GRO Summer Internship Final Report

Waste Indicator Liquid-liquid Extraction Method Validation

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My GRO internship was in EPA's Region 8 Laboratory, Golden, CO, where I worked on methods validation for a specific laboratory method. My project advisor was Mark Burkhardt, and I was mentored by Vincente Marti and David Nguyun. My project aimed to create a standard operating procedure (SOP) that would be used to analyze samples received by the Agency for various analytes that are indicative of waste in water samples. Method validation is essential and necessary for the creation of an SOP; therefore, my work was vital to the creation of an SOP for this extraction method.

The purpose of the project was to validate the performance of an onsite developed liquid-liquid extraction (LLE) at several different concentrations and sample volumes. Subsequently, the sample extracts were analyzed via gas-chromatography mass spectrometry, the data were inputted and processed and the method detection limit (MDL) was calculated. This was repeated several times. After a careful evaluation of several parameters, we were able to identify the three major parameters which play a major role in extraction efficiency.

These parameters were extraction temperature, concentration volume, and blow-down methods. These conclusions were made after careful evaluation of the drastic drop of extraction efficiency when an LLE was run at a lower temperature, allowed to concentrate too far, or was blown down at a rapid rate. After isolating each individual parameter we were able to verify the vital role it played in the entire extraction process, so we standardized the parameter conditions at values at which desired extraction efficiencies were observed.

From the results obtained we were able to validate the LLE extraction at sample volumes 1000 mL and 250 mL. We also studied the extraction time and its effect on the efficiency and investigated whether comparable results are obtainable at a shorter extraction time. The current extraction is run overnight, thus a shorter extraction time would ensure high throughput analysis and allow the laboratory capable of handling a larger influx of samples for analysis.

One of the primary challenges of the internship was learning how to properly run an LLE extraction, as there are several initial setup errors that can severely affect the extraction efficiency. This was a challenge that I was able to overcome with both practice and more careful observation of the extractor setup. Another challenge was to become independent of my project advisor(s). As they became busy with their other work and analyses, I had to learn to pace myself, problem solve on my own and follow a schedule without my advisors' constant supervision. I was able to overcome this by setting up a weekly goal for both the reactions I wanted to run and time to process data and look at results. I eventually came up with a balance for both laboratory time and data processing and analyzing the results from those reactions.

I learned how to set up LLEs and how to properly concentrate and transfer into 1mL vials. However, one of the most valuable skills I learned was how to deal with GC-MS problems. During the internship my group experienced great problems with all of the GC-MS instrumentation. Through observation I learned how to alter the parameters of GC-MS in order to achieve better results when faced with sample extracts with unexpectedly low or high concentrations. I used my prior knowledge of soxhlet extractions and micro-extractions to help understand the mechanism behind the LLE procedure. I used my prior research and findings to help isolate the primary parameters that may be a major influence on the extraction efficiency of the LLE.

My impression of the EPA has shifted dramatically; the staff clearly demonstrates their dedication to their science and the environment. I have an even greater respect for the laboratory director and the staff. Despite the various logistical problems, the staff, both at the analyst and director level, show tremendous teamwork. The teamwork and sense of camaraderie throughout the chemistry staff was admirable and I hope to bring that back to my research group at my university. I have a greater respect for the implementation of the Clean Water Act and other legislation, as I saw first-hand the amount of projects each laboratory processes for numerous analytes of interest.

I learned that there are certain skill sets acquired in the lab that you cannot get from working outside of the industry. I have also learned to appreciate the skills I had prior to the EPA internship as they allowed me to better understand the chemistry I did at the EPA laboratory. I have now firmly decided to pursue a career in academia and would like to continue my environmental work in the research field and study the effect and behavior of chemical species in diverse matrices. I enjoyed the internship as I was able to see the bureaucratic and scientific aspect of the EPA.

I would advise GRO fellows to take their time, pace themselves, and listen while doing their internships with EPA, as the staff are extremely knowledgeable and can offer wise advice and information on their past experiences in diverse fields, in addition to their knowledge of chemical and biological systems.