

Water Contaminant Analysis through Raman Spectroscopy

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ABSTRACT – The objective of this project is to identify and test at least 2 two contaminants of water. Ammonium Nitrate, Sodium Sulfate and Potassium Perchlorate, through a Raman spectrometer. Our group has chosen this project for senior design because not only does it challenge us, but it provided a good mix of optics and photonics, computer, and electrical engineering skills to formulate a solution.

I. INTRODUCTION

As pollution becomes an ever more present problem in our society, people often wonder how their food and drinking water will be affected by these chemicals, pesticides, and contaminants that have become so prevalent in the way things are made. Pesticides cover our grasses and run off into our aquifers, air pollution is absorbed into the rain and falls back down into the oceans and wells. Acid rain is a serious threat to Floridians due to the daily afternoon thunderstorms that we experience. Coupled with the train derailments in Ohio that poisoned the area, the average person is bound to be worried about their overall health faced with all the health hazards that invade our means of life. For that reason, we have decided to develop a Raman spectrometer that will be able to detect the contamination levels of any liquid sample.

People may wonder how their food and drinking water will be affected by these chemicals, pesticides, and contaminants that have become so prevalent.

The goal of this project is to create a Raman spectroscopy platform for identification and measurement of pollutants in water samples. The focus of this project will be identifying contaminants such as heavy metals and organic pollutants. The system will be created to be user-friendly and can be applied to other fields not only the applications related to water.

The team created a specially designed Raman spectroscopy system that originally featured a 785 nm laser that was then switched to a 532nm excitation laser, a fiber optic probe, and a spectrometer to accomplish this objective. The performance of the system was tested using a variety of chemicals that were dissolved in water after it had been calibrated using a mercury line light source.

The finding that we want to show is that the overall system can identify and measure a variety of contaminants in samples of water within our defined spectral range, specifically Ammonium Nitrate, Potassium Perchlorate, and Magnesium Sulfate. The system is suitable for a variety of applications in environmental monitoring and water quality testing since it can identify toxins at different levels of the environment and where people are at the most.

II. NARRATIVE

A. Objective Description

The focus of this project and our design is to develop a tool that is reasonably priced and capable as much as other technologies to determine the type of contaminant and potentially the level of that contaminant within our samples of water.

III. OPTICAL DETAILS

Pictured below are the models of well-plate reader containing well as a conceptual drawing of the spatial heterodyne spectrometer and its fundamental components. A fiber would collect the light scattered from the sample inside of the cuvette and carry that signal to the SHS. Once the laser is focused on the samples it will be illuminated. The light that we expect to see would

be the Raman signal. This will then be imaged upon the detector.

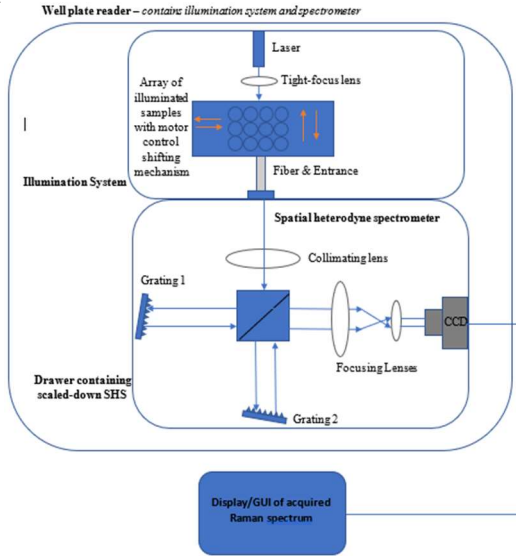


Figure 1 - Illumination system and spectrometer layout

We decided to go with a cage mounting system for ease of use when building and the rods to make sure that everything in the system is always on the same optical axis. As seen below the image of the optical components in a caged system.

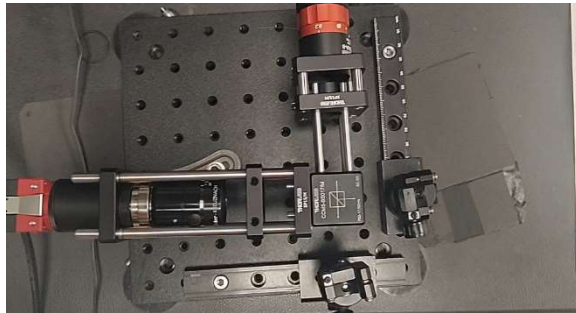


Figure 2 - Spatial heterodyne spectrometer mounted on our cage system.

Our goal is to identify a material's chemical makeup using Raman spectroscopy. This technique can uniquely identify a material using a Raman scattered signal that is collected by a spectrometer.

A laser beam is guided and focused onto a sample via the excitation optical fiber. The light that scatters off the sample is collected and redirected to a spectrometer. A Raman probe is used to send the laser beam towards the sample as well as

collect the light that scatters off it. The scattered light is brought to a collection fiber that is large in diameter compared to the excitation fiber.

The collection fiber has an FC/APC fiber connector at one end such that when connected to the spectrometer there is minimized coupling loss.

The beam that enters via the FC/APC collection fiber is then collimated and brought to a beam splitter. A notch filter is placed on the entrance arm of the spectrometer before the beam splitter to remove laser wavelength with the Raman signal remaining. The split beams are then diffracted from the gratings and recombined to produce an interferogram that is focused onto a detector.

A. Spatial Heterodyne Spectrometer

As mentioned previously, Raman spectroscopy has several advantages in identifying a material's chemical composition. However, the Raman signal scattered off a material is weak. In our research, we determined the Spatial Heterodyne Spectrometer (SHS) would be a unique challenge but would also serve a practical purpose.

The SHS, as a concept, has high throughput, allowing for as much acceptance of the Raman signal possible. There is no entrance slit, commonly seen in other spectrometers such as the Czerny-Turner; an SHS with a one-inch entrance aperture can accept lighter than a spectrometer with a slit size of $10\mu\text{m}$. This device can also achieve high resolution balanced with an appropriate spectral range. This can be illustrated with the below equations:

$$\theta_L = \sin^{-1}\left(\frac{n\lambda}{2d}\right)$$

$$\text{Resolving Power} = 2 \times d \times w$$

$$\text{Resolution: FWHM} = \Delta\lambda = \frac{\lambda}{R}$$

$$\text{Spectral Range} = \frac{N\lambda}{2R}$$

where d is the grating groove density, w is the grating width, λ is the wavelength of the excitation laser source, and N is the number of

pixels along the detector in the horizontal direction.

θ_L is the grating angle with respect to the optical axis such that the first diffracted order is redirected along the path of incidence. Based on our grating 785nm excitation laser, the grating is tilted at 4.6° . A large focus on the design process for our team, based on a selected laser and grating, is based on the tradeoff between spectral range and the resolving power of the spectrometer.

B. Lens Design

Ultimately what we are imaging is the grating across the detector and beam splitter. Considering that our grating is half an inch in size and that the sensing area of the selected detector is inevitably going to be smaller, we needed to design a minifying lens.

The detector was selected based on ease of communication with MATLAB, our means of GUI development and data transfer, as well as quantum efficiency in the desired spectral range and overall spectrometer size.

The spectral range of our spectrometer is approximately 100nm, from 532 to 600nm. The selected detector is approximately 75-85% efficient at visible wavelengths up to 675nm. The number of pixels across the detector is 1,936, with a sensing area of 5.61mm x 3.18mm. With these number of pixels and laser wavelength, the resolution can be as high as 0.1nm.

Considering the area of the sensor and grating size, we decided to construct a lens that has a magnification of $\frac{1}{4}$. All the optics have reasonable transmission efficiencies in the visible region.

Each glass was selected to be one inch in size to be close to the 16mm cage plates used in our system while minimizing optical aberrations that could be more prevalent with smaller optics. There are two achromatic doublets, an iris, and an aspherical lens.

With a total track length of 99.6mm, the effective focal length of the lens is 53.21mm. All optics are to be housed in a lens tube barrel that is to be mounted on the cage system. See the figure below.

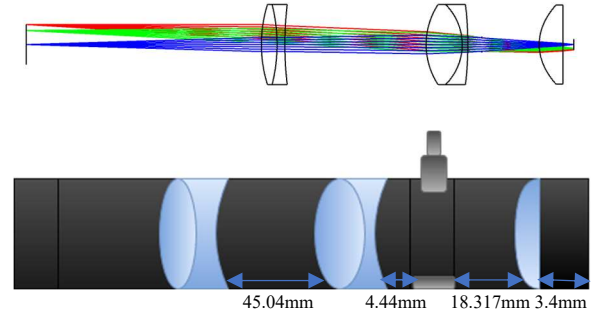


Figure 3 - Lens tube design with lens spacings

C. Optical Testing

The resulting image should be of the two gratings overlapped. The detector should be mounted onto the cage system and centered about the optical axis. It should be emphasized that the gratings are equidistant from the beam splitter and that they should be tilted in the same direction at the same angle such that the first orders are directed back along the incident path.

This can be tested with an alignment laser. A neutral density filter should be placed on the detector should it be used to help with alignment to protect pixels from being damaged. We used IDS Peak to view our images during the alignment process.

By centering the alignment laser on the grating and ensuring that the split beams overlap at the detector, the optical axis can be defined. The fringes that result from a single wavelength laser are equally spaced, or a single spatial frequency, separated by a distance described by the following equation.

$$\nu_F = 4(k - k_L) \tan(\theta_L)$$

where k is the wavenumber of the light incident on the detector.

Using a broadband white light source helped us identify the gratings on the detector. It is crucial to ensure that the light is as collimated as

possible. A collimator can bring our light to this state, at least within the visible region, within the dimensions of our spectrometer. The exit beam diameter is close to 10mm.

Once the light is collimated, one can visually inspect the grating and center the beam about the grating. In the detector view in IDS Peak, as seen in the figure below, there are three elements: the image of the gratings on the reflective and transmission side of the beam splitter, and the original source of light propagating at the entrance. By overlapping all three on the center of the detector, the spectrometer can be fully aligned.

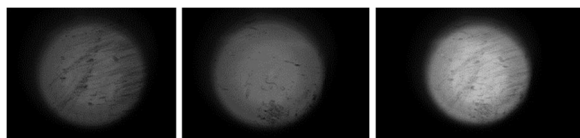


Figure 4 - Grating one (left), grating two (middle), gratings overlapped (right)

Finally, by using a known light source with standardized emission spectra, we can verify the functionality of our spectrometer. A mercury line light source was chosen due to its emission in the visible region. Attaching the mercury light source via an FC/APC fiber optic cable to our collimator, the below image was yielded.

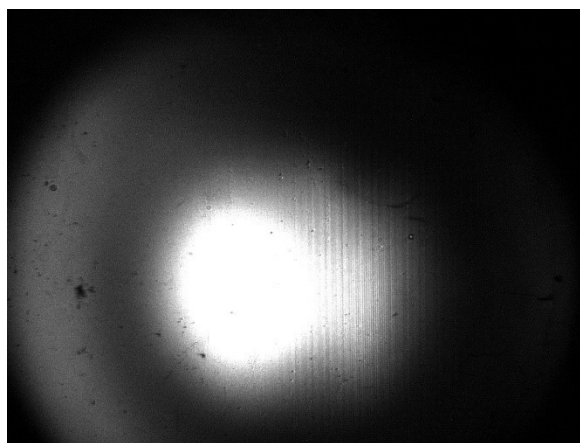


Figure 5 - Interferogram from mercury light source

By Fourier transforming the fringes above, the spectrum of mercury can be obtained. Note that the fringes in the above image are faint. The bright spot towards the middle of the image may be due to the input fiber used in calibration.

Below is an image of the spectrum of mercury obtained by our spectrometer.

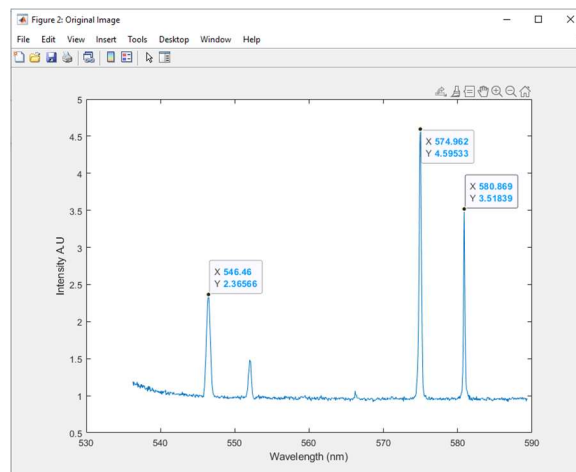


Figure 6 - Fourier transform of mercury fringes results in the below spectrum.

Using a 532nm laser to excite our samples resulted in a spectral range of about 70nm, a resolution of 0.4683nm, and a smaller tilt angle for the gratings.

The chemicals we were able to measure were potassium perchlorate, ammonium nitrate, and sodium sulfate. See the below images.

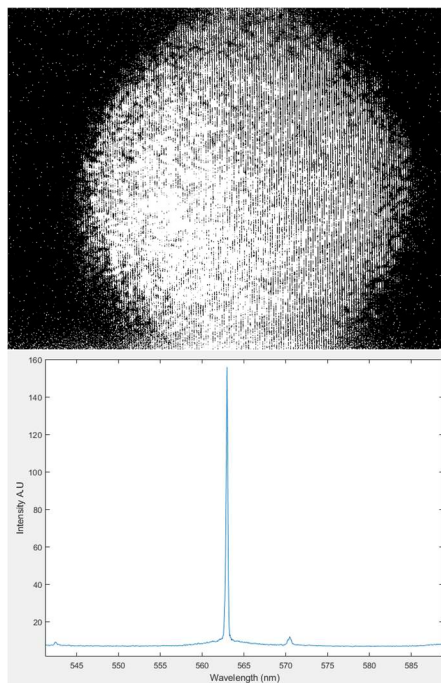


Figure 7 - Resulting fringes and Raman spectrum of potassium perchlorate.

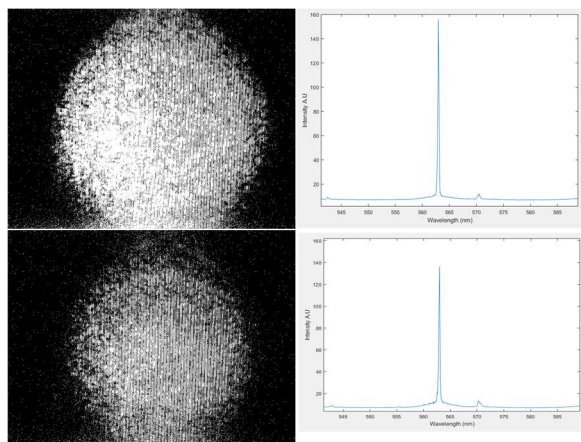


Figure 8 - Top row images potassium perchlorate at higher concentration, bottom row is lower concentration. Resulting peaks show different intensities depending on concentration given the same exposure time.

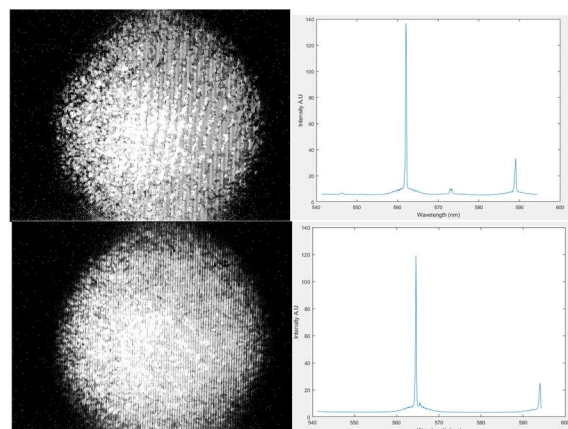


Figure 9 - Top row are higher concentrations of ammonium nitrate dissolved in water. Bottom row are lower concentrations.

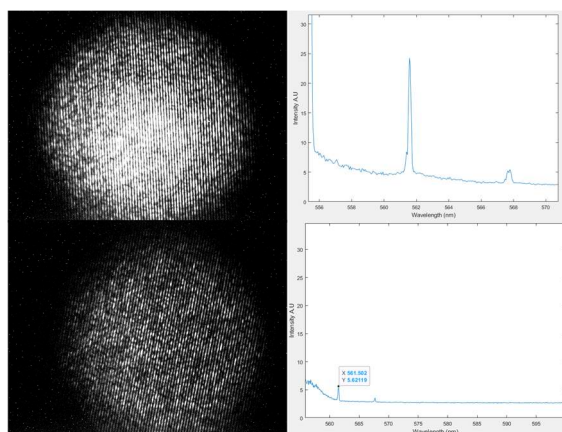
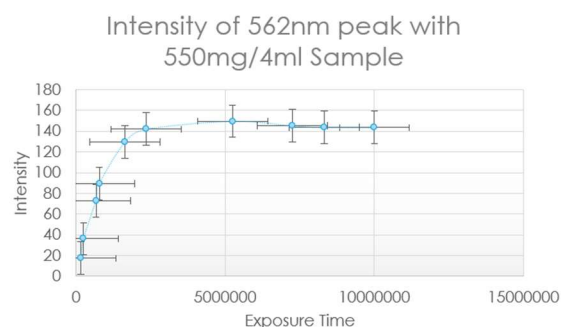


Figure 10 - Top row are higher concentrations of sodium sulfate, bottom row is lower concentration.

In the images of the Raman spectra of the chemicals, we have two sets of images. The top row are images of the fringes from the Raman shifted photons scattered off the chemical interfering with each other in the SHS, and the right image is the Fourier Transform of the fringes, producing the Raman spectrum of the chemical.

You can tell the intensity difference for different concentrations of the chemical dissolved in water based off the intensity of the peaks in the Raman spectrum. The concentration levels used for each chemical were 1750mg/ml and 550mg/ml. Below are tables and plots illustrating the increase in intensity due to increasing concentration level for Potassium Perchlorate.

Concentration Level 2	
Exposure Time (us)	Intensity of 562nm peak
160233	17.3272
251473	36.1045
663573	72.9026
775381	89.304
1639053	129.431
2340773	141.9173
5242008	149.708
7250572	145.3213
8321082	143.6018
10000000	143.502



Concentration Level 1	
Exposure Time (us)	Intensity of 562nm peak
160233	18.3717
251473	42.314
663573	71.8034
775381	91.4056
1639053	132.477
2340773	142.814
5242008	158.23
7250572	156.81
8321082	152.8786
10000000	152.425

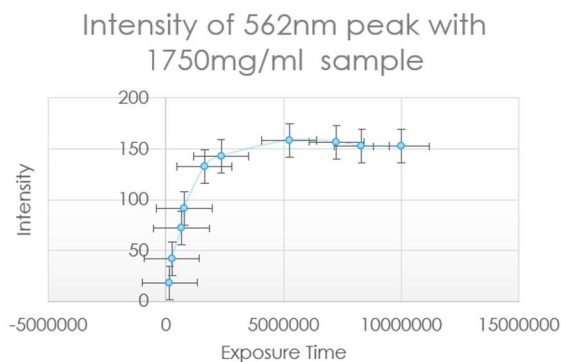


Figure 11 - Table and line graph demonstrating how intensity of fringes and spectrum change as exposure time is increased. The first set of table and line plot is for a KCL04 concentration of 550mg/ml, while the second set is 1750mg/ml.

The below figures illustrate how intensity increases over increasing concentration levels. Note that the first plot is at an integration time of 5.24 seconds and that the second plot is at 10 seconds.

The intensity saturates at a value between 5-6 seconds and begins to decrease as exposure time increases. This is why the intensity appears larger for both concentration levels at a lower exposure time compared to the plot at a 10 seconds exposure time.

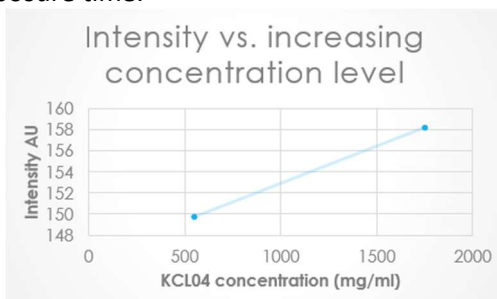


Figure 12 - Intensity versus concentration level of KCL04 at an exposure tie of 5.24 seconds.

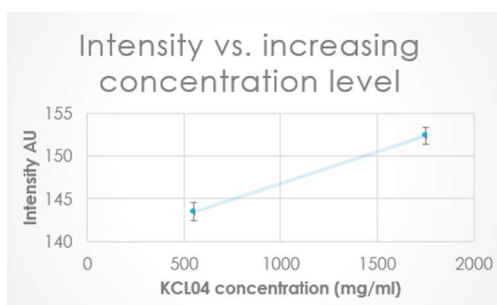


Figure 13 - Intensity vs. concentration level of KCL04 at an exposure time of 10 seconds.

Safety Interlock



Figure 14 - Selected system lock attached to well plate opening.

Our system locks out the user from opening our spectrometer during operation. One of the reasons is because the laser that we are operating is dangerous and can cause injuries to the eyes with even the slightest of exposure. Another reason is that because of the built in interlock on the laser controller, the laser cuts off power when the window is open. This presents a problem; we have many samples that we will be analyzing and opening the enclosure window mid-process leads to interruptions that we want to avoid. These interruptions are also avoided with physically locking the enclosure. Furthermore, having only one system for safety without a backup is not desirable. For these reasons we implemented a safety switch solenoid lock. This will lock the enclosure window during operation and prevent ingress by the user. The model that we decided on was the IDEC HS1L. The primary reasoning for choosing this model is the holding force, with 3000N of holding force it can prevent intrusion by virtually any user. In addition to that, the low current draw during operation and ease of integration made it a good choice. The interlock is also built for industrial purposes and can handle numerous chemicals, some of which may be tested with this spectrometer in the future. The interlock is mounted to the case, and the locking key is mounted to the sliding window. Once the key is in the interlock, the solenoid inside the locking mechanism to be energized, this can be configured in either a normally open or normally closed mode. This simply means that the interlock is either locked when the solenoid is energized, or it is unlocked when the solenoid is

energized. Since we want to conserve energy, we will be unlocking the locking mechanism by energizing it.

A. PCB

The Printed Circuit Board is the main device that not only contains the electrical circuits and devices but also the key integrator of the logic, data, and functioning between many peripherals, and computer with the optical devices. The PCB is comprised of a humidity sensor, a protection latching device, microcontroller, programming connector, fan connector and a PC circuit for data transmission supported by the many power rails from the main PSU that all aid in the proper functioning of the overall system.

Temperature and humidity are critical factors in the performance and accuracy of a spectrometer's measurements for various reasons. These environmental conditions have a direct impact on sample stability, instrument stability, and calibration, which ultimately determine the reliability of the analytical results. Hence it is a concept that should be highly controlled. The reason the HIH6030-021-001 is the sensor of choice is due to the package it comes in as well as pins of the device as they are external and will come in very handy at the time of troubleshooting the PCB. The specifications of the device align perfectly with what we were looking for and the I2C interface protocol to communicate with the microcontroller is supported.

The chip will be programmed through CCS by TI as our microcontroller of choice is the MSP430FR6989. Based on the datasheet, the sensor chip has an I2C address of 0x27 which is sent to commence communication between MCU and sensor. The data that is sent from sensor is 4 bytes. Byte 1 & first half of 2 contain humidity value and second half of byte 2 and byte 3 contain temperature data. Byte 4 is status bit. Raw data is then converted within the code using the datasheet equations for retrieving actual value.

14 bit ADC output for humidity to %RH:

$$\text{Humidity (\%RH)} = \frac{\text{Humidity_14_bit_ADC_output}}{2^{14} - 2} \times 100$$

14 bit ADC output for temperature conversion to °C:

$$\text{Temperature (C°)} = \frac{\text{Temperature_14_bit_ADC_output}}{2^{14} - 2} \times 165 - 40$$

After conversion into decimal, the values are used for manipulating the fan peripheral to deal with the temperature and humidity of the system. A logic circuit is used for this matter using an AND gate connected to a transistor for the purpose of increasing the current of the MCU signal to open/close the transistor for fan usage.

The MSP430FR6989 is a TI microcontroller that is programmed by TI's CCS programming software tool. This MCU was ideal for our system as it contains a variety of GPIO pins that contain protocols that we require such as UART and I2C. One of the biggest issues with having a custom board is the programming of the microcontroller.

To program a microcontroller a specific interface is required which calls for additional components within the board and making sure they are correct as it is a very sensitive concept. The MSP430 is not an exception to this rule, however, there is one simpler alternative which can be bypassed with the use of the development board, MSP430EXPFR6989. The development board is commonly used as a ready-to-go board which has a set of peripherals and a MCU on board ready to be programmed via USB port. However, this board is electrically divided into two sections for a certain reason. For the ability to program other external MSP430FR6989 microcontrollers, the jumper cables connecting the programming interface and the on-board MCU can be disconnected by taking out the jumping cables connecting these two.

The external chip in our custom board requires the SBWTDIO and the SBWTCK from the development board as well as a common ground and power supply between the same. The pins are easily connected through jumper wires into a 4-pin header connector in our custom board going straight into the MCU other than the 47kOhm and 2.2nF pull-up and pull-down resistor and capacitor respectively for the SBWTDIO connection.

Once these pins are properly connected and the development board is USB connected to the PC with CCS programming software the code is ready to be flashed through this process called Spy-by-Wire programming.

To communicate with the PC, we have a USB interface isolated-yet connected to the microcontroller via UART. Information coming and going from and to the PC is shared via USB connection through this interface. The reliability of this circuit was key as USB connections are very sensitive due to them being high speed transfers that require concepts to be implemented such as trace and component placing requirements.

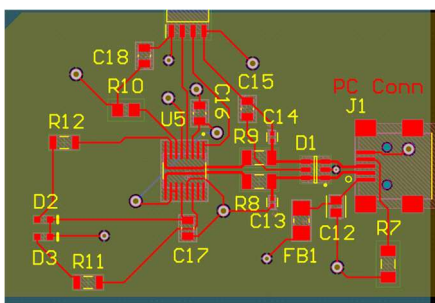


Figure 15 - PCB design layout for temperature and humidity sensing.

The complexity of the USB interface can be seen as a small, isolated part within the custom PCB for the data reliability from the MCU and its peripherals to the external PC.

Supporting the functioning of the PCB we use a main Power Supply Unit (PSU) that provides 24V and up to 5A of which we expect 2-3A to be in use between all the devices within the board with the latch and fan consuming the most current from these.

The PSU is divided into a 12V and a 3.3V rail using two of the same type of buck converters. TI's TPS563300DRLR was the chosen buck converter as it was recommended by the WEBench software and after further review it met all our specs with current being one of the major ones. The supporting passive components and resistor dividers for the desired output voltage rails were tested by the datasheet's

formulas. A physical current loop is also implemented within the custom PCB to keep the impedance reliability as per the manufacturer's specifications.

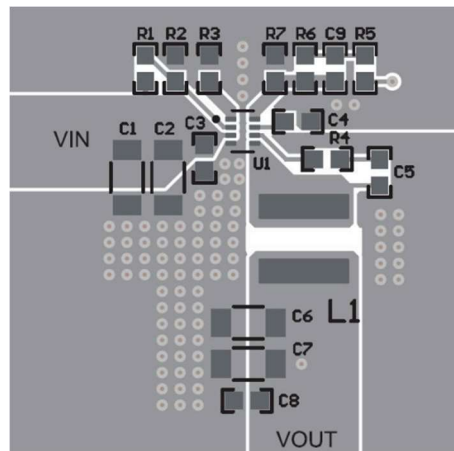


Figure 16 - Section of PCB design.

The USB isolated section of the PCB is being powered through the PC via USB connection. It is denominated as PC5V and PCGND for the common ground coming from the PC into the side of the circuit. This digital power supply is being used as well as the isolation concept from the analog supply on other sections of the board to maintain the data transfer reliability.

IV. DATA ANALYSIS

The data will be collected by the graphical user interface which will have the data from the samples that we are measuring. To determine this, we used different concentration of the contaminants in water. To process the data, software must be run on the user's computer that allows it to interact with the spectrometer. The software will control essentially everything; the laser, the motor that moves the well-plate over the laser, and the spectrometer. The code will also oversee collecting the data, saving the data, translating the data, processing the data, displaying the GUI, displaying the data in the GUI in a way that means something to the user, and having customization options like manually setting the well plate, what communication port the laser, motor, and spectrometer are connected on the computer, etc. To be able to make the software capable of being used by someone

wishing to operate the system we had to create a GUI that was capable of handling multiple inputs. Some other interesting commands that will be used in the GUI is the creation of positioning buttons for the motor. To maintain accurate results, it is sometimes necessary to reset the location of the well plate to ensure that the well-being scanned is the well that we desire. For that reason, we have implemented a “start” and “center” button that will automatically go to the first well in the plate (or the “start”) or the well that is halfway between the “start” and the “end” wells. This start and center will have a default setting, but these values can be edited by the user. The user will also be able to manually move the well plate if they desire a specific well be analyzed.

Once the software has interacted with the system telling it to capture the data from the laser, the data will be captured as an array of images over time, processed using Fourier series calculations, averaged, and displayed as a graph.

```
x = imread('SHS_Test.png').  
F = fftshift(fft2(x))  
imshow(mat2gray(log(1+abs(F))), '  
InitialMagnification',200).
```

The code above takes an image that was generated by the spectrometer reading the data received from shooting the laser into the sample in the cuvette on the well-plate, performs some calculations that include a Fourier's series shift, and then magnified and enhanced to make the image clearer and the spikes of the data more understandable and meaningful to us and the software. The dark image directly below is an example of the image generated by the spectrometer, also called “SHS_Test.png” in the code above. The resulting picture shows a few bright spots in a horizontal line across the center of the page. These points of brightness provide insight as to what compounds are most prevalent in the sample. The brighter the point is in the image, the stronger or taller that peak is, and therefore, means that the substance that is present in the sample and appears at that peak is more prevalent than others.

The calculations, image processing, and the creation of the graph will be automatic to make analysis of the data from the sample significantly easier for the user.

Allowing for the user to quickly determine the wavelengths emerging from the sample and how intense those wavelengths are allowed for the user to be able to quickly determine what compounds/chemicals/substances make up the sample used.

V. CONCLUSION

The purpose of this project is to create an efficient way to find contaminants in water. W.A.R.S has been able to come by a spectrometer prototype, motor, enclosure, various power supplies, and the various cables or other smaller materials needed to complete our system. The largest wait time will be the smaller, more delicate components of our spectrometer as well as the PCB's that first requires, we have all the parts before we can determine whether to build the PCBs ourselves or send them off to be developed by a 3rd-party manufacturer.

The team behind this project has dedicated themselves to creating the best possible machine with the time given. We strive to create a spectrometer that will produce accurate, reliable, and useful results to make the world a healthier place.

ACKNOWLEDGEMENT

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THE ENGINEERS



Julia Augustin-Smith is a graduating senior in Photonics and Optical Engineering. She has plans to move to Dallas, Texas to go into industry and use her skills to move forward in working for a company. After a few years in industry, she plans to get her masters.



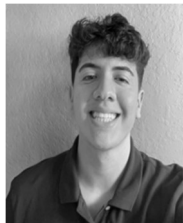
Sebastien Jouhaud is a graduating senior in Computer Engineering. He has plans to complete a master's degree in engineering management to become a more integral part of the leadership team at Lockheed Martin.



Gibran Khalil is graduating with a bachelor's degree in electrical engineering. He hopes to start working in the power and semiconductors industry. He also has plans to pursue a master's degree in electrical engineering. He also has plans to pursue a master's degree in electrical engineering.



George McDonald is graduating with his bachelor's degree in Photonic Science and Engineering. He will be pursuing higher education after spending more time in industry. He thanks Ocean Optics for the fun past 19 months and for the skills, knowledge and experience he's gained with them so far.



Juan Restrepo-Diaz is a graduating senior in Electrical Engineering from Colombia. He will be joining POWER Engineers Inc. shortly after graduation into the power industry as a SCADA/Studies Engineer in Maitland, FL.

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