CCCC and LASER-TEC Educational Raman Spectrometer Demo

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ABSTRACT

Visible and near-infrared spectroscopy is growing at a very fast in many areas, including forestry, medical, agriculture, defense, homeland security and food safety. Raman spectroscopy is also experiencing strong growth, because of its high sensitivity in detection of atmospheric molecules, bomb detection, and early detection of cancer. These growing applications have resulted in increased spectrometer sales, and increased demand for technical talent. This technical talent growth includes a higher demand for photonics/laser technicians in the field of spectroscopy.

To help better prepare graduates of the laser and photonics technology program, Central Carolina Community College, funded by the NSF ATE LASER-TEC grant, developed a module on “Advanced Spectroscopy” for inclusion in the “Laser Applications II” course, to complement the existing “Basic Spectroscopy” module. The “Advanced Spectroscopy” module’s focus is Raman Spectroscopy. Due to the high cost of Raman spectrometers, the module lab did not include a hands-on portion.

Recently, Wasatch Photonics has developed a Raman spectrometer for educational lab use. Students learn from hands-on assembly, alignment and calibration about the internal working of the spectrometer. During the spectrometer lab, students may also use different gratings and light sources.

During this session, LASER-TEC Co-PI Gary Beasley, Central Carolina CC, and Chrys Panayiotou, LASER-TEC PI will demo the spectrometer. A copy of the presentation will be shared with all participants.

Keywords: Raman, Spectroscopy, Spectrometer, Lasers,

1. INTRODUCTION

The Laser and Photonics Technology (LPT) program of Central Carolina Community College (CCCC) is in Lillington, NC, which is in Harnett County, one of the three counties served by CCCC. The laser program was started in 1987 at CCCC**.** It is a two-year, five semester associate degree program. The first-year core classes focus on electronics engineering technology, and the second-year core classes focus on photonics engineering technology.

Though Harnett County is rural, it is located close to Raleigh and the Research Triangle Park (RTP). In addition, CCCC is located relatively close to three major universities, North Carolina State University (NCSU), Duke, and University of North Carolina (UNC), two of which, NCSU and Duke, have engineering programs, including graduate level photonics majors. Several major companies, including Cree, Red Hat and SAS, started in the region. This region is a bed of high-tech industry, which provides placement opportunities for the CCCC LPT graduates. Another technology, which gained popularity in the area was spectroscopy, specifically Raman.

Initially, CCCC LPT graduates were hired by Centice, a spectroscopy start-up, and this interest grew in the area, which eventually led to Wasatch Photonics, originally headquartered in Utah, starting a spectroscopy company in the area, which used Wasatch Photonics diffraction gratings that gave the Wasatch Raman Spectrometer an advantage over other competitor products. Mike Sullivan, CCCC LPT Advisory Committee member, was instrumental in Centice and Wasatch Photonics. Being a very active advisory committee member, Mike helped move the CCCC laser program in the direction to better prepare graduates for the spectroscopy field and started hiring graduates. This has provided a great career opportunity for several CCCC LPT graduates. One problem we encountered in preparing graduates for the Raman spectroscopy industry was the lack of hands-on training students received building and aligning Raman spectrometers.

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With a typical commercial unit, students would only gain experience using the unit, since the commercial units are not made to take apart and experiment with assembling, aligning and calibrating. In addition, there is not an opportunity to try interchanging internal parts to change the configuration.

As Mike continued to review the LPT program, making suggestions for changes for continuous improvement, he came-up with an idea to build an open-system Raman Spectrometer. An open-system would provide a teaching tool for students to gain the hands-on experience of building, aligning, and testing Raman spectrometers. This paper will discuss a new Raman spectrometer under development at Wasatch Photonics for use in education.

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2. LASER-TEC NSF ATE GRANT

Background

The Center for Laser and Fiber Optics Education, LASER-TEC, is a National Science Foundation Advanced Technological Education Center comprised of community and state colleges, universities, high schools and technical centers, trade associations, and laser and fiber optic (LFO) companies.

Headquartered in Fort Pierce, Florida, LASER-TEC started its operation in September 2013 with the goal of developing a sustainable pipeline of qualified laser and fiber optics technicians to meet the industry demand across the southeast region.

To achieve our goal, LASER-TEC is committed to assisting colleges and high schools with creating and offering courses in lasers and fiber optics.

CCCC is a Co-PI in the LASER-TEC grant. The grant funded development of a new “Applications II Course”, which includes a Raman Spectroscopy module. The new course contains modules on photonics applications topics, in areas where graduates are being hired. Two of the new modules cover spectroscopy topics. One is “Basic Spectroscopy”, and the other is “Advanced Spectroscopy”. Each module includes a research/lecture portion, homework and a lab.
The advanced spectroscopy module covers Raman Spectroscopy, which also contains a lab. Up until recently, the lab only covered research topics on Raman Spectroscopy, performed by the students. These spectroscopy modules were added to better CCCC laser program graduates, which several have accepted positions at spectroscopy companies upon graduation. One of the companies is Wasatch Photonics, located in Research Triangle Park, North Carolina. Mike Sullivan, a spectroscopy scientist, who consults for Wasatch, has served on the CCCC laser advisory committee for 15+ years.

3. PROBLEM STATEMENT

Spectroscopy is the science of studying the interaction of electromagnetic energy and matter. One example is the use of light and color to study matter. Numerous, outstanding Internet resources exist for explaining all aspects of spectroscopy. Especially with the rise of spectroscopy companies specializing in spectrometers. Many of these companies, like Ocean Optics, B&W Tek, and Wasatch Photonics have outstanding spectroscopy educational resources on their website. These resources are used to summarize some aspects about the field of spectroscopy, where many CCCC LPT graduates obtain jobs.

Ocean Optics Educational Internet Material – “Spectroscopy Introduction and Applications

Types of Spectroscopy

Spectroscopy is the study of the interaction of light with matter. There are two distinct aspects of this interaction that can be used to learn about atoms and molecules. One is the identification of the specific wavelengths of light that interact with the atoms and molecules. The other is the measurement of the amount of light absorbed or emitted at specific wavelengths. Both determinations require separating a light source into its component wavelengths. Thus, a critical component of any spectroscopic measurement is the breaking up of light into a spectrum showing the interaction of light with the sample at each wavelength. Light interacts with matter in many ways. Two of the most common interactions are light that is absorbed by the atoms and molecules in the sample and light that is emitted after interacting with the atoms and molecules in the sample.

Absorption Spectroscopy

Absorption spectroscopy is the study of light absorbed by molecules. For absorbance measurements, white light is passed through a sample and then through a device (such as a prism) that breaks the light up into its component parts or a spectrum. White light is a mixture of all the wavelengths of visible light. When white light is passed through a sample, under the right conditions, the electrons of the sample absorb some wavelengths of light. This light is absorbed by the electrons so the light coming out of the sample will be missing those wavelengths corresponding to the energy levels of the electrons in the sample. The result is a spectrum with black lines at the wavelengths where the absorbed light would have been if it had not been removed by the sample.

Emission Spectroscopy

Emission spectroscopy is the opposite of absorption spectroscopy. The electrons of the sample are promoted to very high energy levels by any one of a variety of methods (e.g., electric discharge, heat, laser light, etc.). As these electrons return to lower levels, they emit light. By collecting this light and passing it through a prism, the light is separated into a spectrum. In this case, we will see a dark field with colored lines that correspond to the electron transitions resulting in light emission.

Qualitative Spectroscopy

One of the most useful aspects of spectroscopy derives from the fact that the spectrum of a chemical species is unique to that species. Identical atoms and molecules will always have the same spectra. Different species will have different spectra. For this reason, the spectrum of a species can be thought of as a fingerprint for that species. Qualitative spectroscopy is used to identify chemical species by measuring a spectrum and comparing it with spectra for known chemical species to find a match.

As an example, consider the discovery of the element helium. It was first observed, not on the earth, but in the sun! In 1868 the French astronomer Pierre Jules César Janssen was in India to observe a solar eclipse when he detected new lines in the solar spectrum. No element known at that time would produce these lines, so he concluded that the sun contained a new element. This initiated a search for the new element on Earth. By the end of that century, the new element had been identified in uranium ores and was named helium, after the Greek word for the sun (Helios). Today, spectroscopy finds wide application in the identification of chemical species.

Quantitative Spectroscopy

Quantitative spectroscopy is one of the quickest and easiest ways to determine how many atoms or molecules are present in a sample. This is because the interaction of light with matter is a stoichiometric interaction. At any given temperature, the same number of photons will always be absorbed or emitted by the same number of atoms or molecules in a given period of time. This makes spectroscopy one of the few techniques that can provide a direct measure of the number of atoms or molecules present in a sample.

Spectroscopy Instrumentation

A large variety of instruments are used to perform spectroscopy measurements. They differ greatly in the information they provide. What they all have in common is the ability to break light up into its component wavelengths.

Spectroscopes

A spectroscope is the simplest spectroscopic instrument. It functions to take light from any source and disperse it into a spectrum for viewing with the unaided eye. Spectroscopes are useful for determining what wavelengths of light are present in a light source, but they are not very useful for determining the relative amounts of light at different wavelengths. Spectroscopes are most commonly used for qualitative emission spectroscopy.

Spectrometers

A spectrometer is a spectroscope with a meter or detector so it can measure the amount of light (number of photons) at specific wavelengths. It is designed to provide a quantitative measure of the amount of light emitted or absorbed at a particular wavelength. Some spectrometers are constructed so that the wavelength can be varied by the operator and the amount of radiation absorbed or transmitted by the sample determined for each wavelength individually. Others have a fixed light dispersing element (e.g., diffraction grating) that disperses multiple wavelengths of light onto a multi-element detector. Using a spectrometer, it is possible to measure which wavelengths of light are present and in what relative amounts. Spectrometers are common in astronomy where they are used to evaluate light collected by telescopes. They are the only source of information we have about the chemical composition of the universe outside our own solar system.

Spectrophotometers

An instrument that includes a light source is known as a spectrophotometer. It is constructed so that the sample to be studied can be irradiated with light. The wavelength of light incident on the sample can be varied, and the amount of light absorbed or transmitted by the sample determined at each wavelength. From this information, an absorption spectrum for a species can be obtained and used for both qualitative and quantitative determinations. Spectrophotometers measure the amount of light transmitted through a sample. Once the transmission for a sample is measured, it can be converted into other values. Percent transmittance (%T) is the ratio of the transmitted light (I) to the incident light (Io) expressed as a percent."[1]

B&W Tek Educational Internet Material – "Raman Spectroscopy in the Pharmaceutical Industry

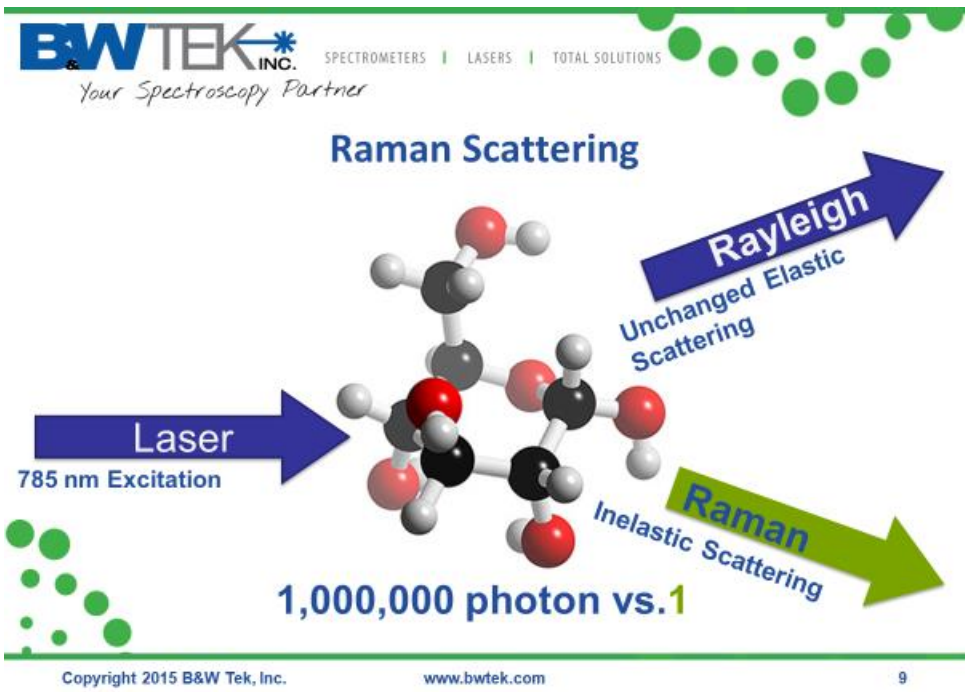

Figure 1 – B&W TEK Raman Scattering Image

Raman spectroscopy is a form of molecular spectroscopy – the scattering of electromagnetic radiation by atoms or molecules. The Raman signal is observed as inelastically scattered light and is an invaluable tool for molecular fingerprinting."[2]

Using a laser source to interact with molecules, two types of scattering may occur. One being Rayleigh, and the other being Raman. The wavelength of the scattered Rayleigh energy is the exact wavelength of the laser energy source. The wavelength of the Raman scattered energy is a different wavelength than the wavelength of the laser energy source, and is in fact a unique wavelength, which is a finger print of the molecule energized by the laser. Now, unfortunately, the amount of Raman energy scattered is extremely small. So, Raman Spectrometer manufacturers had to overcome this obstacle in order to eventually design practical units for the science and industry communities.

“Advantages of Raman Spectroscopy

- Little to no sample preparation required
- Perform analysis directly through transparent containers

  (i.e. plastic bags, glass, etc.)
- Enables both qualitative and quantitative analysis
- Highly selective
- Fast analysis times
- Not sensitive to aqueous absorption bands

Scattering process, not absorption process. Can sample solids, liquids, gases, slurries.

Weak effect (~1 in 10^8 photons are Raman shifted)

- Filtering out Rayleigh (laser) light is critical
- Fluorescence can interfere – especially if have dark samples
- Tradeoff between signal (max w/ bluer laser) and fluorescence (min w/redder laser) is critical decision in many applications

Uses laser light in visible to NIR wavelength region for excitation. Can use glass cells and optics, fiber optics (remote sampling). Allows non-contact sampling: for sterile or hazardous samples; can measure through transparent packaging. Sample size flexible (microns to inches), depending on optics.

Raman Shift is independent of laser output. The excitation wavelength is determined by analytical and sample needs (avoid fluorescence interference with longer wavelength). Raman Intensity depends on laser wavelength. The longer the excitation wavelength the lower Raman peak counts (at same power output). A Raman spectrum is a molecular ‘fingerprint’ that provides structural information. It can identify materials based on the spectrum. Changes in intensity, frequency and peak bandwidth provide valuable information for quantitative and qualitative analysis. A Raman spectrum is a plot of the intensity of Raman scattered radiation as a function of its frequency difference from the incident radiation (usually in units of wavenumbers, cm^{-1}).

Raman has proven to be a promising tool to increase operational capabilities and reduce cost while providing solutions from rapid material identification to process understanding and real-time analysis.”[2]

Raman spectroscopy is used in many areas, and even the same discipline, or area, may have unique and different applications. Here is a list of some examples of areas where it is used.

- Pharmaceutical Industry
  - Correct Prescription Refills
  - Trace Elements
- Homeland Security
  - Poisonous Gases
  - Bomb Detection
- Manufacturing Industry
  - Quality Control
  - Sorting
- Food Industry
  - Contamination
  - Ripeness
- Biomedical Industry
  - Vital Signs Monitoring
  - Early Disease Detection
  - Glucose Monitoring
  - Imaging
  - Dental
- Law Enforcement
  - Drug Identification

Examples of Raman Spectroscopy Applications

"Hyperspectral Imaging - Products for Hyperspectral Imagers include Raman, Fluorescence, IR, Near-IR, and Visible systems. The ability to measure spectra at each point in an image allows detailed molecular analysis of a variety of inhomogeneous samples or scenes. The information obtained through the proposed state-of-the-art spectral imaging instrumentation helps Wasatch customers get the analysis and conclusions they need to make a real difference whether it's in the lab, in factories, or on the front lines. These applications are wide-ranging and far-reaching in their ability to produce valuable information in markets such as:

PATHOLOGY - A hyperspectral image of tissue using computational science can determine disease more accurately for doctor's diagnosis compared to staining methods using visual observation.

PHARMACEUTICAL - Hyperspectral images can reveal vital information that can help improve product quality, cost-effectiveness, and time savings for drug research, development and formulation.

FORENSICS - Hyperspectral imaging technology provides law enforcement with increased discrimination and visualization capabilities that won't destroy the evidence.

STAND-OFF THREAT DETECTION - Hyperspectral imaging is now used for hazardous material detection for government, airport security, and military agencies."[3]

Raman Spectroscopy Application Internet Image

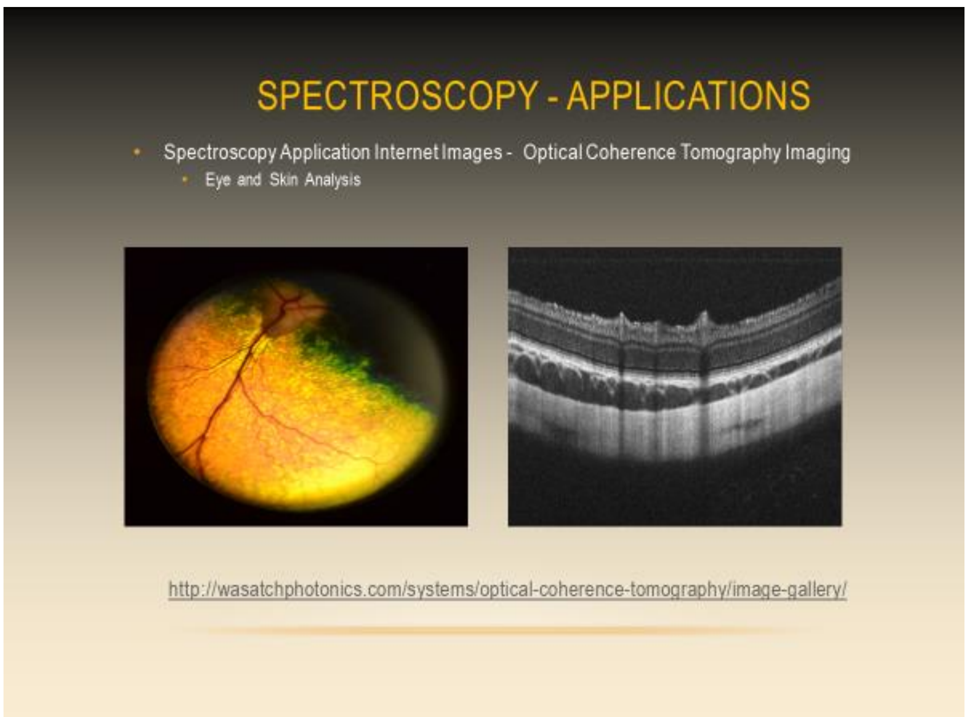

Figure 2 – Spectroscopy Internet Application Image

The Raman spectrometers designed and built by Wasatch are closed systems, with only inputs and outputs, which do not offer teaching opportunities in educational settings, of what makes-up a Raman Spectrometer, or how to build and align one. Because of the low emissions of Raman scattering and delicate operations, commercial Raman spectrometers are closed systems, with a made-to-order configuration. Even if a laser program, such as the CCCC LPT program purchases a Raman spectrometer for using in a lab, students would gain limited experience using it. They would basically just learn how to set-it up and run a sample. They would be preparing to be end user operators, versus learning how to develop, manufacture and test one. Therefore, using a commercial closed system, basically black boxes, with only inputs and outputs, without a hands-on understanding of what exactly makes-up the spectrometer, students are unable to gain hands-on experience building and aligning a spectrometer. This hands-on understanding is invaluable to students pursuing a career in spectroscopy, which is growing, and in need of such talent.

Unless a student interns at Wasatch during his CCCC laser program educational journey, they would not gain the necessary experience to hit the ground running at Wasatch, or other spectroscopy companies hiring graduates, like B&W Tek.

4. SOLUTION - THE WASATCH EDUCATIONAL RAMAN SPECTROMMETER

Mike Sullivan, spectroscopy expert, who now consults for Wasatch, has been a very active member of the CCCC laser program advisory committee for 16+ years. Working with Wasatch, Mike has been the catalyst for several of the laser program graduates to be employees for Wasatch. One of those previous graduates, Joseph Price, is one of the earlier hires. To better prepare our graduates for the main companies hiring our students, like Wasatch, during a previous advisory committee meeting, Mike, and other advisors recommended the CCCC laser program consider implementing modules of curriculum focus in the discipline areas of these companies, like spectroscopy with Wasatch. The LASER-TEC NSF ATE Grant funded the creation of the modules, which makes-up a Photonics Applications II course, which CCCC laser students take during their second year of curriculum study.

Mike was instrumental in coming-up with an idea for a design of an open frame educational Raman Spectrometer, which students could see the internal parts, interchange parts, and align parts of the working system. This will allow students to gain a better understanding of how a Raman Spectrometer works.

During review of the new course modules, Mike's review of the Advanced Spectroscopy module, included an offer to help with development of a Raman Spectrometer lab. This led to the design of the Wasatch Educational Raman Spectrometer.

The new Wasatch Educational Raman Spectrometer is an open-box approach, where faculty and students have access to the internal parts of the spectrometer, including alignment accessibility. Some of the parts may also be changed out with other types of parts, like the grating, filters, and source.

The new Wasatch spectrometer is presently in the prototype stage. When released, this product will be a big step-forward for spectroscopy labs in educational laser programs.

Mike, and another Wasatch employee, Joseph Price, who is a previous CCCC laser graduate, came to CCCC and demonstrated a prototype of the new educational Raman spectrometer to the CCCC laser students at a SPIE student chapter meeting. The following is material of the new spectrometer presented by Mike Sullivan and Joseph Price at a CCCC laser program SPIE student chapter meeting.[4]

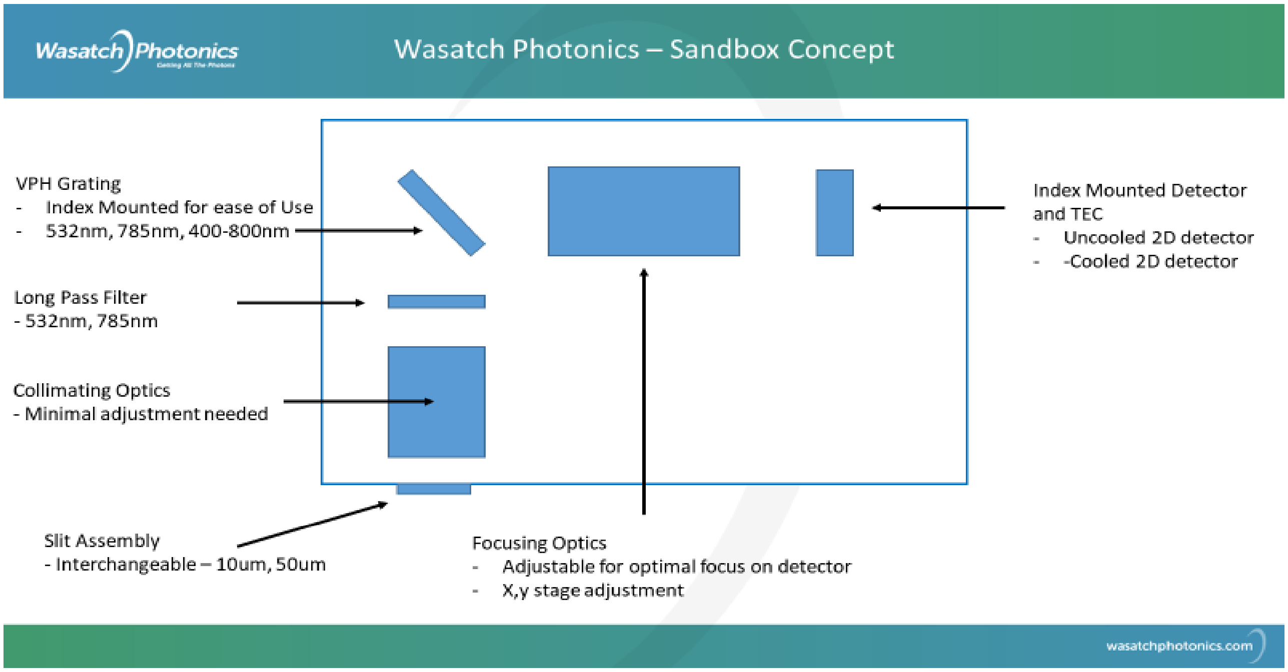

Figure 3 – Block Diagram of the Internal Components, with some having multiple options

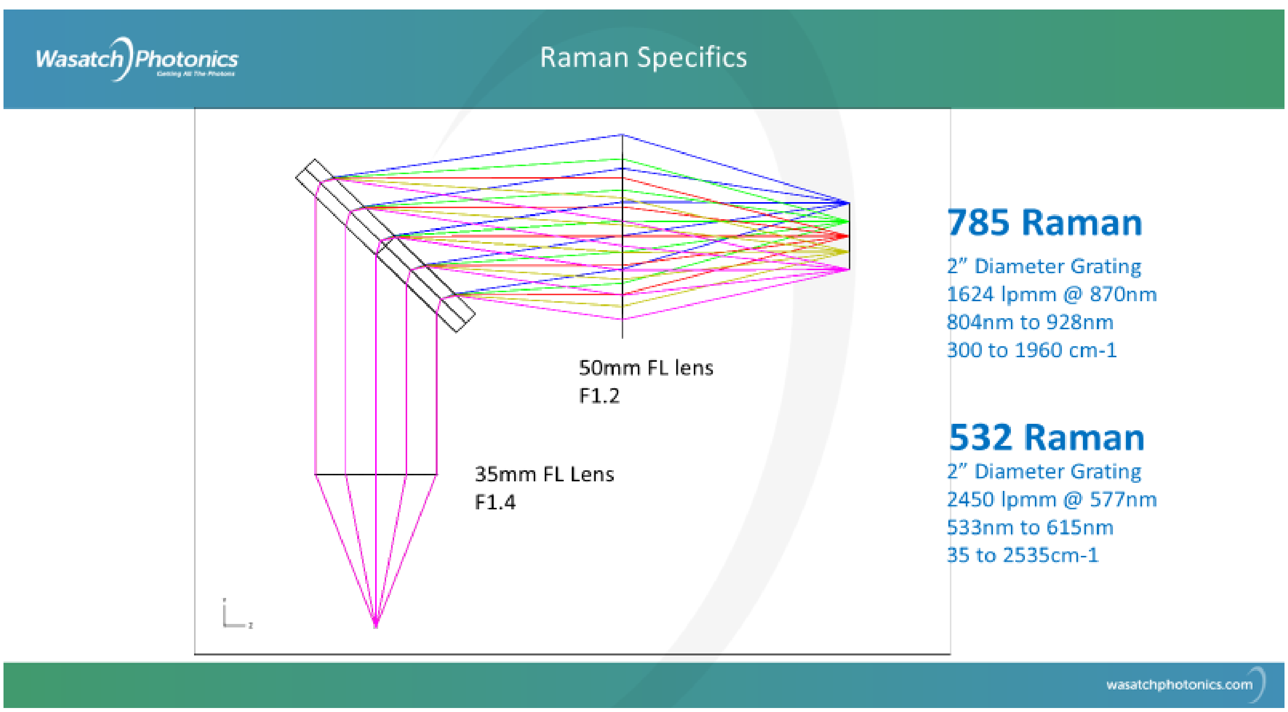

Figure 4 –Diagram of two different Raman sources with different gratings

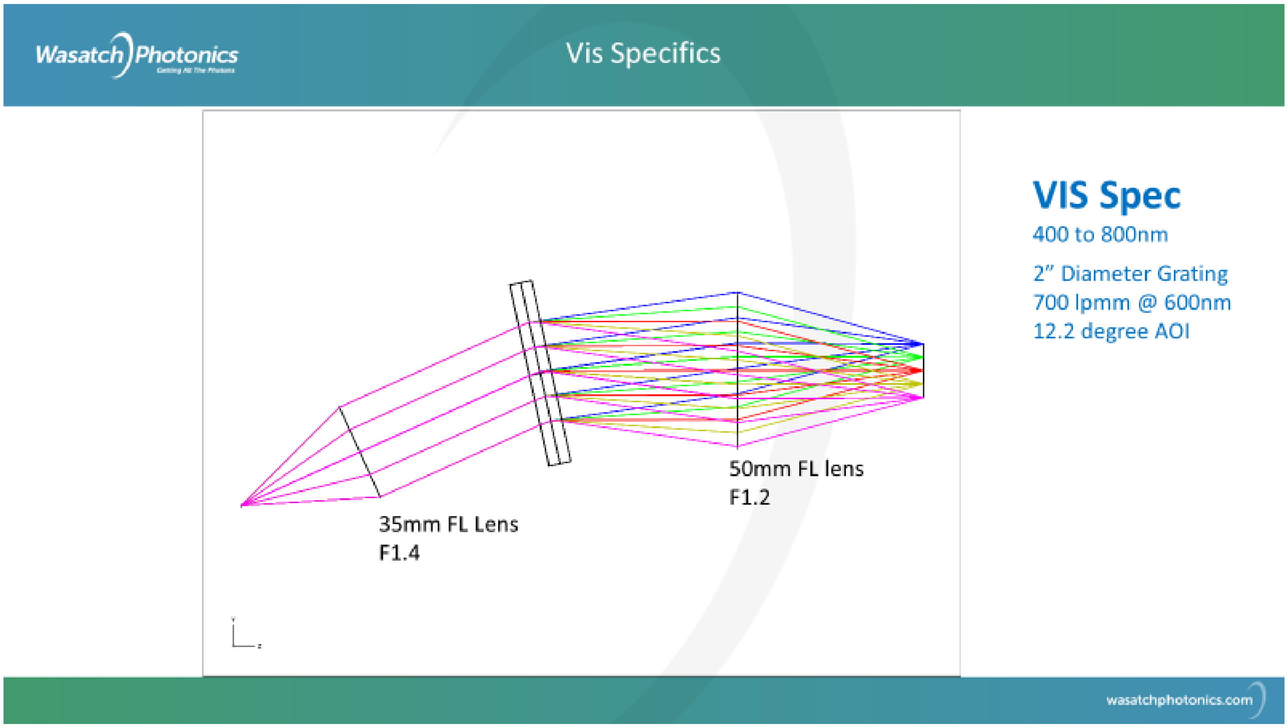

Figure 5 – Diagram of different Visible sources

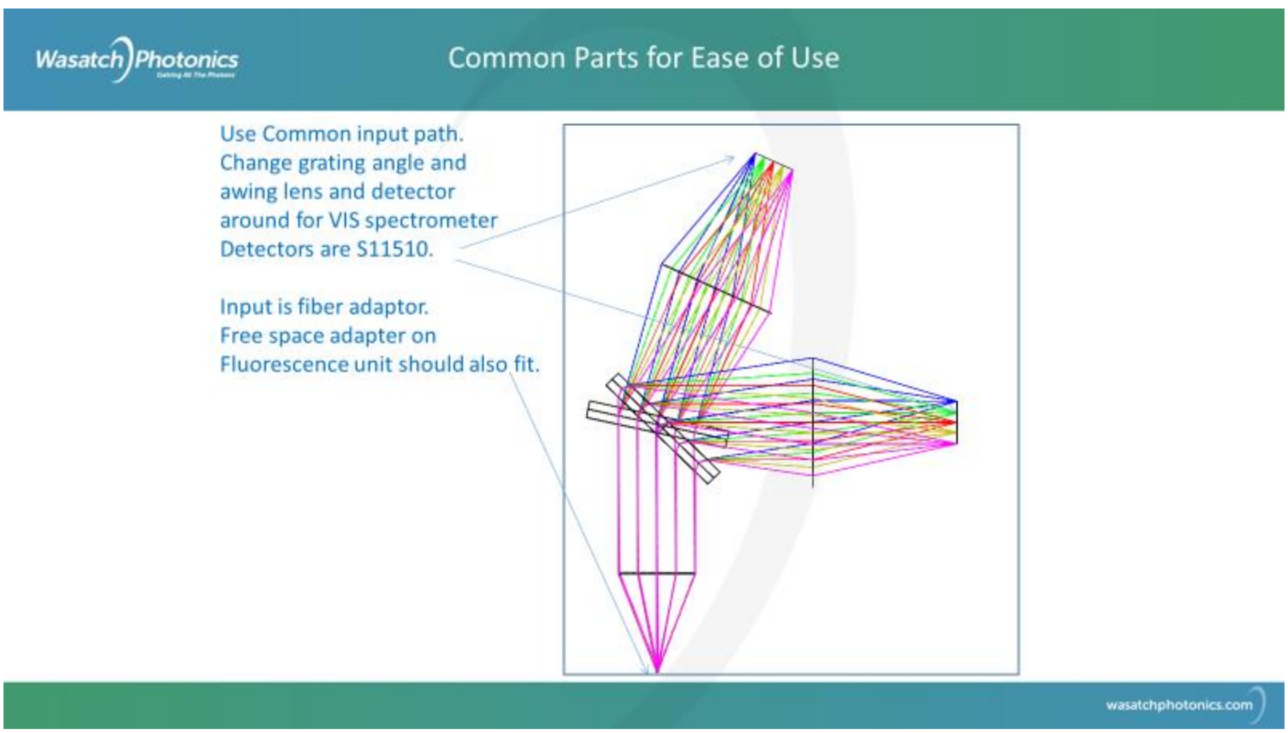

Figure 6 –Diagram of some common parts

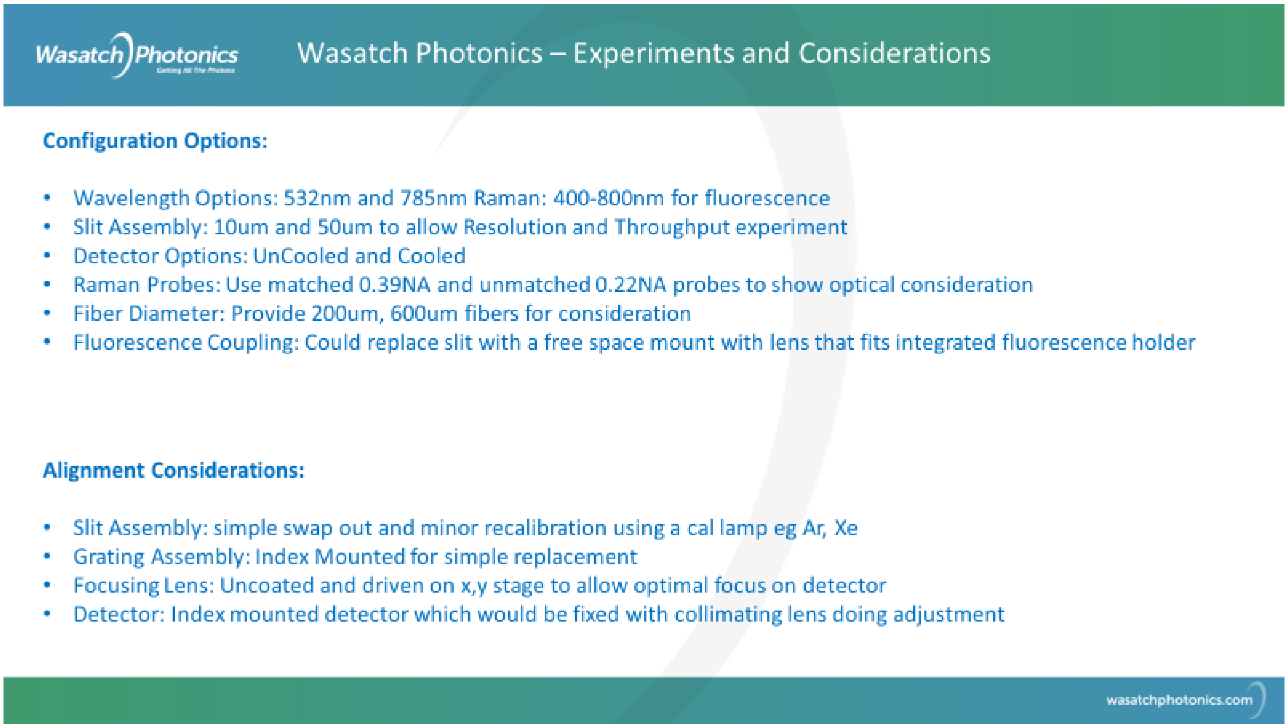

Figure 7 – Experiment Considerations

Figure 8 – Outside Enclosure of Spectrometer

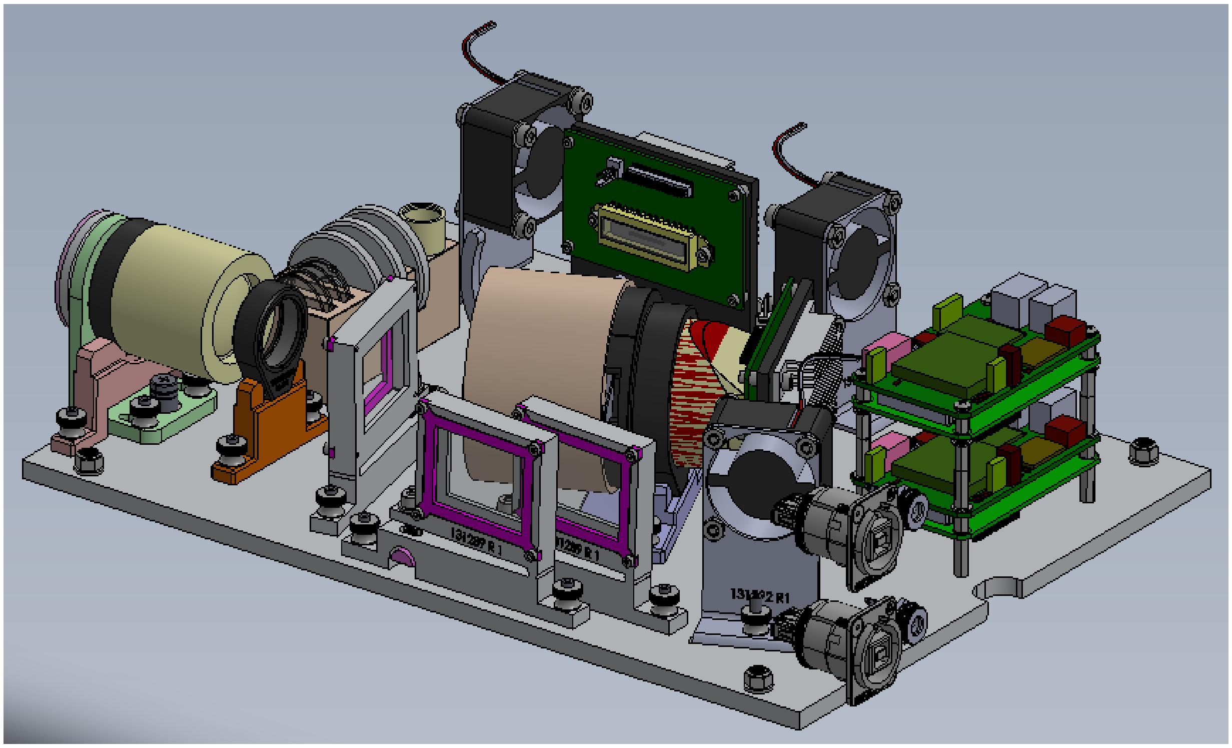

Figure 9 – Sand Box Side View of Raman Spectrometer

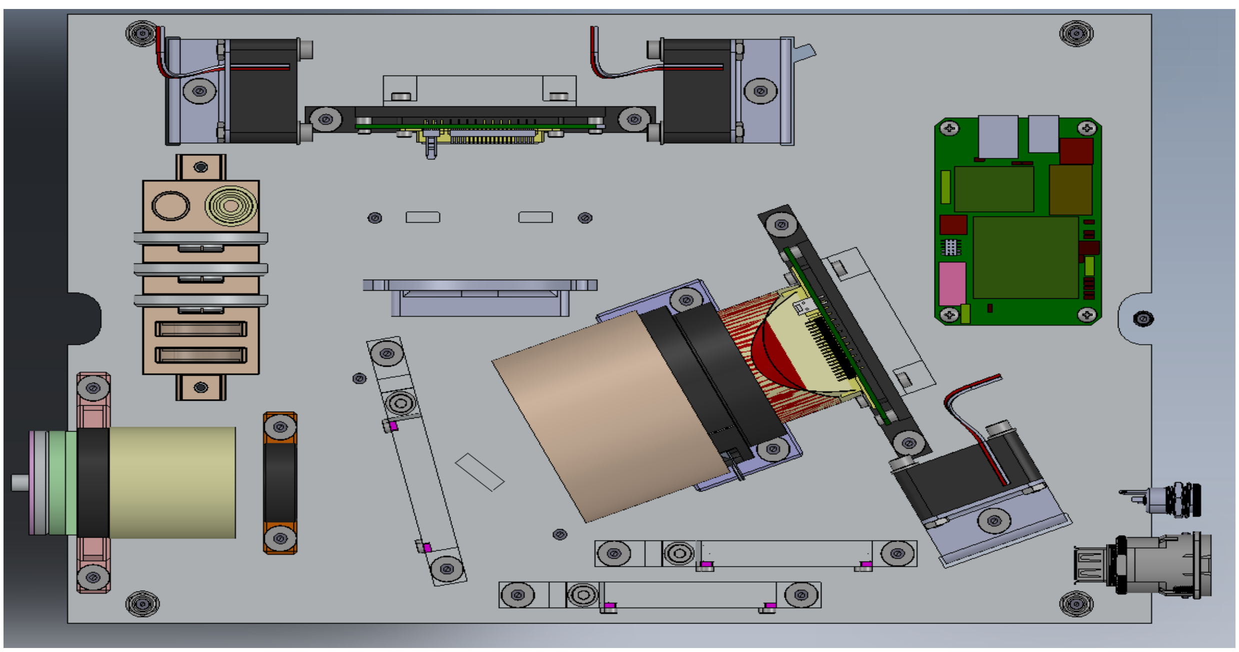

Figure 10 – Sand Box Top View of Raman Spectrometer

5. CONCLUSION

The Wasatch Educational Raman Spectrometer will provide students, studying spectroscopy hands-on experience they would be unable to obtain using closed “black-box” systems. Students will be able to go inside the unit to see how the major components of a Raman spectrometer are positioned and aligned. The educational unit is also being designed for multiple configurations. Options include different slits, detectors, gratings, Raman probes, fiber diameters, and sources. With these different options, several labs would also provide students with a better understanding in different Raman spectroscopy applications.

This new Raman Spectrometer educational unit will provide opportunity for gaining invaluable hands-on experience to students pursuing a career in this field.

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