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Spectroscopy Lab Assistant

Drew Robinson
Western Washington University

COLLEGE OF THE ENVIRONMENT

WESTERN WASHINGTON UNIVERSITY

MAKE WAVES.

Internship Title: Spectroscopy lab Assistant

Student Name: Drew Robinson

Internship Dates: 1/2/2022 - 3/16/2022

STUDENT SIGNATURE [signature]

DATE: 3/16/2022

Characterization of Nanoparticles with Raman Spectroscopy and spICP-MS

Internship goals

I started this internship with the goal of applying the knowledge I've learned in the College of the Environment classes to a project that has real world effects. Through this quarter of running different spectroscopy machines, scheduling meetings, and talking to professors about different projects, I've learned unexpected skills that will be valuable for my continued pursuit of knowledge.

Raman spectroscopy

Raman spectroscopy is a technique that allows for chemical characterization of various materials. On a microscope stage, the sample is set in place and irradiated via a laser. Much of the light produced by the laser is scattered off the sample, unchanged by the sample that it hit. This phenomenon is known as Rayleigh scattering. A small percentage of scattered light is changed because of the chemical makeup of the sample and is reflected at a different wavelength than the laser. This is called Raman scattering and is the key to analyzing what the sample is made of at a molecular level. To filter out Rayleigh light scattering, a filter is applied to the reflected light. The Raman light that makes it through the filter is passed through a prism to partition out the different wavelengths, and then those wavelengths are recorded and analyzed.

The Raman microscope here at Western Washington University can utilize 4 different laser light wavelengths. Different lasers may be used depending sample properties and intended analysis. For the samples we analyzed, an Nd:YAG 532nm laser was used. Samples included epoxy with varying levels of graphene, nylon fishing line, and selenium nanoparticles. One of the most common difficulties with using this machine is making sure that the light analyzed is light that reflected off the sample, and not light that reflected off the glass slide or cover slip. This is one reason that using a microscope to center and adjust the laser is so critical. For epoxies, plastics, and materials with more homogenous chemical makeups, this machine excels. It becomes increasingly difficult to center and analyze the chemical makeup of materials with distinct pockets of different chemicals. For example, analyzing selenium nanoparticles proved to be complex because most of the sample was dried out tween 20, the stabilizing agent used in the production of the Se nanoparticles, while the nanoparticles were too small to analyze. We were unable to record results that picked up the nanoparticles using the 50X microscope lens. For samples with low concentrations of distinct chemical precipitants, a higher-powered lens is much more effective in achieving accurate results. On epoxies and nylon, Raman spectroscopy delivered valuable information. We were able to measure the chemical makeup of not only the surface level components, but also how the materials change with depth. This is an incredibly useful feature for analyzing the degradation of materials.

For my future research in grad school, this could be used for projects like determining the rate of decay of plastics by modified primary decomposers, or characterizing what chemicals are present in decayed wood from saprotrophic fungi. When thinking about future research questions to pursue in grad school, I find it incredibly valuable to have a working knowledge of what tools I could use for analyzing materials. Throughout this quarter, I've gained working knowledge on the uses of Raman spectroscopy, and I can use this knowledge to determine its usefulness in answering the questions I want to ask.

Single Particle ICP-MS

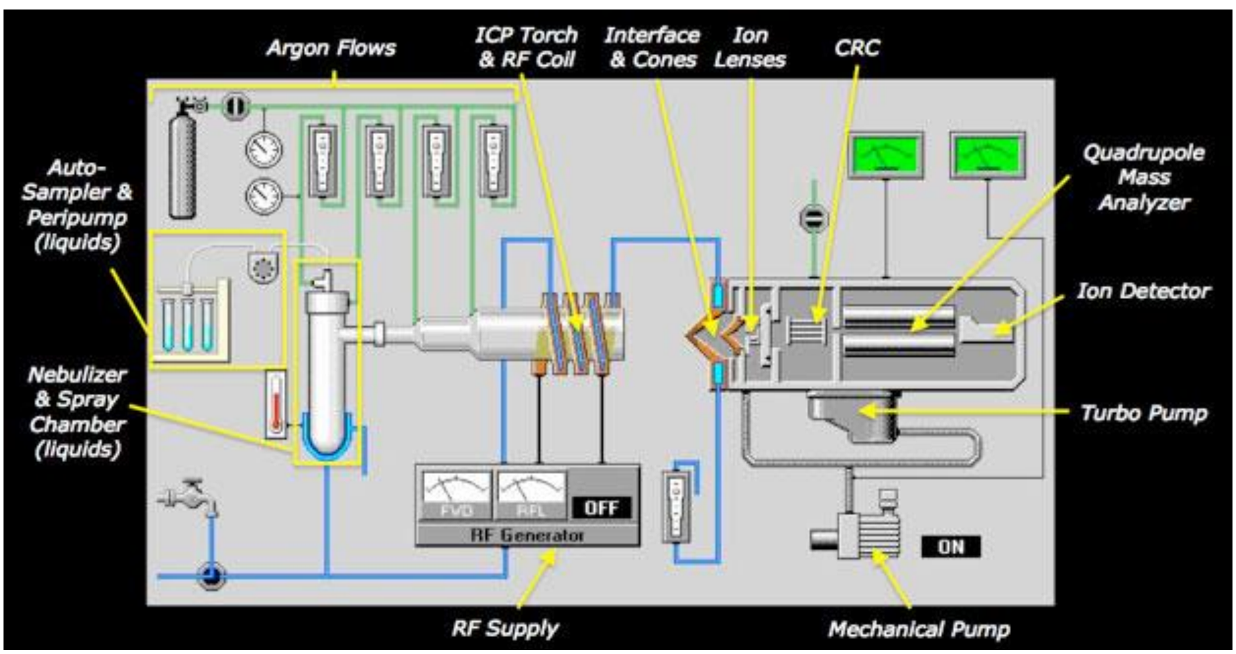

Figure 1. Schematic of an Agilent 7500ce ICP-MS (Jackson School of Geosciences).

Inductively coupled plasma-mass spectrometry (ICP-MS) is a complex machine capable of measuring a variety of different things, from the mass of individual nanoparticles to the elements in aquatic systems and solid rock minerals. Single particle ICP-MS (spICP-MS) works by taking a sample solution that is extremely diluted and pumping the solution from the autosampler into a chamber that nebulizes the liquid by mixing it with argon gas. Some of the aerosolized solution makes it past the spray chamber and is rapidly desolvated in the argon plasma, reducing the solution into the individual ions. The ion beam then passes through two interface cones and is accelerated through the ion lens. This part of the machine is under vacuum to increase the mean free path of the ion beam, allowing it to travel unimpeded to the detector. The ions pass through an omega lens, meant to filter out uncharged atomic and molecular species. Next, the ions pass through the collision reaction cell. This step removes larger polyatomic ions from the sample filtering them through a of collision helium. Large particles are more likely to be hit by the helium gas molecules, which slows them down and prevents them from passing into the next filtering step. The quadrupole mass analyzer is the next step in the mass spectrometer. It separates the ions based on the atomic mass to charge ratio. This is the step that targets the specific elements needed to be measured. For example, the element selenium can have an atomic mass between 74 a.m.u. and 82 a.m.u. depending on the isotope. The most common isotope is selenium-80, but the quadrupole is unable to filter out just the selenium because di-argon ions (Ar_2) will also be present and have a molecular mass of 80 a.m.u. In our analysis, we used the quadrupole mass analyzer to select an atomic mass of 78 a.m.u., because it is the most abundant isotope of selenium capable of being analyzed without interference, accounting for 24 % of naturally occurring selenium. After the specific ions are filtered, they are picked up by the ion detector (Shehu, 2019).

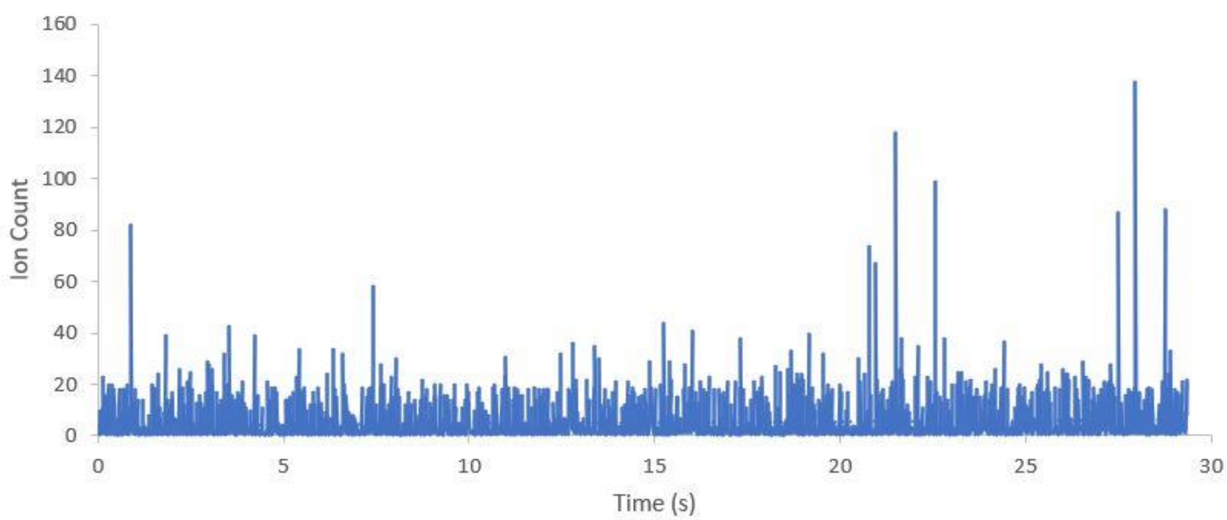

Figure 2. This is an example of raw data output by the ICP-MS in single particle mode. This graph shows how many ions hit the detector in a thousandth of a second. The peaks are individual nanorods. The largest of the peaks are likely to be two nanorods hitting the detector at the same time, and the smaller more abundant ion counts are background noise that made it through all of the filtering steps. This graph shows that while the data is usable, it would be helpful to dilute the sample solutions a bit more.

The ion detector will detect ions that passes through the filter, however background noise in the samples may be present due to the high voltage electronics contributing to an elevated baseline of signal (Figure 2). One of the main limitations of this tool is what elements it can measure. There are 34 elements that the machine is unable to measure because they are present in high amounts in the air or are a noble gas (Brusca,). ICP-MS can be a very valuable tool. It is always important to know the constraints of the tools used in experiments to achieve accurate data.

My time throughout this quarter was spent using the ICP-MS in single particle mode to find the size of selenium nanoparticles and gold nanorods coated with silicon. This was an amazing learning opportunity because my time in the College of the Environment has been spent almost entirely online. I haven't been able to spend much time in a lab and doing mock procedures on a computer program is different than using lab equipment in person. For example, this was my first opportunity actually using a micropipette. Even though they are very simple to use, it takes practice to achieve the precision that is required for lab work like serial dilutions. I was able to test out my accuracy when running a gold calibration curve (Figure 3). During each sample run, it is important to calibrate the machine to get a precise assessment of the elements being analyzed. The gold calibration curve is conducted by taking gold nanoparticles of known size and making acidic solutions with gold concentrations of 0ppb, 1ppb, 5ppb, 10ppb and 50ppb. To achieve an accurate calibration, the solutions need to be as accurate as possible. With my new skill of using a micropipette to perform a serial dilution, I was able to achieve a calibration curve with an R^2 value of 0.9995. Small things like this are essential for grad school in any program dealing with microbiology. The knowledge of how a single particle ICP-MS application could be used is possibly the most useful part of this internship. Measuring trace elements in individual cells has a broad range of applications. Being able to run an ICP-MS will be a valuable tool in any research dealing with bioremediation or biomedical fields.

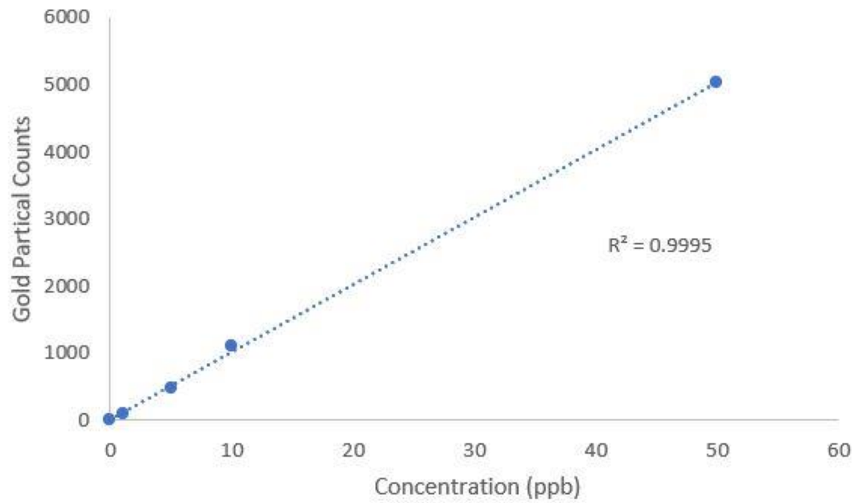

Figure 3. This graph shows a gold calibration curve of standardized gold nanoparticles ran through a single particle ICP-MS.

Collaborative Science and Communication

While it was extremely beneficial to be able to say that I feel able to run these machines enough to run experiments using them, the most impactful aspect of this internship is not something that you can put on a resume. Science communication is an important and often understated part of the process. Most projects especially within the realm of environmental science need collaboration in order to conduct accurate and informative experiments. During this winter while working on projects with Dr. Montaño, I also had the opportunity to meet and help two chemistry professors as well as a microbiology professor and lab manager. With effective communication, projects can be done much quicker because everyone works on the part of the project in their area of expertise thus experiments can be multidisciplinary. This is not without challenges though, the more interdisciplinary the work is the more possibility for individual problems to arise. There are always hiccups in experiments, and great researchers are the ones that can come up with creative solutions.

I plan on going to grad school after finding a lab that is performing research that I find interesting and fits within my moral bounds. If I find myself enjoying the research enough, I will pursue a doctorate. I really enjoy teaching and have dreams of becoming a professor. During my time running ICP-MS and Raman spectroscopy, I was able to learn more about the differences between a research school and a teaching school. There are opportunities and drawbacks to both, and it was helpful to hear an insight on the difference in public vs private grants, ratios of teaching vs research, and how to increase my chances of even getting into programs that would allow me to continue learning in an academic setting.

Conclusion

This internship has given me a chance to look at what it means to do research in a professional setting. I had the opportunity to gain hands on experience in analytical chemistry where, because of the pandemic, my education was lacking. Though this quarter was not what I was expecting, I think it proved to be more valuable in the end. I was able to explore and ask questions about what I want out of a career and am thankful for the opportunity.

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