of the instrument.

## 6 DISCUSSION

### 6.1 Customer Requirements

While our final design fulfills almost all of the customer requirements, there are some aspects that we have identified that can be improved upon. Table 17 shows the status of the customer specifications.

**Table 17. Summary of status of customer requirements**

Customer Requirements	Rank	Status
Automated injection	4	✓
Holds silicon wafer or glass slides	4	✓
Chemically compatible	4	✓
Fast cycle time	4	✓
Fully flexible injection of fluid	4	✓
Multiple solution capability	4	✓
No mixing of solutions	4	✓
Safety enclosure	4	✓
Spinning platform	4	✓
Controllable delivery flow rate	3	✓
Deposit many bilayers (3000)	3	✓
Prevents drips	3	✓
Rinsing capability	3	✓
Automated central control	2	✗
Easy to refill fluids	2	✓
Large fluid capacity	2	✓
Precise volume delivery	2	✓
Variable speed	2	✗
Measure thickness real time	1	✗

### 6.2 Peristaltic Pumps

The peristaltic pumps that we chose to use develop substantial inertial forces during operation and continue spinning after the control signal stops. In concept, we believed that by giving a reverse direction signal and a short impulse signal to rotate in the reverse direction we would be able to immediately stop the forward flow of fluid as well as suck back any drips that formed at the outlet of the tubing. In practice, the pumps will not rotate in the reverse direction until they come to a complete stop, despite the presence of a signal to do so. To overcome this issue with our prototype we performed calibrations for total flow volume based on the commanded flow rate and flow duration as well as the duration of reverse rotation signal needed to achieve the desired suck back.

### **6.3 Fluid Condensation**

Another issue with the design is the fact that substantial fluid vapor and condensation develop on the inside of the spin coater during high numbers of layer depositions, especially at high spin speeds. While we did anticipate some fluid to splash onto the spin coater cover during operation, we did not expect condensation and droplet development on the top of the cover and Loc-Line tubing to present a risk of causing drips onto the substrate. A potential solution for this issue is to activate the nitrogen purge capability of the spin coater creating positive pressure inside the cover and exuding vapors as they form to prevent condensation. With this solution, care would need to be taken to ensure that the solutions being used are not harmful, or the spin coater could be operated inside a fume hood. We did discuss the possibility of making the top air tight and providing a single tube through which gases and vapor could be removed from the spin coater, however, this would be very difficult considering the design of the spin coater cover and hinges. The spin coater has an existing inlet for nitrogen. Furthermore, a simple ventilation system may be incorporated by adding fans on the spin coater lid.

We also briefly discussed the possibility of using a domed shape lid allowing droplets to slide down the surface of the cover to the edges of the coater as oppose to falling directly downward onto the substrate. This would potentially solve the problem of drips from the cover but does not address condensation on the Loc-Line tubing.

### **6.4 Variation of Spin Speed**

We also wanted to incorporate into our design the ability to change spin speeds between different layer depositions. This would add another dimension of versatility to our design and potentially allow different layer thicknesses to be automatically programmed into the creation of a LBL material. We were not able to incorporate this into our prototype due to time constraints and late delivery of modified software from the spin coater supplier, but we designed in the elements needed to create this function. A port was left open on the junction box to install another D-connector to accept the trigger signal from the spin coater. A new LabView program could be written to wait for the trigger to deliver a dose of fluid and a spin coater recipe could be written to change spin speeds before sending the trigger signal. During the last week of the project, we received modified software from SCS that would allow for infinite looping of a series of tasks. By coupling this with a trigger from the spin coater to deposit fluid (which could be read by one of the many open input ports on the DAQ boards), the deposition of each layer could be fully programmable.

### **6.5 Centralized Control**

Because the SCS software was proprietary, there was no way to control both the spin coater and the pumps from the same program. Therefore, central automation was not possible. However, we are able to control the entire process using two programs that can be operated simultaneously.

We tried to keep this system as open as possible in terms of configurability. That is why there are five pumps and solution containers even though currently, tests are only being run using three solutions. Additionally, the code for the control program is open and can be reconfigured if desired. Finally, we left many inputs on the DAQ boards open for use with any other desired input (such as a trigger).

### **6.6 Thickness Measurement System**

A real-time thickness measurement system was not feasible for this design. Cost and time restrictions limited our options to one of several systems produced by Keyence. After several trials with the most suitable laser displacement sensor, we decided that it would not be practical to implement this into our system. For further discussion, see Section 5.5.4.

A possible replacement for a laser displacement system is an ellipsometer integrated with the spin coater. We considered such an option, but the ellipsometer cost of about \$70,000 made this not feasible for this project. Greg Pribil who is an engineer at J. A. Woollam Co., Inc. thinks that it is possible to custom

make an ellipsometer for our application [42]. The ellipsometer would have the advantage of being able to measure both high and low thickness by using laser beams of different wavelengths.

## 7 RECOMMENDATIONS

After researching, manufacturing, and testing our system, we have several recommendations for future work in this area. There are also several things that we would recommend doing differently for the construction of a second-generation prototype.

If another system were to be produced, we recommend that peristaltic pumps with active breaking be investigated. This would allow the pump to be stopped precisely at the end of a control signal, eliminating inertial free-wheeling and the need to calibrate for such effects.

To fix the issue of condensation on the spin coater lid and Loc-Line, we recommend trying to produce films while keeping a positive pressure on the inside of the spin coater using the existing nitrogen purge.

While this system is limited to a constant spin rate for each film, we recommend that the users of the instrument perform tests using the current system and control system to gain a basic understanding of the growth of the films. Later, if it is deemed necessary to vary the spin rate from layer to layer, they can consider implementing communications between the spin coater trigger and the control program.

We would also recommend designing a chuck adaptor that holds smaller substrates of different sizes. This was suggested by our sponsor, after seeing the glass-slide adaptor. This could be helpful in the future for producing films on small or irregularly-shaped substrates.

In order to determine the parameters of the system that produce the optimal films, we recommend a fractional factorial design of experiments approach for each combination of solutions. This will allow efficient determination of the optimal settings for the machine. Possible factors include spin rate, injection rate, injection time, injection angle, injection position, dry time, and the inclusion of a nitrogen purge.

## 8 INFORMATION SOURCES

Information regarding various aspects of this project was collected via scholarly articles, patents, web searches, and communication with the project sponsors.

### 8.1 Spin-assisted LBL Assembly

We toured Prof. Kotov's laboratory to learn about the current method of dip LBL assembly to determine the technical benchmarks. The lab currently uses a machine produced by nanoStrata Inc., which uses a rotating platform to sequentially dip the substrate into the solutions [5][22].

The idea of spin-assisted LBL assembly is not novel. Chiarelli et al. produced nanocomposite materials with as many as 20 bilayers using a manual solution dispensing method as early as 2001[23]. We would like to produce these films in a similar manner, while increasing the production speed and aggregate film thickness by automating the dispensing of solution.

Numerous other studies on spin-assisted LBL assembly of nanomaterials have been conducted by Cho et al. [1], Chiarelli et al. [24], and Heroit and Jones [25]. Additionally, many patents for a variety of spin-coaters exist [26][27][28], and many spin-coaters are commercially available [6][7][8][9][10]. Finally,

Krishnan has developed a mathematical model of spin coating thickness [29].

We also found information on other methods of LBL assembly of nanomaterials. Krogman et al. used a spray LBL technique that was found to be 25 times faster than the traditional dipping technique [30]. Kim et al. are developing a dynamic LBL process using a controlled flow of solutions through a fluidic device [31].

## **8.2 Thickness Measurement System**

Performing a literature search, we found descriptions of a homemade optical device for measuring nanocomposite film thickness on a spin-coating device [25][32]. However, we quickly determined that producing our own optical measurement device is not practical, based on the scope of the project.

The three main types of optical measurement systems that we looked into were interferometers, ellipsometers, and laser displacement systems.

There are many types of interferometers. Interferometer is a well established sensitive measuring instrument for anything that changes the phase of a wave, such as path length or refractive index. Interferometer uses the technique of wave interference to measure the thickness layer. A wave is split into two coherent parts and then combines later to create interference [33],[34]. Therefore, anything that changes the phase of one of the beams shifts the interference from a maximum to a minimum.

Ellipsometry is a popular and powerful optical technique for the investigation of the dielectric properties of thin films. It measures the change of polarization of light, which is reflected off a substrate by using phase information and the polarization state of light [35]. The property of the substrate determines the polarization change. The substrate measured must be composed of a small number of discrete, well-defined layers that are optically homogeneous, isotropic, and non-absorbing. Ellipsometry is an established method of measuring polyelectrolyte layer thickness [36]. No reference measurement is necessary.

The Keyence laser displacement sensor is a digital sensor and it incorporates charge-coupled device (CCD) arrays for sensing position. The measurement principle uses triangulation. The position of the reflected light on the CCD moves as the position of the target changes. The displacement amount of the target is measured by detecting this change [37].

## **9 CONCLUSIONS**

The purpose of this project is to develop a spin-grower to facilitate spin-assisted LBL growth of nanocomposite films. This project is broken down in to three main subsystems: spin coater, flow regulation, and fluid delivery, which are integrated using data acquisition hardware and central computer control. The final design for our prototype is given in Section 5.1, p.21. Engineering drawings for the custom components are shown in Appendix H. Engineering analysis including calibration of pumps have been conducted to ensure that the design meets the required specifications. The process plan for fabrication of the custom components has also been determined in Section 5.4, p.29.

The Keyence laser displacement sensor was tested for real time thickness measurement. We concluded that the inclusion of the laser sensor was not feasible due to the significant drift in measurement from one layer to another.

However, all the rest of the sub-systems and individual components have been fully integrated in to the

system. The pump control algorithm has been written in LabVIEW and is ready for use.

As shown in the mileposts set in the Project Plan and Gantt chart (Appendix A), this project was completed on schedule. The entire system was validated by developing a few thin films of 10 bi-layers. The spin grower is now ready for laboratory use in the creation of various nanocomposite films. Further work on this system may be executed based on the recommendations given in Section 7, p.35.

## **10 ACKNOWLEDGEMENTS**

First, we would like to thank our project sponsors, Prof. Nick Kotov, Prof. John Hart, and Paul Podsiadlo for their guidance and support throughout the project, giving us a work space, and for funding our efforts. We would also like to thank our two section instructors, Dr. Guru Dinda and Prof. Jyoti Mazumder, for providing outside help on this project. We would also like to thank the rest of the section instructors for their informative lectures and Prof. Steve Skerlos for organizing the course. Doctoral student Ming Qin was helpful in providing insight into the quality of our films and for helping us use the ellipsometer in the lab. We also owe thanks to Ruby Sowards, Secretary Senior in the Chemical Engineering Department for her assistance in our purchasing needs. Finally, we would like to thank Bob Coury and Marv Cressey, for their help in the undergraduate machine shop.

## 11 REFERENCES

- [1] Cho, Jinhan, Char, Kookheon, Hong, Jong-Dal, and Lee, Ki-Bong “Fabrication of Highly Ordered Multilayer Films Using a Spin Self-Assembly Method” *WILEY-VCH Verlag GmbH, D-69469 Weinheim, 2001 Adv. Mater. 2001, 13, No. 14, July 18*
- [2] Kotov, Nicholas A., Podsiadlo, Paul, and Hart, A. John, project sponsors. Personal Interview. January 10, 2008.
- [3] Kotov, Nicholas A., “Assembly of Free-Standing Films Using A Layer-By-Layer Process” U. S. Patent 7045087, May 16, 2006
- [4] ME 450 Project List: Winter 2008, p.8.
- [5] nanoStrata, Inc., “nanostrata,” retrieved January 24, 2008. Website: <http://www.nanostrata.com/>
- [6] Laurell, “Spin coater, spin processor, wet etch station, and more - Laurell Technologies Corporation,” retrieved January 24, 2008. Website: <http://www.laurell.com/>
- [7] Specialty Coating Systems, “SCS SPIN COATING SYSTEMS & ACCESSORIES,” retrieved January 24, 2008. Website: [http://www.scscoatings.com/parylene\\_equipment/spin-coaters.aspx](http://www.scscoatings.com/parylene_equipment/spin-coaters.aspx)
- [8] CHEMAT Technology, Inc., “Spin-coater” retrieved January 24, 2008. Website: <http://www.chemat.com/html/spin-coater.html>
- [9] Alfa Aesar, “Thin film spin coater.” Alfa Aesar Product Catalog, p.1655.
- [10] MTI Corporaton, “TC-100 DeskTop Spin Coater 5100RPM with Complete Accessories” retrieved February 21, 2008. Website: <http://www.mtixtl.com/index.asp?PageAction=VIEWPROD&ProdID=590>
- [11] SyringePump.com, “SyringePump.com - NE-500 OEM Syringe Pump” retrieved February 21, 2008. Website: <http://www.syringepump.com/oem.htm>
- [12] Proteus Industries, “Proteus Industries” retrieved February 21, 2008. Website: <http://www.proteusind.com/tofco/tofco.html>
- [13] Watson-Marlow, “Watson-Marlow Bredel Pumps - Watson-Marlow OEM Pumps” retrieved February 21, 2008. Website: <http://www.watson-marlow.com/watson-marlow/p-oem.htm>
- [14] Hart, A. John, “Project #17 The ‘Spin-Grower’” PowerPoint presentation, p.5. January 3, 2008.
- [15] Modularhose.com, “modularhose.com – Loc-Line Modular Hose” retrieved February 21, 2008. Website: <http://www.modularhose.com/>
- [16] SIOS.de, “Laserinterferometerferometric Vibrometer, SP-S Series” retrieved February 21, 2008. Website: [http://www.sios.de/ENGLISCH/PRODUKTE\\_E.HTM](http://www.sios.de/ENGLISCH/PRODUKTE_E.HTM)
- [17] JAWoollam.com, “M-2000 Interferometer” retrieved February 21, 2008. Website: <http://www.jawoollam.com/>

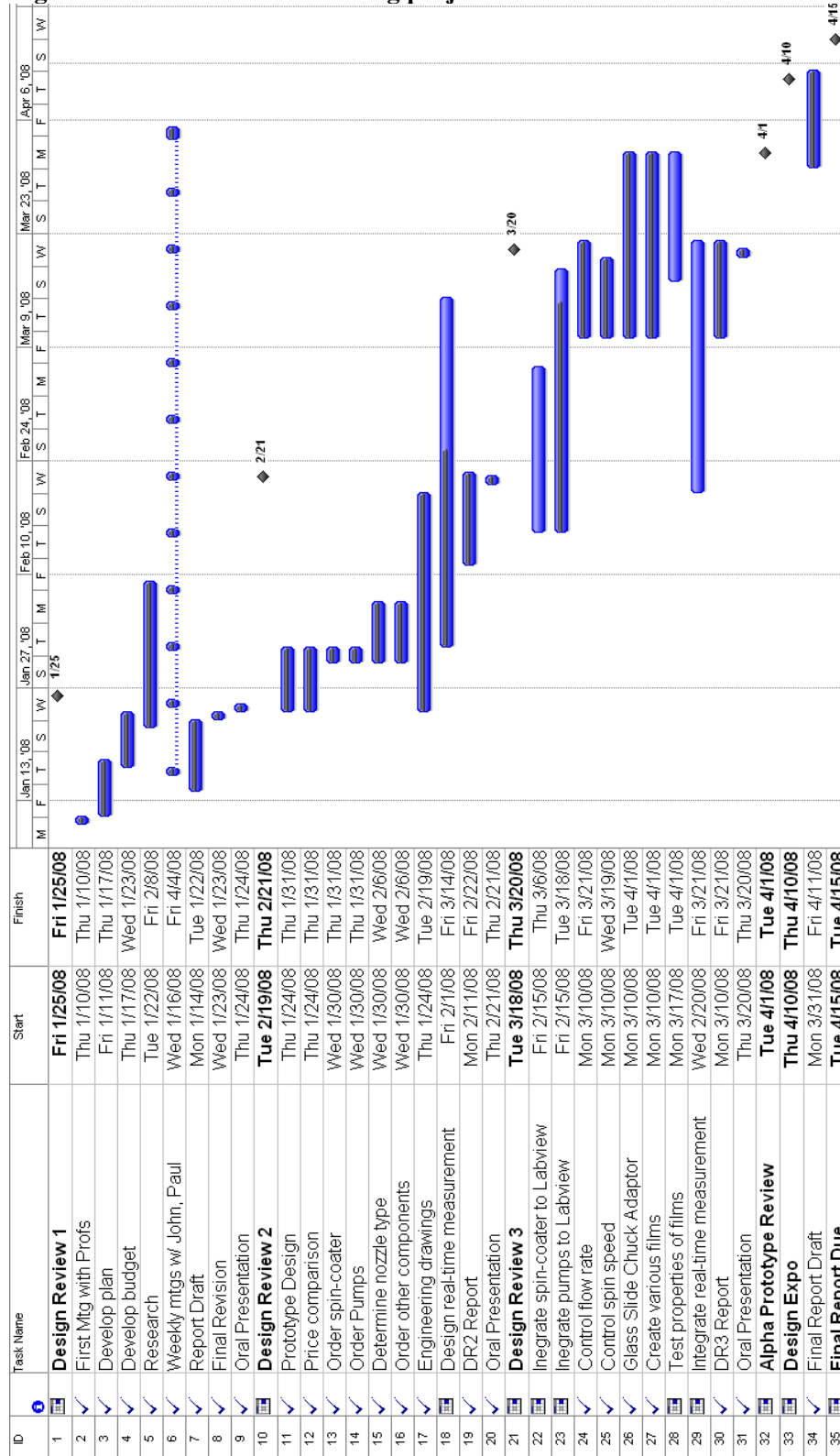
- [18] Keyence.com, "LK-G10 Series Laser Displacement Sensor" retrieved February 21, 2008.  
Website: <http://www.keyence.com/products/vision/laser/laser.php>
- [19] SCS, Inc. September 21, 2004, "Model GP3-8 Desk-Top Precision Spin Coater Operator's Manual"
- [20] Watson-Marlow, "300 Series OEM Systems" Product Catalog, p.4. Retrieved February 21, 2008.  
Website: <http://www.watson-marlow.com/pdfs-global/b-oem300-us-01.pdf>
- [21] National Instruments, "National Instruments - Test and Measurement" retrieved February 21, 2008.  
Website: <http://www.ni.com/>
- [22] Dubas, Stephan T., and Schlenoff, Joseph B., "Combined Vertical and Rotational Motion Indexing Mechanism" U. S. Patent 6460424, Oct. 8, 2002
- [23] Chiarelli, Peter A., Johal, Malkiat S., Casson, Joanna L., Roberts, Jerrad B., Robinson, Jeanne M., and Wang, Hsing-Lin, "Controlled Fabrication of Polyelectrolyte Multilayer Thin Films Using Spin-Assembly" *Adv. Mater.* 2001, 13, No. 15, August 3 Ó WILEY-VCH Verlag GmbH, D-69469 Weinheim, 2001
- [24] Chiarelli, Peter A., Johal, Malkiat S., Holmes, Daniel J., Casson, Joanna L., Robinson, Jeanne M., and Wang, Hsing-Lin, "Polyelectrolyte Spin-Assembly" *Langmuir*, Vol. 18, No. 1, 2002; *American Chemical Society Published on Web 12/06/2001*
- [25] Heriot, Sasha Y., and Jones, Richard A. L., "An interfacial instability in a transient wetting layer leads to lateral phase separation in thin spin-cast polymer-blend films" *Nature Materials* vol 4 October 2005; *Published online 4 September 2005; doi:10.1038/nmat1476*
- [26] Sago, Hiroyoshi, Mizuki, Hideyuki, Kudo, Katsuhiko, Nakayama, Muneo, "Spin-On Method and Apparatus for Applying Coating Material to a Substrate, Including an Air Flow Developing and Guiding Step/Means" U. S. Patent 5238713, Aug. 24, 1993
- [27] Batchelder, William T, "Apparatus and method for spin coating wafers and the like" U. S. Patent 5472502, Dec. 5, 1995
- [28] Hayes, Bruce L., Montanino, Greg, "Spin Coating Bowl" U. S. Patent 5861061, Jan. 19, 1999
- [29] Krishnan, Sriram, "PhD Thesis On the Manufacture of Very Thin Elastomeric Films by Spin-Coating" *Massachusetts Institute of Technology, June 2007*
- [30] Krogman, K. C., Zacharia, N. S., Schroeder, S., and Hammond, P. T., "Automated Process for Improved Uniformity and Versatility of Layer-by-Layer Deposition" *Langmuir* 2007, 23, 3137-3141, *American Chemical Society Published on Web 02/09/2007*
- [31] Kim, Hyong-Jun, Lee, Kangwon, Kumar, Sameer, and Kim, Jinsang, "Dynamic Sequential Layer-by-Layer Deposition Method for Fast and Region-Selective Multilayer Thin Film Fabrication" *Langmuir* 2005, 21, 8532-8538, *American Chemical Society Published on Web 07/28/2005*
- [32] Jones, Richard. Soft Machines. "Understanding structure formation in thin polymer films" published October 19 2005. Retrieved January 24 2007. Website: <http://www.softmachines.org/wordpress/?p=168>

- [33] Costantino, S., Martínez, O.E., and Torga ,J.R. “Wide Band Interferometry for Thickness Measurement” 21 April 2003, Vol. 11, No. 8, OPTICS EXPRESS pp. 952-957
- [34] Bechtold, M.F. , “Coating Thickness Measurement by Interferometry” October 1947, Vol. 37, No. 10, Journal of The Optical Society of America
- [35] Santos, O. , Kosoric, J., Hector, M.P. , Anderson ,P. , and Lindh, L. “Adsorption behavior of statherin and a statherin peptide onto hydroxyapatite and silica surfaces by *in situ* ellipsometry” 15 February 2008, Volume 318, Issue 2, Journal of Colloid and Interface Science pp.175-182
- [36] Fulghum, T.M., Estillore, N. C., Vo, C-D, Armes, S. P., Advincula, R.C. “Stimuli-responsive polymer ultrathin films with a binary architecture: combined layer-by-layer polyelectrolyte and surface-initiated polymerization approach” 22 Jan 2008, MACROMOLECULES, 41 (2): 429-435, ISSN: 0024-9297
- [37] Spinola,C, Vazquez,MJM, Bohorquez, AG, Bonelo, JM, Vizoso,J “Calibration of thickness measurement instruments based on twin laser sensors” 21-23 May 2001, IMTC/2001: 18<sup>th</sup> IEEE Instrumentation and Measurement Technology Conference, pp. 1079-1083
- [38] Daniel J. Inman (2001). *Engineering Vibration*, (2nd Edition) New Jersey: Prentice Hall. ISBN:0-13-017448-3 pp. 367-377
- [39] Ball, Ryan. Phone conversations with Keyence engineer. 3/19/08
- [40] ePanorama.net, “Copper wire”, Retrieved April 14, 2008. Website: [http://www.epanorama.net/documents/wiring/wire\\_resistance.html](http://www.epanorama.net/documents/wiring/wire_resistance.html)
- [41] Qin, Ming. Conversation with PhD Student working in the Kotov Lab. 4/8/08
- [42] Pribil, Greg. Email communication with J. A. Woollam Co., Inc. 2/27/08



# APPENDIX A – GANTT CHART

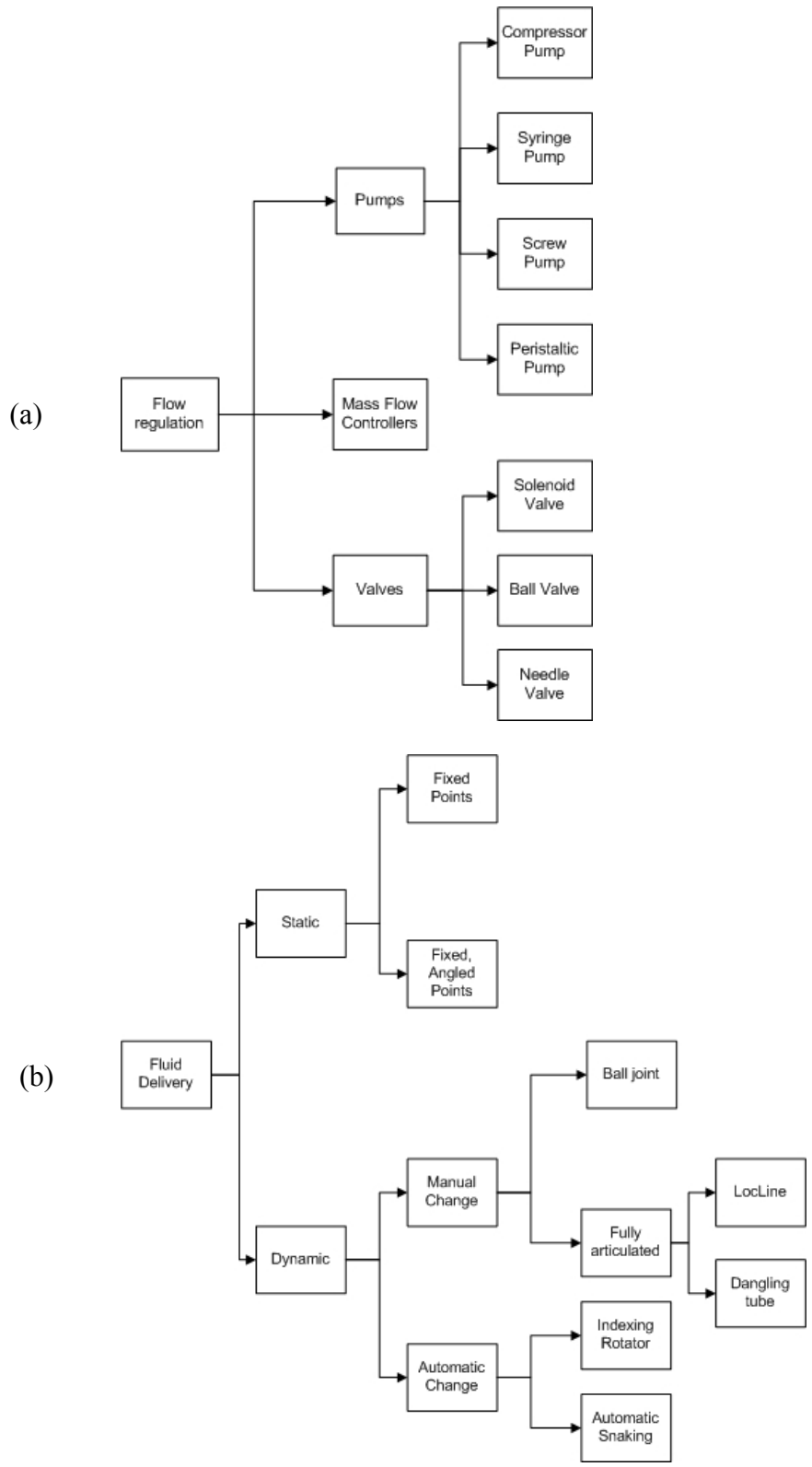
Figure A1. Gantt chart describing project schedule



## APPENDIX B - BRAINSTORMING AND CONCEPT GENERATION

- **Spin Coaters**
  - Build our own
  - Purchase “open source” controller
  - Hack into analog/digital control
  - Work with existing software
  - Communicate via software
  - Communicate via hardware
- **Energy Supply**
  - Outlet Power
  - Probably need DC converter
  - Battery Power
  - Nuclear Power
  - Charged Fluid
  - Get devices with standard power
  - Keep plugs to a minimum
  - One master switch
- **Computer Control**
  - LabView
  - MATLAB/Simulink
  - C++
  - VB
  - NI DAQ
  - 3<sup>rd</sup> Party DAQ
  - Build our own DAQ
  - Wireless
- **Fluid Storage**
  - Open Tanks
  - Closed off tanks
  - Giant syringe
  - Huge vat
  - Store below on cart
  - Store high for gravity feed
  - Snaking/coiled tubes
- **Flow Regulation**
  - Pumps
    - Scroll Pump
    - Screw Pump
    - Syringe Pump
    - Gas Compressor
    - Peristaltic Pump
    - Chain Pump
  - Mass Flow Controller
  - Pressure Vessel
  - Hand Feed
  - Gravity Feed
  - Hydrostatic Pressure
  - Controlled Valve
    - Ball Valve
    - Solenoid
- **Fluid Delivery**
  - Simple fixed points
  - Simple fixed angled points
  - Dangling tube
  - Spray on/mist
  - Uniform Focuser
  - Ball joint
  - Rotating/automatically indexing nozzles
  - Syringe tips
  - Pipette tips
  - Fully articulated coolant hose
  - Coolant hose with tube inside
  - Simple hole
  - Ball valve
  - Solenoid Valve
  - Needle Tip
- **Thickness Measurement System**
  - Interferometry
  - Ellipsometry
  - Laser Displacement Sensor
  - Spectrophotometry
  - CCD Camera
  - Electrical Resistance

**Figure B1. Concept generation trees for flow regulation (a) and fluid delivery (b)**



## APPENDIX C - SPRAY TESTING RESULTS

Figure C1. Results of spray testing

Water Vol (mL)	Time (sec)	Flow rate cm <sup>3</sup> /s	Nozzle Dia (in)	Nozzle Area cm <sup>2</sup>	Velocity cm/s	Speed rpm	Comments
1	1	1	0.02	0.001	808.36	3000	spray on cover 1 inch in
1	2	0.5	0.02	0.001	404.18	3000	spray on cover 1 inch in
10	16	0.625	0.13	0.08	7.89	3000	spray on cover 1.25 inch in
10	2	5	0.13	0.08	63.15	3000	All of top cover
10	8	1.25	0.13	0.08	15.79	3000	spray on cover 1.5 inch in
10	3	3.33	0.13	0.08	42.06	0	No splash on top cover
10	5	2	0.13	0.08	25.26	3000	spray on cover 1.5 inch in
10	1	10	0.13	0.08	126.31	0	All of top cover
							*10 mL deposited at 0 rpm with no splash reved to 3000rpm in 3 sec, no splash on top cover
Target Values							* spray angle approx 35 deg
1	2	0.5	0.0625	0.02	25.26	3000	* Conclusion: 25.3 cm/s is well below the lowest value of testing that caused splash (63 cm/s)

# APPENDIX D – COMPONENT SPECIFICATIONS

Figure D1. Engineering specifications for Keyence LK-G10 Laser Displacement Sensor

## LK-G Series

### Specifications

#### Sensor head

Model	LK-G10/G15	LK-G32/G37		LK-G152/G157	
Mounting mode		Diffused reflection	Specular reflection	Diffuse reflection	Specular reflection
Reference distance	0.39" 10 mm	1.18" 30 mm	0.93" 23.5 mm	5.91" 150 mm	5.81" 147.5 mm
Measuring range <sup>1</sup>	±0.04" ±1 mm	±0.2" ±5 mm	±0.18" ±4.5 mm	±1.57" ±40 mm	±1.54" ±39 mm
Light source	Wavelength	650 nm (visible light), Class II (FDA)			
	Output	0.3 mW max.			
Spot diameter (at reference distance)	Approx. 0.78 x 20 Mil 20 x 500 μm (G15), Approx. 0.78 Mil ø20 μm (G10)		Approx. 1.17 x 33.15 Mil 30 x 850 μm (G37), Approx. 0.17 Mil ø30 μm (G32)		Approx. 4.68 x 68.3 Mil 120 x 1700 μm (G157), Approx. 0.4.68 Mil ø120 μm (G152)
	±0.03% of F.S. (F.S.=±0.04" ±1 mm)		±0.05% of F.S. (F.S.= ±0.2" ±5 mm)		±0.05% of F.S. (F.S.= ±1.57" ±40 mm)
Linearity <sup>2</sup>					
Resolution <sup>3</sup>	0.0008 Mil (0.0004 Mil) 0.02 μm (0.01 μm)		0.002 Mil 0.05 μm <sup>4</sup>		0.02 Mil 0.5 μm <sup>4</sup>
Sampling frequency	20/50/100/200/500/1000 μs (Selectable from 6 levels)				
LED display	Near the center of the measurement. Green lights Within the measurement area: Orange lights Outside the measurement area: Orange flashing				
Temperature characteristics	0.01% of F.S./C (F.S.=±0.04" ±1 mm)		0.01% of F.S./C (F.S.= ±0.2" ±5 mm)		0.01% of F.S./C (F.S.= ±1.57" ±40 mm)
Environmental resistance	Protective construction	IP-67 (IEC60529)			
	Ambient luminance	Incandescent lamp or fluorescent lamp: 10,000 lux max.			
	Ambient temperature	0 to +50°C (32 to 122°F), No condensation			
	Relative humidity	35 to 85%, No condensation			
Resistance to vibrations	10 to 55 Hz, multiple amplitude 0.06" 1.5 mm; two hours in each direction of X, Y, and Z				
Material	Aluminum die-cast				
Weight (including the cable)	Approx. 190 g		Approx. 280 g		Approx. 290 g

- The value is obtained by measuring KEYENCE's standard target (ceramic).
- LK-G10/G15: When the sampling rate is 20μs, the value becomes +0.37(FAR side) to -1mm (NEAR side). LK-G32/G37: When the sampling rate is 20μs, the value becomes +1.8(FAR side) to -5mm (NEAR side) for diffuse reflection, and +1.6mm(NEAR side) to -4.5mm (NEAR side) for specular reflection. LK-G152/G157: When the sampling rate is 20μs, the value becomes -22(NEAR side) to -40mm(NEAR side) for diffuse reflection, and -22(NEAR side) to -39mm(NEAR side) for specular reflection.
- The value is obtained by measuring KEYENCE's standard target (ceramic) with the Standard mode.
- The value is obtained by measuring KEYENCE's standard(SUS) with 4096 times of averaging at the reference distance. The value in parenthesis is the typical resolution obtained by measuring the target with 16384.
- Consult your nearest KEYENCE representative for the Class IIIa type with increased resolution.

#### Controller

Model	All-in-one model	LK-G3001V	
	Separate model <sup>1</sup>	LK-G3001/LK-GD500	
Display	Head compatibility	All LK-G sensor heads are compatible	
	Number of connectable sensors	maximum of 2 units	
	Minimum display unit	0.0004 Mil 0.01 μm	
	Display range	±9999.99 mm to ±9999.99 mm ±9999.99" ±99.9999Mil (Selectable from six levels)	
Refresh rate	10 times/sec		
Terminal block	Analog voltage output	±10 V x 2 outputs, output impedance: 100 Ω	
	Analog current output	4 to 20 mA x 2 outputs, maximum load resistance: 350 Ω	
	Timing input <sup>3</sup>		
	Reset input <sup>3</sup>	For OUT1, non-voltage input	
	Auto-zero input <sup>3</sup>		
	Laser remote interlock input <sup>3</sup>	Non-voltage input	
	Comparator output <sup>2</sup>	For OUT1, NPN open-collector output	
Expansion connector	Alarm output <sup>2</sup>	For OUT1, NPN open-collector output (N.C.)	
	Timing input <sup>3</sup>		
	Reset input <sup>3</sup>	For OUT2, non-voltage input	
	Auto-zero input <sup>3</sup>		
	Program switching input <sup>3</sup>	Non-voltage input x 3 inputs	
	Laser-Off input <sup>3</sup>	For Head A/Head B, non-voltage input	
	Comparator output <sup>2</sup>	For OUT2, NPN open-collector output	
Binary	Alarm output <sup>2</sup>	For OUT2, NPN open-collector output (N.C.)	
	Binary output <sup>2</sup>	Measured data output (21 bits), OUT1/OUT2 selectable, NPN open-collector output	
	Strobe output <sup>2</sup>	NPN open-collector output	
	Binary selector output <sup>2</sup>	NPN open-collector output	
	Binary selector input <sup>3</sup>	Non-voltage input	
RS-232C interface	Measured data output and control input/output (Maximum baud rate: 115200 bit/s, selectable)		
USB interface	In conformity with USB Revision 2.0 Full speed (USB1.1 compatible)		
Major functions	2 OUT simultaneous measurement, Operation, Averaging, Filter, Calibration, Measurement, AUTO ZERO, Sampling frequency setting, Mutual interference prevention, Data storage, 8-program memory, ECO mode, ABLE setting, Target setting, ABLE tuning, Selection of measurement surface of transparent target, Statistics processing, Connection of setting support software, Selectable head-mounting, etc.		
Power supply voltage	24 VDC: 10%, Ripple: 10% (P to P) or less		
Current consumption	500 mA or less with 1 head/600 mA or less with 2 heads		
Ambient temperature	0 to +50°C (32 to 122°F), No condensation		
Relative humidity	35 to 85%, No condensation		
Weight	Approx. 480 g (LK-G3001V), Approx. 370 g (LK-G3001), Approx. 60 g (LK-GD500)		

- LK-G3001 can be operated singly. The measured value display and setting modifications can be performed on the display panel (LK-GD500) or via the setting support software (LK-H1W).
- The rating of the NPN open-collector: 50 mA max (40V max), residual voltage of 0.5 V max
- The rating of non-voltage input: 1 V or less ON voltage, 0.6 mA or less OFF current

#### Extension cable [Cable between the head and controller]

Model	LK-GC2	LK-GC5	LK-GC10	LK-GC30
Cable length	6' 2 m	16.4' 5 m	32.8' 10 m	98.4' 30 m
Weight	Approx. 200 g	Approx. 400 g	Approx. 750 g	Approx. 2000 g

#### Extension cable [Cable for display panel]

Model	OP-51654	OP-51655	OP-51656
Cable length	0.98' 0.3 m	9.8' 3 m	32.8' 10 m

Figure D2. Specs for Watson-Marlow OEM peristaltic pump

## 314 rapid load pumpheads, four rollers



With the same bayonet mounting system, 314 pumpheads may be interchanged with the 313 pumpheads or specified as an alternative head when ordering 300 series OEM pumps. Their four roller design gives higher precision and less pulsation and is suitable for continuous use up to 300 rpm giving flow rates up to 1200 ml/min (intermittent use up to 600 rpm giving flow rates up to 2400 ml/min).

The 314 range of pumpheads includes the 314D pumphead for mounting on either Watson-Marlow 300 series OEM drives or users' own drives having the same drive shaft arrangement, the 314B bare shaft pumphead for drives with a flexible coupling and the 314X extension pumphead for use with the 314D.

The 314D and 314B pumpheads accept up to five extension pumpheads for multi-channel installations depending on the power limit of the drive. A mounting plate which must be incorporated into the installation, is supplied with 314B and 314D pumpheads. Extension pumpheads snap fit directly behind 314D pumpheads. To use 2.4 mm wall thickness tubing, please add the suffix "2" - 314D2

The ordering information below shows the full range of 314 pumpheads as detailed on page 7.

### Ordering information

#### Four roller 1.6 mm wall thickness tubing

Clamp setting	314D	314E	314B	314XB	314CW	314BW
Variable	033.4411.000	033.4431.000	033.4421.000	033.4441.000	033.4451.000	033.4461.000
0.5 - 1.6	033.4411.00c	033.4431.00c	033.4421.00c	033.4441.00c	033.4451.00c	033.4461.00c
3.2	033.4411.00f	033.4431.00f	033.4421.00f	033.4441.00f	033.4451.00f	033.4461.00f
4.8	033.4411.00k	033.4431.00k	033.4421.00k	033.4441.00k	033.4451.00k	033.4461.00k
6.4 - 8.0	033.4411.00n	033.4431.00n	033.4421.00n	033.4441.00n	033.4451.00n	033.4461.00n

#### Four roller 2.4 mm wall thickness tubing

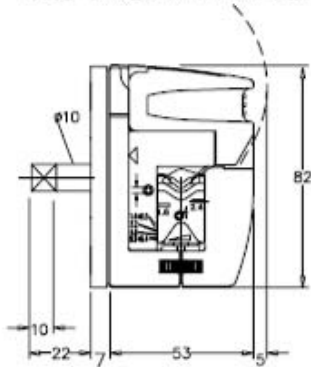
Clamp setting	314D2	314E2	314B2	314XB2	314CW2	314BW2
Variable	033.4511.000	033.4531.000	033.4521.000	033.4541.000	033.4551.000	033.4561.000
0.5 - 1.6	033.4511.00c	033.4531.00c	033.4521.00c	033.4541.00c	033.4551.00c	033.4561.00c
3.2	033.4511.00f	033.4531.00f	033.4521.00f	033.4541.00f	033.4551.00f	033.4561.00f
4.8	033.4511.00k	033.4531.00k	033.4521.00k	033.4541.00k	033.4551.00k	033.4561.00k
6.4	033.4511.00n	033.4531.00n	033.4521.00n	033.4541.00n	033.4551.00n	033.4561.00n

### Flow rates

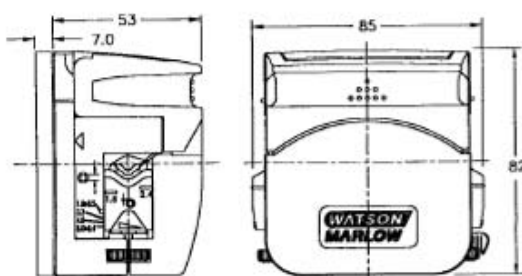
Bore mm	1.6mm (1/16") wall tubing						
	0.5	0.8	1.6	3.2	4.8	6.4	8.0
Bore "	1/32	1/32	1/16	1/8	3/16	1/4	5/16
Flow rate: ml/revolution	0.03	0.06	0.25	0.85	1.9	3.0	4.0
Maximum continuous flow: ml/min	9	18	75	255	570	900	1200
Maximum intermittent flow: ml/min	18	36	150	510	1140	1800	2400

For tube selections, see Tables A and B on page 45.

313B BARESHAFT PUMPHEAD



314D PUMPHEAD



314X EXTENSION PUMPHEAD

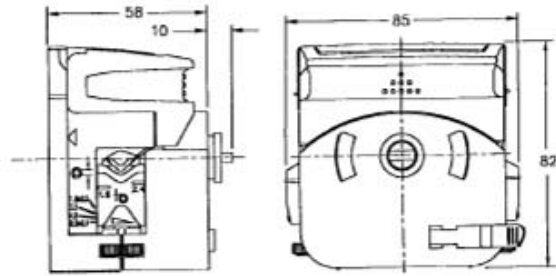
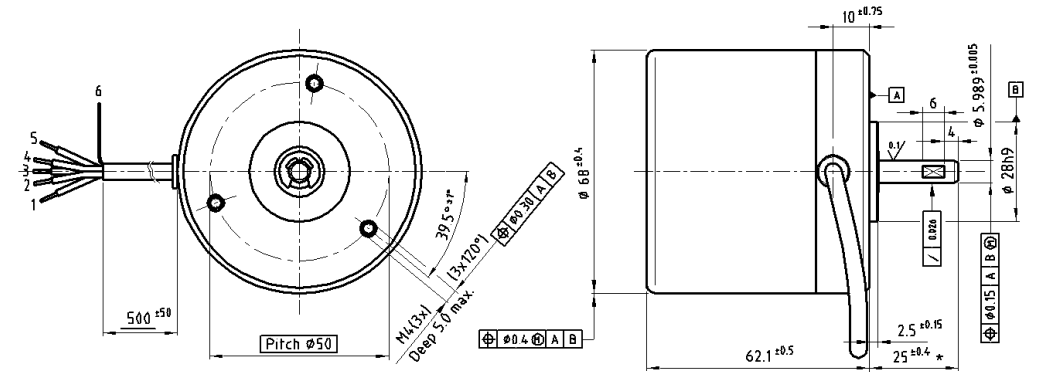


Figure D3. Specs for Premotec motor on Watson-Marlow OEM peristaltic pump

**BL58 EB**                      **Brushless DC motor**                      **35 Watt**

**Dimensional drawing**



**Motor data**

Motor order number	Shaft length 25 mm	4322 016 58001
	Shaft length 20 mm	4322 016 58002 *
Nominal Voltage	[V]	24
No load Speed (V in > 4V)	[rpm]	3650
No load Current (V in > 4V)	[mA]	280
Nominal Current limitation (V in > 4V)	[A]	2.0
Maximum torque	[mNm]	110
Maximum output power	[W]	35
Operating temperature range	[°C]	0 to 90
Thermal resistance from housing to ambient	[K/W]	3.7
Rotor inertia	[kgm <sup>2</sup> ]	120x10 <sup>-6</sup>
Mass of motor	[g]	550

Maximum radial load 20 mm from mounting front (no axial load towards flange)	[N]	40
Maximum axial load - towards flange (no radial load) - from flange	[N]	18
	[N]	10

Thermal motor protection :  
 Motor shuts down if the motor flange temperature reaches approx. 90°C  
 Motor restarts if the flange temperature is cooled down to approx. 80°C

For thermal reasons it is advised to mount the motor on a heat conducting frame if high output power is desired.

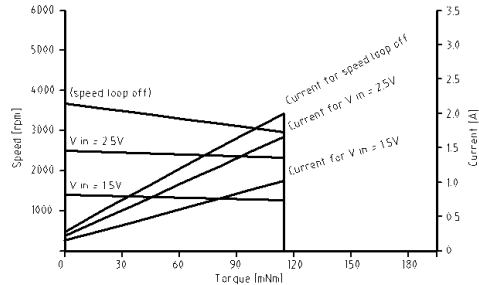
\* Shaft 20 mm for combination with gearboxes.

**Electrical Connection**

Lead no.	Lead colour	Function	Description
1	brown	FW/RV	Direction control input : 'High' CW, 'Low' CCW (shaftside) [do not leave this Lead floating]
2	white	V in	Input voltage (setpoint) for speed loop Resulting speed approx. 1000 rpm/V V in > 4 V : motor at full speed, speedloop off (open loop)
3	green	FG	Frequency generator output, 36 ppr ; R out = 4k Ohm (approx)
4	black	GND	Motor return, ground (0 V)
5	red	Vp	Motor supply voltage +24 V (min 14 V - max 30 V)
6	bare	shield	Shield for cable and connected to motor housing

	min.	typ.	max.
<b>Lead 1</b>			
input 'high'	[V]	4.1	5
input 'low'	[V]	0	1.9
abs. max./min. input	[V]		±30
<b>Lead 2</b>			
abs. max./min. input	[V]		±30
<b>Lead 3</b>			
output 'high', not loaded	[V]	4.0	4.5 5.0
output 'low', not loaded	[V]	0	0.1 0.2

**Performance curve**



**Product combinations**

- \* Gearbox S64A                      \* Gearbox P50A
- \* Gearbox S69A                      \* Gearbox P59A

**Options**

- \* Square mounting flange                      \* Speed loop with frequency input
- \* Shaft diameter, 7 or 8 mm                      \* Protection class upto IP67DS

**Features**

- \* Adjustable speed loop
- \* Direction control input (forward / reverse)
- \* Frequency Generator output (speed sensing)
- \* Thermal motor protection
- \* Long life (20.000 hours)
- \* Low EMI
- \* Protection class IP54

**PREMOTEC**

PRECISION MOTOR TECHNOLOGY BV                      Internet : www.premotec.com - E-mail : sales@premotec.com  
 Precision Motor Technology b.v. - Kerkeplaat 16 - 3313 LC - Dordrecht - The Netherlands - Tel. : +31 78 621 99 40 - Fax. : +31 78 621 48 28

**Figure D3. Specs for SCS spin coater**

The SCS G3 Spincoat series sets the standard in operating precision and programming flexibility, with a high level of rotation accuracy and repeatability, along with precise acceleration and deceleration control. The G3, coupled with the SCS Multi-Dispense, enables research and development laboratories to easily and efficiently develop and refine coating applications for a variety of uses.

**SCS G3 Spincoat Series**

The SCS Spincoat series accurately applies liquid coating materials – such as photoresists, polyimides, metallo-organics, dopants and silica films on planar substrates. The non-programmable SCS G3-8 performs single-step coating profiles. The programmable G3P models store and execute up to 30 programs with 20 steps each, which are easily entered on the front-panel LCD display and keypad. Optional PC interface software allows external programming, profile storage, diagnostics, vacuum on/off, slow speed centering, and programmable home position.



**Program Specifications and Profile**

Figure 1 shows a representative example of a coating cycle that is easily and quickly programmed, saved and executed. Each dwell portion of the cycle can be defined in the range of 0 to 9,999 RPM (4,000 RPM max for the 15-inch model), with ramp times from 0.1 to 25.5 seconds. A single step may have a dwell time of up to 999 seconds, and coating cycles are interruptible by the operator at any time. Ramp-up time is dependent on the chuck size and substrate weight.

Control Panel Features	Non-Programmable	Programmable
Recipe Number		✓
Speed	✓	✓
Remaining Process Time		✓
Message Line	✓	✓
Arrow Control	Ramp Up/Down, Dwell	Right, Left, Up, Down
Start and Stop Buttons	✓	✓
Mode, Clear and Enter Buttons		✓

Characteristic	Range	Tolerance
Rotational Speed	0 to 9,999 RPM <sup>1</sup> in 1 RPM increments	± 1 to 3 FPM full scale
Acceleration/Deceleration Time <sup>2</sup>	0.1 to 25.5 seconds in 0.1 second increments	± 0.05 seconds
Dwell Time	0 to 999 seconds in 1 second increments	± 0.05 seconds
Dispense Time (optional feature)	10 to 25.5 seconds in 0.1 second increments	± 0.05 seconds
Acceleration/Deceleration Linearity		± 0.1 percent

1. 4,000 RPM maximum for the 15-inch bowl unit.  
2. Size and weight of substrate will affect acceleration values.

**Figure 1**

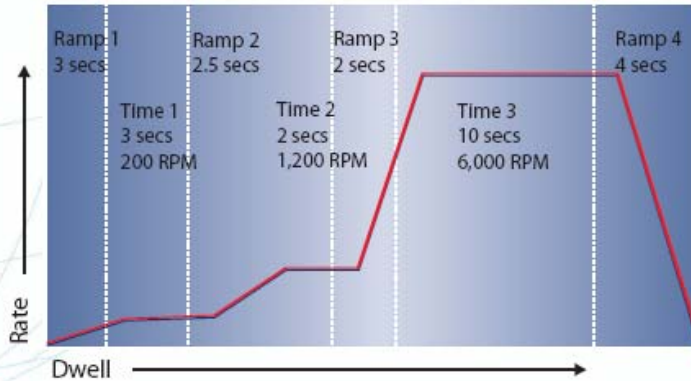




Figure D4. Specs for NI-6008 DAQ

Low-Cost Multifunction DAQ for USB

Specifications

Typical at 25 °C unless otherwise noted.

Analog Input

Absolute accuracy, single-ended

Range	Typical at 25 °C (mV)	Maximum (0 to 55 °C) (mV)
±10	14.7	128

Absolute accuracy at full scale, differential<sup>1</sup>

Range	Typical at 25 °C (mV)	Maximum (0 to 55 °C) (mV)
±20	14.7	128
±10	7.73	84.8
±5	4.28	58.4
±4	3.59	53.1
±2.5	2.56	45.1
±2	2.21	42.5
±1.25	1.70	39.9
±1	1.53	37.5

Number of channels..... 8 single-ended/4 differential  
 Type of ADC..... Successive approximation

ADC resolution (bits)

Module	Differential	Single-Ended
USB-6008	12	11
USB-6009	14	13

Maximum sampling rate (system dependent)

Module	Maximum Sampling Rate (kS/s)
USB-6008	10
USB-6009	48

Input range, single-ended..... ±10 V  
 Input range, differential..... ±20, ±10, ±5, ±4, ±2.5, ±2, ±1.25, ±1 V  
 Maximum working voltage..... ±10 V  
 Overvoltage protection..... ±35 V  
 FIFO buffer size..... 512 B  
 Timing resolution..... 41.67 ns (24 MHz timebase)  
 Timing accuracy..... 100 ppm of actual sample rate  
 Input impedance..... 144 k  
 Trigger source..... Software or external digital trigger  
 System noise..... 0.3 LSB<sub>rms</sub> (±10 V range)

Analog Output

Absolute accuracy (no load)..... 7 mV typical, 36.4 mV maximum at full scale  
 Number of channels..... 2  
 Type of DAC..... Successive approximation  
 DAC resolution..... 12 bits  
 Maximum update rate..... 150 Hz, software-timed

Output range..... 0 to +5 V  
 Output impedance..... 50 Ω  
 Output current drive..... 5 mA  
 Power-on state..... 0 V  
 Slew rate..... 1 V/μs  
 Short-circuit current..... 50 mA

Digital I/O

Number of channels..... 12 total  
 8 (P0..0..7->)  
 4 (P1..0..3->)  
 Direction control..... Each channel individually programmable as input or output  
 Output driver type  
 USB-6008..... Open-drain  
 USB-6009..... Each channel individually programmable as push-pull or open-drain  
 Compatibility..... CMOS, TTL, LVTTTL  
 Internal pull-up resistor..... 4.7 kΩ to +5 V  
 Power-on state..... Input (high impedance)  
 Absolute maximum voltage range..... -0.5 to +5.8 V

Digital logic levels

Level	Min	Max	Units
Input low voltage	-0.3	0.8	V
Input high voltage	2.0	5.8	V
Input leakage current	-	50	μA
Output low voltage (I = 8.5 mA)	-	0.8	V
Output high voltage (push-pull, I = -8.5 mA)	2.0	3.5	V
Output high voltage (open-drain, I = -8.5 mA, nominal)	2.0	5.0	V
Output high voltage (open-drain, I = -8.5 mA, with external pull-up resistor)	2.0	-	V

Counter

Number of counters..... 1  
 Resolution..... 32 bits  
 Counter measurements..... Edge counting (falling edge)  
 Pull-up resistor..... 4.7 kΩ to 5 V  
 Maximum input frequency..... 5 MHz  
 Minimum high pulse width..... 100 ns  
 Minimum low pulse width..... 100 ns  
 Input high voltage..... 2.0 V  
 Input low voltage..... 0.8 V

Power available at I/O connector

+5 V output (200 mA maximum)..... +5 V typical  
 +4.85 V minimum  
 +2.5 V output (1 mA maximum)..... +2.5 V typical  
 +2.5 V output accuracy..... 0.25% max  
 Voltage reference temperature drift..... 50 ppm/°C max

<sup>1</sup>Input voltages may not exceed the working voltage range.

BUY ONLINE at ni.com or CALL (800) 813 3693 (U.S.)

## APPENDIX E – PROJECT BILL OF MATERIALS

### Bill of Materials

Component	Manufacturer	Description	Qty.	Unit	Unit Price	Total Price
<b>Spin system</b>						
Spin coater	SCS	G3P-8 Spin Coater, 115V/60Hz	1	ea	\$4,797.00	\$4,797.00
Chuck	SCS	Vacuum Chuck, Type CS (SST), 3" Diameter	1	ea	\$650.00	\$650.00
Chuck Adaptor		1/4" Delrin adaptor for vacuum chuck to hold 1"x3" glass slides	1	in	\$25.00	\$6.25
Vacuum pump	SCS	K1 Vacuum Pump, 115V/60Hz	1	ea	\$618.00	\$618.00
<b>Fluid Delivery System</b>						
Peristaltic pump	Watson-Marlow	OEM 314VDL/D variable speed pumps with brushless DC motor	5	ea	\$670.00	\$3,350.00
Tubing	Watson-Marlow	Bioprene	10	m	\$8.01	\$80.10
Fluid Container	Corning	2 Liter bottle	5	ea	\$10.00	\$50.00
Line/Nozzle	Loc-Line	Flexible Hosing	5	ea		\$31.35
<b>Control System</b>						
Computer	HP		1	ea	FREE!	\$0.00
Spin coater software			1	ea	\$399.00	\$399.00
Computer software		Windows XP, Labview	1	ea	FREE!	\$0.00
18 gauge Wire cable		For serial cables	30	ft	\$20.00	\$20.00
18 gauge Wire	RadioShack		45	ft		\$5.99
Wire connectors	RadioShack	9 pin D-sub serial connectors	20	ea	\$1.99	\$39.80
9 pin hoods	RadioShack	Hoods to cover the D-sub connectors	10	ea	\$2.99	\$29.90
Heat shrink	RadioShack	To cover any open wires	1	ea		\$8.00
Switch	Gardner Bender	10 amp	2	ea	\$3.98	\$7.96
Barrier Strip	RadioShack	6 position	2	ea	\$2.39	\$4.78
Barrier Strip	RadioShack	8 Position	1	ea	\$2.39	\$2.39
Jumper Strip	RadioShack	8 Position	3	ea	\$1.99	\$5.97
24VDC Power Source	Acopian	Gold Box 24VDC/10A	1	ea	\$135.00	\$135.00
Extension Cord			1	ea	\$6.99	\$6.99
USB Cable	IOGEAR	4-port USB 2.0 Hub	1	ea	\$14.99	\$6.99
Outlet	Leviton	for vacuum pump	1	ea	\$2.99	\$2.99
Power Strip			1	ea	\$14.99	\$14.99
DAQ board	National Instruments	NI USB-6008	3	ea	\$143.10	\$429.30
<b>Support System</b>						
Aluminum sheet		14 Gauge	12	ft <sup>2</sup>	\$3.12/ft <sup>2</sup>	\$37.50
Bolts	Crown Bolt	#8-32 x 3/8" (includes washers and nuts)	32	ea		\$4.16
Lock washers	Crown Bolt	#8	40	ea		\$2.08
Washers	Crown Bolt	1/2" zinc washers for Loc-line	5	ea		\$1.99
Aluminum angle		1" Angle	10	ft2	\$0.60/ft	\$6.36
Plexiglas		18" x 24"	3	ft <sup>2</sup>	\$4.49/ft <sup>2</sup>	\$13.49
Grommets	Dantona		1	ea		\$2.99
Rubber feet	Shepherd	1" anti-skid pads	8	ea		\$4.58
Rivets	Crown Bolt	1/8" x 1/4"	40	ea		\$4.97
Backing washers	Crown Bolt	1/8"	40	ea		\$1.79
<b>Other</b>						
Vinyl Tubing		3/4" tubing for waste	1	ea	\$14.99	\$14.99
Loc Line Pliers	Loc-Line	Pliers to snap together Loc-Line	1	ea	\$15.00	\$15.00

Total **\$10,812.65**

# APPENDIX F – FLOW REGULATION FULL PUGH CHART

		Products (Flow Regulation: Peristaltic Pumps)															
		Watson-Marlow 300S OEM with Brushless DC motor		Barnant Multichannel Pump		Watson-Marlow OEM		Watson-Marlow multichannel		Masterflex L/S single channel		instech P256 OEM pump		Longer BT100-L multichannel		Masterflex Rapidload B/T Pumps	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
Selection Criteria	weight	5	0.75	2	0.3	5	0.75	2	0.3	4	0.6	5	0.75	2	0.3	3	0.45
Ease of integration to central control	1.5	4	0.6	2	0.3	4	0.6	2	0.3	4	0.6	4	0.6	2	0.3	4	0.6
Chemically compatible	5	5	0.25	5	0.25	5	0.25	5	0.25	5	0.25	5	0.25	5	0.25	5	0.25
Controllable delivery flow rate	5	3	0.15	5	0.25	2	0.1	5	0.25	5	0.25	2	0.1	5	0.25	3	0.15
Easy to refill fluids	5	5	0.25	5	0.25	4	0.2	4	0.2	5	0.25	4	0.2	5	0.25	5	0.25
Multiple solution capability	5	2	0.1	2	0.1	2	0.1	2	0.1	2	0.1	2	0.1	2	0.1	2	0.1
Precise volume delivery	5	4	0.2	5	0.25	3	0.15	5	0.25	5	0.25	3	0.15	5	0.25	4	0.2
Prevents drips	5	5	0.25	2	0.1	4	0.2	2	0.1	2	0.1	4	0.2	2	0.1	5	0.25
Safety	10	4	0.4	5	0.5	4	0.4	4	0.4	5	0.5	4	0.4	5	0.5	4	0.4
Availability	10	5	0.5	4	0.4	4	0.4	4	0.4	5	0.5	2	0.2	3	0.3	5	0.5
Price	20	4	0.8	2	0.4	5	1	2	0.4	2	0.4	5	1	2	0.4	4	0.8
Total Score	100	4.25		3.1		4.15		3.2		3.4		4.05		3		3.95	
Rank		1		7		2		6		5		3		8		4	
Purchase?		No		No		No		No		No		No		No		No	
		Products (Flow Regulation: Others)															
		Toftco Flow Meters USF2005		Laurell DCV-1B		NE-500 OEM											
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score										
Selection Criteria	weight	4	0.6	5	0.75	3	0.45										
Ease of integration to central control	1.5	4	0.6	5	0.75	4	0.6										
Chemically compatible	5	2	0.1	5	0.25	5	0.25										
Controllable delivery flow rate	5	4	0.2	3	0.15	3	0.15										
Easy to refill fluids	5	5	0.25	1	0.05	4	0.2										
Multiple solution capability	5	2	0.1	3	0.15	5	0.25										
Precise volume delivery	5	4	0.2	4	0.2	4	0.2										
Prevents drips	5	3	0.15	5	0.25	2	0.1										
Safety	10	4	0.4	4	0.4	3	0.3										
Availability	10	2	0.2	3	0.3	2	0.2										
Price	20	1	0.2	1	0.2	4	0.8										
Total Score	100	3		3.45		3.5											
Rank		-		-		-											
Purchase?		No		No		No											

# APPENDIX G – FLUID DELIVERY SYSTEM FULL PUGH CHART

		Concepts											
Selection Criteria	weight	Fixed Vertical Injection		Uniform Focus		Ball Joint		Fully Articulating Tube		Revolving Nozzles		Fixed Angled Injection	
		Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score	Rating	Weighted Score
Repeatability of delivery position	15	5	0.75	4	0.6	4	0.6	3	0.45	3	0.45	5	0.75
No mixing of solutions	15	3	0.45	4	0.6	4	0.6	4	0.6	5	0.75	4	0.6
Ability to vary outlet velocity	7	3	0.21	3	0.21	2	0.14	2	0.14	3	0.21	3	0.21
Ease of integration into lid	6	4	0.24	2	0.12	1	0.06	5	0.3	1	0.06	4	0.24
Complexity	6	5	0.3	1	0.06	3	0.18	5	0.3	1	0.06	5	0.3
Low Cost	9	5	0.45	2	0.18	1	0.09	4	0.36	1	0.09	5	0.45
Chemical Compatibility	15	5	0.75	4	0.6	4	0.6	4	0.6	4	0.6	5	0.75
Freedom of deposition location	11	1	0.11	3	0.33	5	0.55	5	0.55	2	0.22	1	0.11
Freedom of deposition angle	11	1	0.11	1	0.11	3	0.33	5	0.55	1	0.11	1	0.11
Ease of integration with flow regulation system	5	4	0.2	2	0.1	2	0.1	4	0.2	1	0.05	4	0.2
Total Score	100		3.57		2.91		3.25		4.05		2.6		3.72
Rank			3		5		4		1		6		2
Continue?			No		No		No		Develop		No		No

# APPENDIX H – DIMENSIONED DRAWINGS

Figure H1. Dimensioned drawing of modified spin coater cover top

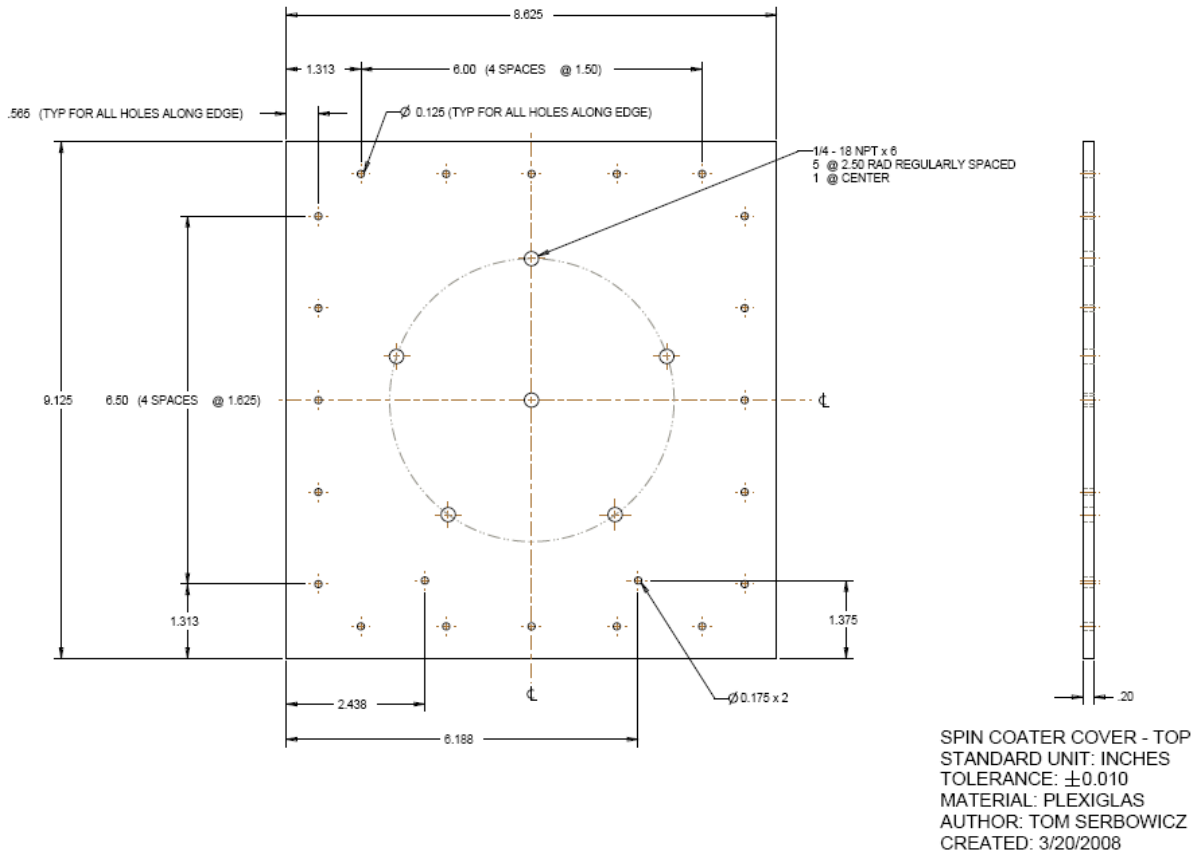
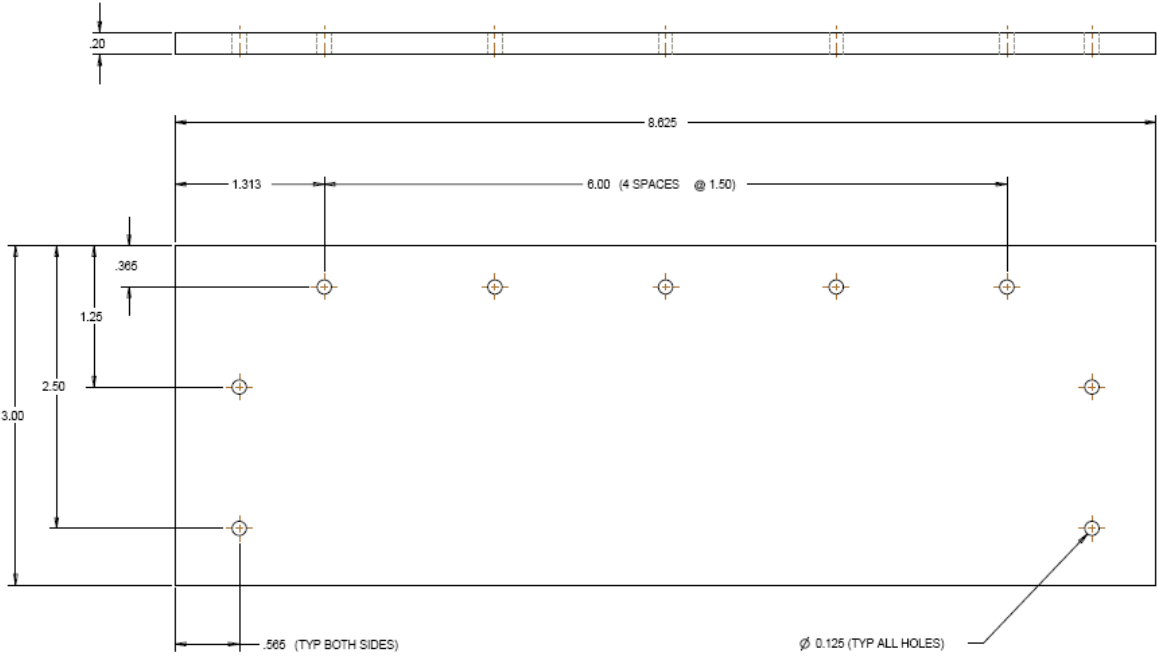
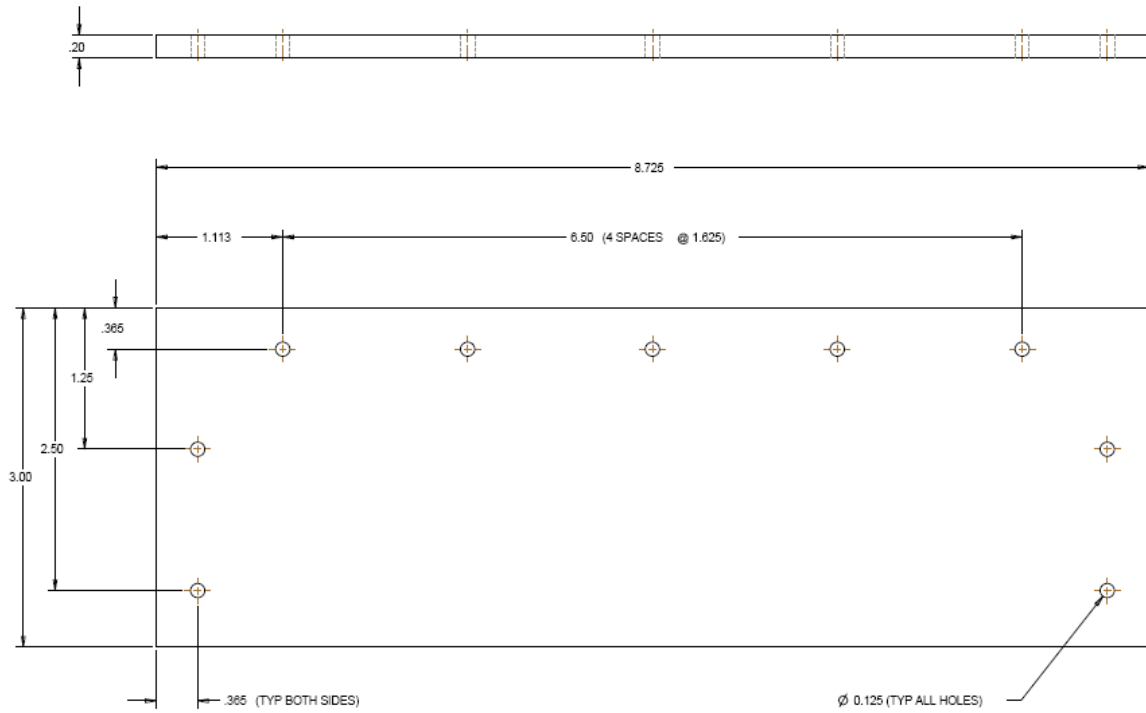


Figure H2. Dimensioned drawing of modified spin coater cover side



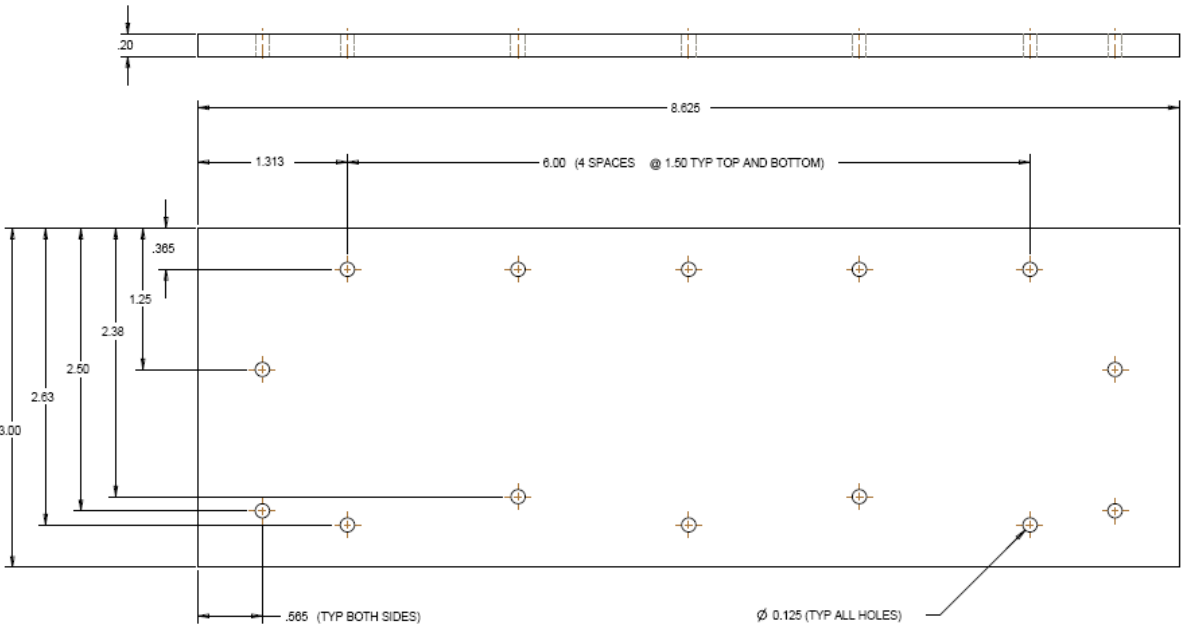
SPIN COATER COVER - SHORT SIDE FRONT  
STANDARD UNIT: INCHES  
TOLERANCE: ± 0.010  
AUTHOR: TOM SERBOWICZ  
CREATED: 3/20/2008

**Figure H3. Dimensioned drawing of modified spin coater cover front**



SPIN COATER COVER - LONG SIDE  
STANDARD UNIT: INCHES  
TOLERANCE:  $\pm 0.010$   
AUTHOR: TOM SERBOWICZ  
CREATED: 3/20/2008

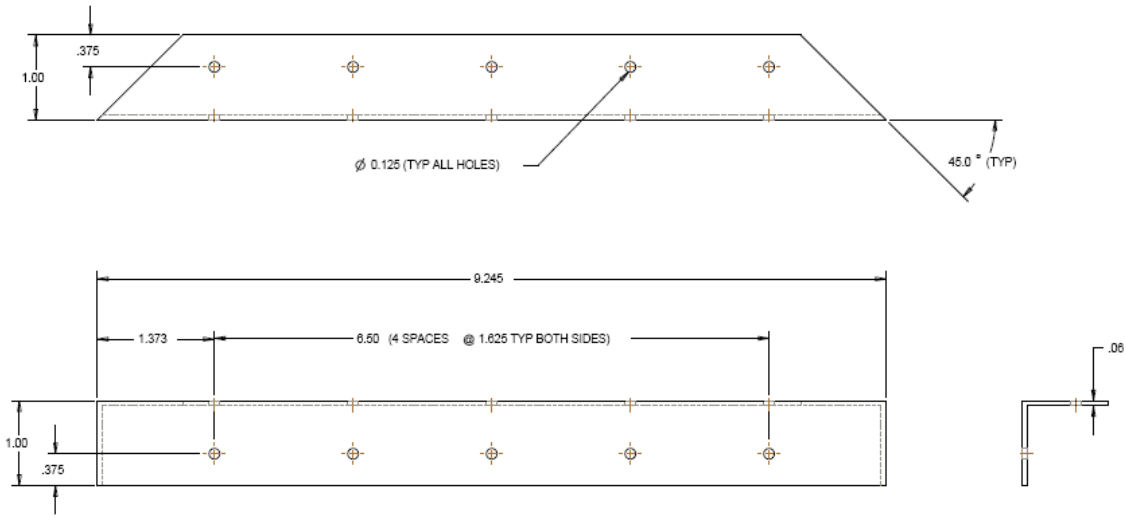
**Figure H4. Dimensioned drawing of modified spin coater cover back**



SPIN COATER COVER - SHORT SIDE BACK  
 STANDARD UNIT: INCHES  
 TOLERANCE:  $\pm 0.010$   
 AUTHOR: TOM SERBOWICZ  
 CREATED: 3/20/2008

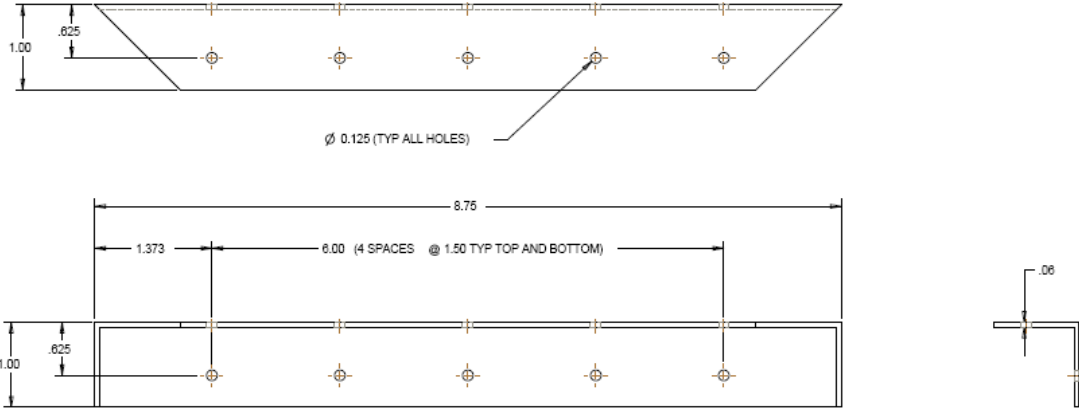


**Figure H5. Dimensioned drawing of modified spin coater cover long corner brackets**



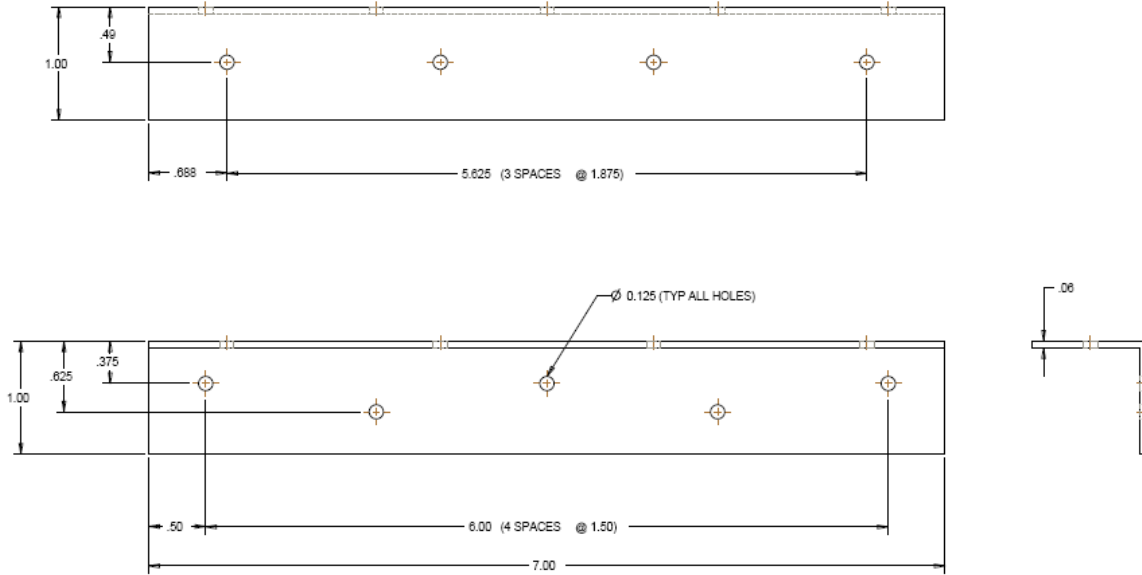
SPIN COATER COVER - LONG CORNER  
STANDARD UNIT: INCHES  
TOLERANCE:  $\pm 0.010$   
MATERIAL: ALUMINUM  
AUTHOR: TOM SERBOWICZ  
CREATED: 3/20/2008

**Figure H6. Dimensioned drawing of modified spin coater cover short corner brackets**



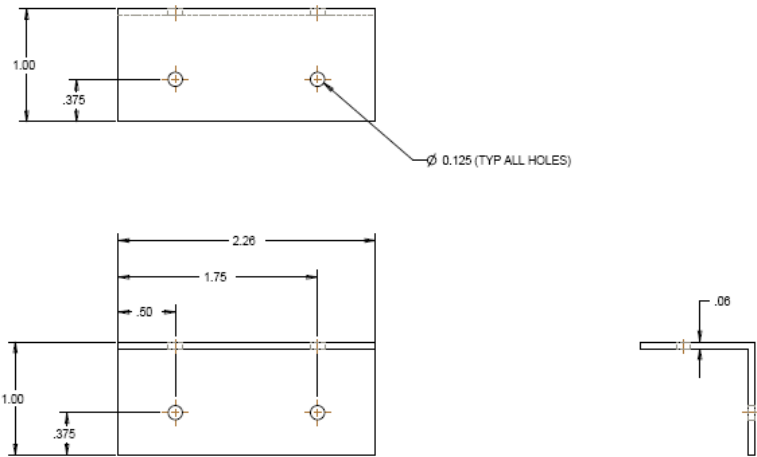
SPIN COATER COVER - SHORT CORNER  
STANDARD UNIT: INCHES  
TOLERANCE: ± 0.010  
MATERIAL: ALUMINUM  
AUTHOR: TOM SERBOWICZ  
CREATED: 3/20/2008

**Figure H7. Dimensioned drawing of modified spin coater cover hinge bracket**



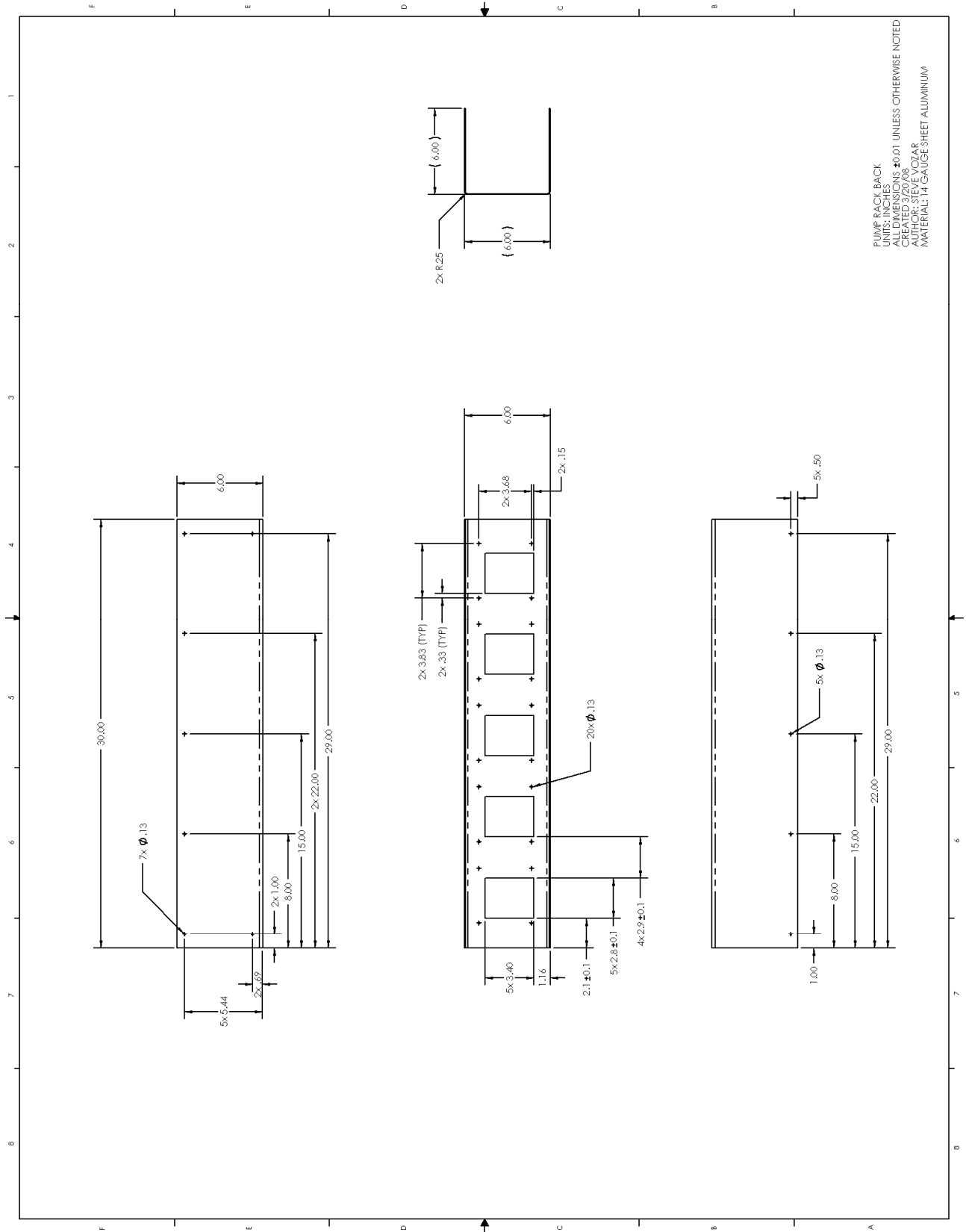
SPIN COATER COVER - HINGE BRACKET  
STANDARD UNIT: INCHES  
TOLERANCE:  $\pm 0.010$   
MATERIAL: ALUMINUM  
AUTHOR: TOM SERBOWICZ  
CREATED: 3/20/2008

Figure H8. Dimensioned drawing of modified spin coater cover side bracket

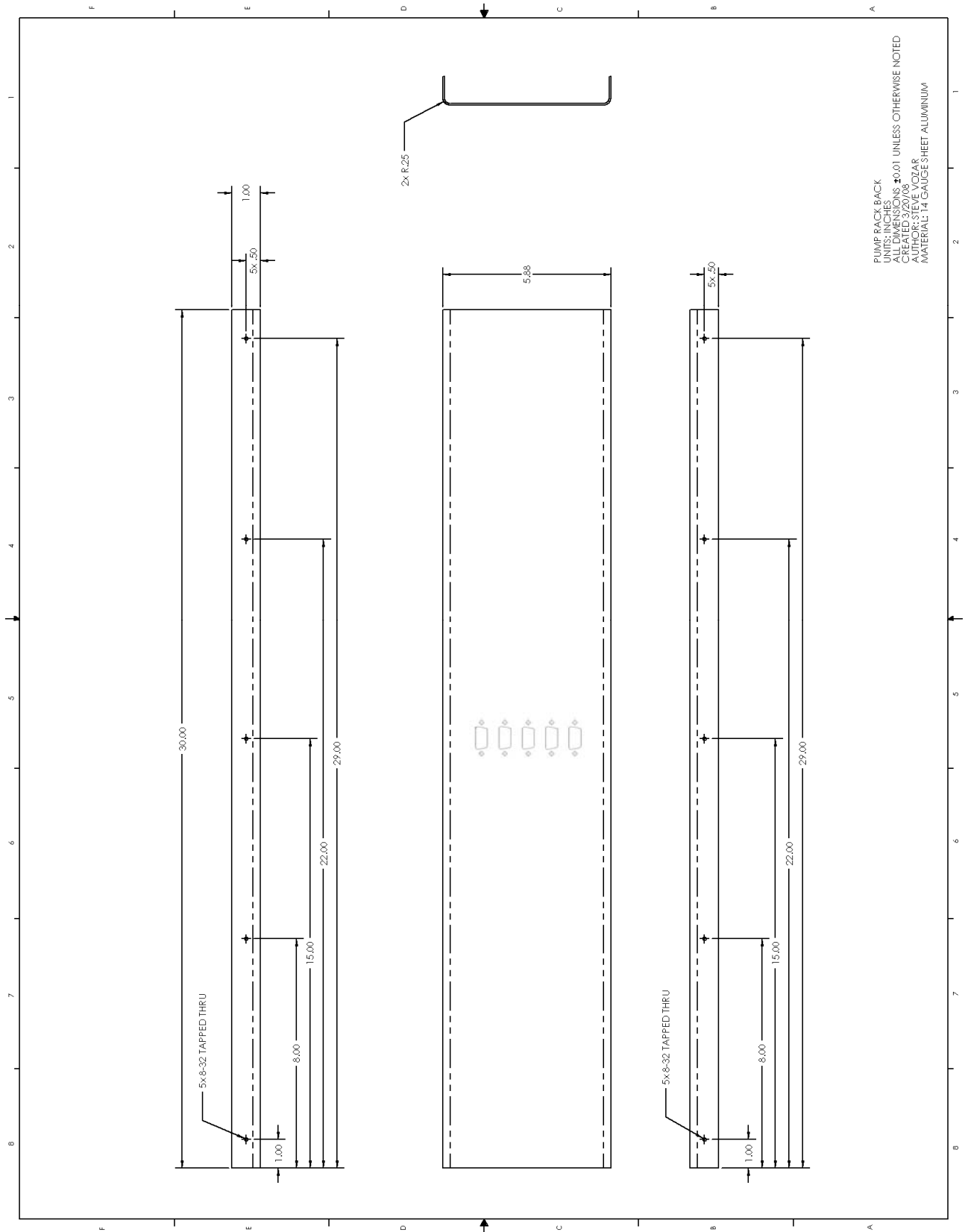


SPIN COATER COVER - SIDE CORNER  
STANDARD UNIT: INCHES  
TOLERANCE: ± 0.010  
MATERIAL: ALUMINUM  
AUTHOR: TOM SERBOWICZ  
CREATED: 3/20/2008

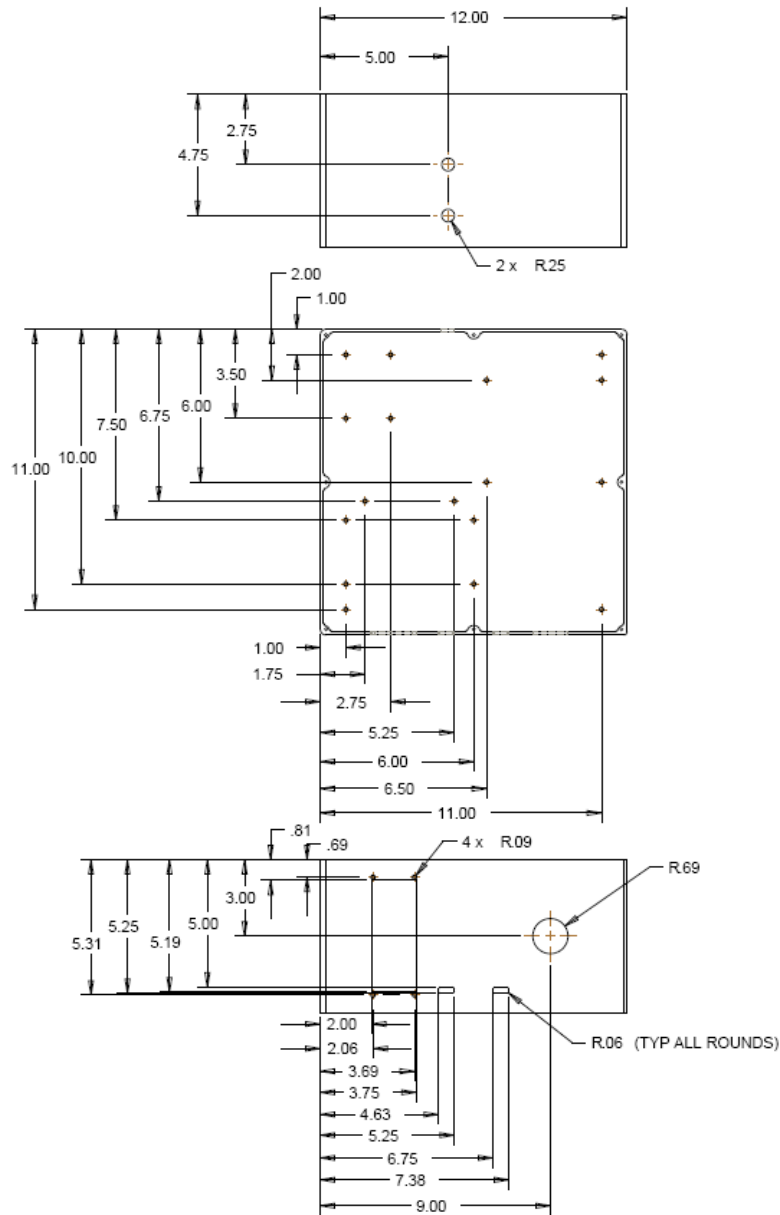
**Figure H9. Dimensioned drawing of pump rack front**



**Figure H10. Dimensioned drawing of pump rack back**

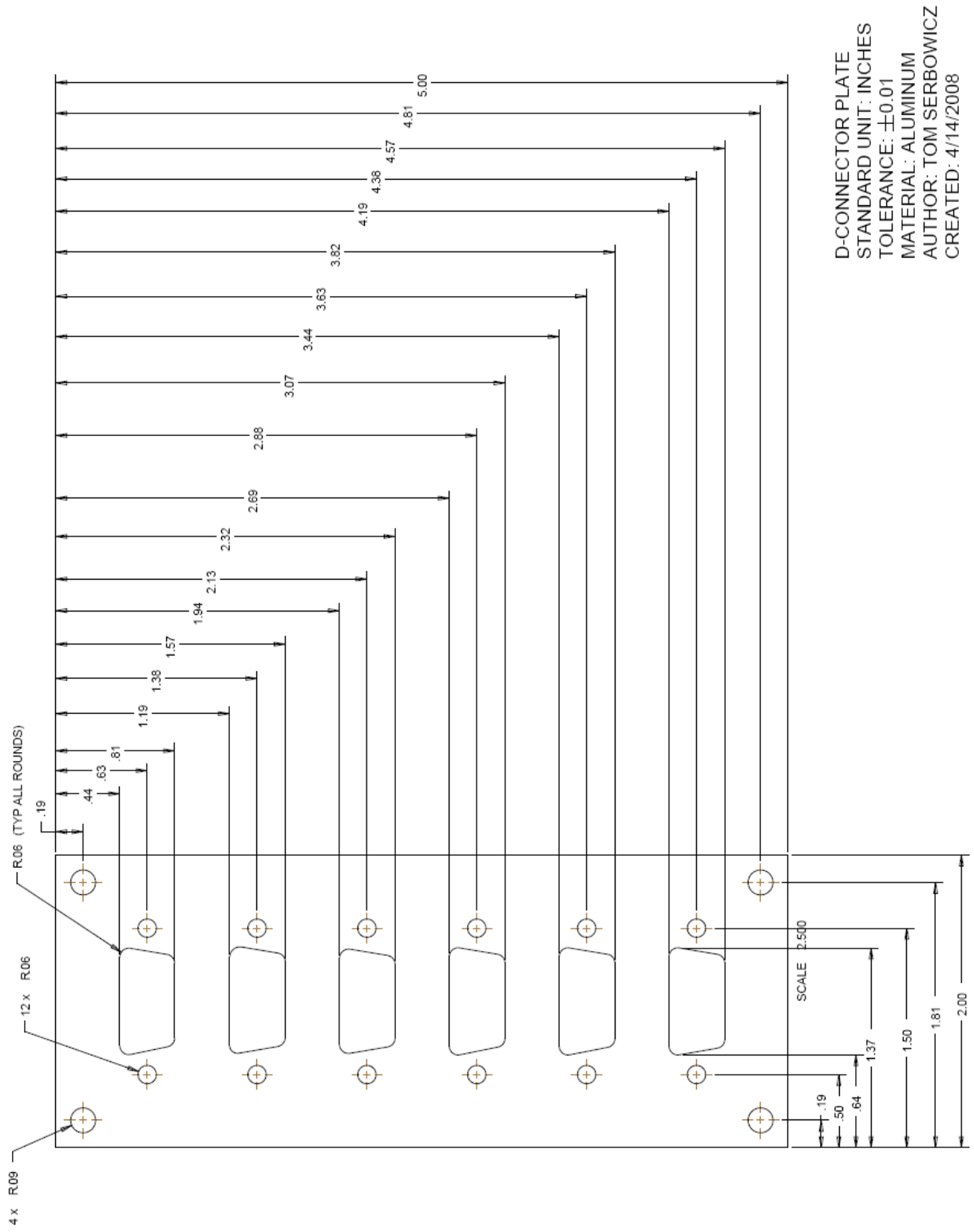


**Figure H11. Dimensioned drawing of junction box modifications**



JUNCTION BOX MODIFICATIONS  
 STANDARD UNIT: INCHES  
 TOLERANCE:  $\pm 0.01$   
 MATERIAL: PVC  
 AUTHOR: TOM SERBOWICZ  
 CREATED: 4/14/2008

**Figure H12. Dimensioned drawing of D-connector plate for junction box**







## **APPENDIX I – DESIGN CHANGES SINCE DR3**

Summary of design changes since Design Review #3:

- 1) Decided not to pursue Laser Displacement Sensor
- 2) Added D-connector holes on pump rack
- 3) Had to fabricate own cables instead of using RS232 interface
- 4) Switched from crimp-type D-connectors to solder D-connectors
- 5) Added chuck adaptor for glass slides

Note: Most components were already fabricated at the completion of Design Review #3. Therefore, we did not make very many changes to our system.

## APPENDIX J – DESIGN ANALYSIS ASSIGNMENTS

### Appendix J1. Material Selection Assignment

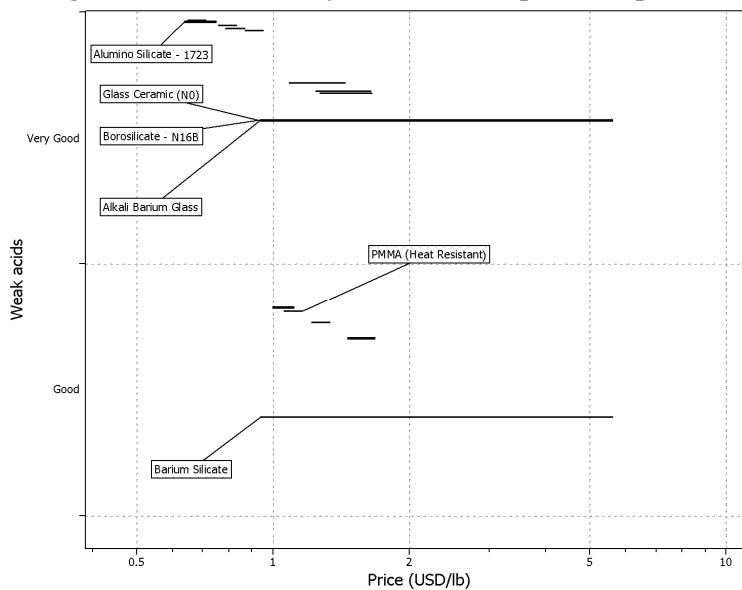
Our two materials for the material selection assignment are those for the modified cover and the pump rack. The function of the modified cover is to enclose the spin coater and house the fluid delivery tubes. The objective for making the cover is to machine it ourselves in the shop. Constraints for the modified cover include being transparent, chemically compatible, and safely enclose substrates spinning up to 10,000 rpm. The constraints on each selection criteria for the material are shown in Table J1 below.

**Table J1. Selection criteria for modified spin coater cover**

Stage	Selection Criteria	Constraint
1	Weak Acid	Good or very good
2	Optical	Transparent
3	Price	Maximum 1.5 USD/lb

The top five Choices were PMMA (Plexiglas), Aluminum Silicate, Glass Ceramic, Barium Silicate, and Borosilicate. Figure J1 shows the top five choices while comparing the price to resistance of the material to weak acid. From this graph it appears Alumino Silicate would be the optimal choice, however, the most readily available material is Plexiglas. Therefore we selected Plexiglas as the material for the modified spin coater cover.

**Figure J1. Weak acidity resistance vs. price for possible modified cover materials**



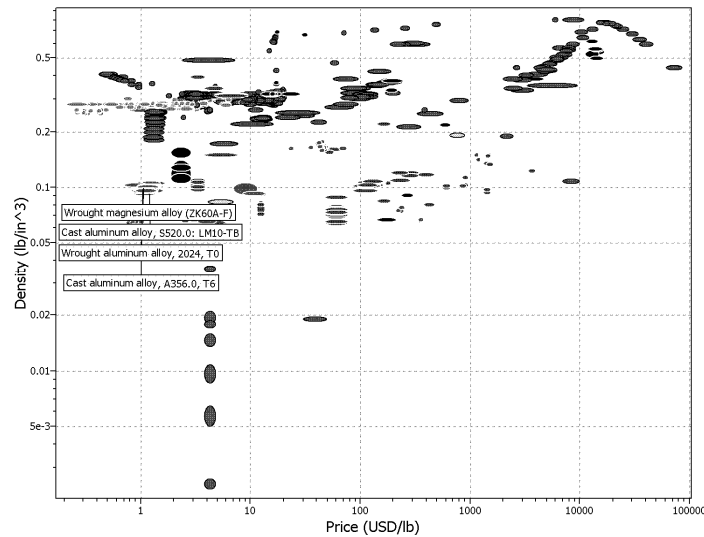
The pump rack was designed to house all five pumps with easy access to the solution containers. We also planned to machine this in the available shops. The pump rack must be easily portable, sturdy, and aesthetically pleasing. The constraints on each selection criteria for the material are shown in Table J2, p.67.

**Table J2. Selection criteria for the pump rack**

Stage	Selection Criteria	Constraint
1	Price	Maximum 2 USD/lb
2	Density	Max 0.1 lb/in <sup>3</sup>
3	Young's Modulus	Min 5x10 <sup>6</sup> psi
4	Optical	Opaque

The top five choices for the pump rack are aluminum 2024, aluminum 6061, cast aluminum, magnesium alloy, and PVC. Figure J2 shows the possible materials for the pump rack while comparing density and price. We chose 14 gauge sheet aluminum 6061 because it met the criteria and was easily obtained and machined.

**Figure J2. Density vs. price for possible pump rack materials**



### Appendix J2. Design for Assembly

The component of our system that we chose to analyze with the Design for Assembly method was the pump rack. The original design consisted of a main body piece and a back panel held on with screws that tighten into threaded holes in the body of the rack. The goal of this method for attaching the back panel was to allow the screws to be tightened without having to reach inside the long rack, and to allow quick and easy access in the case that a pump would need to be replaced or an electrical connection repaired. The five pumps bolt onto the front face of the body with the motor and gear box inside rectangular cutouts. Each pump requires four bolts with a lock washer and nut to be held securely in place. Four rubber feet were also incorporated into the design to help eliminate vibration and noise and to provide a secure, slip-free base. The two rear feet are fastened using two of the screws that hold the back panel in place, and the two front feet are secured with screws that tighten into their own threaded holes at the front two corners of the body. Five serial port D-connectors also mount to the inside of the back panel with special internally threaded hex head studs and nuts. Each D-connector requires two studs for mounting, and the connectors are fastened prior to mounting the back panel.

Using the test for minimum number of parts and ease of assembly guidelines, we determined four areas where the design could be improved. For a production redesign, we incorporated a “snap-in-place” design for the back panel, eliminating 10 fasteners and 10 washers and greatly reducing the assembly time of the back panel. The D-connectors could have threaded holes eliminating the need for nuts with the hex head studs. The rubber feet can be held in place with adhesive. Also, the separate lock washers could be

eliminated from the pump mounts while maintaining the same function by using nuts with attached lock washers. These changes improved the design efficiency from 24% to 43%, reducing assembly time from 840 s to 458 s.

**Table J3. Pump rack assembly steps before redesign**

1	2	3	4	5	6	7	8	9			
Part ID No	Number of times the operation is carried out consecutively	Two digit manual handling code	Manual handling time per part	Two digit manual insertion code	Manual in sertion time per part	Operation time (seconds) (2) * [(4) + (6)]	Operation cost (cents) 0.4 * (7)	Figures for estimation of theoretical minimum parts	Pump rack		
1	1	20	1.8	00	1.5	3.3	1.32	1	Main body		
2	1	20	1.8	01	2.5	4.3	1.72	1	Back panel		
3	5	30	1.95	06	5.5	37.25	14.9	5	Pump		
4	4	11	1.8	38	6	31.2	12.48	4	Rubber Feet		
5	8	11	1.8	38	6	62.4	24.96	0	Panel bolt		
6	10	03	1.69	00	1.5	31.9	12.76	0	Washer		
7	20	11	1.8	06	5.5	146	58.4	20	Pump bolt		
8	20	02	1.88	48	8.5	207.6	83.04	20	Nut		
9	20	04	2.18	10	4	123.6	49.44	0	Lock washer		
10	5	30	1.95	06	5.5	37.25	14.9	5	D-connector		
11	10	11	1.8	06	5.5	73	29.2	10	Hex-head stud		
12	10	04	2.18	38	6	81.8	32.72	0	Small nut		
							<b>839.6</b>	<b>335.84</b>	<b>66</b>	design efficiency = (3*NM)/TM	<b>0.24</b>
							TM	CM	NM		

**Table J4. Pump rack assembly steps after redesign**

1	2	3	4	5	6	7	8	9			
Part ID No	Number of times the operation is carried out consecutively	Two digit manual handling code	Manual handling time per part	Two digit manual insertion code	Manual in sertion time per part	Operation time (seconds) (2) * [(4) + (6)]	Operation cost (cents) 0.4 * (7)	Figures for estimation of theoretical minimum parts	Pump rack		
1	1	20	1.8	00	1.5	3.3	1.32	1	Main body		
2	1	20	1.8	30	2	3.8	1.52	1	Back panel		
3	5	30	1.95	06	5.5	37.25	14.9	5	Pump		
4	4	11	1.8	30	2	15.2	6.08	4	Rubber Feet		
7	20	11	1.8	06	5.5	146	58.4	20	Pump bolt		
8	20	12	2.25	48	8.5	215	86	20	Nut		
10	5	30	1.95	06	5.5	37.25	14.9	5	D-connector		
11	10	11	1.8	38	6	78	31.2	10	Hex-head stud		
							<b>457.8</b>	<b>183.12</b>	<b>66</b>	design efficiency = (3*NM)/TM	<b>0.43</b>
							TM	CM	NM		

### Appendix J3. Design for Environmental Sustainability

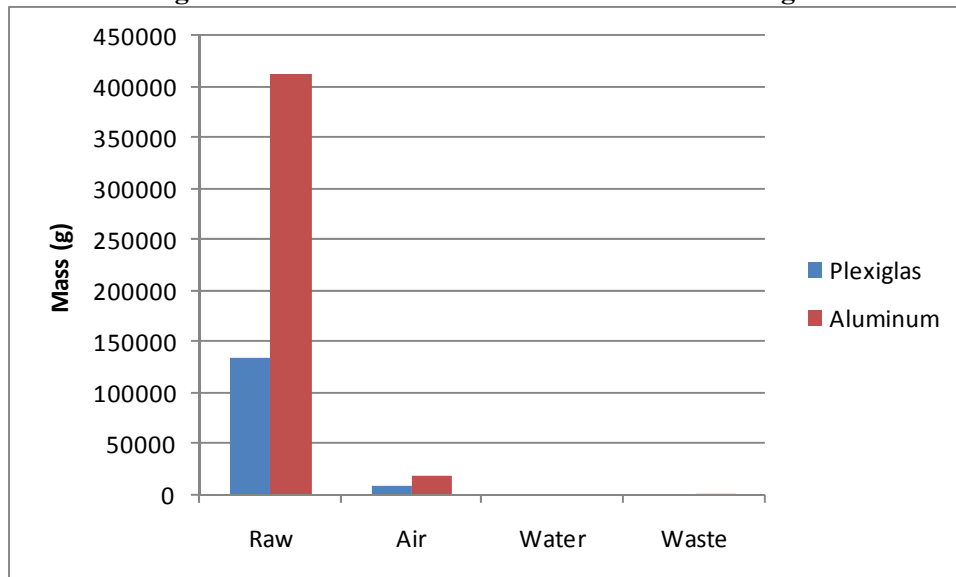
Design for environmental sustainability was done for two of the selected materials for our project shown in Table J5. Using SimaPro, the following charts were created to compare the environmental impact from the two materials: Total emission (Fig. J3), relative impact in disaggregated damage categories (Fig. J4), normalized score in human health, eco-toxicity, resource categories (Fig. J5), and single score comparison (Fig. J6). Based on these results, we see that the raw material of both aluminum and Plexiglas will have the highest impact on mass emissions. Water and waste emissions are negligible in comparison.

Looking at the normalized score for both materials, aluminum has a much higher impact, with the most important category being resources. Both materials are negligible for eco-toxicity, and have a low impact on human health. Because aluminum is a mined material, it makes sense that the largest impact is in resources, while Plexiglas is a polymer and is manufactured. From Fig. J6, it is clear that aluminum has a higher impact on the EcoIndicator 99 scale. The value of 6.4 for aluminum is over twenty times the damage classification than the value of 0.3 for Plexiglas. If this product were in high demand, it would be beneficial for the environment to reconsider the use of aluminum as a selected material. Because there will only be one product made for lab use, it is appropriate to use aluminum because of its availability and ease of machining.

**Table J5. Materials selected for design for environmental sustainability**

Material	Mass (kg)
Aluminum 6061	2.21
PMMA (Plexiglas)	1.4

**Figure J3. Total Emissions of Aluminum and Plexiglas**



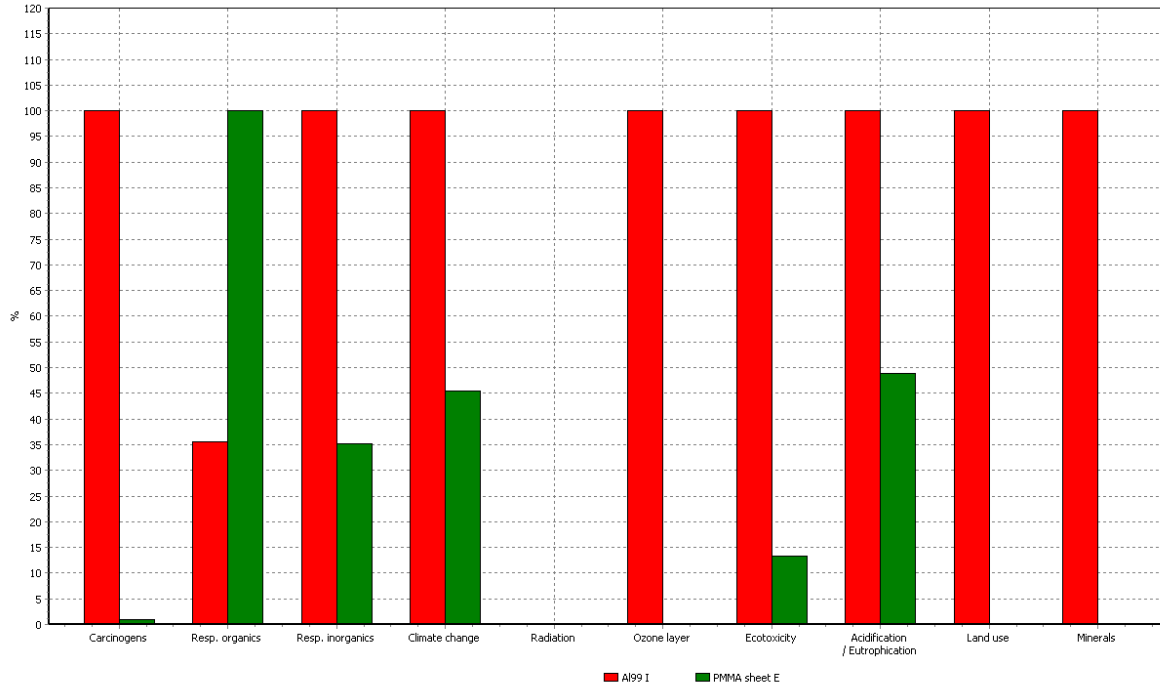
# Figure J4. Impact assessment of Aluminum and Plexiglas

SimaPro 7.1 Educational  
Project: Des for Env Sustainability

Impact assessment

Date: 4/5/2008 Time: 3:37:41 PM

Title: Comparing 2.2 kg 'Al99 I' with 1.4 kg 'PMMA sheet E'  
 Method: Eco-indicator 99 (I) V2.02 / Europe EI 99 I/A  
 Indicator: Characterization  
 Skip categories: Never  
 Relative mode: Non



Comparing 2.2 kg 'Al99 I' with 1.4 kg 'PMMA sheet E'; Method: Eco-indicator 99 (I) V2.02 / Europe EI 99 I/A / characterization

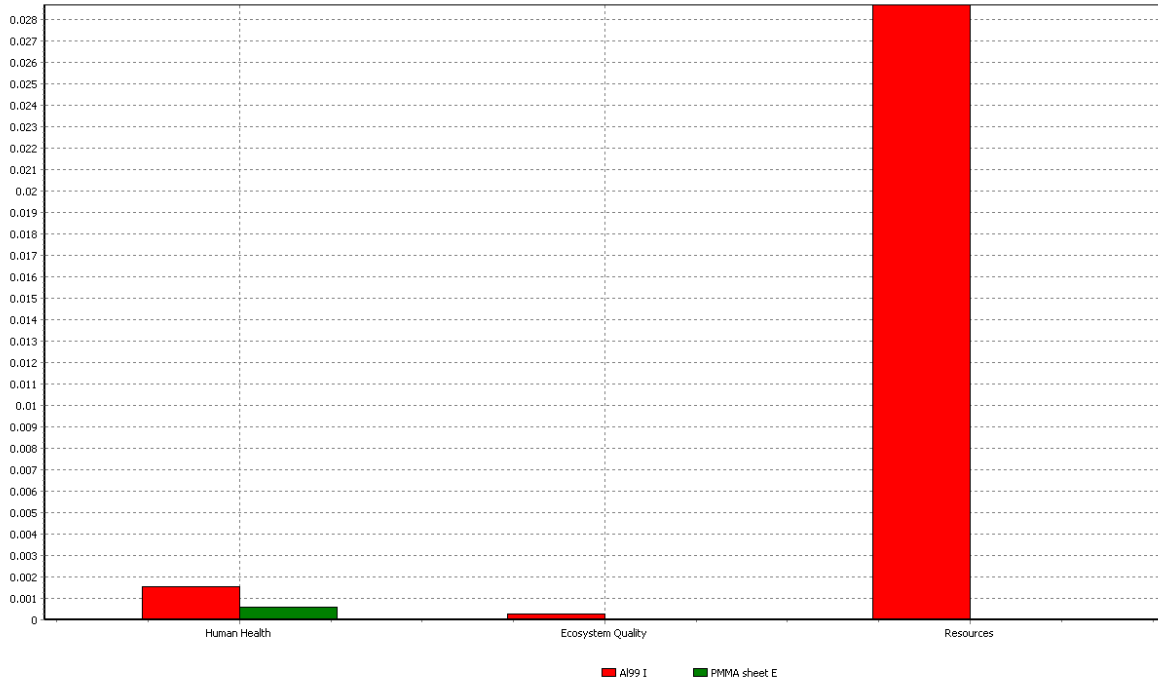
# Figure J5. Normalized score in human health, eco-toxicity, and resource for aluminum and Plexiglas

SimaPro 7.1 Educational  
Project: Des for Env Sustainability

Impact assessment

Date: 4/5/2008 Time: 3:38:07 PM

Title: Comparing 2.2 kg 'Al99 I' with 1.4 kg 'PMMA sheet E'  
Method: Eco-indicator 99 (I) V2.02 / Europe EI 99 I/A  
Indicator: Normalization  
Per impact category: Yes  
Skip categories: Never  
Relative mode: Non



Comparing 2.2 kg 'Al99 I' with 1.4 kg 'PMMA sheet E'; Method: Eco-indicator 99 (I) V2.02 / Europe EI 99 I/A / normalization



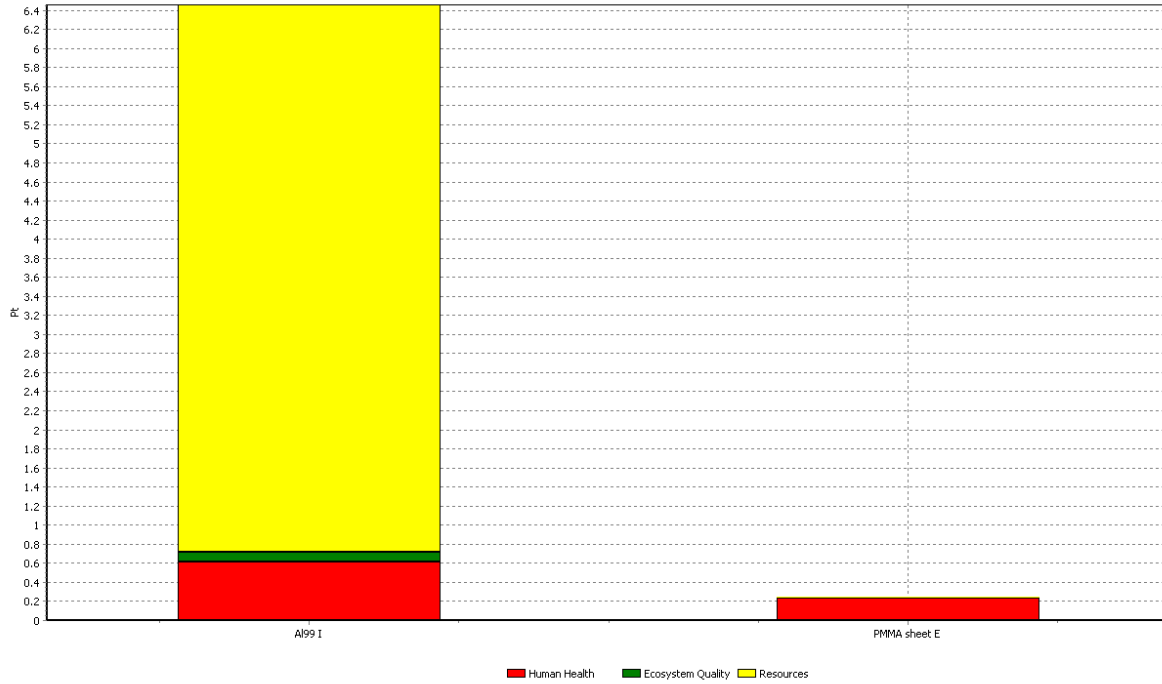
**Figure J6. EcoIndicator 99 single score comparison of aluminum and Plexiglas**

SimaPro 7.1 Educational  
Project: Des for Env Sustainability

Impact assessment

Date: 4/5/2008 Time: 3:38:39 PM

Title: Comparing 2.2 kg 'Al99 I' with 1.4 kg 'PMMA sheet E'  
Method: Eco-indicator 99 (I) V2.02 / Europe EI 99 I/A  
Indicator: Single score  
Per impact category: Yes  
Skip categories: Never  
Relative mode: Non



**Appendix J4. Design for Safety**

The report below shows the risk assessment for the final design including major risks and who is at risk. Using DesignSafe we were able to maintain or reduce all risk levels, with none of the remaining risks above moderate. A few risks were unable to be reduced by design, and therefore our project will include warning labels as well as instructions on how to operate all equipment.

Risk assessment focuses more towards human risk of operating our project. Another tool for assessing risk is FMEA (failure modes and effects analysis). FMEA focuses towards the potential failures of the product. Because our product will be used in a laboratory, it was more useful to perform a risk assessment for our project to keep the operators safe while using the machine.

We have designed our system with acceptable risks with respect to function and safety. The potential hazards could still occur if someone neglects the safety features. It would be impossible for this system to have zero risk because of the chemicals used and high speed of the rotating objects. We recommend to anyone using our product to read all instructions before operating.

**designsafe Report**

Application: Spin-Grower Safety Analyst Name(s): Matt Bachner  
 Description: Product Identifier: Spin-Grower Company: UM  
 Assessment Type: Detailed Facility Location: 3417 GGB (Kotov laboratory)  
 Limits:  
 Sources: not sure yet

Guide sentence: When doing [task], the [user] could be injured by the [hazard] due to the [failure mode].

User / Task	Hazard / Failure Mode	Initial Assessment		Risk Reduction Methods /Comments	Final Assessment		Status / Responsible /Reference
		Severity Exposure Probability	Risk Level		Severity Exposure Probability	Risk Level	
All Users All Tasks	mechanical : pinch point When inserting the tube into the peristaltic pump, fingers could get caught if pump begins rotating.	Serious Occasional Unlikely	Moderate	warning label(s), standard procedures	Serious Occasional Negligible	Moderate	On-going [Daily] User
All Users All Tasks	mechanical : break up during operation When running the spin coater, be sure to follow safety instructions of vacuum pump on and lid closed. Do not bypass safety features	Slight Frequent Unlikely	Moderate	fixed enclosures / barriers, interlocked switches, E-stop control	Slight Frequent Negligible	Low	On-going [Daily] User
All Users All Tasks	electrical / electronic : energized equipment / live parts While power is on, injury could result from touching live wires.	Catastrophic Remote Unlikely	Moderate	prevent energy buildup, fixed enclosures / barriers	Catastrophic Remote Unlikely	Moderate	Complete [4/6/2008] User
All Users All Tasks	electrical / electronic : shorts / arcing / sparking When removing serial cables with power on, sparking could occur.	Serious Occasional Possible	High	fixed enclosures / barriers screw down hoods	Serious Remote Negligible	Low	Complete [4/6/2008] User
All Users All Tasks	electrical / electronic : water / wet locations When refilling the solutions, do not spill liquid onto pumps or electrical equipment.	Serious Remote Negligible	Low	fixed enclosures / barriers	Serious Remote Negligible	Low	On-going [Daily] User

Page 1

User / Task	Hazard / Failure Mode	Initial Assessment		Risk Reduction Methods /Comments	Final Assessment		Status / Responsible /Reference
		Severity Exposure Probability	Risk Level		Severity Exposure Probability	Risk Level	
All Users All Tasks	electrical / electronic : unexpected start up / motion When running labview, the computer could be slow sending the signal, especially after a recent startup.	Slight Remote Unlikely	Low	instruction manuals	Slight Remote Unlikely	Low	On-going [Daily] User
All Users All Tasks	fire and explosions : sparks When starting the pumps, loosely connected cables could cause sparks or wrong operation of pumps.	Serious Remote Negligible	Low	fixed enclosures / barriers	Serious Remote Negligible	Low	Complete [4/6/2008] User
All Users All Tasks	confined spaces : confined spaces When in the lab, be aware of others in the lab	Slight Occasional Possible	Moderate	safety glasses, special clothing	Slight Occasional Possible	Moderate	On-going [Daily] User
All Users All Tasks	environmental / industrial hygiene : wastewater contamination When removing waste, use proper disposal containers and do not pour down sink	Slight Frequent Negligible	Low	fixed enclosures / barriers labeled waste container	Slight Remote Unlikely	Low	On-going [Daily] User
All Users All Tasks	chemical : skin exposed to toxic chemical When using chemicals, the user could be injured by the strong acids or bases used if skin contact occurs.	Serious Remote Unlikely	Moderate	special clothing, gloves	Serious Remote Unlikely	Moderate	On-going [Daily] User
All Users All Tasks	fluid / pressure : liquid / vapor hazards When system is running, evaporated solutions are airborne and could be inhaled.	Minimal Occasional Negligible	Low	separate hazard / people in time or space	Minimal None Probable	Low	On-going [Daily] User

### Appendix J5. Manufacturing Process Selection

The spin grower was created for the use in a lab and therefore does not have a very high demand. For this assignment we will assume that other research labs could use our machine, and assume a production volume of 100 machines. The two materials selected are aluminum to make the pump rack and Plexiglas to make the cover.

Table J5 shows the selection criteria for the aluminum to make the pump rack. The three options for metals from these criteria are stamping, press forming, and micro-blanking. For producing 100 pump racks we would recommend stamping the metal to form its shape and cut the holes. Some holes will later need to be tapped.

**Table J5. Selection criteria for aluminum shaping**

Stage	Selection Criteria	Constraint
1	Shape	Flat sheet
2	Mass	Max 5 lbs
3	Thickness	Max 0.1 inches
4	Process	Primary shaping process

For the Plexiglas to make 100 spin coater covers, it is recommended to cut the material in a laser cutter as we did for this project. Table J6 shows the criteria used to determine an appropriate joining method. The choices available for joining the sheets are adhesives of epoxy, acrylic, polyester, and polysulfide. We recommend epoxy adhesive joining process because of its quick drying time, strength, and chemical compatibility.

**Table J6. Selection criteria for Plexiglas joining**

Stage	Selection Criteria	Constraint
1	Material	Composites
2	Join Geometry	Butt joining
3	Function	Water tight

## APPENDIX K – ELECTRICAL CONNECTIONS

Figure K1. Pinout diagram for male connectors

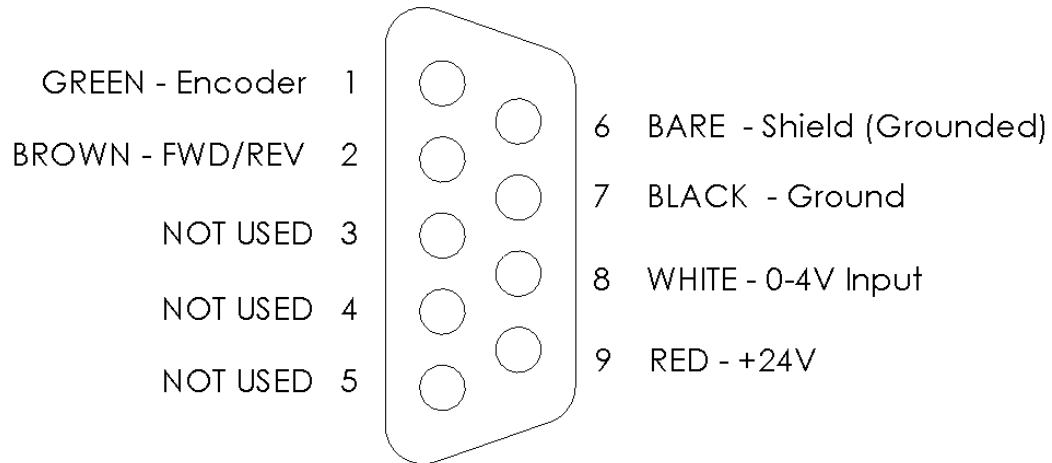
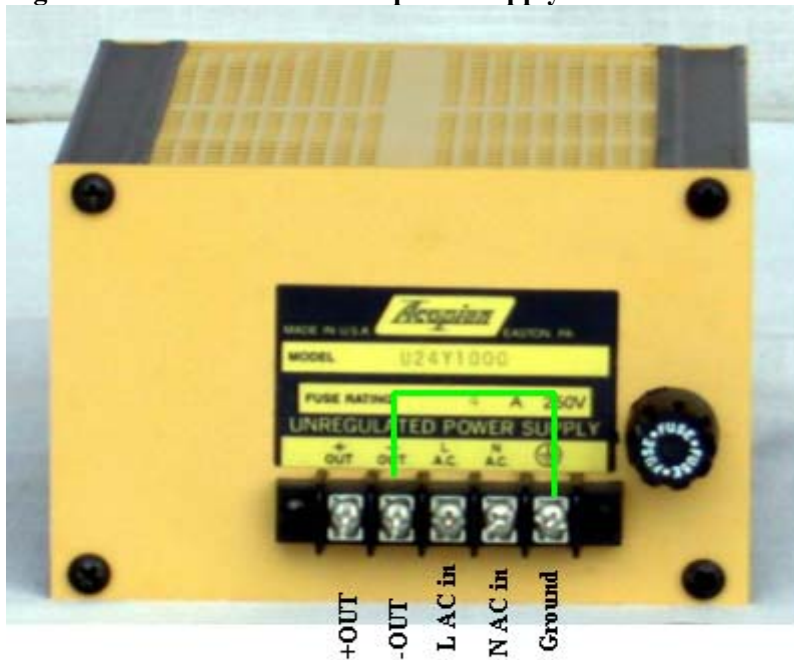


Figure K2. Connections to DC power supply



Note: -OUT Connected to Ground

### Notes:

- 1) Brown (FWD/REV), Red (+OUT/+24V), Black (-OUT/Ground), and Shield are common to all pumps, connected via barrier strips and jumper strips.
- 2) Shield, Ground, and Signal Ground are all common to outlet ground
- 3) Green (Encoder) is not used, but is connected for possible future use

## APPENDIX L – INSTRUCTIONS FOR USE

This is a basic manual explaining how to use the “Spin Grower” instrument. Separate instructions for the individual components can be found with the instrument documentation. This assumes that you have read the instructions for and properly set up the SCS Spin Coater and the peristaltic pumps.

### Connections:


- 1) Plug the vacuum pump into the outlet on the control box. Connect the female end of the USB connector to the control box, and the male end to the computer. Connect each pump to the corresponding port on the control box with the cables provided.

### WARNING:


**NEVER CONNECT OR DISCONNECT CABLES TO OR FROM THE CONTROL BOX OR PUMPS WHILE THE CONTROL BOX IS SWITCHED ON. DOING SO MAY LEAD TO AN ELECTRICAL ARC JUMPING BETWEEN PINS, WHICH CAN DAMAGE THE INSTRUMENT AND/OR CAUSE SERIOUS INJURY.**

- 2) Thread one end of each of the bioprene tubes through the holes in the container caps. Thread the other ends through the Loc-Line tubing into the spin coater. It may be easier to disconnect the last (nozzle) link of the Loc-Line, thread it through the other links, and then pull it through the nozzle. Then reconnect the link to the rest of the Loc-line.
- 3) Clamp the bioprene tubes into the corresponding peristaltic pumps.
- 4) When you want to use the pumps, switch on the ‘Power’ switch on the front of the control box.

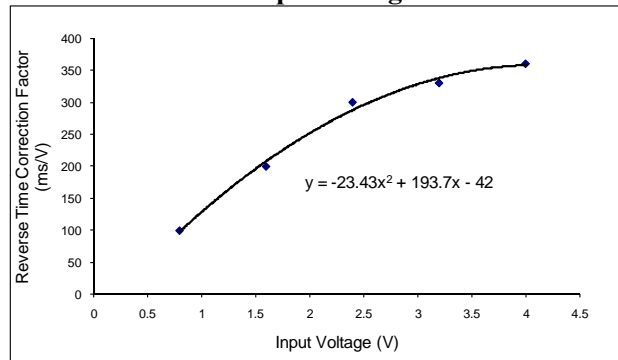
### Purging the Lines:

- 1) To purge the lines of air, run the program ‘Normal Output.vi’, and continuously run  the vi. Set a container inside the spin coater to catch the purging fluid. Set the program to Pump 1, set the output rate to about 2V, and then wait for the line to purge. Then set the output rate to zero, wait for the pump to stop running, and dab off any drips from the line with a paper towel. Repeat this for each of the remaining pumps.

### Calibrating the Instrument:

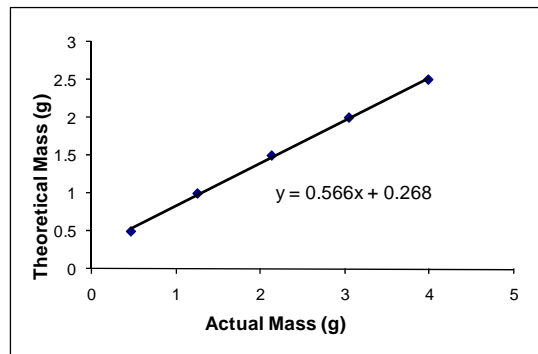
- 1) The pump must be calibrated for proper reverse time after fluid deposition so that there are no drips. Open the program ‘No Drip Calibration.vi’ and set the injection time to 2 seconds. For injection rates of 15, 30, 45, 60, and 75 mL/min, run  different reverse times (with liquid being deposited into a beaker) and record the lowest reverse time for which the pump completely prevents all drips. Plot the injection rate vs. the reverse time and fit a second-order trend line to the data (see template at ‘reverse calibration.xls’) The coefficients of the equation should be put into the table in the block diagram of the program ‘Pump Control.vi’. Repeat this for each pump.

**Figure L1. Calibration curve between input voltage and Reverse Time correction factor**

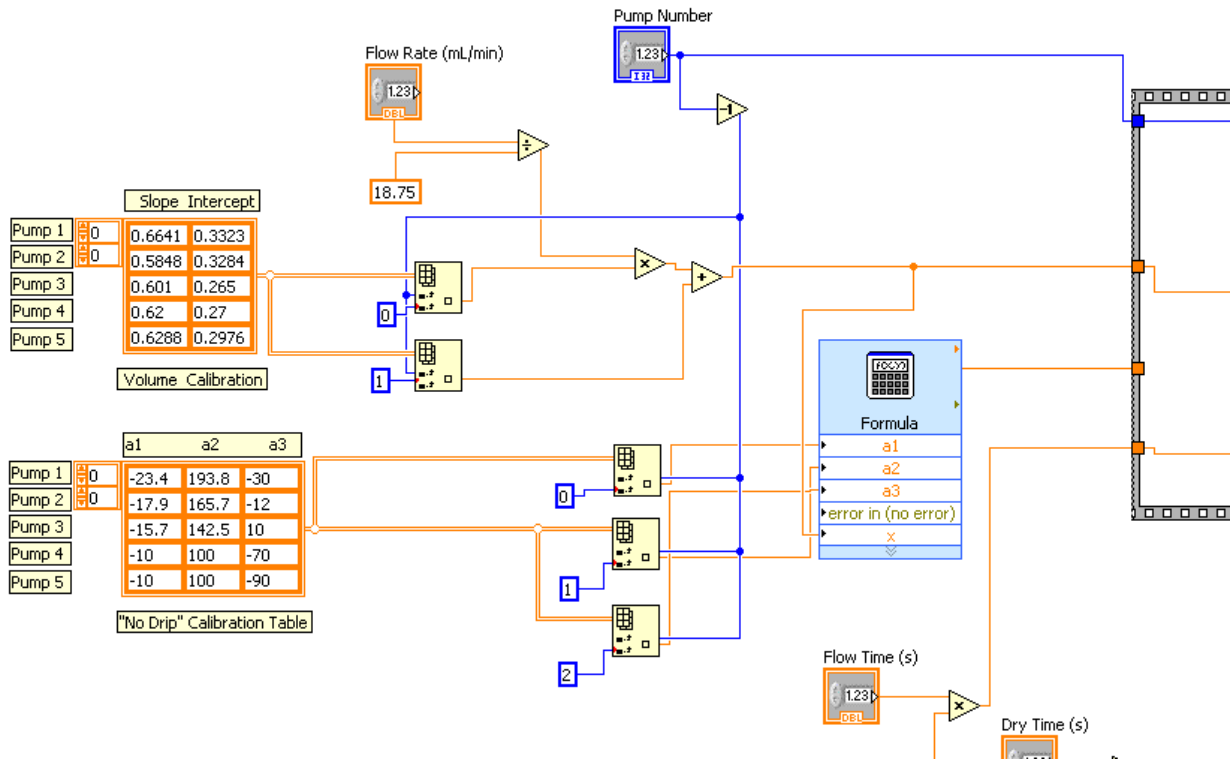


- 2) The pumps must also be calibrated to inject the proper amount of fluid. Using distilled water as the injection fluid, open the program 'Pump Control.vi'. Open the block diagram for of the vi, and set all the values of the first column of the volume calibration table to 1, and the second column values to 0. Set pump 1 to inject at 15, 30, 45, 60, and 75 mL/min for a time of 2 seconds. For each run, inject the water into a beaker, and determine the mass of the injected liquid. Plot the actual mass vs. the expected mass and fit a linear trend line through the data (see template at 'volume calibration.xls'). Change the value of the first column to the slope of the line, and the value of the second column to the intercept of the line. Test to make sure the proper volume is being deposited, and reiterate the calibration if necessary. Repeat this for each pump.

**Figure L2. Calibration curve theoretical mass and actual mass of injected water**



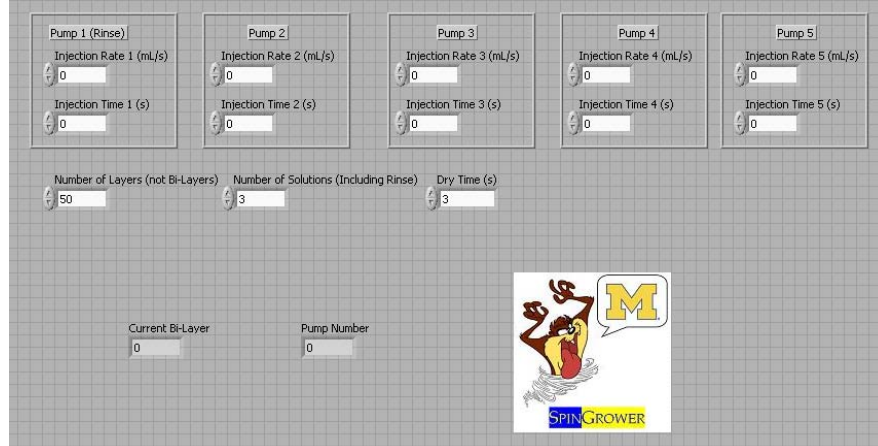
**Figure L3. Portion of block diagram of ‘Pump Control.vi’ showing Volume Calibration and “No Drip” Calibration tables**








**Making a film:**

- 1) Position the Loc-line tubing to the desired fluid deposition location, center the substrate onto the chuck in the spin coater, and then close the spin coater lid.
- 2) Load the recipe with the desired spin rate and duration into the spin coater.
- 3) Open the program ‘Spin Grower.vi’, and input the parameters for the film (Each solution’s injection rate and time, the dry time between layers, the number of solutions, and the number of layers desired).

**Figure L4. Front Panel of ‘Spin Grower.vi’, showing the input parameters**



- 4) Turn on the vacuum switch on the control box, and start the spin coater.
- 5) Press the run button () on the top of the vi, and let the program run through.
- 6) If a sequence needs to be aborted, wait until a time where the substrate is drying (no pump injection), and hit the abort button () on the vi.
- 7) To perform an emergency stop, switch off power to the pumps on the control box (**NOT THE VACUUM**), abort () the vi, set all injection rates to zero, run () the vi again, and then abort () it again. Turn the pumps back on. This way, no signal voltage is being supplied to the pumps when you turn them back on.

**Troubleshooting:**

- 1) If the pumps don't work, check to make sure that the power on the control box is on.
- 2) If one pump does not work, but the others do, it might be a problem with the cable. **TURN THE CONTROL BOX POWER OFF.** Switch the cable for the nonworking pump with the cable for a working pump, turn the control box power back on, and see if the cable needs to be fixed. If the problem is in the cable, check the cable for continuity and shorts with a multi-meter. **NEVER CONNECT OR DISCONNECT CABLES TO OR FROM THE CONTROL BOX OR PUMPS WHILE THE CONTROL BOX IS SWITCHED ON.**