Project Final Report

I. Project Information

Title: Investigating the role of groundwater in pollutant transport to Nu`uuli Pala Lagoon, American Samoa

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Organization: American University (Washington, DC) and University of Hawai`i

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II. Executive Summary

Nu`uuli Pala Lagoon on the island of Tutuila, American Samoa, is an ecologically valuable aquatic and wetland habitat that is threatened by development in the surrounding watershed. This project aimed to quantify groundwater-borne nutrient and pollutant fluxes into the Pala, assess how these fluxes vary in space and time, and explore whether groundwater-borne nitrogen inputs supported primary productivity of macroalgae in the Pala. Two sampling trips were conducted in August 2022 and March 2023. Concentrations of two natural groundwater tracers (radium and radon), nutrients (nitrate + nitrite, ammonium, and phosphate), dissolved metals, pharmaceuticals, hydrogen and oxygen isotopes of water, and nitrogen isotopes in macroalgal tissue were measured. The residence time of water in the Pala was estimated using radium isotope ratios. We are still working on calculating submarine groundwater discharge and associated pollutant fluxes into the Pala; however, preliminary results indicate that these fluxes are highest on the western and northern shores of the Pala.

III. Purpose

A. Overarching goal(s) of the project

The project focused on answering the following questions:

- How large are groundwater-borne pollutant fluxes into Pala Lagoon and how do they compare to surface water inputs?
- How do SGD and associated pollutant inputs to the lagoon vary in space and time?
- Is primary production in the lagoon supported by anthropogenic nitrogen pollution, and if so, what is the dominant source and transport pathway?

B. Hypotheses and objectives of the project.

We hypothesized that groundwater-borne nutrient and pollutant fluxes would be substantial in terms of their impact on water quality and the lagoon's ecology, although we did not have specific quantitative hypotheses about the magnitude of the fluxes. Because the streams flowing into the Pala are relatively small and most of them only flow intermittently, we hypothesized that fluxes of water, nutrients and pollutants from SGD would be greater than from surface water flow. Additionally, we hypothesized that SGD-borne nutrient and pollutant inputs would be higher at low tide and during the rainy season (possibly with a lag to account for groundwater flow time from recharge areas to the coast) and that primary production in the lagoon would be at least partially supported by anthropogenic nitrogen pollution delivered via SGD.

IV. Approach

A. Detailed description of the work that was performed.

Overview

Two research trips to American Samoa were conducted, one in August 2022 and one in March 2023. Each trip was approximately 2 weeks in length. During each trip, the following research activities were completed: (1) Point sampling in the Nu'uuli Pala Lagoon and its watershed, including lagoon surface water, spring water, porewater, streams, and the ocean immediately beyond the lagoon mouth, (2) Continuous spatial surveys of radon in lagoon surface water using a boat (3) Time series of radon and other water quality measurements to assess variability related to the daily tidal cycle (4) Collection and filtration of large-volume samples of lagoon surface water, springs, and streams for radium analysis (5) Collection of macroalgae samples from the lagoon bottom and shoreline to find the $\delta^{15}N$ signature (March 2023 only).

1. Point sampling

The goal of point sampling in and around the Nu'uuli Pala Lagoon was to be able to find the pathways that SGD is entering the lagoon. This was done by sampling water from the lagoon's surface, springs, porewater, streams, and ocean water directly past the mouth of the lagoon. This sampling technique is beneficial to aid in locating where nutrients, pollutants, and other identifiers from land are entering the watershed. For point sampling measurements, water samples for nutrient testing, trace metals, radon, and radium were collected. Water quality measurements were also collected using a YSI.

2. Continuous spatial surveys

Continuous spatial surveys were conducted for the purpose of identifying and quantifying hotspots of SGD in the Nu'uuli Pala Lagoon. This was done by taking a kayak around the perimeter of the lagoon as well as crossing through the middle. Spatial surveys occurred in both the wet season (March 2023) as well as the dry season (August 2022) to analyze the differences in SGD between the two seasons. Typically, samples for nutrient analysis, trace metal, water quality measurements (YSI), and radon were collected.

3. Time series of radon and other water quality measurements

The objective of performing time series measurements of both radon and other water quality measurements was to analyze and quantify the connection between the tidal cycle and SGD input. Time series measurements were conducted in two ways (1) using the spray Chamber attachment on the DURRIDGE RAD7 Radon detector to collect continuous radon measurements and (2) collecting other water quality samples roughly every other hour of the tidal cycle. At some locations, radon measurements had to be taken using the Big Bottle System on the DURRIDGE RAD7 Radon detector due to low tide.

Tidal cycle sampling was conducted to find the differences in water tracers between high and low tides. This was conducted in 2 locations in August of 2022 and 3 locations in March of 2023. Generally, at coastal sites, the DURRIDGE

RAD7 Radon detector was stationed with the Spray Chamber attachment to collect continuous measurements. When possible, the RAD7 was positioned on the shore. The RAD7 was monitored in 15-minute intervals for 6-8 hours to ensure the device ran properly. In addition to continuous radon measurements, the YSI and other water tracers were collected every 2-3 hours at each location.

In August of 2022 at site N02, the tide fell after 4.5 hours, and continuous measurements with the Spray Chamber attachment were no longer feasible. To conclude the N02 tidal cycle radon measurements, 2 Big Bottle system measurements were gathered and run on the RAD7. YSI measurements and other water tracers sampling were not affected by the low tide. At site WSP03, continuous radon measurements were collected for 5 hours, with approximately 4 measurements per hour. This resulted in 21 total radon measurements.

In March of 2023, at the NW-TS site, the RAD7 with the Spray Chamber attachment was placed inside a kayak that was anchored to the Pala floor near the shore. This was to prevent the need to switch to the Big Bottle sampling method due to the shifting waterline over the sampling period. Continuous radon measurements occurred for 7 hours at this location with water tracers being sampled every 2-3 hours. For location WSP03, the RAD7 with the Spray Chamber attachment was placed on shore and continuous radon measurements were taken for 9 hours which resulted in 38 measurements. The system was monitored every 15 minutes and water tracers were gathered every 2-3 hours. At location N02, sampling occurred every 2 hours between 10:00 and 16:00. This resulted in having 4 radon measurements. These were collected by kayaking to the location and collecting samples in Big Bottles to run on the RAD7. In August 2022, the N02 location was measured closer to the shore than in March 2023, which was sampled in the mouth of the inlet.

4. Radium analysis

The goal of radium sampling in this project was to provide an additional SGD tracer as well as identify the residence time of water in the Pala Lagoon. The specific objectives were to (1) investigate the decrease in radium activity from the shore to the middle of the Pala and (2) compare the radium isotopic ratios to find the residence time.

Radium samples were collected in 20-L containers at locations on the island including mid-Pala, springs, streams, and along the shoreline. There were 21 radium samples collected in August 2022 and 17 in March 2023. The exact volume of water collected was determined using a luggage scale and the density of water to convert mass to volume. After weighing the samples, the water was then gravity filtered through a cartridge at a rate ≤1 liter/minute (Garcia-Solsona et al., 2008). This generally occurred immediately after weighing the sample but at maximum occurred within 24 hours. This cartridge contained 7 grams of magnesium oxide (MnO_2) coated acrylic fiber, which captures and extracts the radium from the water, obtained from Scientific Computer Instruments (South Carolina). Once the entire sample had been filtered, the fibers were then rinsed with radium-free water. This step is performed to remove any sea salt or particles that may remain on the fibers (Garcia-Solsona et al., 2008). To create radium-free water, tap water was filtered through a magnesium-coated fiber to capture and remove the radium in the tap water. Once the fibers had been rinsed with radium-free water, the fibers were then air-dried until there was a water content between 40-110% of their dried mass. These fibers were then analyzed within 10 days on a Radium Delayed Coincidence Counting (RaDeCC) manufactured by Scientific Computer Instruments (South Carolina). A second run occurred 3-6 weeks after sample collection to find the thorium correction following the methods Garcia-Solsona et al. paper article (Garcia-Solsona et al., 2008).

Activities and analytical uncertainties of ^{223}Ra and ^{224}Ra were calculated in RStudio based on the methods explained by Garcia-Solsona et. al (2008). In March, the relative uncertainty % with radium 223 ranged from 17.80% to 37.60% with a median value of 25.50%. The relative uncertainty % with radium 224 ranged from 9.93% to 23.90% with a median value of 15.46%. In August, the relative uncertainty percent with radium 223 ranged from 20.16% to 122.21% with a median value of 27.46%. The relative uncertainty percent with radium 224 ranged from 10.16% to 142.71% with a median value of 15.89%.

5. Macroalgae samples

The purpose of collecting macroalgae samples from the lagoon bottom and shoreline was to analyze nitrogen isotopes in the samples to identify which sample locations had the most nitrogen input from land sources. Sampling was attempted in August but was unsuccessful due to the turbidity of the lagoon and the lack of visible macroalgae on the shoreline or the lagoon bottom. In March, the collection was successful, and 20 samples were gathered in total. Algae were sampled by snorkeling and removing primary producers in the Pala by hand. Algae was also collected by walking on the shoreline and gathering samples. At each sample location, a visual assessment of the species present was performed and samples were gathered from 3 distinct plants of each species and placed in a WhirlPak bag. Bags were kept frozen. Before processing the algae, samples were thawed in the fridge for 3 days and then rinsed with tap water followed by a rinse of deionized water. Each sample location and species within the sample location was placed into individual weigh boats and placed into a 60℃ oven for 72 hours to dry.

Dried samples were stored in a Sanplatec Dry Keeper while waiting to be processed. Once ready to package samples, pieces of each plant were placed in a porcelain mortar and ground with a matching porcelain pestle. After the sample has become a fine powder, it was transferred into a silver capsule. For calcareous macroalgae (*Halimeda*), 0.002 grams of dried material was placed into silver capsules. While still open, drops of 2N HCl are added to the fine powder until it stops bubbling. Specimens were left in the hood overnight and then capsules were carefully formed into balls. The samples were sent to Hong Kong University for $\delta^{15}N$ analysis.

Analytical Methods: The following methods were used in conjunction with point sampling, spatial surveys and time series measurements.

1. ***On-site water quality measurements (YSI):*** A YSI Professional Plus handheld probe (Yellow Springs, OH, USA) was used to measure salinity, temperature, dissolved oxygen, and pH at multiple sampling site types in both August 2022 and March 2023. In the field, the YSI probe was placed in multiple streams, springs, porewater, lagoon surface water, shoreline water, and ocean water. After around 30 seconds, measurements were recorded in the field notebook with the sample location, date, and time collected.

2. ***Radon:*** Radon concentrations were measured using a RAD7 radon detector (Durridge, Billerica, MA) along with the 250-mL or 2-L (Big Bottle) RAD-H2O accessories for grab samples or the RAD-AQUA spray chamber accessory for *in situ* measurements. Grab samples were run within 3 days of collection and concentrations were adjusted for the delay time between collection and analysis as described in the product manual.

3. ***Nutrients (Nitrate, nitrite, ammonia, and phosphate):*** Nutrients were sampled to locate where and how much pollution is entering the watershed. This is important to understand the extent of degradation and eutrophication in the Nu'uuli Pala Lagoon. Throughout both sampling periods, nutrients in water samples were collected from streams, groundwater, shoreline, and the Pala. Water was filtered using a 0.2 μm syringe filter. This water was then placed into an amber high-density polyethylene (HDPE) bottle. Throughout each sampling day, bottles were kept in an ice-filled cooler. At the end of each sampling day, bottles were then placed in the freezer. At the end of both August 2022 and March 2023 sampling trips, frozen bottles were relocated to the American University laboratory where they remained in the freezer until analysis was performed on the EasyChem Multi-Parameter Discrete Analyzer (EZKem, Hood River, OR). Protocol from the manufacturer was followed to perform colorimetric analysis which was established from methods from the United States Environmental Protection Agency (USEPA) methods.

4. ***Hydrogen and oxygen isotopes:*** Stable isotopes of hydrogen and oxygen were used to gain insight into the seasonality and elevation of groundwater recharge. This information is useful for understanding the Pala’s hydrology and the sources and transport of pollutants into it. It will also help local residents and environmental managers predict and understand climate change impacts, which may indirectly affect SGD by altering rainfall, evapotranspiration, and sea level. Samples for hydrogen and oxygen isotope analysis were collected in 20-mL glass bottles, making sure that no air bubbles were trapped in the bottle, kept cold (in a refrigerator or cooler) until analysis, and analyzed at the University of Hawai`i. Stable isotopes were analyzed by the Biogeochemical Stable Isotope Facility at the University of Hawaiʻi at Mānoa. Stable isotope analyses for δ18O and δD were performed using a Picarro L1102-i wavelength scanned cavity ring down spectroscopy (WS-CRDS) relative to V-SMOW.

5. ***Pharmaceuticals***: Concentrations of three pharmaceuticals/micropollutants (caffeine, sulfamethoxazole, and carbamazepine) were measured in samples from streams, groundwater, the shoreline and the middle of the Pala. These pharmaceuticals are indicative of sewage pollution and may also negatively impact aquatic life. Samples were filtered through 0.2 μm filters into amber glass bottles and kept cold until analysis. Micropollutant samples were stored refrigerated at 4°C – 8 °C and analyzed within one month of collection at the coastal geochemistry and hydrology lab at University of Hawaiʻi (contact: H. Dulai, hdulaiov@hawaii.edu). Carbamazepine (CBZ), sulfamethoxazole (SMX), and caffeine (CAF) were analyzed using Enzyme-Linked Immunosorbent Assay (ELISA) kits purchased from Abraxis, LLC. Separate microtiter wells coated with compound specific antibody received protocol prescribed volumes (20 microliter –100 microliter) of water samples, standards, and blanks. Enzyme conjugate and antibody solutions were added to each well in a kit specific order. After a prescribed incubation time, the solution was discarded, the wells were washed with wash solution, and a color solution was added to each well and incubated for 30 minutes. The developed color intensity was inversely proportional to the amount of compound of interest in the sample. The color development was

stopped with the addition of a color stop solution. The plate with the wells was then analyzed within 15 minutes. The absorbance at 450 nm was determined for each well using a microplate reader (photometer Model 4303, Abraxis). 100% of the standards and blanks and 10 % of the samples were run in duplicates. Based on 90% of the sample absorbance to blank absorbance ratio (B/Bo%) the kit sensitivities ranged 0.021 ng/mL – 1.1 ng/mL.

6. ***Dissolved metals and trace elements***: For a suite of dissolved trace metal analysis, including uranium (U), vanadium (V), chromium (Cr), lead (Pb), nickel (Ni), Mo, Ba, Re, Fe, Cs, Mn, and copper (Cu), water was filtered in the field and acidified in a clean laboratory to pH ~1.8 using ultrapure 6 M HCl (Seastar Baseline) to a final acid concentration of 0.024 M. Acidified samples were analyzed similarly to the procedure in Shiller (2003). Briefly, prior to analysis, samples were slightly diluted (1.5×) by addition of ultrapure nitric acid (Seastar Baseline) containing known amounts of In, Sc, and Th as internal standards. The diluted samples were approximately 0.16 M in nitric acid and 2 ppm in the internal standards. Trace elements were then determined using a sector-field inductively coupled plasma–mass spectrometer (ICP–MS, Thermo-Fisher Element 2) at the University of Southern Mississippi Center for Trace Analysis (CETA). A low-flow (100 μL/min) self-aspirating nebulizer (Elemental Scientific, Omaha, NE, USA) and Teflon spray chamber were utilized. Cs, Re, Pb, and U were determined in low resolution (as masses 133, 187, 208, and 238, respectively) and the other elements were determined in medium resolution (51 V, 52 Cr, 55 Mn, 56 Fe, 60 Ni, 63 Cu, 66 Zn, 85 Mo, 88 Sr, and 137 Ba). Calibration was done with external standards made in 0.16 M ultrapure nitric acid. The standards were independently checked against U.S. Geological Survey standard reference waters and an in-house consistency standard was also measured as a check on long-term stability of the standards. In was used for instrumental drift correction and sensitivity check, with the other internal standards serving as a check on the In. Sample acidification and other preparations for analysis were carried out in a laminar-flow clean bench.

Calculations and data analysis

1) Spatial mapping and analysis: We added all our data to a geographic information system (GIS) using ArcGIS Pro. The goal of this was to allow us to see spatial trends and correlations, perform spatial analysis, and create maps of our data.

2) Pala residence time calculation: We used $^{224}Ra/^{223}Ra$ isotope ratios of groundwater and Pala water to estimate the residence time of water in the Pala using the method of Moore et al. (2000).

3) Submarine groundwater discharge: Submarine groundwater discharge was estimated using a non-steady-state mass-balance approach. Radium and radon were used as natural groundwater tracers. The characteristics of the discharging groundwater endmember were estimated based from spring samples. The mass balance model accounted for Rn evasion to the atmosphere, diffusion from sediments, and advective flow related to ebbing or flooding tide.

4) Pollutant fluxes: Pollutant fluxes were estimated by multiplying pollutant concentrations in discharging groundwater (springs) by the submarine groundwater discharge. When significant spatial variability in groundwater quality occurred, we used different groundwater endmember concentrations for different parts of the lagoon.

B. Project management: In this section, we list individuals and/or organizations actually performing the work and how it was done.

Karen Knee (American University) was the principal investigator (PI). She planned and carried out the study, supervised students associated with the project, and completed all necessary reporting. Karen Knee was primarily responsible for radium, radon, and nutrient analysis.

Kiho Kim (American University) and Henrietta Dulai (University of Hawai`i) were co-PIs. They assisted with planning and carrying out the study and student supervision. Kiho Kim was primarily responsible for the macroalgae analysis and Henrietta Dulai assisted with radium and radon analysis and was primarily responsible for hydrogen and oxygen stable isotopes, micropollutants, and dissolved metals.

Nina Mewborne (American University) is a graduate student completing an MS in Environmental Science. She performed field work and lab work, as well as much of the data analysis and writing that went into this final report.

TreVaughn Ellis (American University), Hailey Pantaleo (University of Hawai`i), and Annie Chien (University of Hawai`i) assisted with field work.

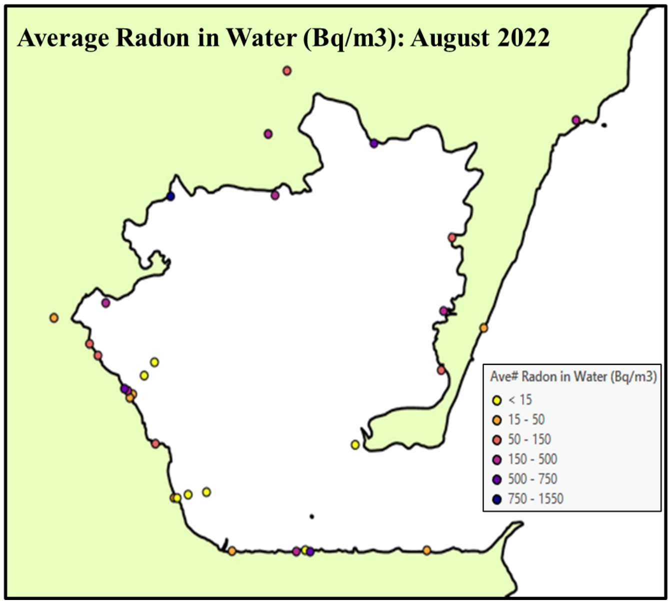
Casey TeBeest (University of Hawai`i), Alisha Gill (NOAA), Kelley Anderson Tagarino (University of Hawai`i Sea Grant, American Samoa Community College), Tolo Hoching (Village of Nu`uuli), and Chris Shuler (University of Hawai`i) provided logistical advice and support, including assisting with field sampling.

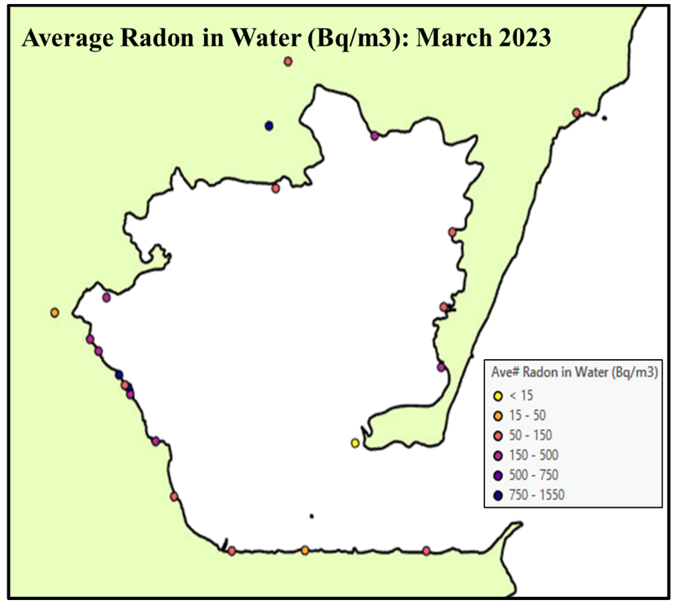
V. Findings

A. Actual accomplishments and findings.

Although the pandemic delayed this project, we were eventually able to complete all the planned sampling. Some of our key findings were:

1) Radon was elevated at near the shoreline of the Pala, indicating submarine groundwater discharge. This SGD signal was spatially variable, with the highest radon concentrations observed along the western shoreline, where many springs were present, and at the inlet on the north shoreline of the Pala.

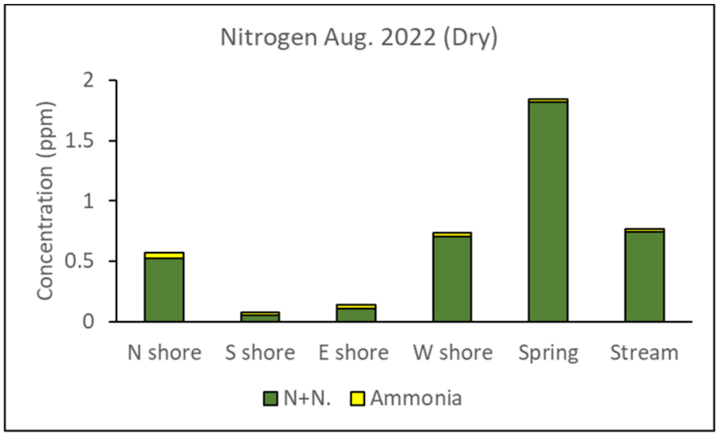


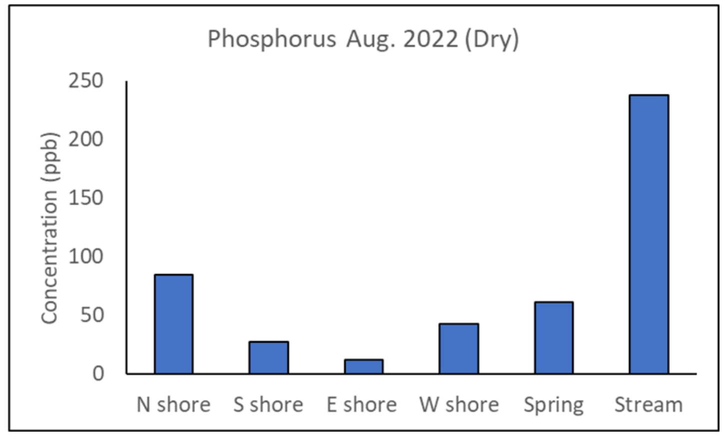


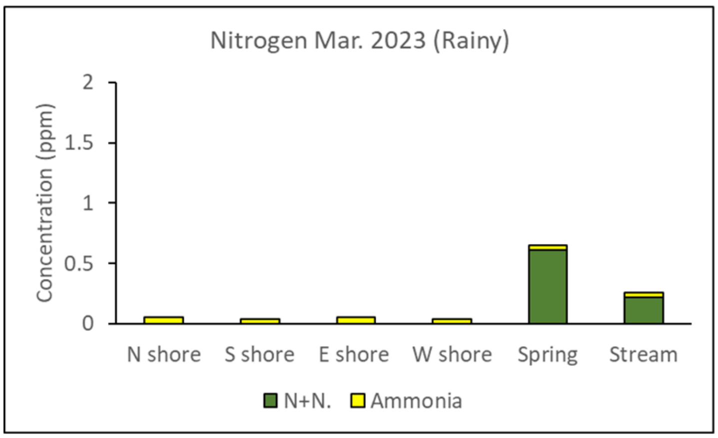
2) Radium isotope ratios indicated that the residence time of water in the Pala was longer in August (dry season) than in March (rainy season), was on the order of 1-2 weeks, and varied with location within the Pala.

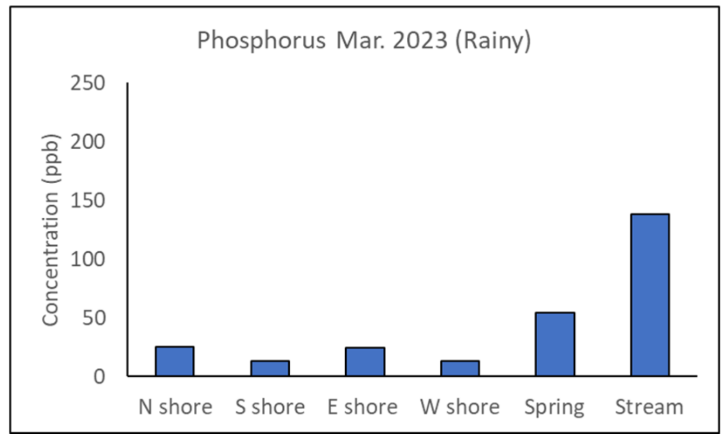
	Groundwater activity ratio	Pala activity ratio	Estimated residence time (d)
Aug-22	10.6-26.5	3.1-13.0	<16
Mar-23	6.9-21.6	7.6-13.9	<8

3) Nutrient (dissolved inorganic nitrogen and orthophosphate) concentrations displayed distinct patterns of variability related to site type and season. Concentrations of both DIN and orthophosphate were higher across the board in August 2022 (dry) compared to March 2023 (rainy). This seasonal difference was especially pronounced for nitrate + nitrite. Springs had the highest DIN concentrations, followed by streams and, in March 2023, the northern and western parts of the Pala's shoreline. In contrast, streams had the highest phosphate concentrations.

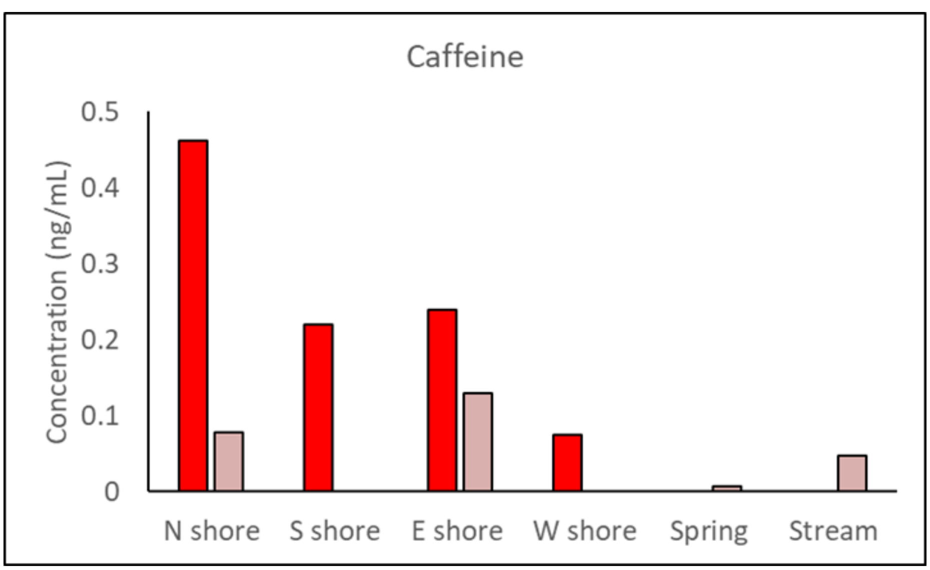


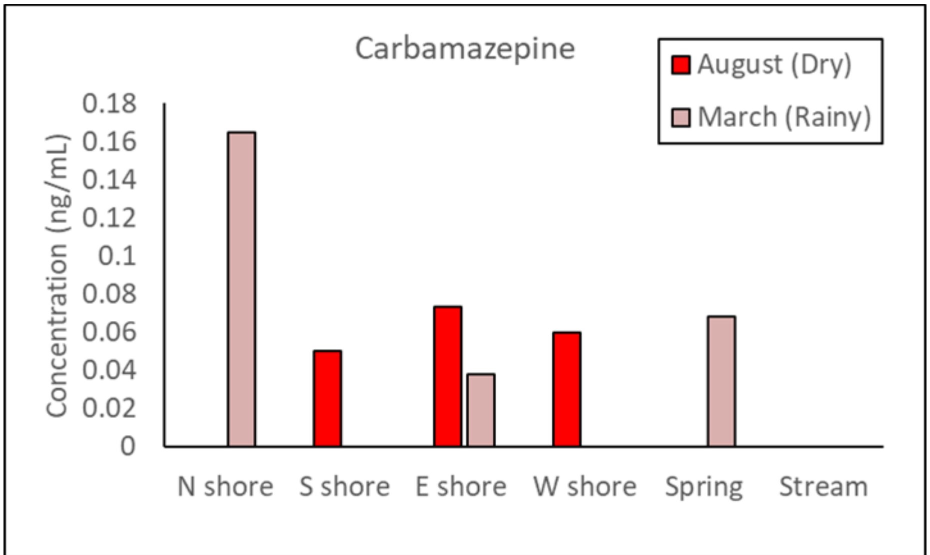


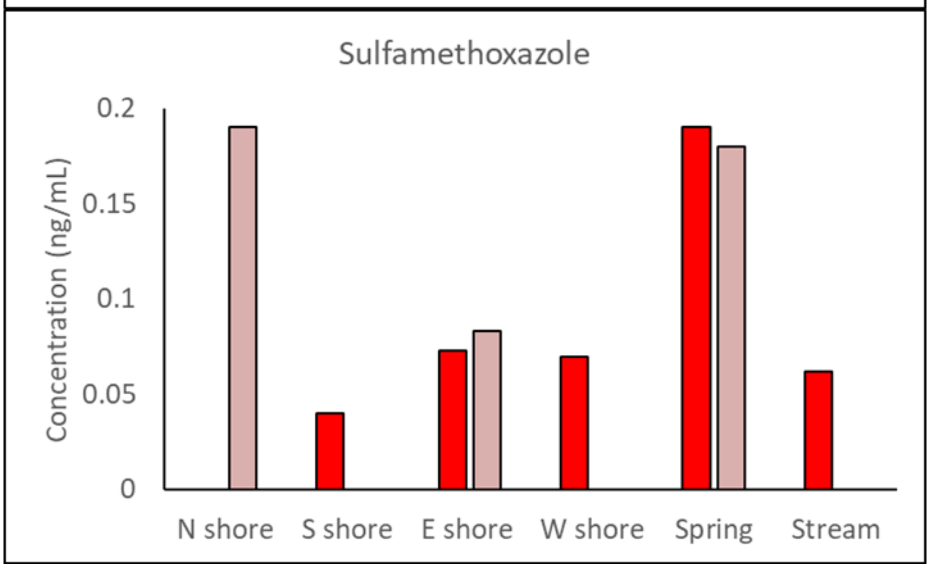




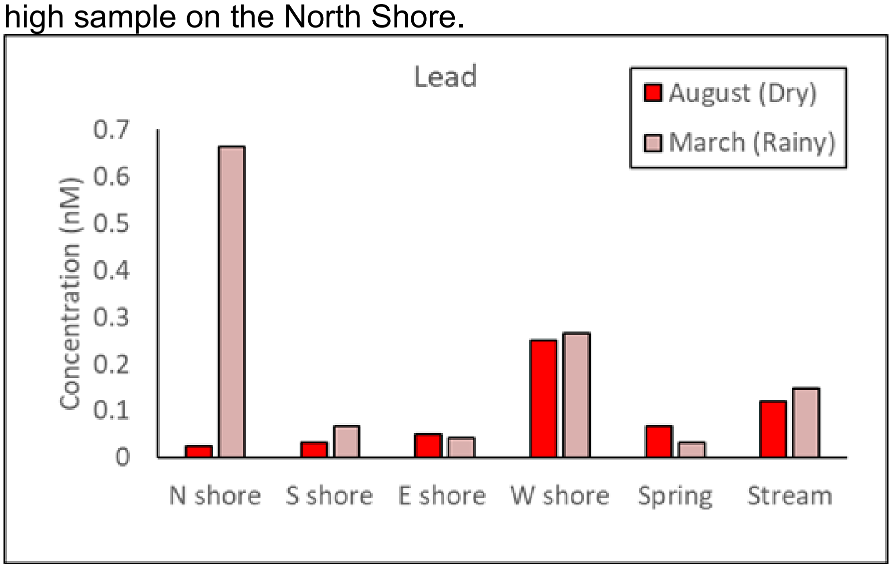
4) Caffeine, carbamazepine and sulfamethoxazole were detected in all sample types at concentrations of up to 0.5 ng/mL (caffeine) and 0.2 ng/mL (carbamazepine and sulfamethoxazole). There were not any distinct patterns of variability related to site type or season. These concentrations reflect some level of sewage pollution in many different site types around the Pala and are broadly similar to what has been observed in other coastal areas with anthropogenic impact.

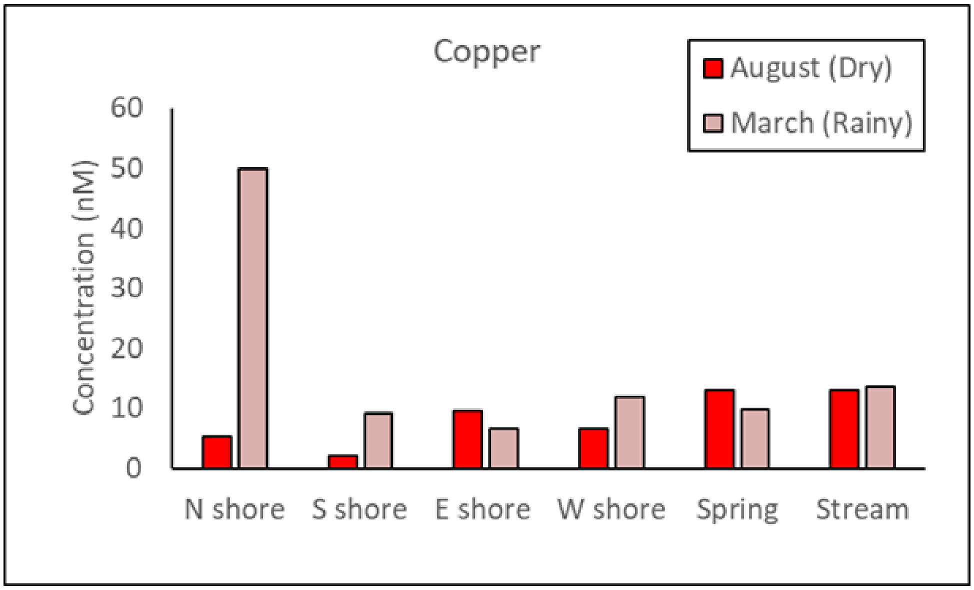






5) Concentrations of a suite of metals (Mo, Ba, Re, Pb, V, Fe, Ni, Cu, Cs, U, Cr, Mn, Zn, Sr, Na, Mg, S, Ca, K, Sr) were measured. We are still analyzing this data set, but there appear to be some interesting spatial trends. For example, the north and western shores of the Pala and streams had higher lead concentrations, while copper concentrations were relatively constant across different site types except for one very high sample on the North Shore.





B. Problems

The only problem that influenced our data collection was the lack of macroalgae in August 2022. This reduced the number of macroalgal samples we were able to collect and prevented us from drawing any conclusions about seasonal variation in nutrient sources to algae.

C. Description of need, if any, for additional work.

We are currently completing data analysis and writing for this project. Most of the data for this project will be incorporated into Nina Mewborne's master's thesis, which will be completed by May 2024. We anticipate submitting one or more publications to peer-reviewed scientific journals shortly thereafter.

When we were in American Samoa interacting with the community, many people expressed concern about the potential effects of pollution (e.g. heavy metals) on fish that are used for human consumption. Thus, we feel that there is a need for more research on how dissolved metals in the water move through aquatic food webs in the Pala and bioaccumulate in fish that are used for human consumption.

There are also many parts of American Samoa (both on Tutuila and other islands) where no measurements of submarine groundwater discharge and related nutrient and pollution inputs have been made. We feel that there is a need to do similar studies in more locations within the territory to get a better idea of how groundwater flow affects coastal water quality.

VI. Applications

A. Outputs

1) **New fundamental or applied knowledge**: We have generated new knowledge about submarine groundwater discharge and water quality in Nu`uuli Pala Lagoon, American Samoa.
2) **Scientific publications**: We have not produced any publications yet, but we are currently working on writing up the results and plan to have a manuscript ready to submit by summer 2024.
3) **Data**: We are still working on data analysis and quality control. Once all the data are finalized, we will make the data available as an

appendix to the publication(s) based on that data, or in some other format.

4) **Patents**: None
5) **New methods and technology**: None
6) **New or advanced tools (e.g. models, biomarkers):** None
7) **Workshops**: We held three workshops for local stakeholders in American Samoa, two during the August 2022 trip and one during the March 2023 trip. Once our analyses are finalized, we are planning to offer another workshop (probably by Zoom, unless we end up doing more work on American Samoa or are able to find funding to support travel just for the workshop) to share our results with local stakeholders and help them think through how these results can be used in coastal management.
8) **Presentations**: Karen Knee presented a lightning talk and Nina Mewborne presented a poster about this work at the Coastal and Estuarine Research Federation (CERF) biennial meeting in Portland, OR in November 2023. Nina Mewborne also gave presentations about this work at the American University Environmental Science departmental seminar in October 2023 and at a summer student research showcase in August 2023.
9) **Outreach activities/products (e.g. website, newsletter articles):**
   This research was featured in two articles on the American University website, "Going Green: AU Environmental Scientists Work to Save Our Planet" and "Research Protecting Some of the World's Most Threatened Wetlands". Once we have finalized our results, we are planning to work with a Samoan-English translator to prepare bilingual (Samoan-English) materials about our work for distribution in American Samoa.

B. Management outcomes - To the best of our knowledge, this project has not had any management outcomes yet because we are still finalizing our data analysis and have not yet shared our results with local stakeholders. We are planning to do this within the next several months and we can provide updates about if/how our results are being used to inform management decisions if that information would be welcome.

VII. Evaluation

All of our goals and objectives relating to field and lab work have been accomplished. We are still working on data analysis and writing, but we anticipate that those goals will be accomplished in spring or summer 2024.

VIII. Data Management

Karen Knee contacted the NCEI by email on 12/20/2023 to inquire about how to format and submit the data. According to NOAA's Data and Publication Sharing Directive, data archiving should be done in a timely fashion, which means by the time papers using the data are published or within two years of the end of the funding period. We anticipate being able to post the data in a timely fashion.

IX. References

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Prepared by:

[signature] 12/22/23

Signature of Principal Investigator Date