STUDENT SUMMER INTERNSHIP TECHNICAL REPORT

In-Situ Data Collection, Sampling, and Water Quality Monitoring in Tims Branch Watershed, Savannah River Site, SC

DOE-FIU SCIENCE & TECHNOLOGY
WORKFORCE DEVELOPMENT PROGRAM

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ABSTRACT

Tims Branch is a stream that flows through the U.S. Department of Energy's Savannah River Site (SRS) in Aiken, South Carolina, into Upper Three Runs and then ultimately into the Savannah River. Aqueous discharges from nuclear fuel and target fabrication during the Cold War included heavy metals and radionuclides, notably depleted uranium and nickel. This wastewater entered Tims Branch via the A-014 outfall tributary which originates from the SRS M Area. Tims Branch has also received treated groundwater from an air stripping process since 1985 through this outfall tributary. The air stripping process was upgraded in 2007 to remove mercury using tin (II) chloride – the tin reduces inorganic mercury to elemental mercury allowing removal by air stripping. The mercury concentration in the water discharged to Tims Branch was lowered from approximately 250 ng/L (ppt) to approximately 10 ppt. Oxidized tin (Sn) from the treatment is released to the stream ecosystem. Since 2007, researchers from DOE National Laboratories and universities have monitored the stream to help assure that the release of oxidized Sn does not adversely impact the ecosystem.

This internship aimed for collection of water samples for heavy metals analysis and for biofilm sample collection for Sn concentration analysis. Water samples were collected and analyzed using an ICP-MS, following similar methods from a 2016 sampling trip. Biofilm samples were collected and analyzed by a XRF, following similar methods from a 2011 study. Results of the water samples show expected results. Heavy metals that are associated with the suspended particles in the water column accumulated over the past 60 years in an area known as Steed Pond. Sn concentrations in the biofilm indicate no accumulation is occurring. An interesting research topic could be to understand where the Sn is fractioning too.

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1. INTRODUCTION

The Savannah River Site (SRS) is a U.S. Department of Energy (DOE) nuclear complex facility. SRS is a 310-square-mile area found in western South Carolina, bordering Georgia along the Savannah River. Since operations began in the 1950s, SRS's main goal was to manufacture the elements and materials needed for expansion of the United States nuclear arsenal as well as environmental stewardship (Kilgo, 2005). Over the years, contaminants from nuclear processing activities have entered the SRS environment. Applied research for environmental cleanup and remediation has been of the highest concern.

The Savannah River National Laboratory (SRNL) and the Savannah River Ecology Laboratory (SREL) have both been critical for environmental cleanup efforts of the SRS. The overall research of this report focuses on the Tims Branch, which originates on the SRS, flows into Upper Three Runs, and then ultimately into the Savannah River. Cleanup is a priority because the Savannah River is the major drinking water supply for 1.5 million people between Georgia and South Carolina. This report will cover two main aspects of the summer internship research: the accumulation of inorganic tin in biofilm samples compared to a research study done in 2011 by Amaury Betancourt (a previous DOE Fellow) and water sampling for data to be used in solute transport models.

Tin (Sn) accumulation is of concern because of its unknown distribution and effects on the surrounding environment (Looney et al., 2012). Groundwater from the A/M area of the SRS has been treated by an air stripper, the M-1 air stripper, since 1985 to remove chlorinated solvents and then discharged the effluent through the A-014 outfall. During the period from 1985 until 2007, low concentration mercury associated with the contaminated groundwater were released to the A-014 Outfall and a tributary of Tims Branch. In 2007, tin (II) chloride (stannous chloride) was added to the treatment process to chemically reduce the mercury into an elemental form for removal in the air stripper and to lower the amounts of mercury entering the stream. The tin (II) chloride treatment worked to lower the mercury to acceptable levels, approximately 10 ppt, but discharged Sn into the A-014 tributary and possibly to Tims Branch (Looney et al., 2010).

In 2011, Betancourt conducted a study to research the fate and distribution of Sn in the A-014/ Tims Branch ecosystem. The study considered several different sites along the A-014 tributary and Tims Branch. Three types of samples were taken at different sites for Sn analysis: water, sediment, and biofilm. This report will address the concentration of Sn found in the biofilm samples analyzed by Betancourt as a comparative analysis of Sn accumulation between 2011 and 2017.

Discharge of heavy metals from the A/M area of SRS has resulted in approximately 44,000 kg of depleted uranium (U) being released into the Tims Branch watershed during the main operations of SRS. Most of the U accumulation in the sediments is in an area called Steed Pond; an area where a wooden dam built by a farmer allowed the water to accumulate and, therefore, the U accumulated on the sediments in the area. In 1984, the dam broke, leaving the U contaminated sediments exposed.

The U contaminated sediments are, for the most part, inert. Mobilization of this radioactive material is not of much concern during base flow of Tims Branch. Mobilization concerns arise when storm events occur. It is approximated that about 1500% to 2800% more U is mobilized from storm events than during base flow (Batson et al., 1996). For this reason, water quality data is needed for the creation of a hydrological model to estimate the mass flux of U mobilization after storm events.

2. EXECUTIVE SUMMARY

This research work has been supported by the DOE-FIU Science & Technology Workforce Initiative, an innovative program developed by the U.S. Department of Energy's Environmental Management (DOE-EM) and Florida International University's Applied Research Center (FIU-ARC). During the summer of 2017, a DOE Fellow intern, Ron Hariprashad, spent 12 weeks doing a summer internship at the Savannah River Site under the supervision and guidance of Dr. John C. Seaman from SREL and Dr. Brian B. Looney from SRNL. The intern's project was initiated on May 22, 2017, and continued through August 11, 2017 with the objective of in-situ data collection for the development, calibration, and application of hydrologic and contaminant transport models of the Tims Branch watershed. The student's project consisted of five major components, including:

1. Cross section profiling along the Tims Branch stream
2. Collecting water, sediment and biota samples
3. Field measurement of water quality parameters and flow characteristics
4. Laboratory analysis of water, sediment, and biota
5. Implementing long-term monitoring station/s for flow discharge

3. RESEARCH DESCRIPTION

Field Sampling

Field sampling occurred over three days: June 14, June 15, and July 10, 2017. The first two sampling trips included water samples, water quality measurements, flow measurements, cross-sectional profiling, and biofilm collection. The final sampling day included just biofilm sampling, to replace the first biofilm samples which were stored incorrectly. In total, there were eight sampling sites between the A-014 outfall and the Tims Branch-Upper Three Runs confluence.

Site Selection

Sampling sites were selected along the A-014 tributary and Tims Branch stream based on the ease of accessibility. Specifically, three sites were selected on the A-014 tributary and five were selected on Tims Branch (Table 1).

Table 1. Field Sampling Sites

Location ID	Landmark	Latitude	Longitude	Elevation (m)
Site 1	A-014 outfall	33.28711	-81.69721	112
Site 2	A-014/A-011 confluence	33.29205	-81.70107	88
Site 3	Weir, culvert, riprap	33.32485	-81.71822	104
*Site 4	Wetland area above confluence of A-014 and Tims Branch	33.31727	-81.71508	83
Site 5	TIMS04 (SRR sampling site)	33.34035	-81.7177	76
Site 6	Steed Pond	33.33308	-81.73305	72
Site 7	Old train tracks	33.33175	-81.72164	53
Site 8	Old USGS sampling site	33.33186	-81.72732	-

**Site 4 is upstream of the A-014/Tims Branch confluence and is therefore the control site.*

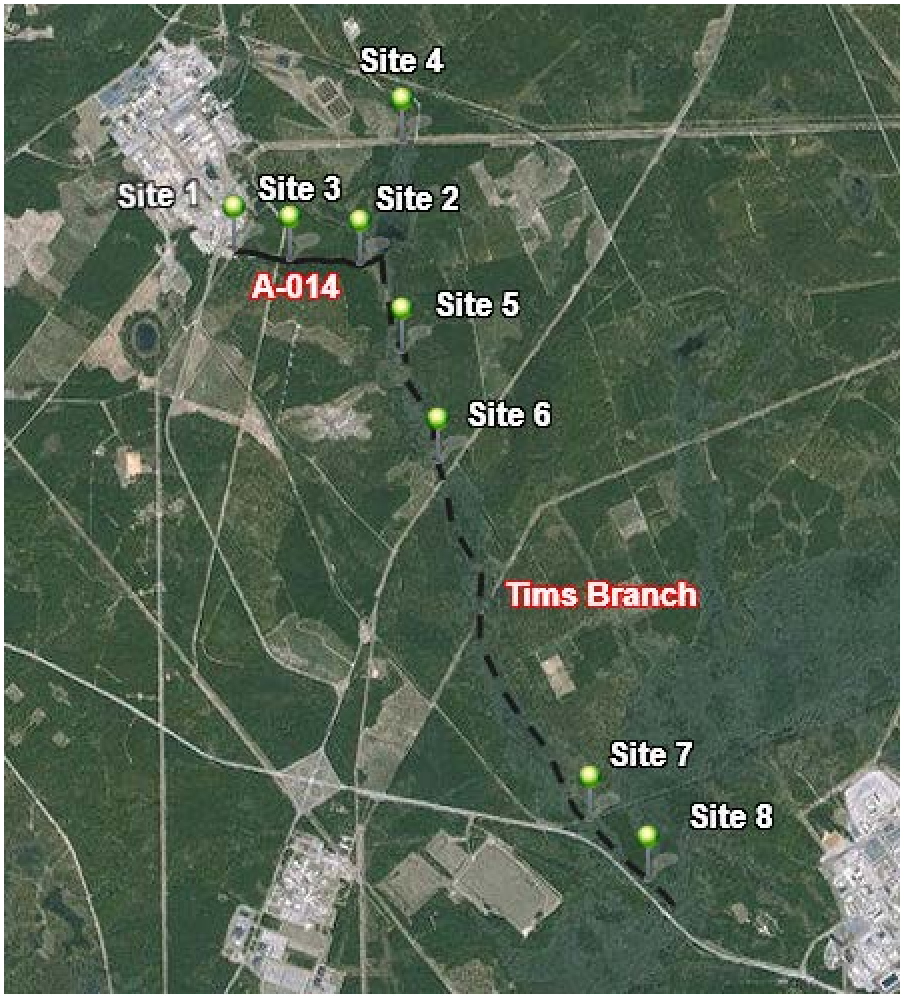


Figure 1. Sampling locations during summer 2017 internship.

Sample Collection

Water samples were taken from each site using 125-mL plastic bottles. A volume of 125 mL was collected so that enough sample was available for both unfiltered and filtered samples. The bottles were rinsed three times with stream water from the respective site at which the sample was taken. Then, a final water grab sample was taken. The bottles were placed in a cooler with ice to keep them from growing algae and other microorganisms until they were surveyed by SRS Radiological Protection and cleared for lab work at SREL.

Biofilm samples were collected by gently cutting off parts of vegetation which included a thin layer of biofilm. Locating biofilm was more successful in areas where a log or other sort of “dam” laid across the stream, allowing the vegetation and their roots to float in the water. Special care was taken to not disturb the biofilm; therefore, scissors were used to cut the vegetation instead of pulling out the whole plant from its roots. Once the desired vegetation was cut, the samples were placed in a plastic bag labeled with the respective site location. These bags were also placed in a cooler with ice but for the opposite reason of the water samples. Biofilm starts dying once they are removed from the water column; the ice was used to slow the mortality rate. Sampling for biofilm was repeated on July 10 because the first sampling trip resulted in the prolonged storage of the biofilm samples. The second sampling trip was successful because the samples were correctly surveyed for radiation and placed in the ovens the same day (specifics on the lab work will be mentioned in the *lab processing* section).

Water quality, flow measurements, and cross-sections were taken at each sampling site in hopes of collecting data which may be used in future hydrological models. Water quality was taken at all sites. Flow measurements were not possible at Sites 4 and 5 because the water depth was too shallow for a reading. Cross-sections were taken at each site except Site 4 because Tims Branch became a wetland in this area. The table below summarizes the samples and/or parameters that were measured at each site.

Table 2. Summary of Site Samples and Data Collection

Location ID	Water Sample	Biofilm Sample	Water Quality	Flow Measurement	Cross-section
Site 1	x	x	x	x	x
Site 2	x	x	x	x	x
Site 3	x	x	x	x	x
*Site 4	x	x			
Site 5	x		x	x	x
Site 6	x	x	x		x
Site 7	x	x	x	x	x
Site 8	x	x	x	x	x

Lab Processing

Water Samples

Each sampling site had 125 mL of water taken and the sample was immediately placed in the ice cooler, as mentioned in the *sample collection* section. The samples were then surveyed by the SRS Radiological Protection team for possible radiation. Once the samples were surveyed, they were returned to SREL the same day. At SREL, the samples were acidified with 2% nitric acid to prevent any further microbial growth. A 10-mL unfiltered sample was taken from each sample bottle and placed into a 10-mL autosampler tube for analysis by a Nexion 300X ICP-MS at SREL. Another 10-mL sample was taken using a 0.22 µm pore size syringe filter and then placed into a 10-mL autosampler tube for ICP-MS analysis as well. This was the filtered sample. The samples were given to SREL Analytical Services for analysis.

Biofilm Samples

Biofilm samples were also surveyed for possible radiation by SRS Radiological Protection and returned to SREL the same day. Once received, the samples were removed from their bags and placed onto trays and put into drying ovens at 60 °C for 24 hours. After drying, the samples were placed back into their respective bags. Biofilm was collected by lightly squishing the bags and allowing the biofilm to flake off the vegetation. The biofilm was then disaggregated using a mortar and pestle. The biofilm was disaggregated until it was a consistent powder and any larger vegetation (if any found its way into the pestle) was removed. The powdered biofilm samples were placed into XFR sample cups to be analyzed by a Niton XL 3t GOLDD+ XRF analyzer at SRNL.

4. RESULTS AND ANALYSIS

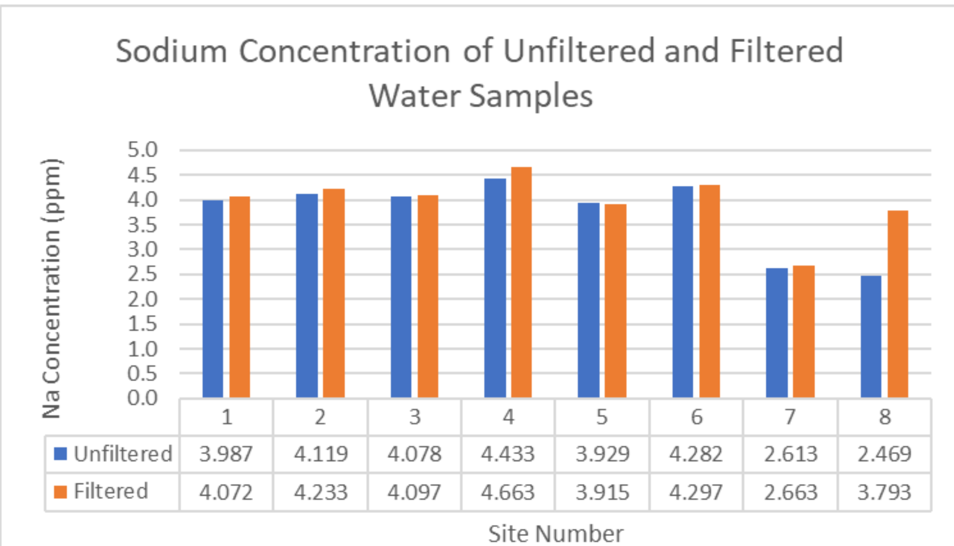
The objective of the field sampling was to collect data for the development of hydrological models and to understand the fate and distribution of Sn. The first section of the results presents an analysis of the water sample data which can be used later for a solute transport model. The second section presents an analysis of the biofilm samples for Sn concentration. Water quality data, cross-sectional measurements, and a description of long term stream monitoring is included in Appendix A.

Water Samples

Results

The water samples were analyzed for a total of 15 elements. Six were macro-elements (Na, Mg, Al, K, Ca, and Fe) and 9 were microelements (Mn, Ni, Cu, Zn, As(1), and As(2), Cd, Pb, and U).

Filtered samples were designated with a sample name ending with an "A". The macro-elements showed an increase in their concentration after being filtered. This is common because the concentration before being sampled was so minuscule that the water sample picked up some of the element from the filter's fibers. As an example, Figure 1 shows an increase in Na for each filtered sample except at Site 5, where the concentration decreased from 3.929 ppm to 3.915 ppm. Because of this, the macro-elements results are only included in Appendix A.



	1	2	3	4	5	6	7	8
Unfiltered	3.987	4.119	4.078	4.433	3.929	4.282	2.613	2.469
Filtered	4.072	4.233	4.097	4.663	3.915	4.297	2.663	3.793

Figure 2. Na concentration increased in the water samples after filtering except for Site 5.

A SRM river water standard was used verify ICP-MS results. The standard's actual concentrations, readings from the ICP-MS, calculated percentage error, and microelement concentrations are all found in Table 3.

Table 3. ICP-MS Reading for River Standard and Site Water Samples

Sample ID	Mn 55 (ppb)	Ni 60 (ppb)	Cu 63 (ppb)	Zn 66 (ppb)	As-2 75 (ppb)	Cd 111 (ppb)	Pb 208 (ppb)	U 238 (ppb)	As-1 75 (ppb)
River SRM Reading	40.228	25.750	89.975	59.137	7.748	4.067	13.222	26.492	7.800
River SRM CAV	40.390	25.320	85.750	55.640	8.075	3.992	12.101	25.350	8.075
Error %	0.4%	1.7%	4.7%	5.9%	4.2%	1.8%	8.5%	4.3%	3.5%
1	5.823	0.408	0.168	3.248	0.029	0.003	0.087	0.029	0.028
1A	5.380	0.457	0.088	8.678	0.036	0.008	0.076	-0.013	0.027
2	2.275	0.414	0.304	4.887	0.117	0.001	0.095	0.067	0.109
2A	1.111	0.280	0.166	10.708	0.063	-0.002	0.092	0.000	0.063
3	2.687	0.541	0.356	5.822	0.178	0.021	0.077	0.043	0.162
3A	2.077	0.388	0.200	11.803	0.061	0.034	0.093	-0.006	0.066
4	51.575	1.315	5.124	13.085	1.451	-0.002	0.725	1.893	1.480
4A	11.916	0.969	3.745	12.184	1.133	0.028	0.347	1.006	1.101
5	24.285	8.648	0.854	5.097	0.413	0.023	0.612	19.959	0.413
5A	6.175	3.988	0.315	0.022	0.228	0.008	0.041	2.369	0.223
6	92.966	19.135	0.887	1.517	1.336	0.031	0.301	9.400	1.410
6A	17.303	15.299	0.567	-0.175	0.714	0.021	0.028	4.342	0.680
7	122.218	5.072	0.220	0.701	1.434	0.004	0.227	2.715	1.464
7A	38.107	3.503	0.145	-1.140	0.653	0.009	0.005	0.937	0.648
8	92.612	4.575	0.251	0.986	1.128	0.004	0.217	2.466	1.153
8A	19.862	3.166	0.230	-0.786	0.640	0.038	0.030	0.908	0.668

Analysis

Concentrations of the unfiltered samples followed a correlation as expected. Following the concentration of U is an excellent example of how the microelements are distributed throughout Tims Branch. Along A-014, U levels are below 0.1 ppb. U, and other heavy metals, do not appear to accumulate on the A-014 sediments because of the steep land gradient and severe erosion in the 1950s and 1960s, which deposited the contaminated sediments downstream to Tims Branch. U levels increase as the sampling sites enter Tims Branch. At the control site, Site 4, 1.893 ppb of U is observed. High levels of U are observed at Sites 5 and 6. Site 5 has 19.9 ppb and Site 6 has 9.4 ppb of U. It is surprising that Site 5 has twice the amount of U as Site 6 because Site 6 is officially known as Steed Pond. One explanation is that Site 5 could have possibly been included in the ponding of water when the farmer’s dam was still standing. Today, Site 5 is a labeled SRR sampling site, known as TIMS04. Downstream, at Sites 7 and 8, U levels drop to 2.72 ppb and 2.47 ppb, respectively. Figure 3 demonstrates the flux of U, starting at A-

014 outfall, to the Steed Pond area, and ending at the Tims Branch/Upper Three Runs confluence. Figure 4 illustrates the flux of U in the water column after filtering the samples.

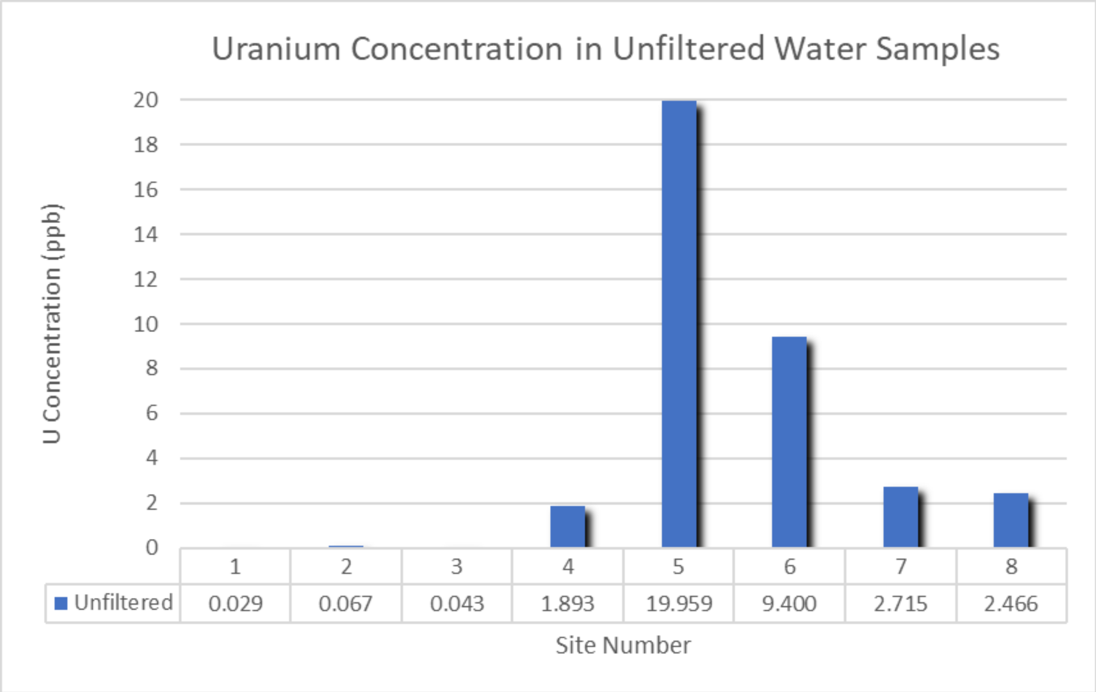


Figure 3. U concentrations in unfiltered water samples at all eight sites.

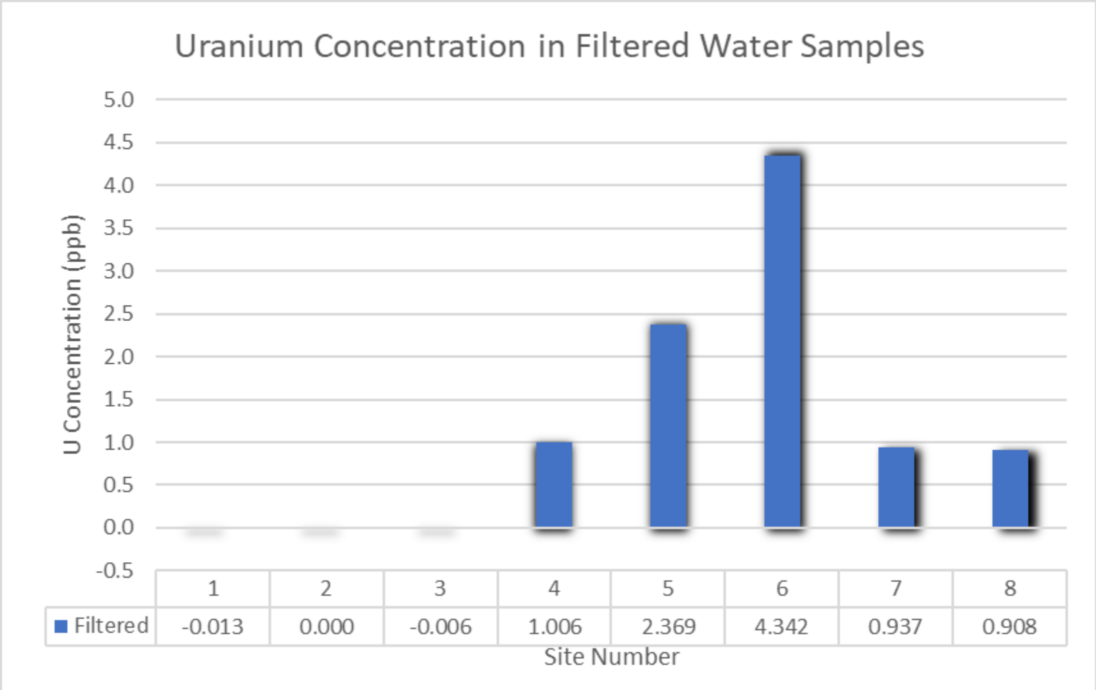


Figure 4: U concentration in filtered water samples at all eight sites

Filtering the water samples provided insight into the association of elements with the suspended particles. Again, following U will provide a good understanding of the microelements along the area of the study. Sites along the A-014 tributary show that essentially all of the U was associated with the suspended particles, as 100% of the U was removed by filtering. Sites 1 and 3 had 0 ppb after filtering because having a negative concentration of an element is not possible. The percentage of U associated with suspended particles then ranged from 47% at Site 4 to 88% at Site 5, as shown in Figure 5.

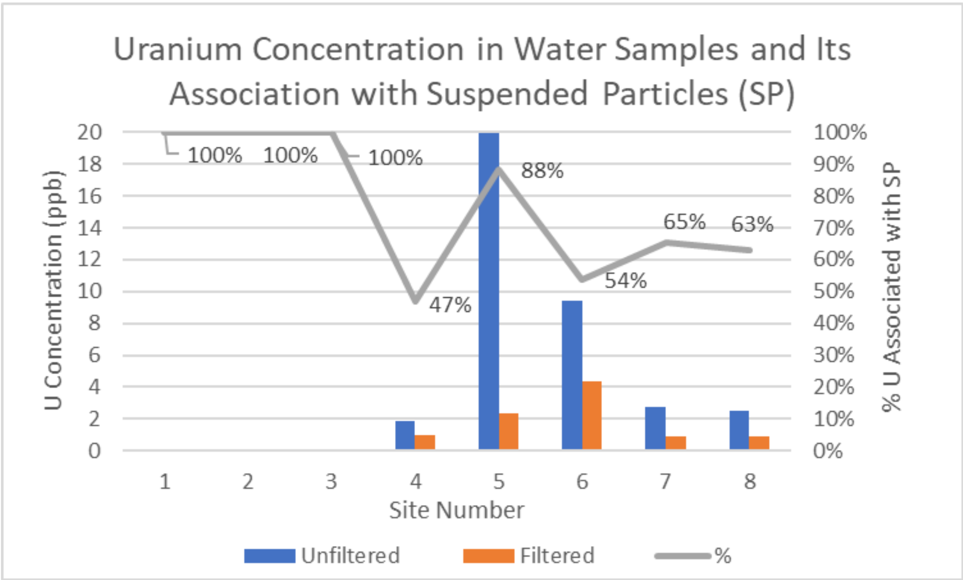


Figure 5. Percentage of U that was associated with suspended particles.

Biofilm Samples

Results

After the biofilm samples were disaggregated and placed in the XRF sample cups, Site 6 did not have enough biofilm to get an accurate reading. Therefore, only Sites 1, 2, 4, 5, 7, and 8 had enough biofilm for analysis.

When running the XRF analysis, the same Sn standards created by Betancourt back in 2011 were used to create a calibration curve. Sn is an extremely inert element, so it was acceptable to use the same standard from six years ago.

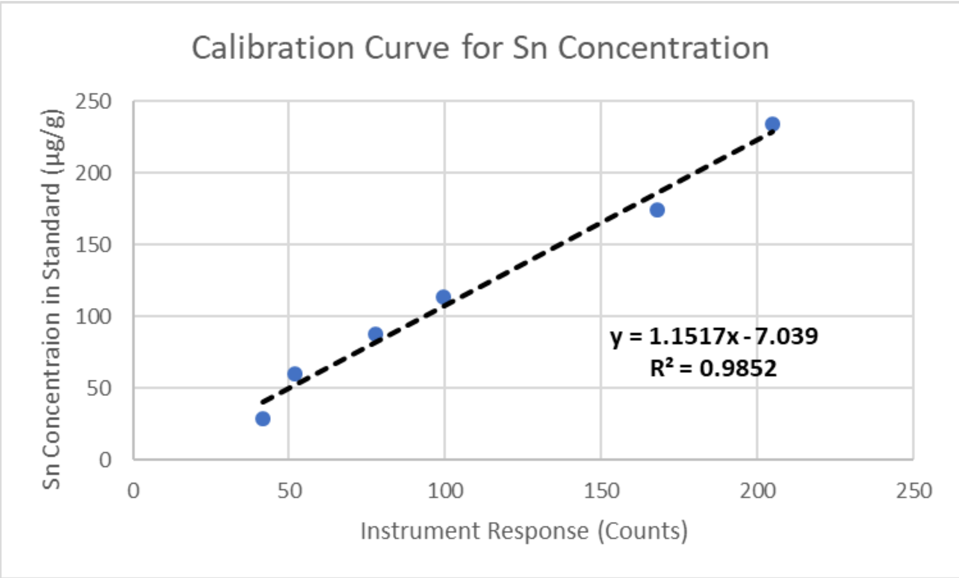


Figure 6. Calibration curve for Sn concentration on XRF.

What is important to take away from the calibration curve is the y-intercept equation. This equation can now be used to find the actual concentration of a field sample by the number of counts that the XRF reports. Counts are the x-value and Sn concentration is the y-value (Table 4).

Table 4. Actual Sn Concentration after using the Calibration Curve for all Six Sites

Site	Instrument Response	Sn Concentration (µg/g)
1	2399.75	2756.753
2	48.57	48.899
4	13.35	8.336
5	0	0
7	0	0
8	0	0

Analysis

To analyze the Sn accumulation data, the sample locations must be coordinated with Betancourt's sample locations from 2011 (Table 5).

Table 5. Comparison of Betancourt's Biofilm Sampling Sites to the Current Sampling Sites

Current Site	Sn Concentration (µg/g)	Distance from A-014 Outfall (ft.)	Betancourt's Site	Sn Concentration (µg/g)	Distance from A-014 Outfall (ft.)
1	2757	5	1	10640	5
2	49	3700	2	9737	20
4*	8		5	2071	750
5	0	6700	6*	0	
7	0	23000	9	585	3700
8	0	25700	13	0	10500

**Control site*

Figure 6 and Figure 7 illustrate the sampling locations of the current internship versus Betancourt's sampling locations. Current site locations are abbreviated to "CS" for "current site" and are in green pins. Betancourt's site locations are abbreviated to "BS" for "Betancourt's site" and are in blue pins.

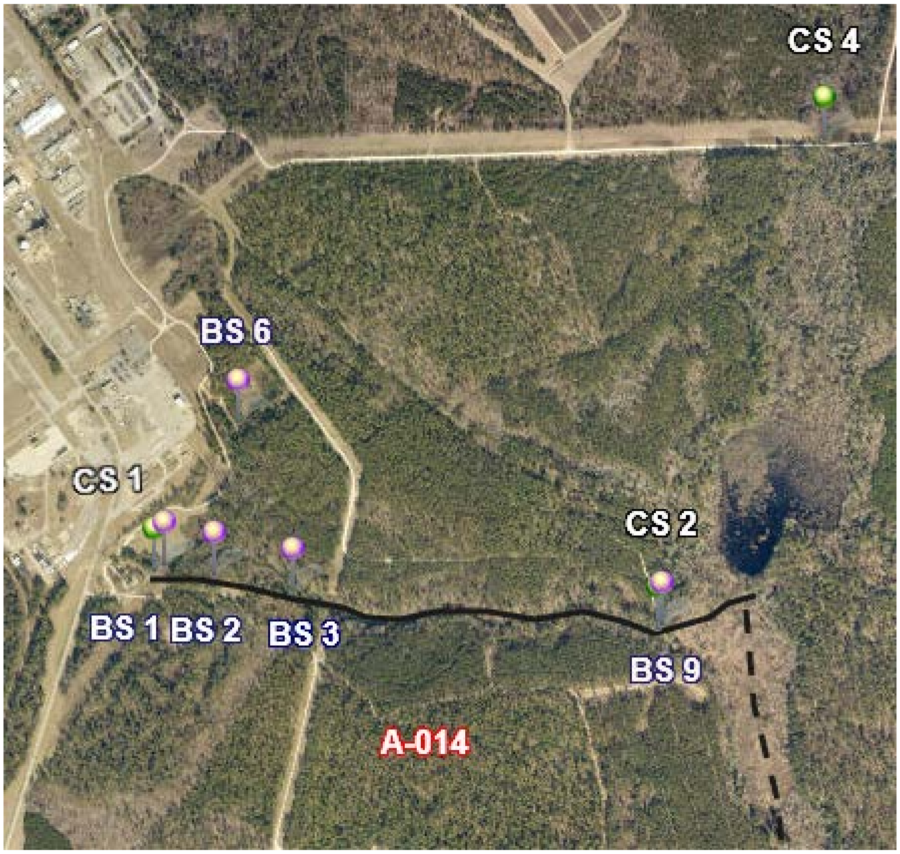


Figure 7. Biofilm sampling locations along A-014 tributary.

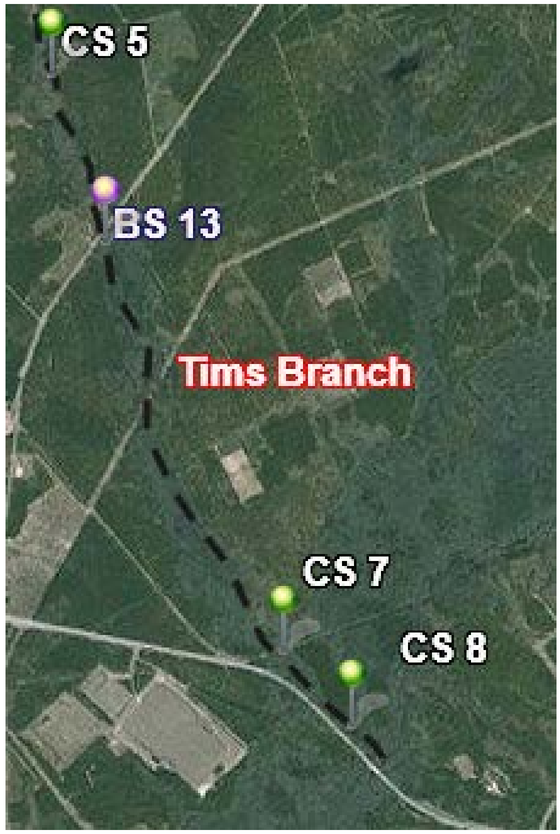


Figure 8. Biofilm sampling locations along Tims Branch.

From the data, the Sn concentrations in the biofilm between 2011 and 2017 have dropped significantly. At the A-014 outfall, Sn decreased 74.1%, from 10,640 ppm to 2,756 ppm. At the A-014/A-011 confluence, Sn decreased 91.6%. Along Tims Branch, there was no detection of Sn. It is important to mention that the XRF did report values of 12 ppm for Betancourt's Sites 6 and 13 but this is effectively zero given the accuracy of the device. The current Sites 5, 7, and 8 were given a reading of "limit of detection" (LOD), effectively zero.

The overall decrease in Sn concentration along A-014 is quite surprising. It was assumed that the Sn would accumulate or have reached a steady-state concentration after six years. One explanation is that there was an increase of rain in 2017. An increase of rainfall would mean that there was a smaller time frame between flushing of the biofilm. More flushing events would not allow the biofilm to accumulate Sn to the levels of Betancourt's samples. It is also interesting that there was no Sn found on the Tims Branch stream, even just downstream of the A-014/Tims Branch confluence. With the assumption that Sn would have reached a steady-state concentration along A-014, the mass flux of Sn should have carried over to Tims Branch, which did not happen.

5. CONCLUSION

Water sampling was successful during this internship. The data results from the samples were interpreted and found to be as expected. Microelements were found in the samples and accumulated at the area where they historically have been found to accumulate, the Steed Pond area on Tims Branch. Filtering the water samples also provided results that were expected; the microelements were highly associated with the suspended particles in the samples. This data will be useful for solute transport models in the future.

Biofilm sampling was also successful during this internship. The results, however, were surprising. Sn concentrations dropped significantly along the A-014 tributary and showed no signs of reaching the Tims Branch stream. Although the results are surprising, this is an excellent opportunity for further research on multiple fronts. For one, this internship sampling project was only the second sampling effort in the A-014/Tims Branch for Sn concentrations and distribution. The first effort was Betancourt’s in 2011. A more in-depth sampling project including water, sediment, and biofilm sampling at more points along the A-014 tributary would validate the question of where the Sn is fractioning. Another front is the ongoing research into nanoparticles and their activity in the environment. The mass flux Sn from the M-1 air stripper is known, as well as the water outflow. This would be an ideal case for further investigation because of the known parameters and the recent data indicating that the Sn is not accumulating into the biofilm.

6. REFERENCES

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APPENDIX A.

Water Quality

Water quality data was collected from seven of the eight sampling sites using a YSI multiparameter meter. No data was collected at Site 4 because the water depth was too shallow to insert the sonde into the water. At each sampling site, the sonde was placed into the stream and three logs were taken; one minute passed between each logging event. The three logs were then averaged; the averaged water quality log readings are summarized below in Table 6.

Table 6. Average of Water Quality Data

Site	Temperature (°C)	Specific Conductance (mS/cm)	Conductance (µS/cm)	Salinity (ppt)	DO_{sat} (%)	DO (mg/L)	pH	pHmV
1	22.18	0.052	48.67	0.0267	0.125	1.0867	7.50	111.93
2	23.59	0.265	68.00	0.0300	0.121	1.0233	7.41	117.50
3	23.28	0.140	135.33	0.0633	0.109	0.9300	6.66	161.65
4	-	-	-	-	-	-	-	-
5	22.53	0.029	28.00	0.0100	0.153	1.3233	5.87	126.53
6	21.78	0.031	29.67	0.0100	0.175	1.5433	5.76	132.63
7	21.80	0.022	21.00	0.0100	0.206	1.8100	6.09	121.70
8	20.97	0.021	19.00	0.0100	0.178	1.5850	5.96	120.90

Cross-Sections

Cross-sectional profiles were taken at four sites along Tims Branch: Site 4, 5, 7, and 8. Cross-sections for Sites 1, 2, 3, and 6 were all taken during a sampling trip last summer by project team members from FIU.

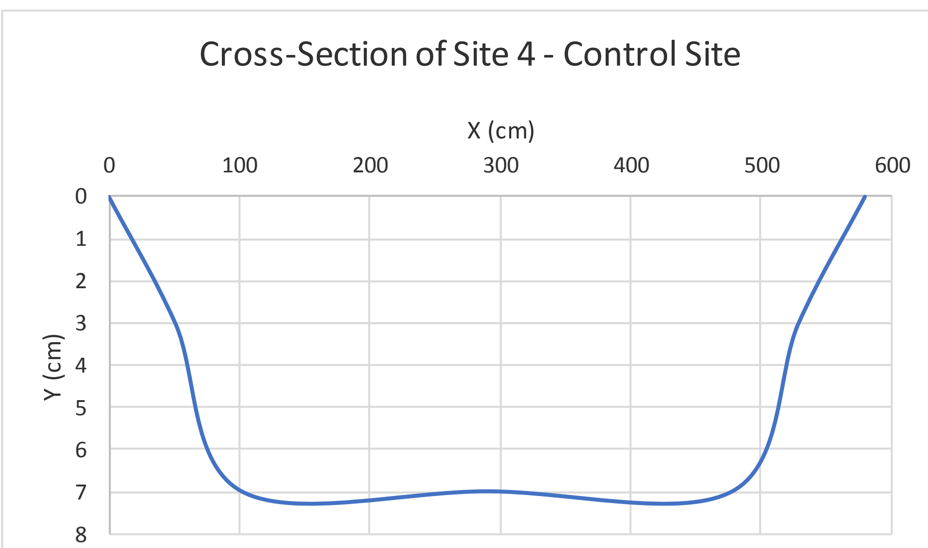

Figure 9. Cross-section of site 4.

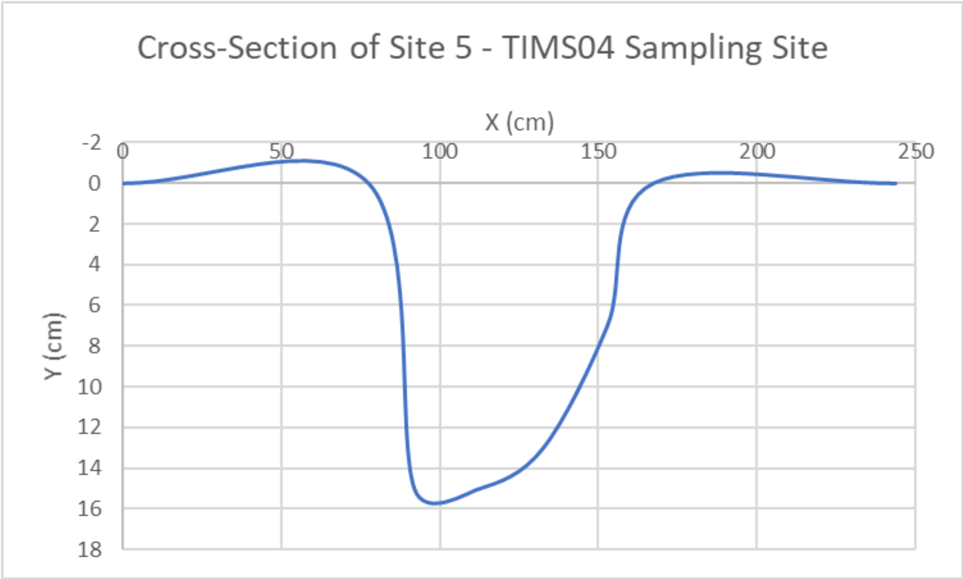

Figure 10. Cross-section of site 5.

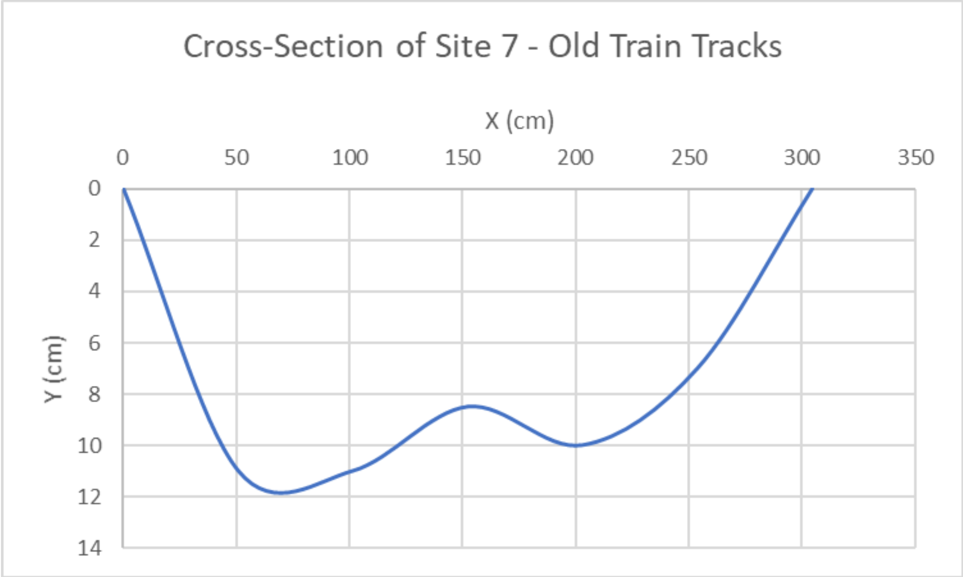

Figure 11. Cross-section of site 7.

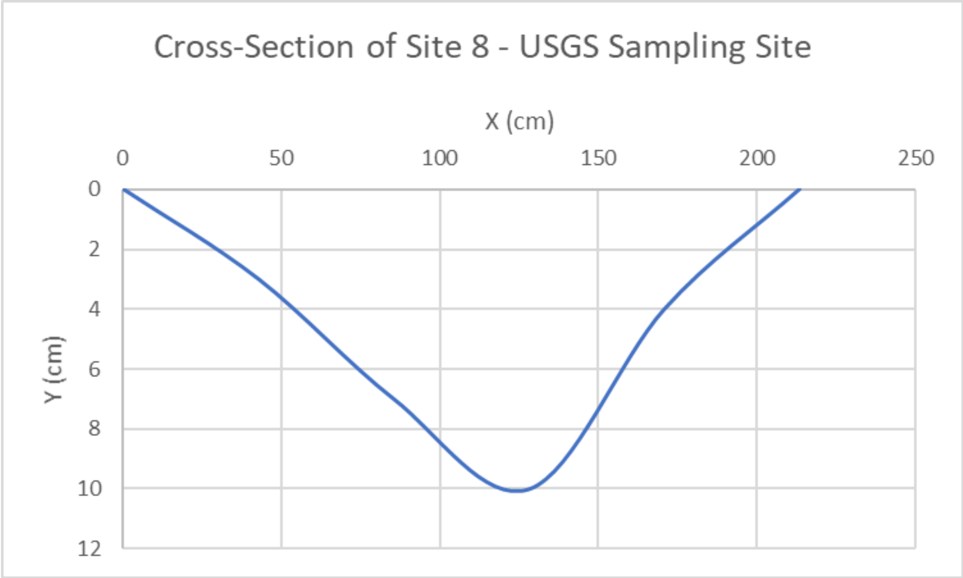

Figure 12. Cross-section of site 8.

Table 7. X and Y Data for Each Cross-Section in Centimeters (cm)

Site 8		Site 7		Site 5		Site 4	
x	y	X	y	x	y	x	y
0	0	0	0	0	0	0	0
42.7	3	50.8	11	77.45	0	50.8	3
85.4	7	101.6	11	91.9	15	101.6	7
128	10	152.4	8.5	112.2	15	289.6	7
170.7	4	203.2	10	132.5	13	477.6	7
213.4	0	254	7	152.8	7	528.4	3
		304.8	0	167.25	0	579.1	0
				243.8	0		

Long-Term Monitoring

Human sampling of Tims Branch has been effective for data collection but is insufficient alone because most contaminant transport occurs during storm events. Sampling of Tims Branch requires participation from SRS radiological protection personnel but field work isn’t performed during storm events for safety reasons. This presents a dilemma to researchers at SRS who need data collected from Tims Branch during storm events. The solution is the field installation of an automated sampling system called an ISCO. The selected site for field installation was located just downstream of the Steed Pond area because contaminant concentrations in the water samples is expected to be elevated during/after storm events (Figure 12).

Figure 13. Location of ISCO deployment.