

A Baseline Chemical Contaminants Assessment of Sediment from the Nu'uuli Pala Lagoon, American Samoa



NOAA National Centers for Coastal Ocean Science
Stressor Detection and Impacts Division

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A Baseline Chemical Contaminants Assessment of Sediment from the Nu'uuli Pala Lagoon, American Samoa

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ABSTRACT

Thirteen marine sediment samples were collected as part of an effort to characterize and assess chemical contamination in the Nu'uuli Pala Lagoon on the island of Tutuila, American Samoa. Overall, concentrations of organic contaminants in sediment from the Nu'uuli Pala Lagoon are low as compared to other studies conducted by NOAA's National Status and Trends (NS&T) Program, and are similar to those measured in the nearby Faga'alu watershed and bay. The only organic contaminants measured in the Pala Lagoon that exceeded any known guidelines or thresholds was for total DDT, and then only the Effects Range Low. PBDEs in the Pala Lagoon also appear to be elevated as compared to other relatively lower population coastal US areas. The concentrations of trace and major elements had a number of exceedances of established guidelines, including arsenic, chromium, copper, nickel, and zinc. Nickel was the only metal that exceeded both the ERL and ERM, pointing towards likely adverse impacts to the Pala Lagoon.

INTRODUCTION

This report contains the results of a chemical contaminants assessment of the Nu'uuli Pala Lagoon Special Management Area (SMA) located on the island of Tutuila, American Samoa. This characterization was conducted by the National Oceanic and Atmospheric Administration's (NOAA) National Centers for Coastal Ocean Science (NCCOS), National Status and Trends (NS&T) Program, with funding from NOAA's Coral Reef Conservation Program (CRCP), and in partnership with the American Samoa Environmental Protection Agency (ASEPA), the American Samoa Coral Reef Advisory Group (CRAG), and the American Samoa Community College (ASCC). NOAA's NS&T Program measures a standard suite of 193 chemical contaminants in US coastal waters, including the Great Lakes and coral reef systems, and maintains a database of previous chemical contaminants studies that provides a baseline against which we can compare data from this study. An additional 299 organic chemical contaminants were also measured in addition to the NS&T standard suite.

BACKGROUND

Study Site: Climate, Population, and Geography

Climate conditions in American Samoa are relatively stable throughout the year, consisting of a warm and humid tropical environment. Although there is technically a rainy season (October to April) and a dry season (May to September), rainfall is common throughout the year. Annual rainfall averages range from 320 to 750 cm (NPS, 2015) depending on elevation with mountainous areas receiving more rain.

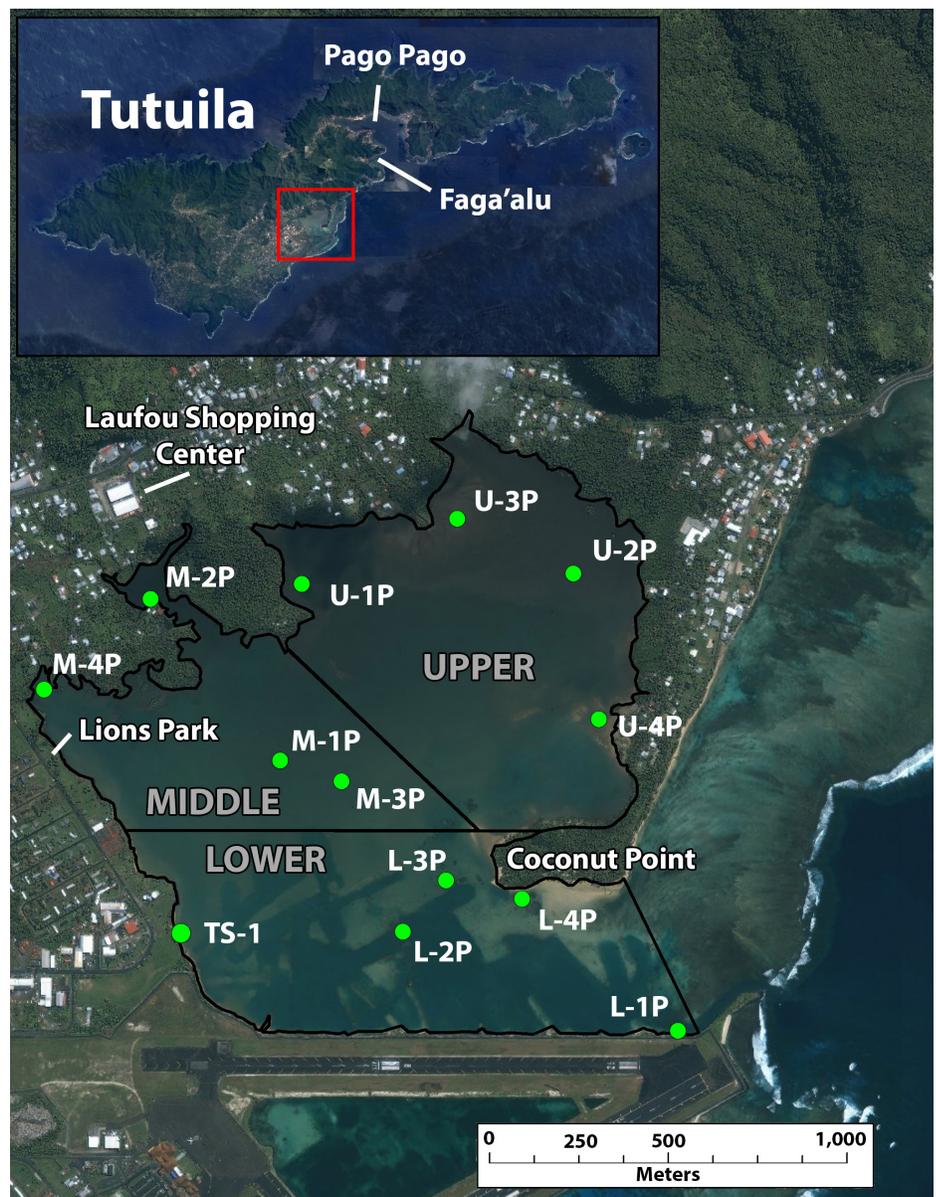


Figure 1. Map of the Nu'uuli Pala Lagoon showing sediment sampling locations and strata.

The Nu'uuli Pala Lagoon watershed drains the most populous area of the island of Tutuila, including the village of Nu'uuli, and parts of Tafuna, Faleniu, Malaeimi, and Mesepa among other areas. The combined population of these villages as of 2011 was estimated at 15,424, or approximately 28% of the total population of American Samoa (ASG, 2011). The Nu'uuli Pala Lagoon SMA, referred to as just the Pala Lagoon hereafter, is located on the southern coast of the island of Tutuila, just north of the Pago Pago International Airport (Figure 1). It is comprised of 13% emergent vegetation (including mangroves), 2% coral, and 77% uncolonized sediments (NOAA, 2009). During the 1960s, the lagoon's natural circulation patterns were heavily altered through the creation of the airport (Scott, 1993). The construction of the runways directly affected the Pala Lagoon through the removal of dredge material to create new land, and through the artificial restriction of ocean water exchange through the narrow channel between the airport runway and Coconut Point. The lagoon was further impacted in the 1960s by the conversion of approximately 33% of the original mangrove swamp to dry land (NOAA, 2009).

Land based sources of pollution (LBSP) have the potential to negatively impact coral reef ecosystems. The Pala Lagoon has multiple potential sources of LBSP including: runoff from roads, poorly functioning septic systems/cesspools, the airport, a jail, a history of piggeries, and low intensity agriculture (e.g. bananas). This study quantifies the magnitude and distribution of pollution in the lagoon and serves as a baseline against which future impacts can be measured.

Overview of the Chemical Contaminants

Quantifying the concentrations of over 400 chemical contaminants in sediment allows us to describe the nature of the pollution present, make hypotheses about their sources and fate in the environment, and begin to document potential stresses that the observed concentrations can cause in the marine environment, by comparing them to accepted guidelines. Each class of contaminant analyzed for this project is discussed below.

Polycyclic Aromatic Hydrocarbons.

This class of oil-related compounds, also referred to as PAHs, are usually less abundant in crude oil than saturated hydrocarbons and include one or more aromatic benzene rings in their structure (NRC, 1985). PAHs can be associated with the use and combustion of fossil fuels and other organic materials. Additional natural sources of PAHs can include decay of organic material (vegetation) and forest fires. The complete list of PAHs analyzed in this study can be found in Table 1. The PAHs analyzed here are two to six ring aromatic compounds. PAHs can bioaccumulate in both aquatic and terrestrial organisms and many individual compounds are toxic. Some compounds such as benzo[a]pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k] fluoranthene, dibenzo[a,h]anthracene, and indeno[1,2,3-c,d]pyrene, are likely carcinogenic (ATSDR, 1995).

Polychlorinated Biphenyls.

Made up of 209 individual congeners (Table 1), Polychlorinated biphenyls (PCBs) are a chemical contaminant class that was manufactured in the US starting in 1929 through 1977, before they were banned in 1979 as a result of their toxicity. These chemicals were sold in a number of congener mixtures, referred to as Aroclors, and had many industrial uses such as in electrical transformers, pesticides, paints, and heat transfer liquids. PCBs are highly persistent in the environment and have the ability to bioaccumulate in both marine life and humans. Because of their slow degradation and wide use during their manufacture period, PCBs are a commonly measured marine contaminant. Fish exposed to PCBs have been shown to exhibit reduced reproductive viability, including vertebral anomalies, as well as experiencing growth deficits (EPA, 1997).

Organochlorine Pesticides.

This class of chemicals includes 31 individual analyzed components for this study (Table 1), the most well-known being the pesticide dichlorodiphenyltrichloroethane (DDT), which had very wide spread usage beginning in the 1950s. Large scale DDT use was banned (with limited exceptions for indoor applications) in the 1970s because of its environmental persistence. Organochlorine pesticides are generally neurotoxins and are known to be endocrine disruptors (Rogen and Chen, 2005). DDT and its metabolites have been shown to cause thinning of bird egg shells, resulting in substantial decreases in reproductive success in many bird species (Lincer, 1975). Other non-target species affected by DDT and its metabolites include multiple fish species, shrimp, and crayfish.

Table 1. List of legacy chemical contaminants and trace and major elements analyzed as part of this study. PAHs = polycyclic aromatic hydrocarbons; HCH = hexachlorocyclohexane; DDMU = 1-chloro-2,2-(p-chlorophenyl)ethylene; DDD = dichlorodiphenyldichloroethane; DDE = dichlorodiphenyldichloroethylene; DDT = dichlorodiphenyltrichloroethane; PCB = polychlorinated biphenyl.

| PAHs | Organochlorine Pesticides | PCBs | Elements |
|------------------------------|----------------------------|-------------|----------------|
| cis/trans Decalin | Aldrin | PCB1 | PCB180 |
| C1-Decalins | Dieldrin | PCB7/9 | PCB183 |
| C2-Decalins | Endrin | PCB8/5 | PCB185 |
| C3-Decalins | Heptachlor | PCB15 | PCB187 |
| C4-Decalins | Heptachlor-Epoxyde | PCB16/32 | PCB189 |
| Naphthalene | Oxychlorane | PCB18 | PCB191 |
| C1-Naphthalenes | Alpha-Chlordane | PCB22/51 | PCB194 |
| C2-Naphthalenes | Gamma-Chlordane | PCB24/27 | PCB195/208 |
| C3-Naphthalenes | Trans-Nonachlor | PCB25 | PCB196/203 |
| C4-Naphthalenes | Cis-Nonachlor | PCB26 | PCB199 |
| Benzothiophene | Alpha-HCH | PCB28 | PCB200 |
| C1-Benzothiophenes | Beta-HCH | PCB29 | PCB201/157/173 |
| C2-Benzothiophenes | Delta-HCH | PCB31 | PCB205 |
| C3-Benzothiophenes | Gamma-HCH | PCB33/53/20 | PCB206 |
| C4-Benzothiophenes | DMU | PCB40 | PCB209 |
| Biphenyl | 2,4'-DDD | PCB41/64 | PCB136 |
| Acenaphthylene | 4,4'-DDD | PCB42/59/37 | PCB138/160 |
| Acenaphthene | 2,4'-DDE | PCB43 | PCB141/179 |
| Dibenzofuran | 4,4'-DDE | PCB44 | PCB146 |
| Fluorene | 2,4'-DDT | PCB45 | PCB149/123 |
| C1-Fluorenes | 4,4'-DDT | PCB46 | Cadmium (Cd) |
| C2-Fluorenes | 1,2,3,4-Tetrachlorobenzene | PCB47/48/75 | Chromium (Cr) |
| C3-Fluorenes | 1,2,4,5-Tetrachlorobenzene | PCB49 | Copper (Cu) |
| Carbazole | Hexachlorobenzene | PCB52 | Iron (Fe) |
| Anthracene | Pentachloroanisole | PCB56/60 | Lead (Pb) |
| Phenanthrene | Pentachlorobenzene | PCB66 | Manganese (Mn) |
| C1-Phenanthrenes/Anthracenes | Endosulfan II | PCB70 | Mercury (Hg) |
| C2-Phenanthrenes/Anthracenes | Endosulfan I | PCB74/61 | Nickel (Ni) |
| C3-Phenanthrenes/Anthracenes | Endosulfan Sulfate | PCB81 | Selenium (Se) |
| C4-Phenanthrenes/Anthracenes | Mirex | PCB82 | Silicon (Si) |
| Dibenzothiophene | Chlorpyrifos | PCB83 | Silver (Ag) |
| C1-Dibenzothiophenes | | PCB84 | Tin (Sn) |
| C2-Dibenzothiophenes | | PCB85 | Zinc (Zn) |

Trace and Major Elements.

Sixteen trace and major elements were analyzed in sediment for this study (Table 1). Each of these elements are naturally occurring to some extent, though aluminum, iron, and silicon are classified as crustal metals and comprise the major elements for this study. Trace metals occur naturally at much lower levels in marine and terrestrial sediment, though anthropogenic activities, such as mining and manufacturing, can lead to artificially elevated levels. Improper waste disposal of items like batteries can also lead to elevated levels of trace metals in the environment. Though some trace elements are essential micronutrients, they can also be toxic at relatively low levels. Previous research (Whitall et al., 2014) showed that some metals (arsenic, chromium, copper, and nickel) can accumulate in coral tissues.

Arsenic is a naturally occurring metal that can exist in both organic and inorganic forms. Arsenic has a number of applications ranging from use in agriculture, including pesticides and herbicides, to pharmaceuticals (Eisler, 1988). Arsenic is a known carcinogen, mutagen, and teratogen, with adverse effects observed in plants, humans, and aquatic mammals and invertebrates (Eisler, 1988; Novellini et al., 2003).

Chromium has multiple human uses including as a component of stainless steel, in electroplating of metals, and as an industrial catalyst (RCS, 2014). Studies of the effects of chromium in the environment have shown reduced growth in fish, decreased reproductive success in sea urchins, and reduced survival in small crustaceans (Novellini et al., 2003; Eisler, 1986).

Copper is a widely used trace metal that, due to its high conductivity properties, is used in many electronic applications. Copper is also a major component of automotive parts including brake pads. Copper also has uses as a component of anti-fouling paints and other biocides. Copper is an essential nutrient for many plants and animals, but at high exposure levels it has been shown to have adverse effects ranging from deleterious effects on reproduction and development in invertebrates (Eisler, 1998), and sea urchins (Edullantes and Galpate, 2014; Dermeche et al., 2012; and Novellini et al., 2003). For corals, concentrations of copper above 20 µg/L have been shown to impact fertilization success and at concentrations above 75 µg/L reproductive rates dropped to one percent or below (Reichelt-Brushett and Harrison, 2005).

Nickel is a naturally occurring trace element in the earth's crust. Human activities such as mining and use in metal plating, coins, and batteries have served to concentrate nickel levels and result in potential increased levels in the environment. Elevated levels of nickel have been shown to have adverse effects on both marine invertebrates and fish, as well as coral larvae mortality (Novellini et al. 2003; Hunt et al., 2002; Goh, 1991).

Zinc is an essential micronutrient for both plants and animals, but like many other naturally occurring trace elements, it can be toxic at higher levels. Zinc has industrial applications in the creation of the metal alloys bronze and brass, paints, rubber vulcanization, ceramics, fertilizers, batteries, and in corrosion control in drinking water systems (EPA, 2005). Elevated levels of zinc in the environment have been shown to be toxic to aquatic invertebrates and fish, but less so to birds and mammals (Novellini et al., 2003; Edullantes and Galapate, 2014; Besser and Lieb, 2007; USDOJ, 1998).

Multi Residue Pesticides and Human Use Pharmaceuticals.

In addition to the standard suite of 193 contaminants (described above and listed in Table 1), another 299 individual chemical contaminants were analyzed. These additional chemicals (listed in Tables 2 and 3) include a number of current use and legacy pesticides and human use pharmaceuticals. Note that there is some overlap between these lists though different analytical methods were used. These chemicals have a wide array of uses and applications along with many potential environmental endpoints and effects. The effects of many of these chemicals to aquatic organisms are not currently well understood. For this study, we focus only on those chemicals that were detected: Amphetamine, triclocarban, DEET (N,N-diethyl-meta-toluamide), clotrimazole, perfluorooctane sulfonate (PFOS), perfluorooctanesulfonic acid (PFOA), chlorpyrifos, beta-endosulfan, alpha hexachlorocyclohexane (HCH), alpha chlordane, trans-nonachlor, and dieldrin. For many of the Multi Residue Pesticides and Human Use Pharmaceuticals measured for this study, no sediment quality guidelines currently exist.

Polybrominated Diphenyl Ethers (PBDEs).

This class of chemicals represents an environmentally persistent set of flame retardant chemicals and is comprised of 10 individual homologues (mono through deca PBDE). Table 3 lists the PBDE congeners that were analyzed as part of this study. PBDEs have been in use since the 1960s with commercial production in the US beginning in the late 1990s (Siddiqi

Table 2. List of human use pharmaceuticals, personal care products, and perfluorinated organic compounds that were analyzed as part of this study. DEET = N,N-diethyl-m-ta-toluanide.

| Human Use Pharmaceuticals and Personal Care Products | | | |
|---|----------------------|------------------------|--|
| 1,7-Dimethylxanthine | Ciprofloxacin | Fluocinonide | Oxazepam |
| 10-hydroxy-amitriptyline | Citalopram | Fluoxetine | Oxolinic Acid |
| 2-Hydroxy-ibuprofen | Clarithromycin | Fluticasone propionate | Oxycodone |
| 4-Epianhdrochlorotetracycline | Ischlorotetracycline | Medroxyprogesterone | Oxytetracycline [OTC] |
| -[EACTC] | -[ICTC] | -Acetate | Triclocarban |
| 4-Epianhdrotetracycline [EATC] | Clinafloxacin | Furosemide | Penicillin V |
| 4-Epichlorotetracycline [ECTC] | Clonidine | Gemfibrozil | Prednisolone |
| 4-Epioxytetracycline [EOTC] | Clotrimazole | Glipizide | Prednisone |
| 4-Epitetracycline [ETC] | Cloxacillin | Glyburide | Promethazine |
| Acetaminophen | Cocaine | Hydrochlorothiazide | Paroxetine |
| Albuterol | Codeine | Hydrocodone | Penicillin G |
| Alprazolam | Colchicine | Hydrocortisone | Propoxyphene |
| Amitriptyline | Cotinine | Ibuprofen | Propranolol |
| Amlodipine | Cyclophosphamide | Iopamidol | Ranitidine |
| Amphetamine | Daunorubicin | Lincomycin | Rosuvastatin |
| Amsacrine | DEET | Lomefloxacin | Roxithromycin |
| Anhydrochlorotetracycline [ACTC] | Dehydronifedipine | Melphalan | Sarafloxacin |
| Anhydrotetracycline [ATC] | Demeclocycline | Meperbamate | Sertraline |
| Atenolol | Desmethyldiltazem | Metformin | Simvastatin |
| Atorvastatin | Diatrizoic acid | Methylprednisolone | Sulfachloropyridazine |
| Azathioprine | Diazepam | Metoprolol | Sulfadiazine |
| Azithromycin | Digoxigenin | Metronidazole | Sulfadimethoxine |
| Benzoyllegonine | Digoxin | Miconazole | Sulfamerazine |
| Benzotropine | Diltiazem | Minocycline | Sulfamethazine |
| Betamethasone | Diphenhydramine | Moxifloxacin | Sulfamethizole |
| Bisphenol A | Doxorubicin | Naproxen | Sulfamethoxazole |
| Busulfan | Doxycycline | Norflouxacin | Sulfanilamide |
| Caffeine | Drosiprenone | Norflouxetine | Sulfathiazole |
| Carbadox | Enalapril | Norgestimate | Tamoxifen |
| Carbamazepine | Enrofloxacin | Norverapamil | Teniposide |
| Gefotaxime | Erythromycin-H2O | Oflloxacin | Tetracycline [TC] |
| Chlortetracycline [CTC] | Etoposide | Ormetoprim | Theophylline |
| Cimetidine | Flumequine | Oxacillin | Thiabendazole |
| Perfluorinated Compounds | | | |
| | | | Pentafluorobenzoic acid (PFBA) |
| | | | Perfluoro-n-pentanoic acid (PFPeA) |
| | | | Perfluorohexanoic acid (PFHxA) |
| | | | Perfluoroheptanoic acid (PFHpA) |
| | | | Perfluorooctanoic acid (PFOA) |
| | | | Perfluorononanoic acid (PFNA) |
| | | | Perfluorodecanoic acid (PFDA) |
| | | | Perfluoroundecanoic acid (PFUnA) |
| | | | Perfluorododecanoic acid (PFDoA) |
| | | | Perfluorotributanesulfonic acid (PFTrBS) |
| | | | Perfluorohexanesulfonic acid (PFHxS) |
| | | | Perfluorooctanesulfonic acid (PFOS) |
| | | | Perfluorooctanesulfonamide (PFOSA) |

Table 3. List of multi residue pesticides and polybrominated diphenyl ethers (PBDEs) analyzed as part of this study.

| Multi Residue Pesticides | | | | Polybrominated diphenyl ethers (PBDEs) |
|---------------------------------|----------------------|---------------------|--------------------|---|
| Tecnazene | Nonachlor, cis- | Desethylatrazine | Chlorpyrifos | PBDE-1 PBDE-37 PBDE-166 |
| Hexachlorobenzene | alpha-Endosulphan | Simazine | Fenitrothion | PBDE-2 PBDE-75 PBDE-183 |
| Quintozene | beta-Endosulphan | Atrazine | Malathion | PBDE-3 PBDE-71/49 PBDE-181 |
| Heptachlor | Dieldrin | Ametryn | Parathion-Ethyl | PBDE-10 PBDE-47 PBDE-190 |
| HCH, alpha | 2,4'-DDD | Metribuzin | Chlorpyrifos-Oxon | PBDE-7 PBDE-66 PBDE-202 |
| HCH, gamma | 4,4'-DDD | Cyanazine | Disulfoton Sulfone | PBDE-11 PBDE-77 PBDE-201 |
| HCH, beta | 2,4'-DDE | Hexazinone | Ethion | PBDE-8 PBDE-100 PBDE-204 |
| HCH, delta | 4,4'-DDE | Phorate | Phosmet | PBDE-12 PBDE-119 PBDE-197 |
| Chlorothalonil | 2,4'-DDT | Terbufos | Azinphos-Methyl | PBDE-13 PBDE-99 PBDE-198/199/203/200 |
| Aldrin | 4,4'-DDT | Diazinon-Oxon | Permethrin | PBDE-15 PBDE-116 PBDE-196 |
| Dacthal | Captan | Diazinon | Cypermethrin | PBDE-32 PBDE-118 PBDE-205 |
| Octachlorostyrene | Perthane | Disulfoton | | PBDE-30 PBDE-126 PBDE-194 |
| Chlordane, oxy- | Endrin | Fonofos | | PBDE-17 PBDE-85 PBDE-195 |
| Heptachlor Epoxide | Endosulphan Sulphate | Dimethoate | | PBDE-25 PBDE-155 PBDE-208 |
| Chlordane, gamma | Mirex | Chlorpyrifos-Methyl | | PBDE-33 PBDE-154 PBDE-207 |
| Chlordane, alpha | Methoxychlor | Parathion-Methyl | | PBDE-28 PBDE-153 PBDE-206 |
| Nonachlor, trans- | Endrin Ketone | Pirimiphos-Methyl | | PBDE-35 PBDE-138 PBDE-209 |

et al., 2003; Hardy, 2002) and still continues today for certain congeners. The European Union (EU) banned the use of PentaBDE and OctaBDE commercial mixtures in 2004 (BSEF, 2007) with US chemical manufacturers also voluntarily halting production. As of 2013, both the EU (banned) and the US (voluntarily phased out) have also halted production of DecaBDE.

PBDEs are commonly used in the manufacture of polyurethane foam for mattresses and other padded furniture and carpets, as well as TV casings. Thus, marine debris and improper disposal of household goods can become sources of PBDEs in the marine environment. Atmospheric deposition of PBDEs is also possible through combustion of PBDE containing products. There currently are no established guidelines or thresholds for levels of PBDEs in sediments or tissues. PBDEs have been found to bioaccumulate in aquatic species, especially marine mammals (Johnson et al., 2005).

Bacterial Indicator.

In this study we analyzed the bacterium *Clostridium perfringens* as an indicator of fecal pollution in the marine environment. *C. perfringens* occurs naturally in the intestines of many mammals including humans, dogs, and bats, and is a common cause food borne illnesses. While *C. perfringens* are not a chemical contaminant, it allows us to further quantify land-based sources of pollution entering the Pala Lagoon. The presence of *C. perfringens* in the marine environment can have multiple sources including leaking sewage infrastructure and domestic and wild animals in the watershed.

METHODS

Field collections

Sediment samples (n=13) in the Pala Lagoon SMA were collected using kayaks to reach each site (Figure 2). Sites were randomly selected using ArcGIS within three predetermined strata classified as Upper, Middle, and Lower (Figure 1). Four samples were collected from each area to allow for statistical comparison among strata. An additional targeted sample was collected (TS-1) as an area of interest, though it is not included in the statistical strata comparison analysis for this study. Individual sediment sample collection was achieved using a telescoping pole and custom designed sample jar holder to allow for fresh certified pre-cleaned 250ml IChem jars to collect sediment from each site. All field personnel wore nitrile gloves to avoid contaminating samples. In an effort to quantify loading of land-based sources of pollution entering the Pala Lagoon, surface salinity was measured using a hand-held refractometer at each sediment collection site. Samples were labelled and placed on ice in a cooler until they could be transferred to a freezer at the conclusion of each sampling day. A subset of each sample was placed into a Whirl-pak™ bag for *C. perfringens* and grain size analysis and refrigerated upon the conclusion of each sample day. Samples were shipped under chain-of-custody and arrived in good condition to TDI Brooks International in College Station, TX for analytical services.

All laboratory analysis were performed using protocols from the NS&T Program by TDI-Brooks, International or their subcontractor, AXYS Analytical. Detailed descriptions of NS&T protocols, including quality assurance/quality control (QA/QC) and analytical methods



Figure 2. Field collection at site L-3P, facing the Pago Pago Airport runway fence line.

used can be found in Kimbrough et al., 2006. Laboratory analysis methods specifically for AXYS related analytical results (current use pesticides and human use pharmaceuticals) are proprietary and confidential. The method names used for this study were MLA-035 REV.07.04 and MLA-070 REV.07.04. Contact information for further references is: AXYS Analytical Services Ltd, 2045 Mills Road W., Sidney, BC, Canada, V8L 5X2. Tel. (250) 655-5800, fax (250) 655-5811.

NOAA numerical sediment quality guidelines (SQG) developed by Long and Morgan (1990) and Long et al. (1995), known as Effects Range Median (ERM), and Effects Range-Low (ERL), each express statistically derived contamination levels above which toxic effects can be expected. These guidelines express statistically derived levels of chemical contamination in surficial sediments below which effects to benthic organisms were rarely (<10%) expected (ERL) and above which toxic effects would be expected to be observed with at least a 50% frequency (ERM). The ratio of the ERM value to the sediment concentration for each chemical, or sum of chemicals such as total PAHs, is called the ERM quotient or ERMq (Long et al., 1998). This quotient expresses how close measured concentrations are to the established ERM level on a zero to one scale. A quotient of one or greater means the concentrations are at or above the ERM. This also normalizes the ERMs for different chemicals to a common scale. By averaging the mean ERMq of contaminants it is possible to express a measure of contamination across the entirety of all analytes. Previous studies by Hyland et al. (1999) suggest that mean ERMq values of 0.1 in southeast U.S. coastal waters represent a threshold above which degradation in benthic communities start appearing. The mean quotient of the ERMs and contaminant concentrations have been calculated on a site-by-site basis.

Statistical analysis for this report was conducted using JMP statistical software version 12.1.0. Data from this study were not normally distributed, therefore nonparametric Wilcoxon Signed Rank (Kruskal-Wallis where more than two groups were compared) statistics were used to determine statistical differences among strata and contaminants. An alpha of 0.05 was used for determining significant results.

Results for this study are compared with those from Whitall and Holst (2015) due to the fact that the many of the same chemicals were analyzed using the same methodology and analytical laboratory. The location of Faga'alu is shown in Figure 1 (inset).

RESULTS

Salinity

Mean salinity for the Pala Lagoon was 27.33 ± 9.25 (standard deviation, same throughout the rest of this report) PSU. Maximum salinity of 36 PSU was recorded at site L-4P while minimum salinity of 10 PSU was measured at both M-2P and M-4P (Figure 3).

Lower salinity numbers point toward the potential for increased land-based runoff, though it is important to note that significant rainfall events occur regularly and

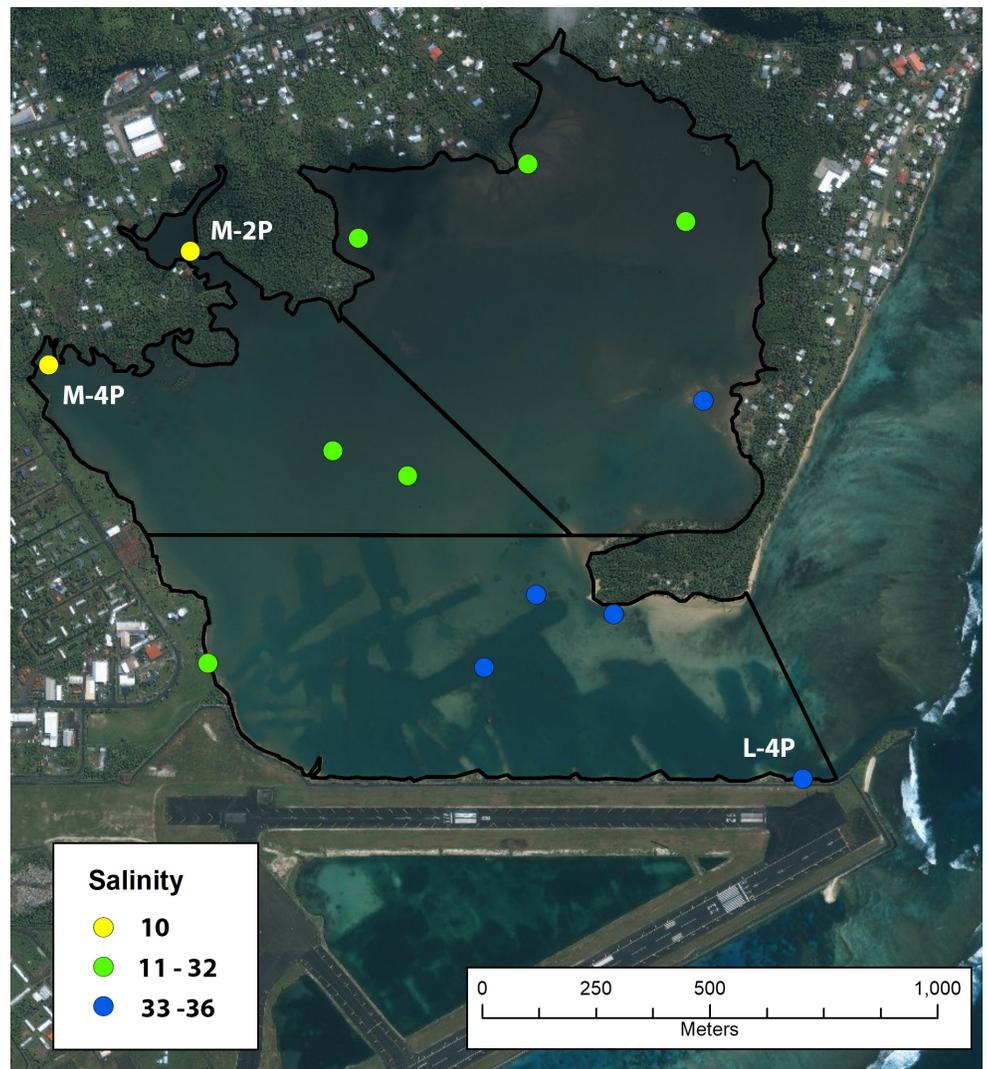


Figure 3. Measured salinity (PSU) during sediment collection.

could potentially affect the salinity results as only surface measurements were collected. That said, the distribution of lower salinity sites are significantly correlated ($\alpha=0.05$) to distance from freshwater inputs into the Pala Lagoon (nonparametric Wilcoxon Signed Rank = 0.0002) suggesting that salinity values that were measured for this study are primarily driven by freshwater runoff and tidal influence instead of direct deposition of rainfall.

Grain Size and Total Organic Carbon (TOC)

Fine grained sediments, such as silt and clay, have higher surface areas and typically higher organic carbon content that increases the adsorption rate of organic chemical contaminants. Conversely, sand and gravel have smaller surface areas and a lower affinity for organic chemical contaminants. As such, organic chemical contaminants tend to accumulate in sediments with high organic carbon content as compared to sediments low in organic carbon. The charge structure on the surface of clay particles also tends to attract and bind metals as well.

Sand and gravel (shell and coralline rubble) were the dominant grain size fraction in the sediment sampled from the Pala Lagoon. In all but two samples, M-2P and U-1P, sand and gravel comprised over 50% of the total content, with a maximum fraction of 97.32% at site L-1P, the site closest to the mouth of the Lagoon. The highest percent fines (%silt + %clay) was measured at site U-1P at 61.47%. A nonparametric Wilcoxon (Rank Sums, $\alpha=0.05$) analysis indicated that percent gravel (shell and coralline rubble) varied significantly among the Lower stratum versus the Middle and Upper strata (Chi Square = 0.0274). There were no other significant differences between other grain size categories and strata.

The mean percent total organic carbon (TOC) in the sediments collected from the Pala Lagoon was $3.05 \pm 2.94\%$, and ranged from a low of 0.9% at site L-4P to a high of 11.56% at site M-2P. There was no significant difference between percent TOC and strata.

Polycyclic Aromatic Hydrocarbons (PAHs)

Total PAHs include the sum of all 64 individual PAHs, sulfur-containing aromatics, and decalins listed in Table 1. The mean concentration for the total PAHs for this study was 68.44 ± 143.71 ng/dry g. The median concentration of total PAHs was 16.47 ng/dry g. The maximum concentration of total PAHs was 506.25 ng/dry g at site M-4P while the minimum concentration of 2.69 ng/dry g was found at site L-2P (nonparametric Wilcoxon Signed Rank, Chi-square > 0.9001, $\alpha=0.05$; Figure 4).

Comparison with other data.

There was no statistically significant difference between the total PAHs measured by Whitall and Holst (2015) in the Faga'alu watershed and bay and those measured in the Pala

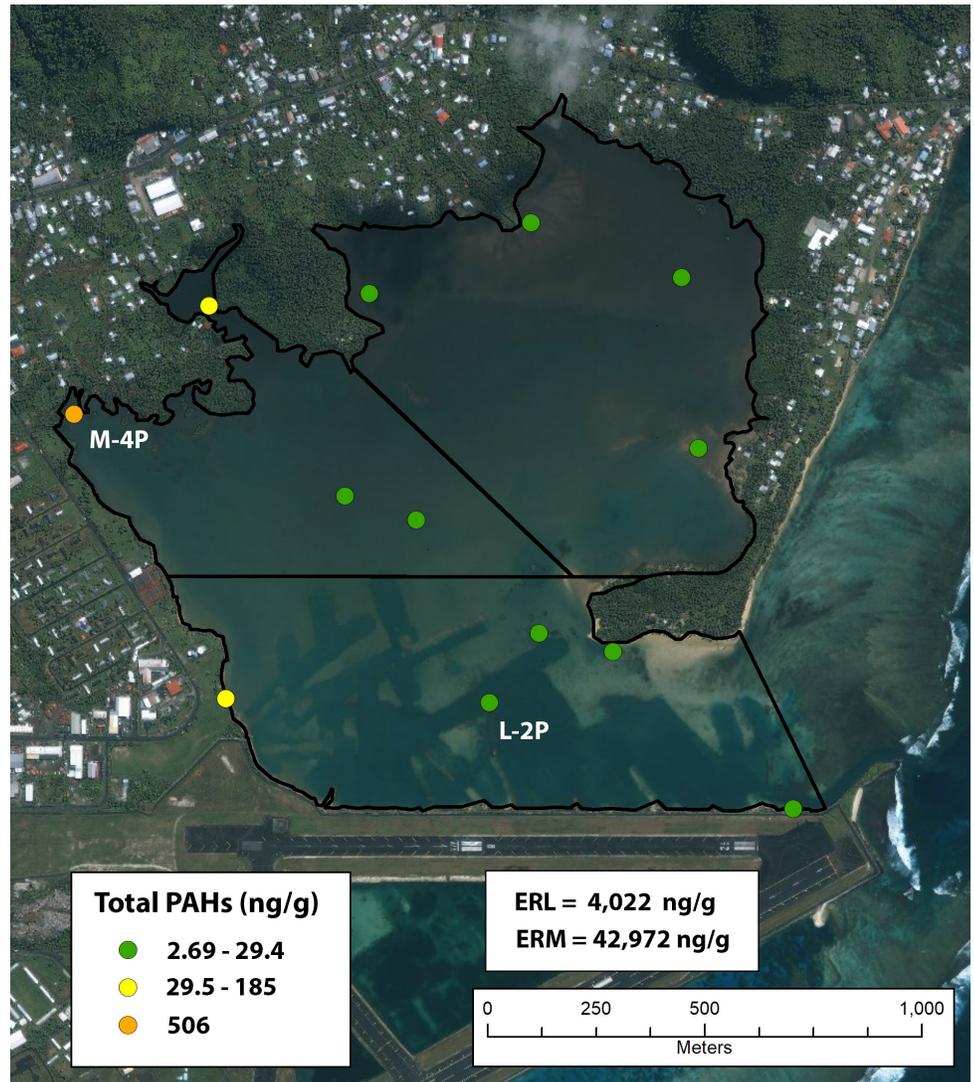


Figure 4. Map of total polycyclic aromatic hydrocarbon (PAH) concentrations (ng/g dry weight) measured in sediment from the Pala Lagoon. ERL = Effects Range Low, ERM = Effects Range Median.

Lagoon in this study (Table 4). The highest measured concentration of total PAHs in sediment from Faga'alu (2,097 ng/dry g) was an order of magnitude higher than the highest concentration measured in the sediment from the Pala Lagoon (506 ng/dry g).

NOAA Sediment Quality Guidelines for Total PAHs.

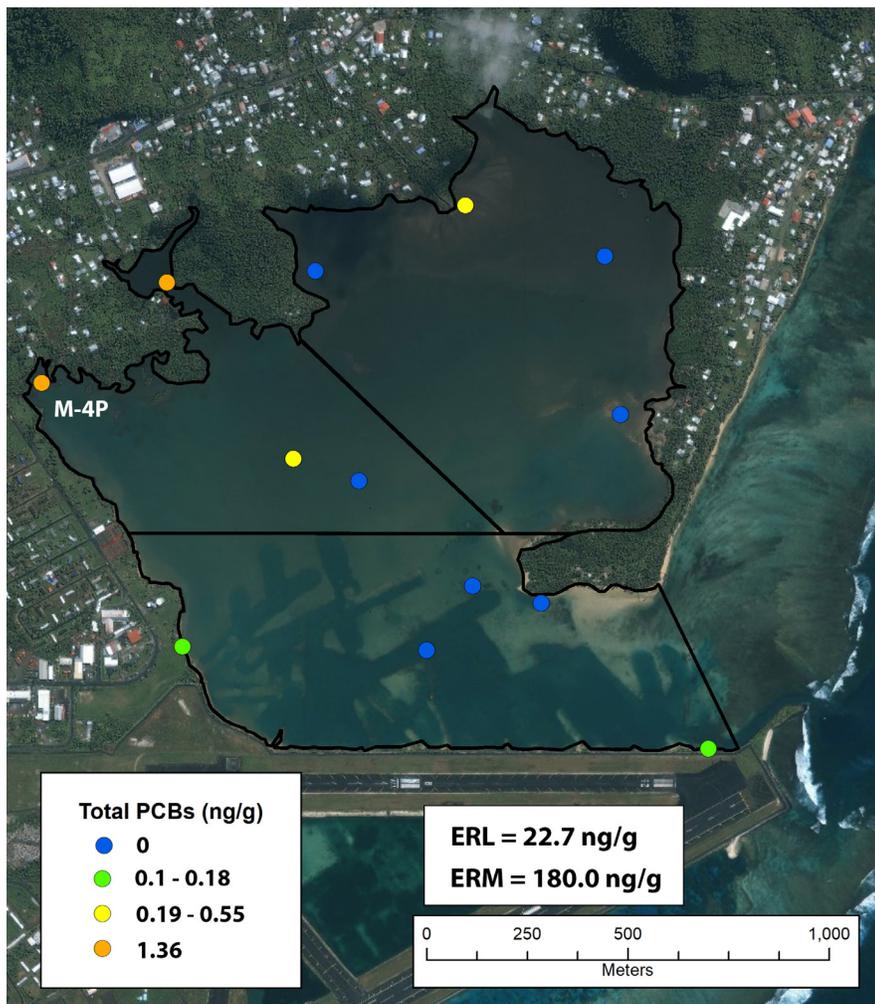
The NS&T Program has developed effects-based, numeric guidelines that allow for the estimation of likely toxic effects of certain sediment contaminants (Long et al., 1998). Values between the ERL and ERM thresholds represent concentrations at which toxic effects occasionally may be observed (NOAA NS&T, 1998). ERL and ERM values for total PAHs (n=64) are shown in Figure 4. No sediment samples from the Nu'uuli Pala Lagoon exceeded the ERL of 4,022 ng/g.

ERL and ERM guidelines have also been calculated for a number of individual PAHs. These guidelines are shown in Table 5. No sediment samples analyzed for this study exceeded any of the ERL or ERM thresholds for individual PAH compounds.

Polychlorinated Biphenyls (PCBs)

Total PCBs analyzed as part of this study refers to the sum of all 81 congeners listed in Table 1. For this study only total PCBs are reported. The mean concentration of total PCBs for the Pala Lagoon was 0.29 ± 0.49 ng/dry g while the median concentration was 0. The maximum concentration for total PCBs in this study was 1.36 ng/dry g at site M-4P while the

multiple sites across the Lagoon had non-detections for total PCBs. There were multiple locations where no PCBs were detected (Figure 5). There were no differences among strata for PCB concentrations.



The NS&T mean concentration for sediments nationwide is 13.7 ng/dry g which is an order of magnitude higher than the highest measured value from the Pala Lagoon. Additionally, NOAA ERL and ERM guidelines for total PCBs (sum = 18 congeners) are 22.7 ng/dry g and 180.0 ng/dry g, respectively, and both are at least an order of magnitude higher than any measured value from the Pala Lagoon.

Comparison with other data.

Total PCBs concentrations measured by Whitall and Holst (2015) in sediment from the Faga'alu watershed and bay were significantly higher than those in the Pala Lagoon (nonparametric Wilcoxon Rank Sums, $\alpha=0.05$, Chi-square > 0.0001; Table 4). The highest concentration of total PCBs from Faga'alu of 92.89 ng/dry g was an order of magnitude greater than the highest measured concentration of total PCBs in sediment from the Pala Lagoon (4.75 ng/dry g).

Dichlorodiphenyltrichloroethane (DDT)

Total DDT for this study represents the

Figure 5. Map of total polychlorinated biphenyl (PCB) concentrations (ng/g dry weight) measured in sediment from the Pala Lagoon. ERL = Effects Range Low, ERM = Effects Range Median.

Table 4. Comparison of chemical contaminants in sediment between the Nu'uuli Pala Lagoon and Faga'alu Bay and watershed. ERL = Effects Range Low.

| Analyte | Nu'uuli Pala Lagoon | Faga'alu | Chi Square |
|------------|---------------------|---------------|------------|
| Aluminum | No difference | No difference | - |
| Iron | No difference | No difference | - |
| Silicon | No difference | No difference | - |
| Arsenic | Higher (8) | Lower (3) | 0.0006 |
| Cadmium | Lower (0) | Higher (0) | 0.0078 |
| Chromium | Higher (6) | Lower (2) | 0.0417 |
| Silver | Lower (0) | Higher (4) | 0.0195 |
| Antimony | No difference | - | - |
| Copper | No difference | - | - |
| Lead | No difference | - | - |
| Manganese | No difference | - | - |
| Mercury | No difference | - | - |
| Nickel | No difference | - | - |
| Selenium | No difference | - | - |
| Tin | No difference | - | - |
| Zinc | No difference | - | - |
| Total PCBs | Lower (0) | Higher (3) | 0.0001 |
| Total PAHs | No difference | - | - |
| Total DDT | No difference | - | - |

Note: Values in parentheses denote number of ERL exceedences

Table 5. Comparison of selected individual PAH concentrations to NOAA National Status and Trends (NS&T) Program historical data and sediment quality guidelines. ERL = Effects Range Low, ERM = Effects Range Median.

| Compound | Sites | | | | | | | | | | | | | NS&T Statistics and Guidelines | | | |
|------------------------|-------|------|------|------|------|------|------|-------|------|------|------|------|------|--------------------------------|-----------------|------|-------|
| | L-1P | L-2P | L-3P | L-4P | M-1P | M-2P | M-3P | M-4P | TS-1 | U-1P | U-2P | U-3P | U-4P | Median | 85th Percentile | ERL | ERM |
| Acenaphthylene | ND | ND | 1.00 | 0.27 | 0.10 | 0.72 | 0.06 | 2.44 | 0.80 | 0.19 | 0.09 | 0.14 | 0.14 | 2.1 | 15.1 | 44 | 640 |
| Anthracene | 0.16 | 0.02 | 0.13 | 0.58 | 0.13 | 1.37 | 0.05 | 5.67 | 1.32 | 0.30 | 0.14 | 0.19 | 0.14 | 3.4 | 38.7 | 85.3 | 1,100 |
| Naphthalene | 0.36 | 0.35 | 0.71 | 0.83 | 0.34 | 3.54 | 0.40 | 10.30 | 0.50 | 1.10 | 0.69 | 0.96 | 0.44 | 3.7 | 27.6 | 160 | 2,100 |
| Benzo-a-pyrene | 0.24 | ND | 0.42 | 1.37 | 0.52 | 5.60 | 0.22 | 16.40 | 7.25 | 0.91 | 0.38 | 0.78 | 0.54 | 14.7 | 127 | 430 | 1,600 |
| Dibenzo(a,h)anthracene | 0.25 | ND | ND | 0.33 | ND | 4.28 | ND | 2.80 | 1.32 | 0.70 | 0.30 | ND | 0.24 | 5 | 23.8 | 63.4 | 260 |

All concentrations are in ng/dry g. ND = Non Detect

sum of 2,4'-DDT, 4,4'-DDT and their metabolites 2,4'-DDE, 4,4'-DDE, 2,4'-DDD, 4,4'-DDD, and 1,1-Bis(p-chlorophenyl)-2-chloroethene (DDMU). The results from this analysis can be seen in Figure 6. The mean concentration of total DDT for the Pala Lagoon was 0.44 ± 1.30 ng/dry g while the median value was 0.02 ng/dry g. The maximum measured concentration of total DDT was 4.75 ng/dry g at site M-4P. There were no statistically significant differences among strata for total DDT concentrations.

The NS&T mean concentration for total DDT in sediments nationwide is 3.11 ng/dry g, which is below only the highest concentration measured in the Pala Lagoon. Site M-4P also exceeded the ERL of 1.58 ng/dry g total DDT, but did not exceed the ERM of 46.1 ng/dry g. The ratio of DDT to DDE and DDD, one way to estimate how degraded DDT is in the environment, was 72% DDT, 8% DDE, and 20% DDD at site M-4P. Though this suggests a relatively 'fresh' source of DDT entering the Pala Lagoon, in the marine environment DDT and its metabolites degrade very slowly, on the order of decades (Chattopadhyay and Chattopadhyay, 2015). Additionally, the relatively low concentrations measured, along with the low organic carbon content of sediments in the Pala Lagoon limit the ability to make more precise conclusions as to the relative age of the total DDT present.

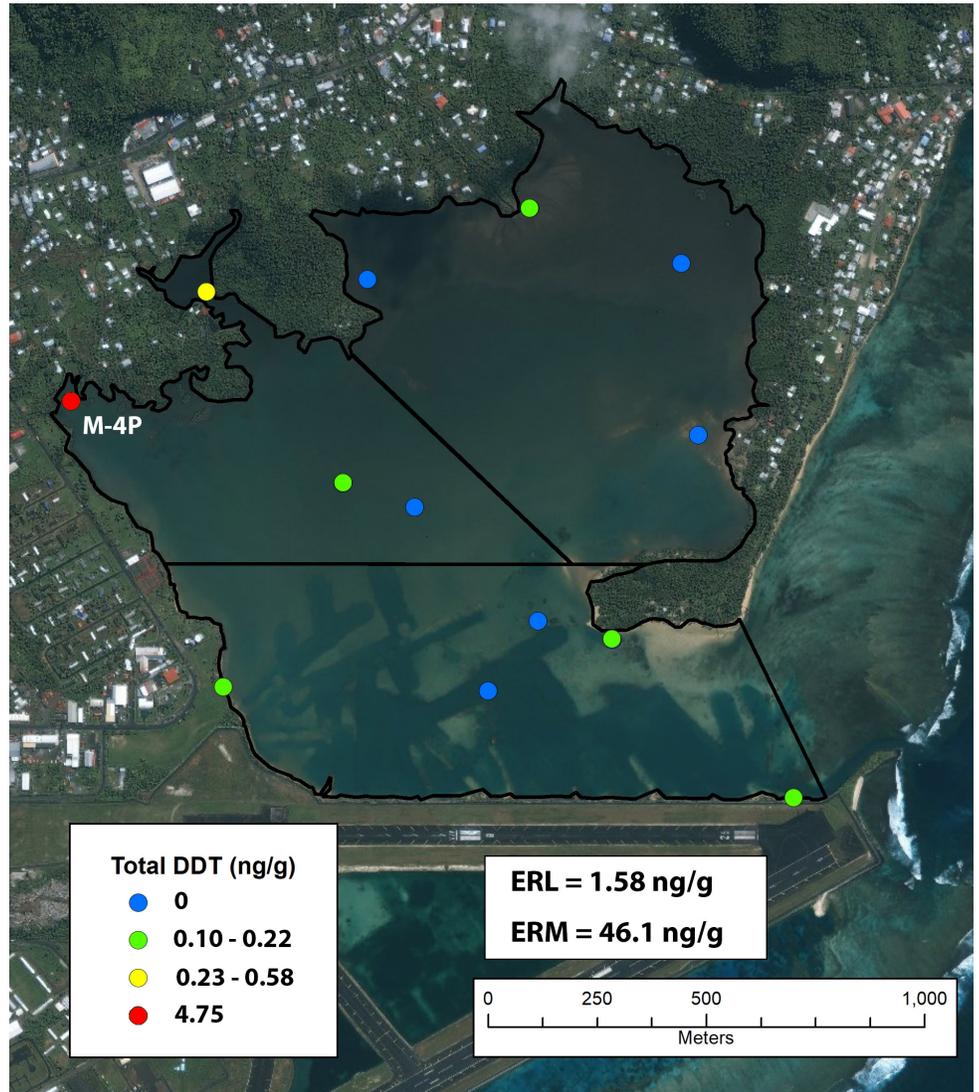


Figure 6. Map of total DDT concentrations (ng/g dry weight) measured in sediment from the Pala Lagoon. ERL = Effects Range Low, ERM = Effects Range Median.

Comparison to other data.

There were no statistically significant differences between total DDT concentrations in sediment from the Pala Lagoon and those measured by Whitall and Holst (2015) in the watershed and bay of Faga'alu (nonparametric Wilcoxon Signed Rank, Chi-square > 0.8102, $\alpha=0.05$; Figure 4). The highest concentration of total DDT in Faga'alu (2.29 ng/dry g) was comparable to the highest concentration of total DDT measured in the Pala Lagoon (1.36). An additional 24 individual organochlorine pesticides were analyzed as part of this study (Table 1). However, there were no detections of any of these chemicals and no additional summary of their potential impacts is included in this report.

Polybrominated Diphenyl Ethers (PBDEs)

As discussed previously, PBDEs are a class of flame retardant chemicals that are common contaminants in the environment. PBDEs preferentially bind to organic containing sediment, and therefore the low amount of TOC measured in sediment from the Pala Lagoon (high of 11.56% at site M-2P; see previous section on TOC) points toward a similarly low ability for sediments in the Lagoon to trap and accumulate PBDEs. Even so, relatively high levels of PBDEs, as compared

to historical NS&T data for the coastal US (Kimbrough et al., 2009) were measured in the Pala Lagoon.

The mean concentration of total PBDEs (sum of 51 congeners) for this study was 10.87 ± 25.50 ng/dry g while the median concentration was 3.22 ng/dry g. The maximum concentration measured was 94.99 ng/dry g at site M-4P while it was not detected in sediment at site L-1P. The maximum value at site M-4P was the 4th highest concentration ever measured by the NS&T Program (Kimbrough et al., 2009) for locations including coastal Alaska, Hawai'i, the continental Pacific coast, the continental Atlantic coast, the Gulf of Mexico, and in the Great Lakes. Figure 7 shows the comparison of total PBDE concentra-

tions from this study and the NS&T historical database. Other than total PBDEs measured at site M-4P, concentrations of PBDEs in sediment in the rest of the Pala Lagoon appear to be low. When compared to mean PBDE data from around the world (sum of subset of 8 congeners) compiled by Zhang et al. (2016), the median concentration for the Pala Lagoon (2.42 ng/dry g for the same subset of 8) falls well below the higher means for China and Korea, but are comparable to those recorded for the Philippines and the south and central San Francisco Bay in 2007 (Table 6), an urban area with approximately 7.15 million people (US Census Bureau, 2013). As of 2010, the population estimate for all of American Samoa, including all outlying islands, was 67,380 (ASG, 2011), or roughly 0.94% the population of the San Francisco Bay area.

Multi Residue Pesticides, Human Use Pharmaceuticals, and Perfluorinated Compounds

Multi residue pesticides, human use pharmaceuticals, and perfluorinated compounds represent a diverse range of organic contaminants that only recently have begun to be measured in the marine environment. This group of chemicals for this study included 141 individual human use pharmaceuticals and personal care products, 62 individual current use and legacy pesticides, 13 perfluorinated compounds, and 51 multi residue pesticides (Tables 2 and 3). Currently there are no sediment guidelines or thresholds determining environmentally relevant concentrations or concentrations at which potentially adverse effects may occur to marine organisms. Given the lack of guidelines, for this study we simply report the concentrations measured. Due to the high cost of analysis, only a subset of the thirteen total collected samples were analyzed for these organic contaminants, six for perfluorinated compounds and three of those six for human use pharmaceuticals and multi residue pesticides.

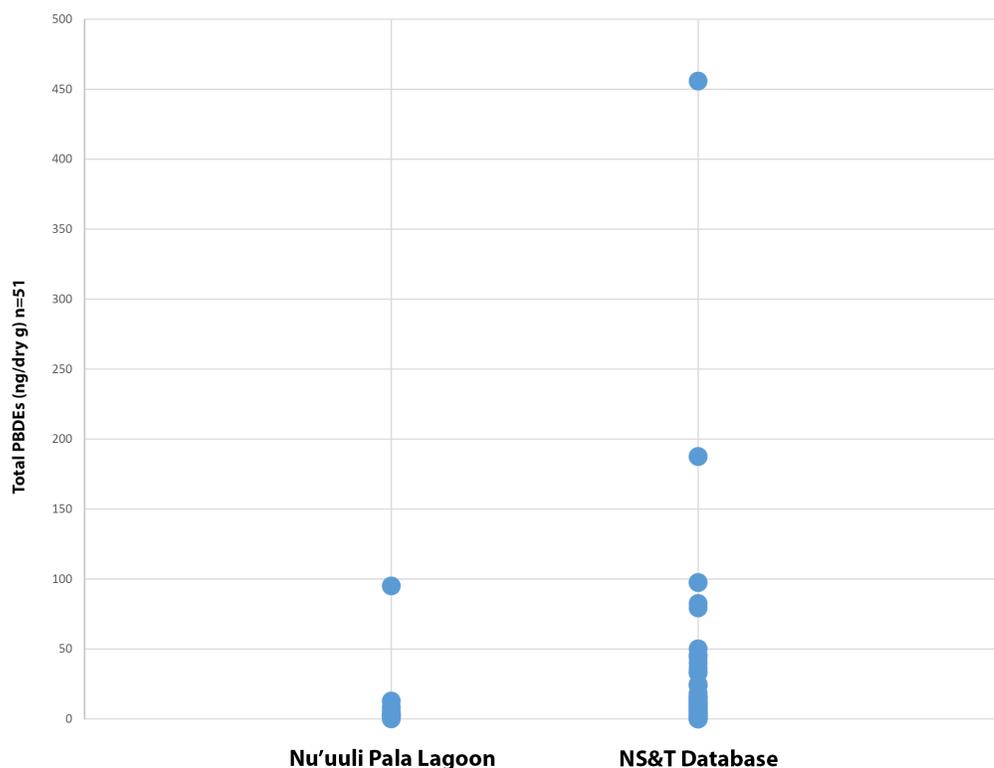


Figure 7. Comparison of all PBDE data (n=51) from both the Nu'uuli Pala Lagoon and the NS&T database. NS&T database includes Mussel Watch historical sites.

Table 6. Comparison of worldwide median PBDE (n=8) concentrations in sediments with the median concentration of PBDEs (n=8) in sediment from Nu'uuli. MWP = NOAA Mussel Watch Program.

| Region | Median (ng/g) | Source |
|------------------------|---------------|-----------------------|
| Macao, China | 26.85 | Zhang et al. 2016 |
| Hong Kong, China | 7.56 | Zhang et al. 2016 |
| Ulsan Bay, South Korea | 31.17 | Zhang et al. 2016 |
| Busan Bay, South Korea | 77.5 | Zhang et al. 2016 |
| Tokyo, Japan | 46 | Zhang et al. 2016 |
| Philippines | 2.36 | Zhang et al. 2016 |
| San Francisco Bay area | 3.84 | Zhang et al. 2016 |
| Coastal US (MWP) | 0.2 | Kimbrough et al. 2009 |
| Nu'uuli (2017) | 2.42 | -- |

Table 7. Results from multi residue pesticide, human use pharmaceutical, and perfluorinated compound analysis for sediment from the Nu'uuli Pala Lagoon. All concentrations in ng/dry g. DEET = N,N-Diethyl-meta-toluamide, PFOS = Perfluorooctanesulfonic acid, PFOA = Perfluorooctanoic acid, HCH = hexachlorocyclohexane, N/A = Not analyzed

| Site | Amphetamine | Triclocarban | DEET | Clotrimazole | PFOS | PFOA |
|------|-------------|--------------|------|--------------|-------|-------|
| L-3P | N/A | N/A | N/A | N/A | -- | -- |
| M-2P | 2.84 | -- | 15.7 | 2.5 | -- | -- |
| M-4P | 1.87 | 14.8 | 8.12 | 10.7 | 0.337 | -- |
| U-1P | 1.72 | -- | 4.07 | 0.674 | -- | -- |
| U-2P | N/A | N/A | N/A | N/A | 0.223 | -- |
| U-4P | N/A | N/A | N/A | N/A | 0.318 | 0.107 |

| Site | Chlorpyrifos | Beta-Endosulfan | HCH, alpha | Chlordane, alpha | Nonachlor, trans- | Dieldrin |
|------|--------------|-----------------|------------|------------------|-------------------|----------|
| L-3P | 0.035 | -- | -- | -- | -- | -- |
| M-2P | -- | 1.22 | -- | -- | -- | -- |
| M-4P | 0.058 | -- | 0.01 | 0.251 | 0.332 | 0.026 |
| U-1P | -- | -- | -- | -- | -- | -- |
| U-2P | -- | -- | -- | -- | -- | -- |
| U-4P | -- | -- | -- | -- | -- | -- |

A summary of all these results are included in Table 7. Of the total 299 additional chemicals analyzed for this study, only twelve were detected in sediment from the Pala Lagoon. These were amphetamine, triclocarban, DEET, clotrimazole, PFOS, PFOA, chlorpyrifos, beta-endosulfan, alpha-hexachlorocyclohexane (HCH), alpha chlordane, trans-nonachlor, and dieldrin. Amphetamine, triclocarban, and clotrimazole are all human use pharmaceuticals while DEET is a well-known insect repellent. Amphetamine is central nervous system stimulant pharmaceutical while triclocarban is an antibacterial chemical and a common component of antibacterial soaps. Clotrimazole is an antifungal drug commonly included in many over-the-counter antifungal products. The presence of these chemicals in the sediments of the Pala Lagoon are most likely indicators of the presence of both grey water (non-sewage) and human waste reaching the marine environment of the Pala Lagoon. The presence of the insect repellent DEET is also not surprising in a tropical environment where wide spread usage of mosquito repellent is common. The presence of both PFOS and PFOA, an environmentally persistent class of chemicals found in products such as Teflon™ and Scotchgard™, is also not surprising due to their high solubility in water which can lead to significant transport via surface and ground waters.

Chlorpyrifos is a current use pesticide commonly used in commercial agriculture and may still be in use on the island of Tutuila. Beta-endosulfan, alpha-HCH, alpha-chlordane, trans-nonachlor, and dieldrin are all banned pesticides that are classified as persistent organic pollutants that do not easily breakdown in the environment. The presence of these chemicals in the sediment of the Pala Lagoon is likely from past usage prior to the individual bans that currently exist, and not due to ongoing application.

The three sites selected for the full suite of chemical analysis were chosen as they were deemed to be the sites most likely to contain measurable levels of organic contaminants (based on preliminary grain size and TOC analysis along with their proximity to freshwater inputs to the lagoon). The fact that so few chemicals were detected in sediments may be a factor of the low organic content of the sediments instead of a complete lack of these chemicals in the Pala Lagoon. A follow up study of fish or other marine animals (invertebrates), or an analysis of the dissolved phase of the water in the Pala Lagoon could further shed light on the presence or absence of a larger suite of CECs in the environment.

Trace and Major Elements

Detailed results for aluminum, iron, and select elements that had elevated concentrations are included below. Summary results for all sixteen trace and major elements are included in Table 8.

Aluminum, Iron, and Silicon

The island of Tutuila is comprised of five Pliocene-to-Pleistocene volcanoes, with the central part of the island dominated by a basaltic-to-andesitic shield volcano (Addison et al., 2006; Macdonald, 1968). Weathering of basaltic volcanic rock produces oxisolic soils, which are low in mineral content but high in iron and aluminum oxides. In locations with large amounts of rainfall, such as American Samoa, silicate clays are known to leach from basaltic volcanic rock and produce poor soil fertility. Andisetic deposits, derived from ash and cinder accretion, also have high aluminum and iron content (HDOH, 2012).

Based on the geology of the island of Tutuila, we expect to see high amounts of aluminum, iron, and silicon in marine sediment due to erosion. A comparison of the correlation between these and other trace metals can be used to determine approximately

how much of a given metal's presence in marine sediments can be attributed to crustal erosion. Because of these relationships, poor correlations between aluminum, iron, and silicon and other trace metals can point to potential anthropogenic sources behind elevated concentrations. For this study, although silicon had the highest number of significant correlations with all the other trace metals (R-square > 0.70), based on previous established methods, aluminum was selected for estimating the amount of erosional influence on trace metal concentrations (Table 9). Iron showed almost identical patterns to aluminum, but with slightly less significant results. The correlation between aluminum and iron had an adjusted R-Square value of greater than 0.8949 for sediment in the Pala Lagoon, indicating a very close relationship between the two crustal metals. More detailed information for aluminum, iron, and silicon are included below.

The mean concentration of aluminum in sediment in the Pala Lagoon was $25,428.46 \pm 23,619.38$ $\mu\text{g/g}$ while the median concentration was 14,800 $\mu\text{g/g}$. The maximum concentration of aluminum was 68,400 $\mu\text{g/g}$ at site M-4P and the minimum concentration was 1,160 $\mu\text{g/g}$ at site L-2P (Figure 8).

Table 9. Correlations between trace and major elements for this study. Green = high correlation (R-square > 0.70), Orange = low correlation (R-square < 0.70).

| | Al | Fe | Si |
|----|------|------|------|
| Al | -- | 0.89 | 0.97 |
| As | 0.37 | 0.47 | 0.26 |
| Cd | 0.35 | 0.09 | 0.41 |
| Cr | 0.80 | 0.89 | 0.83 |
| Cu | 0.62 | 0.32 | 0.71 |
| Fe | 0.89 | -- | 0.84 |
| Hg | 0.35 | 0.25 | 0.31 |
| Mn | 0.79 | 0.90 | 0.75 |
| Ni | 0.35 | 0.37 | 0.46 |
| Pb | 0.34 | 0.12 | 0.49 |
| Sb | 0.49 | 0.24 | 0.54 |
| Si | 0.97 | 0.84 | -- |
| Sn | 0.71 | 0.40 | 0.77 |
| Zn | 0.96 | 0.77 | 0.97 |

Table 8. Summary data for trace and major elements sampled for this study. All units in $\mu\text{g/g}$. ND = Non-detection.

| Element | Mean | Standard Dev | Median | Max |
|----------------|---------|--------------|---------|---------|
| Aluminum (Al) | 25,428 | 23,619 | 14,800 | 68,400 |
| Antimony (Sb) | 0.286 | 0.239 | 0.24 | 0.874 |
| Arsenic (As) | 12.0 | 6.7 | 13.0 | 24.1 |
| Cadmium (Cd) | 0.036 | 0.090 | 0 | 0.286 |
| Chromium (Cr) | 94.1 | 90.2 | 47.2 | 276 |
| Copper (Cu) | 12.73 | 13.31 | 7.67 | 50.50 |
| Iron (Fe) | 39,910 | 39,053 | 19,400 | 115,000 |
| Lead (Pb) | 6.47 | 6.61 | 3.16 | 24.70 |
| Manganese (Mn) | 385 | 333 | 269 | 1,040 |
| Mercury (Hg) | 0.01240 | 0.01480 | 0.00827 | 0.05600 |
| Nickel (Ni) | 44.2 | 42.7 | 25.8 | 123.0 |
| Selenium (Se) | ND | ND | ND | ND |
| Silicon (Si) | 55,765 | 51,839 | 29,000 | 162,000 |
| Silver (Ag) | ND | ND | ND | ND |
| Tin (Sn) | 2.19 | 2.55 | 1.03 | 9.55 |
| Zinc (Zn) | 101.6 | 105.3 | 48.1 | 332 |

The mean concentration of iron in sediment in the Pala Lagoon was $39,910.77 \pm 39,053.37$ $\mu\text{g/g}$ with a median concentration of 19,400 $\mu\text{g/g}$. The maximum concentration of iron of 115,000 $\mu\text{g/g}$ was measured in sediment at site U-1P while the minimum concentration of 3,890 $\mu\text{g/g}$ was measured at site L-2P.

For silicon, the mean concentration for the Pala Lagoon was $55,765.38 \pm 51,838.91$ $\mu\text{g/g}$ with a median concentration of 29,000 $\mu\text{g/g}$. The maximum concentration of 162,000 $\mu\text{g/g}$ was measured at site M-4P while the minimum concentration of 3,580 $\mu\text{g/g}$ was measured at site L-2P.

Comparison with other data.

There were no statistically significant differences between aluminum, iron, or silicon concentrations measured in the sediment from the Pala Lagoon and those measured by Whitall and Holst (2015) for the Faga'alu watershed and bay (Table 4). This points to comparable levels of erosion between the two bays. The means, standard deviations, and

Table 10. Summary statistics for select sediment contaminants in the Nu'uuli Pala Lagoon and Faga'alu watershed and bay. Faga'alu data from Whitall and Holst (2015).

| Analyte | Faga'alu n=17 | Nu'uuli Pala Lagoon n=13 |
|-----------------------|------------------------------|------------------------------|
| TOC % | 1.35 ± 0.81 (2.85) | 3.05 ± 2.94 (11.56) |
| Total PAHs ng/dry g | 14.35 ± 501.25 (2,097) | 77.34 ± 141.26 (506) |
| Total PCBs ng/dry g | 14.35 ± 29.06 (92.89) | 0.44 ± 1.30 (4.75) |
| Total DDT ng/dry g | 0.23 ± 0.54 (2.29) | 0.29 ± 0.49 (1.36) |
| Aluminum µg/g | 25,683 ± 28,816 (72,400) | 25,428 ± 23,619 (68,400) |
| Arsenic µg/g | 4.44 ± 2.90 (11.5) | 12.04 ± 6.67 (24.1) |
| Cadmium µg/g | 0.10 ± 0.09 (0.31) | 0.04 ± 0.09 (0.29) |
| Chromium µg/g | 39.47 ± 46.42 (191) | 101.91 ± 89.49 (276) |
| Copper µg/g | 8.53 ± 9.67 (37.7) | 13.74 ± 13.38 (50.5) |
| Iron µg/g | 28,484 ± 29,827 (103,000) | 39,911 ± 39,053 (115,000) |
| Nickel µg/g | 35.13 ± 50.66 (211) | 44.21 ± 42.66 (123) |
| Silver µg/g | 0.49 ± 0.81 (2.74) | Non-detect |
| Zinc µg/g | 109.69 ± 119.72 (416) | 101.58 ± 105.35 (332) |
| C. perfringens (CFUs) | 301.88 ± 432.26 (1,722) | 258.96 ± 359.98 (1,183.8) |

*Values in paranthesis represent maximum measured concentrations

Comparison with other data.

Arsenic concentrations measured in the Pala Lagoon were significantly higher than those measured by Whitall and Holst (2015) in the watershed and bay of Faga'alu (nonparametric Wilcoxon Rank Sums, $\alpha=0.05$, Chi-square > 0.0006; Table 4). Despite the significant difference between the two waterbodies, the highest concentration of arsenic in sediments from the Pala Lagoon (24.1 µg/g) is comparable with the highest concentration of arsenic measured in sediment from the Faga'alu watershed and bay (11.5 µg/g; Table 10). Arsenic concentrations in the Pala Lagoon were also slightly higher than those measured in Pago Pago Harbor (CH2MHill, 2007), which had concentrations ranging from 4.54 to 14.58 µg/g and a mean of 10.46 µg/g.

Chromium

Concentrations of chromium in sediment from the Pala Lagoon ranged from a maximum of 276 µg/g at site U-1P to a minimum of 0.4 µg/g at site L-2P. The mean concentration of chromium was 94.11 ± 90.19 µg/g with a median concentration of 47.2 µg/g. Six of the thirteen samples exceeded the ERL sediment guideline threshold of 81 µg/g (Figure 10).

maximum concentrations for both aluminum and iron in both waterbodies are presented in Table 10.

Arsenic

Arsenic is a naturally occurring metal in the earth's crust, though anthropogenic influences such as mining have served to concentrate and increase the amount present in the environment. The mean concentration of arsenic for this study was 12.04 ± 6.68 µg/g and a median concentration of 13.00 µg/g. The maximum concentration of arsenic in sediment from the Pala Lagoon was 24.1 µg/g at site U-1P while the minimum concentration was 2.82 µg/g at site L-1P. Eight of the thirteen sites sampled for arsenic exceeded the ERL of 8.2 µg/g (Figure 9) indicating potential adverse effects at those sites. None of the sites sampled exceeded the ERM of 70 µg/g.

A bivariate analysis of arsenic and aluminum showed a significant relationship ($F > 0.0160$), however the adjusted R-square value, or in this case an estimate of how much of the variation in concentrations of arsenic could be attributed to crustal erosion (aluminum) was low at only 0.3711.

Additionally, a non-parametric Wilcoxon test (Rank Sums) showed that arsenic concentrations in the Upper and Middle Pala Lagoon are significantly higher than those of the Lower Pala Lagoon (Chi-Square > 0.0335).

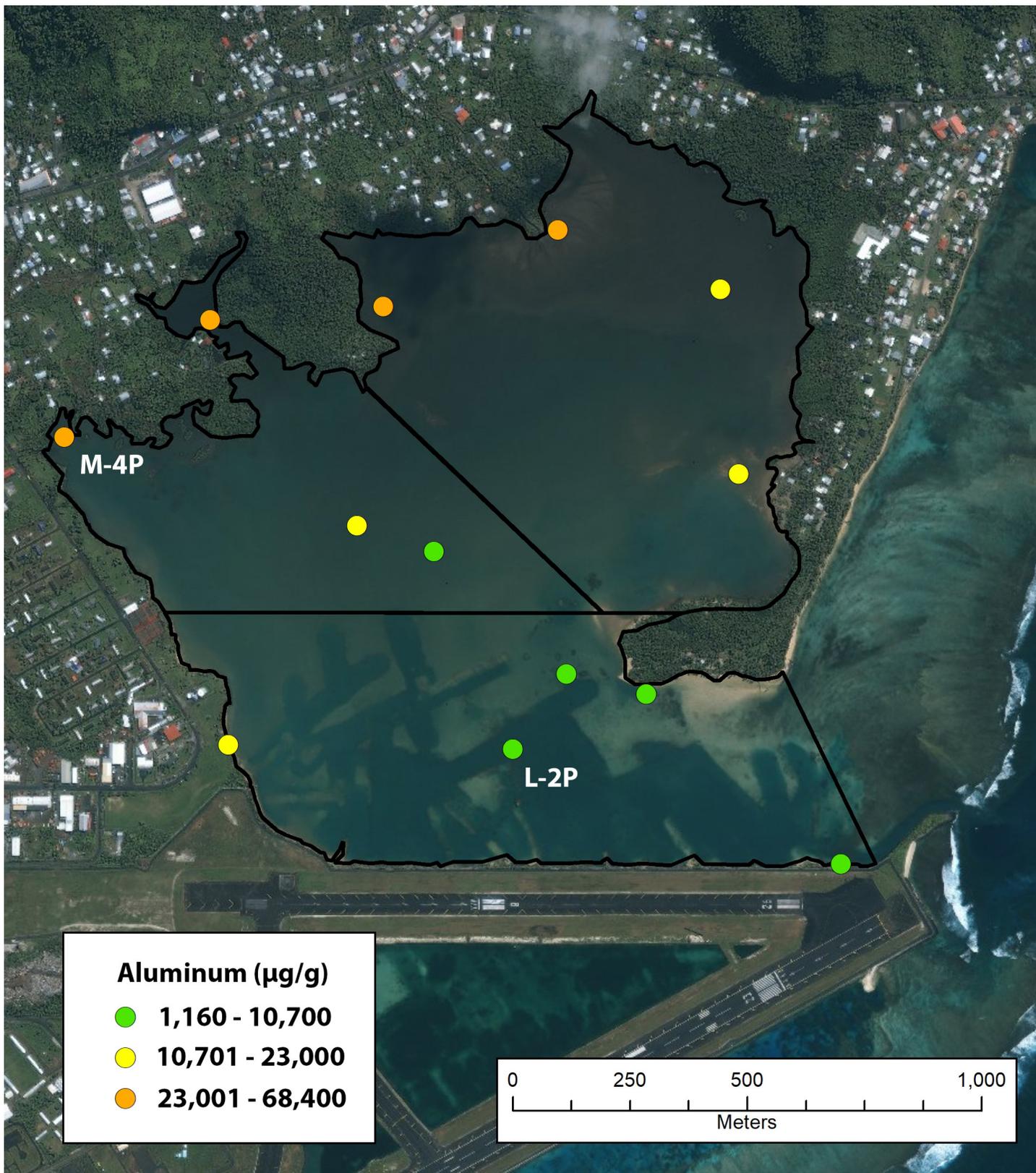


Figure 8. Aluminum concentrations ($\mu\text{g/g}$ dry weight) in sediment from the Nu'uuli Pala Lagoon.

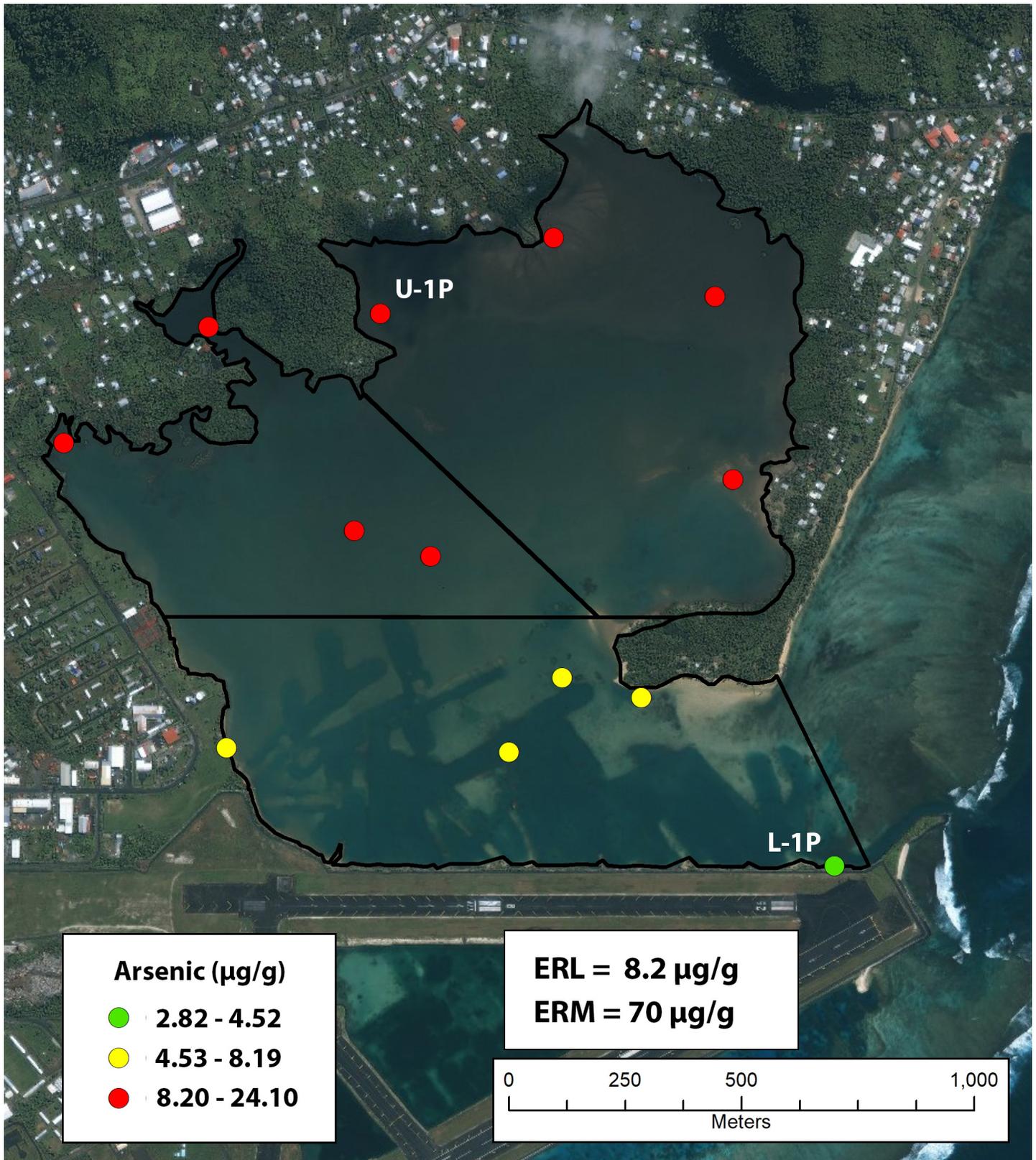


Figure 9. Arsenic concentrations ($\mu\text{g/g}$ dry weight) in sediment from the Nu'uuli Pala Lagoon. Sites in red exceed the Effects Range Low (ERL). ERM = Effects Range Median.

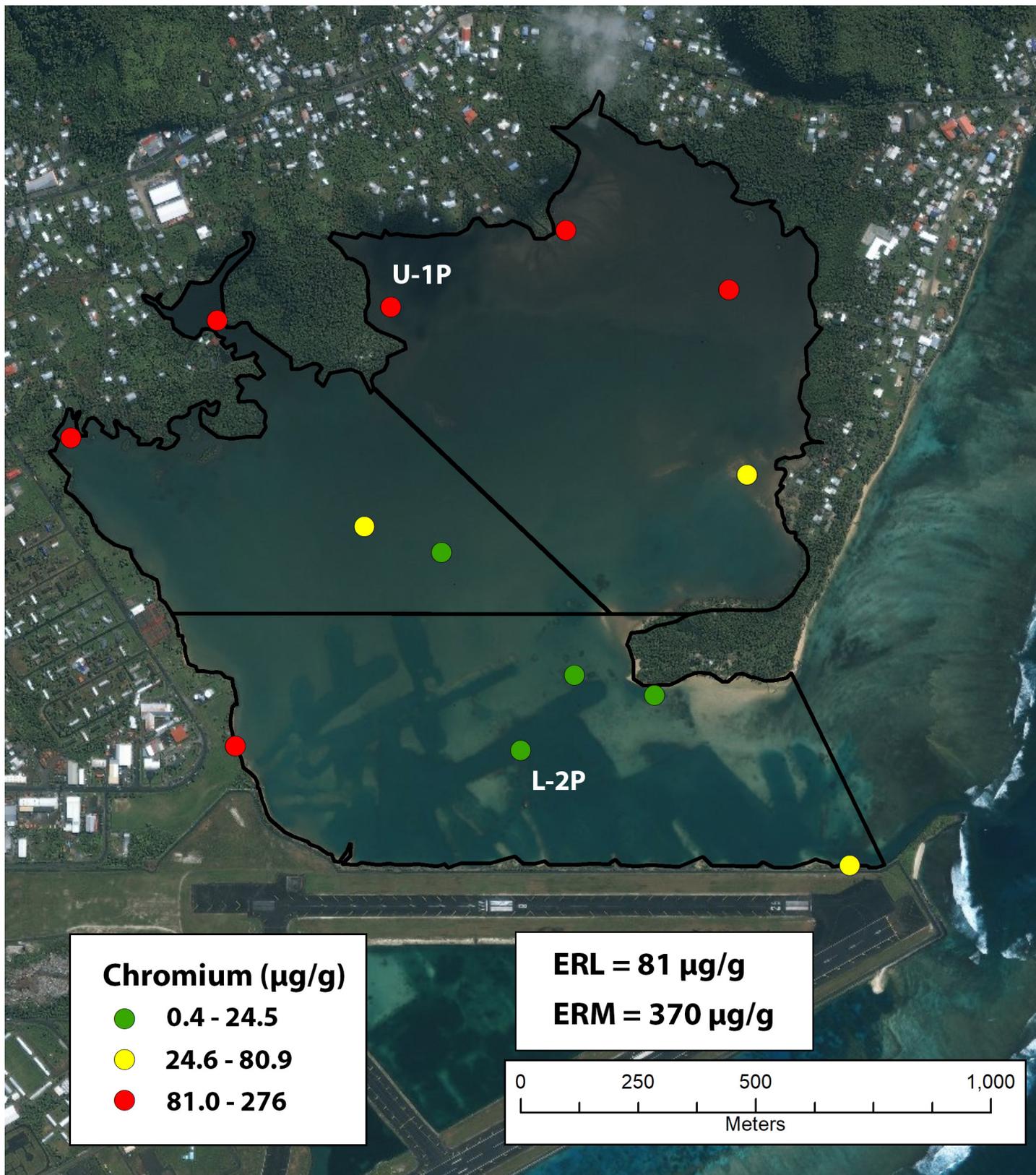


Figure 10. Chromium concentrations ($\mu\text{g/g}$ dry weight) in sediment from the Nu'uuli Pala Lagoon. Sites in red exceed the Effects Range Low (ERL). ERM = Effects Range Median.

None of the samples exceeded the ERM.

A bivariate analysis of chromium and aluminum showed a strong significant correlation ($F > 0.0001$) with an adjusted R-square value of 0.8029, indicating that the majority of chromium measured in the sediment in the Pala Lagoon could be the result of erosion.

A non-parametric Wilcoxon Rank Sums analysis showed that when the Upper and Middle stratum are combined they are significantly higher ($\alpha=0.05$, Chi-Square > 0.0272) than the Lower Pala Lagoon. There are no statistically significant differences between all three strata when analyzed individually.

Comparison with other data.

Chromium concentrations measured in the Pala Lagoon were significantly higher than those measured by Whitall and Holst (2015) in the watershed and bay of Faga'alu (nonparametric Wilcoxon Rank Sums, $\alpha=0.05$, Chi-square > 0.0417 ; Table 4). Despite the significant difference between the two waterbodies, the highest concentration of chromium in sediments from the Pala Lagoon (276 $\mu\text{g/g}$) is comparable with the highest concentration of arsenic measured in sediment from the Faga'alu watershed and bay (191 $\mu\text{g/g}$; Table 10). Chromium concentrations in the Pala Lagoon were also higher than those measured in the sediment of the Pago Pago Harbor (CH2MHill, 2007), which had a mean concentration of 56.03 $\mu\text{g/g}$ and a range of 17.9 to 85.7 $\mu\text{g/g}$.

Cadmium

The mean concentration of cadmium in sediment from the Pala Lagoon was 0.04 ± 0.09 $\mu\text{g/g}$ with a median concentration of 0 $\mu\text{g/g}$. The maximum concentration of 0.29 $\mu\text{g/g}$ was measured at site M-4P. Cadmium was only detected at two sites within the Pala Lagoon, M-4P and M-2P (Figure 11). No sediment samples exceeded the ERL or ERM for cadmium. Despite having only two detections, a bivariate analysis of cadmium concentrations versus aluminum showed a significant correlation ($F > 0.0188$), though the adjusted R-square value was low at 0.3542 indicating that the cadmium measured in the sediment of the Pala Lagoon likely is not from crustal erosion. There are no statistically significant differences among strata for cadmium concentrations in sediment.

Comparison with other data.

Cadmium concentrations measured by Whitall and Holst (2015) in sediment from the Faga'alu watershed and bay were significantly higher than those measured in the Pala Lagoon (nonparametric Wilcoxon Rank Sums, $\alpha=0.05$, Chi-square > 0.0078 ; Table 4). Despite the significant difference between the two waterbodies, the highest concentration of cadmium in sediments from the Pala Lagoon (0.29 $\mu\text{g/g}$) is comparable with the highest concentration of cadmium measured in sediment from the Faga'alu watershed and bay (0.31 $\mu\text{g/g}$; Table 10).

Copper

The mean concentration of copper in the Pala Lagoon sediments was 12.73 ± 13.31 $\mu\text{g/g}$ with a median concentration of 7.67 $\mu\text{g/g}$. The maximum concentration of copper of 50.5 $\mu\text{g/g}$ was measured at site M-4P while the minimum concentration of 0.65 $\mu\text{g/g}$ was measured at site L-2P. Only one of the thirteen sediment samples exceeded the ERL (Figure 12) and none exceeded the ERM. A bivariate analysis of copper and aluminum showed a statistically significant correlation ($F > 0.0008$) with an adjusted R-square value of 0.6248. There are no statistically significant differences among strata for copper.

Comparison with other data.

There were no statistically significant differences between copper concentrations measured in the sediment from the Pala Lagoon and those measured by Whitall and Holst (2015) for the Faga'alu watershed and bay (Table 4). The highest concentration of copper in sediment from the Faga'alu watershed and bay (37.7 $\mu\text{g/g}$) is also comparable to the highest concentration measured in sediment from the Pala Lagoon (50.5 $\mu\text{g/g}$; Table 10). Sediment copper concentrations in the Pago Pago Harbor (CH2MHill, 2007) were an order of magnitude higher than those measured in sediment from this study. Pago

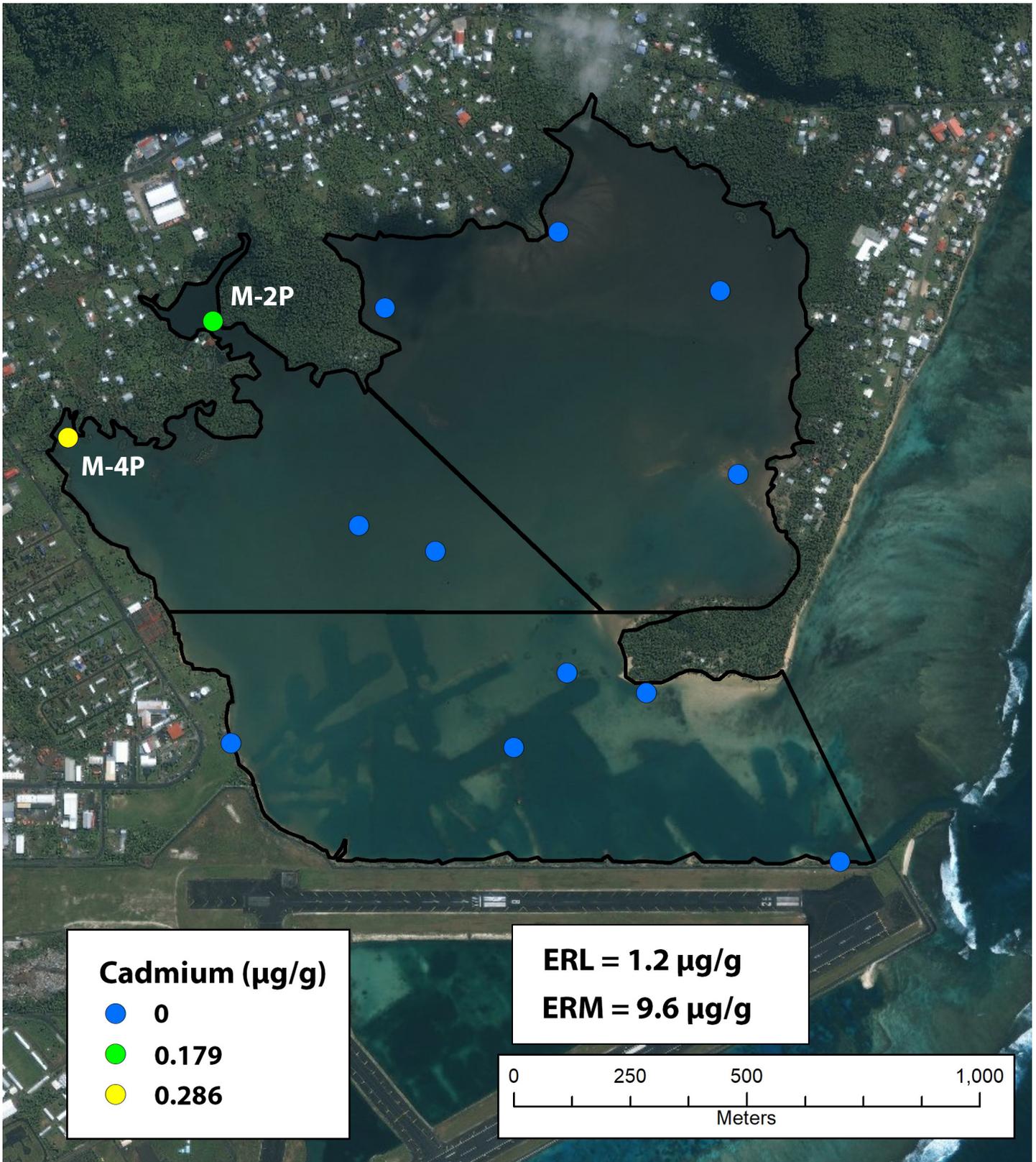


Figure 11. Cadmium concentrations (µg/g dry weight) in sediment from the Nu'uuli Pala Lagoon. ERL = Effects Range Low, ERM = Effects Range Median.

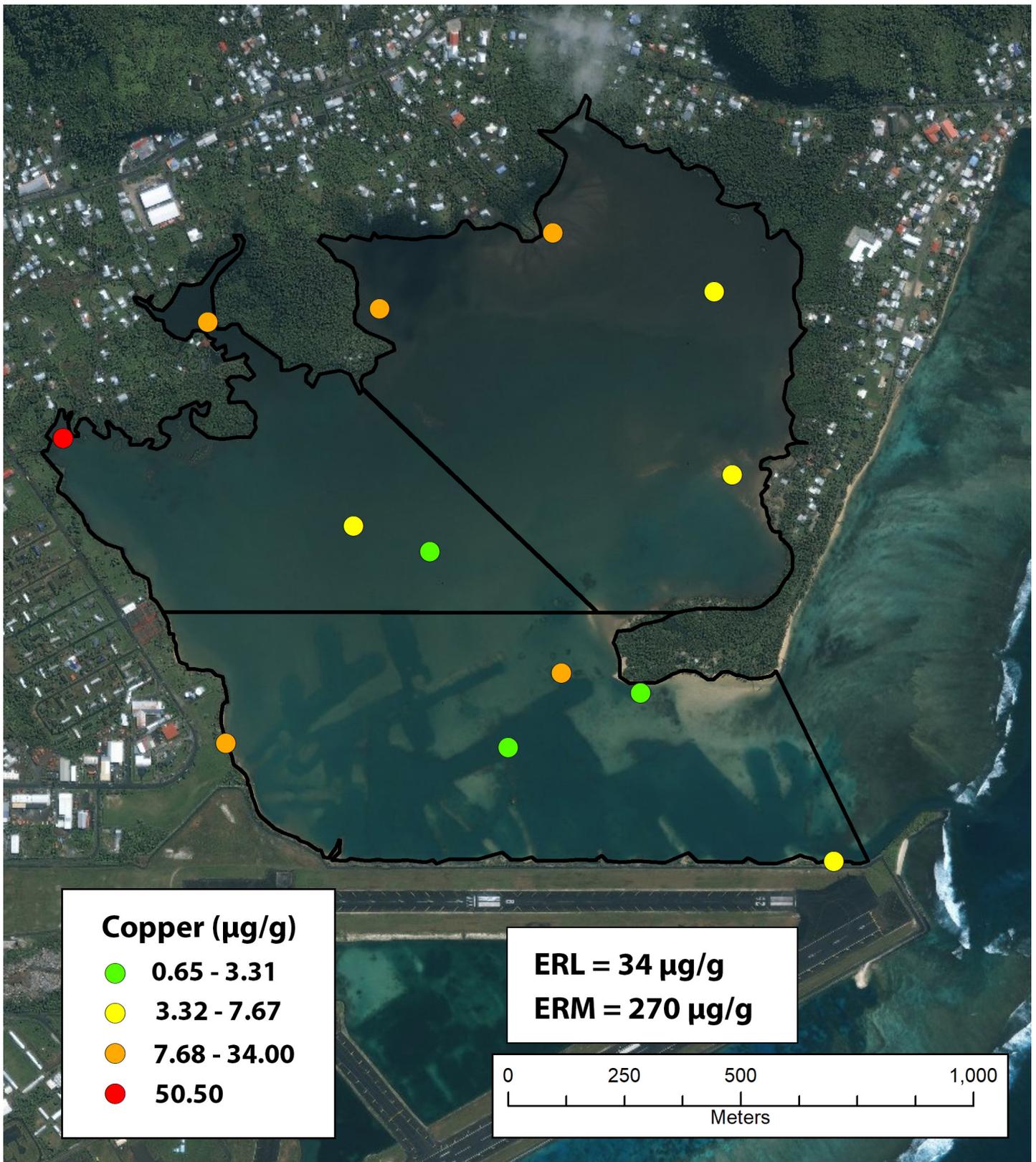


Figure 12. Copper concentrations ($\mu\text{g/g}$ dry weight) in sediment from the Nu'uuli Pala Lagoon. Sites in red exceed the Effects Range Low (ERL). ERM = Effects Range Median.

Pago Harbor copper concentrations ranged from 4.82 to 564 $\mu\text{g/g}$ with a mean concentration of 211.3 $\mu\text{g/g}$. The higher copper concentrations in sediment from the Pago Pago Harbor are not surprising due to the amount of boat traffic in that area as compared to the Pala Lagoon, with copper being a common component of anti-fouling boat paints.

Nickel

Concentrations of nickel in the sediment from the Pala Lagoon ranged from a maximum of 123 $\mu\text{g/g}$ at site TS-1 and a minimum of 2.53 $\mu\text{g/g}$ at site L-2P. The mean concentration of copper was 44.21 ± 42.66 $\mu\text{g/g}$ with a median concentration of 25.8 $\mu\text{g/g}$. Five sites exceeded the ERL for nickel while an additional four sites exceeded both the ERL and the ERM (Figure 13). A bivariate analysis of nickel and aluminum showed a statistically significant correlation ($F > 0.0191$), however the adjusted R-square (0.3525) indicates that the majority of nickel in the Pala Lagoon is not likely related to crustal erosion. There are no significant differences between any strata for nickel concentrations.

Comparison with other data.

There were no statistically significant differences between nickel concentrations measured in the sediment from the Pala Lagoon and those measured by Whitall and Holst (2015) for the Faga'alu watershed and bay (nonparametric Wilcoxon Rank Sums, $\alpha=0.05$, Chi-square > 0.3909 ; Table 4). The highest concentration of nickel in sediment from the Faga'alu watershed and bay (211 $\mu\text{g/g}$) is also comparable to the highest concentration measured in sediment from the Pala Lagoon (123 $\mu\text{g/g}$; Table 10). Nickel concentrations in the Pala Lagoon were slightly higher than those measured in sediment from the Pago Pago Harbor (CH2MHill, 2007) which had a mean of 40.2 $\mu\text{g/g}$ and a range of 10.4 to 62.5 $\mu\text{g/g}$.

Zinc

The mean concentration of zinc in sediment from the Pala Lagoon was 101.58 ± 105.35 $\mu\text{g/g}$ with a median concentration of 48.1 $\mu\text{g/g}$. The maximum concentration of 332 $\mu\text{g/g}$ was measured at site M-4P while the minimum concentration of 6.52 $\mu\text{g/g}$ was measured at site L-2P. Four of the thirteen sediment samples analyzed for zinc exceeded the ERL of 150 $\mu\text{g/g}$ (Figure 14). No samples exceeded the ERM.

A bivariate analysis of zinc concentrations versus aluminum showed a very high significant correlation ($F > 0.0001$) with an adjusted R-square value of 0.9626 indicating that the majority of zinc in the sediment of the Pala Lagoon potentially driven by erosion.

A non-parametric Wilcoxon Rank Sums analysis of logNormal zinc concentrations showed a statistically significant difference between the combined Upper/Middle strata and the Lower strata ($\alpha=0.05$, Chi Square > 0.0107), where the combined Upper/Middle strata was significantly higher than the Lower strata.

Comparison with other data.

There were no statistically significant differences between sediment from the Pala Lagoon and those measured by Whitall and Holst (2015) for the Faga'alu watershed and bay for zinc concentrations (Table 4). The highest concentration of zinc in sediment from the Faga'alu watershed and bay (416 $\mu\text{g/g}$) is also comparable to the highest concentration measured in sediment from the Pala Lagoon (332 $\mu\text{g/g}$; Table 10). Concentrations of zinc measured in the sediment from Pago Pago Harbor (CH2MHill, 2007) were higher than those measured in the Pala Lagoon. The Pago Pago Harbor sediment had zinc concentrations that ranged from 33.4 to 1,810 $\mu\text{g/g}$ and a mean concentration of 778.9 $\mu\text{g/g}$.

Bacterial Indicator

The bacterial indicator *Clostridium perfringens*, a surrogate for measuring human and animal waste inputs to the environment, was detected in every sediment sample collected in from the Pala Lagoon. The mean concentration of *C. perfringens* was 258.96 ± 359.98 colony forming units per gram (CFU/g) with a median concentration of 119.6 CFU/g. The maximum concentration of *C. perfringens* of 1,183.8 CFU/g was measured at site M-2P, located just south of the Laufou shopping center, while the minimum concentration of 9.1 CFU/g was measured at site L-1P, located at the north side of the end of the Pago Pago airport runway (Figure 15).

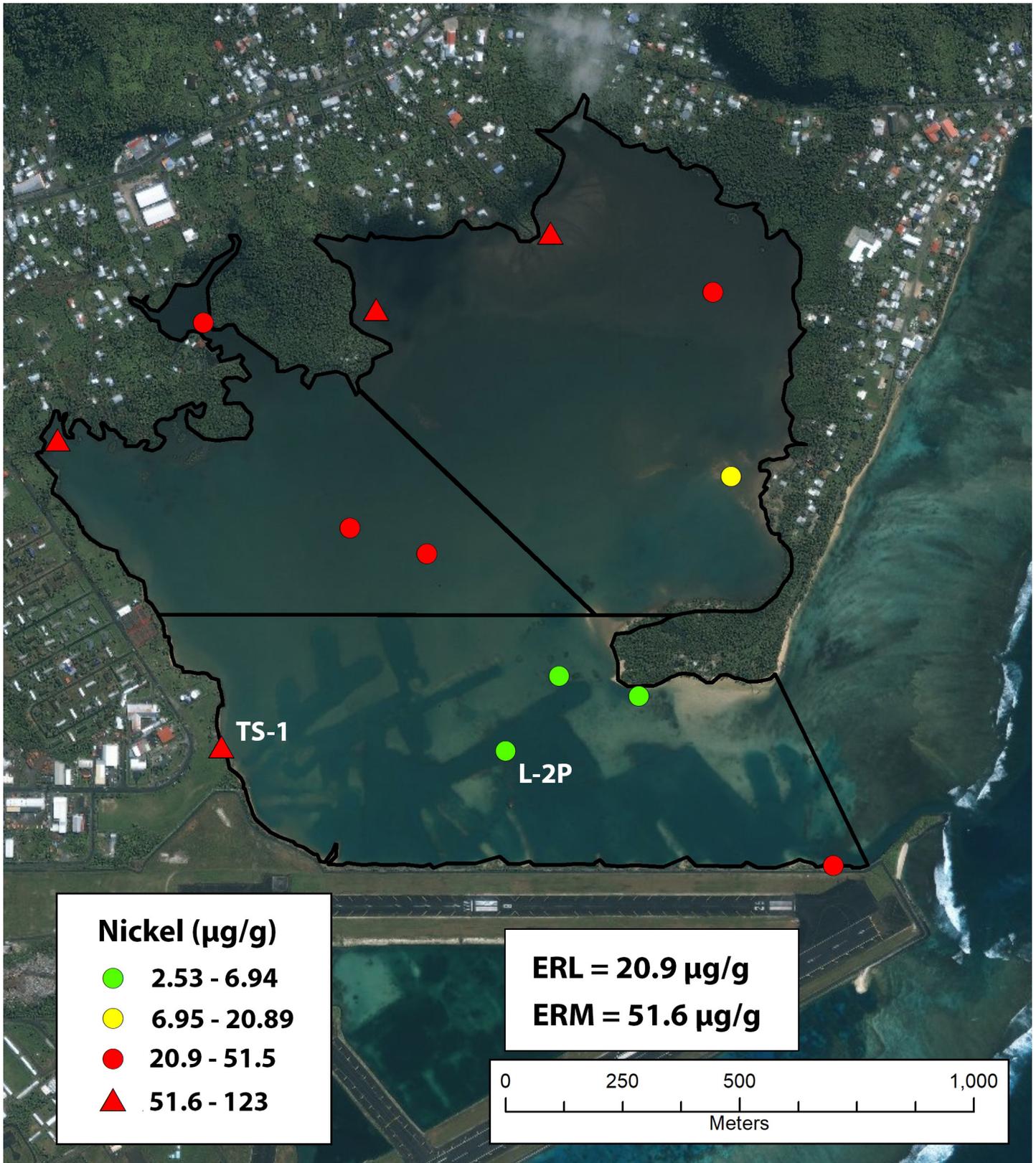


Figure 13. Nickel concentrations ($\mu\text{g/g}$ dry weight) in sediment from the Nu'uuli Pala Lagoon. Sites in red exceed the Effects Range Low (ERL). Sites denoted by triangles exceed the Effects Range Median (ERM).

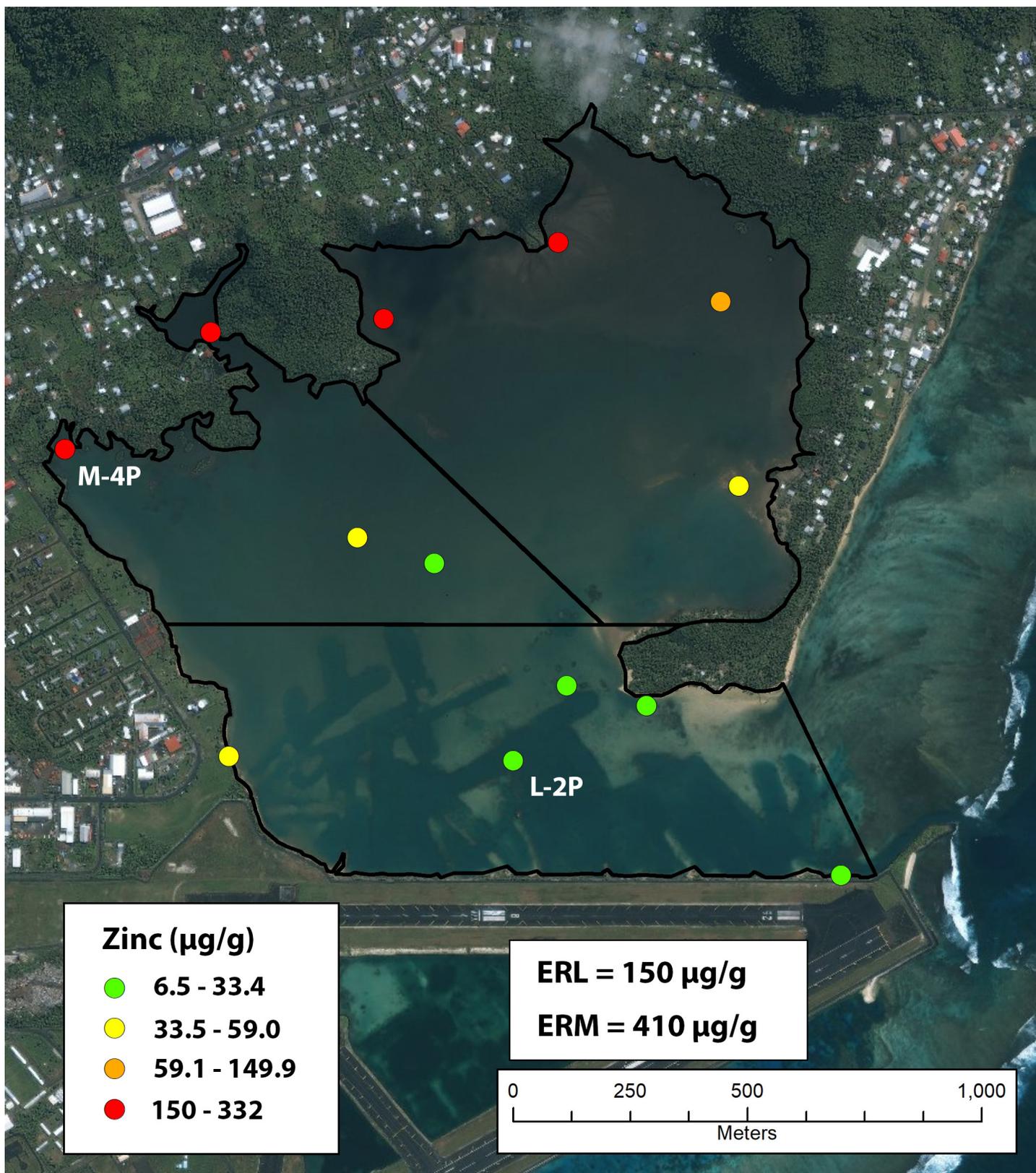


Figure 14. Zinc concentrations ($\mu\text{g/g}$ dry weight) in sediment from the Nu'uuli Pala Lagoon. Sites in red exceed the Effects Range Low (ERL). ERM = Effects Range Median.

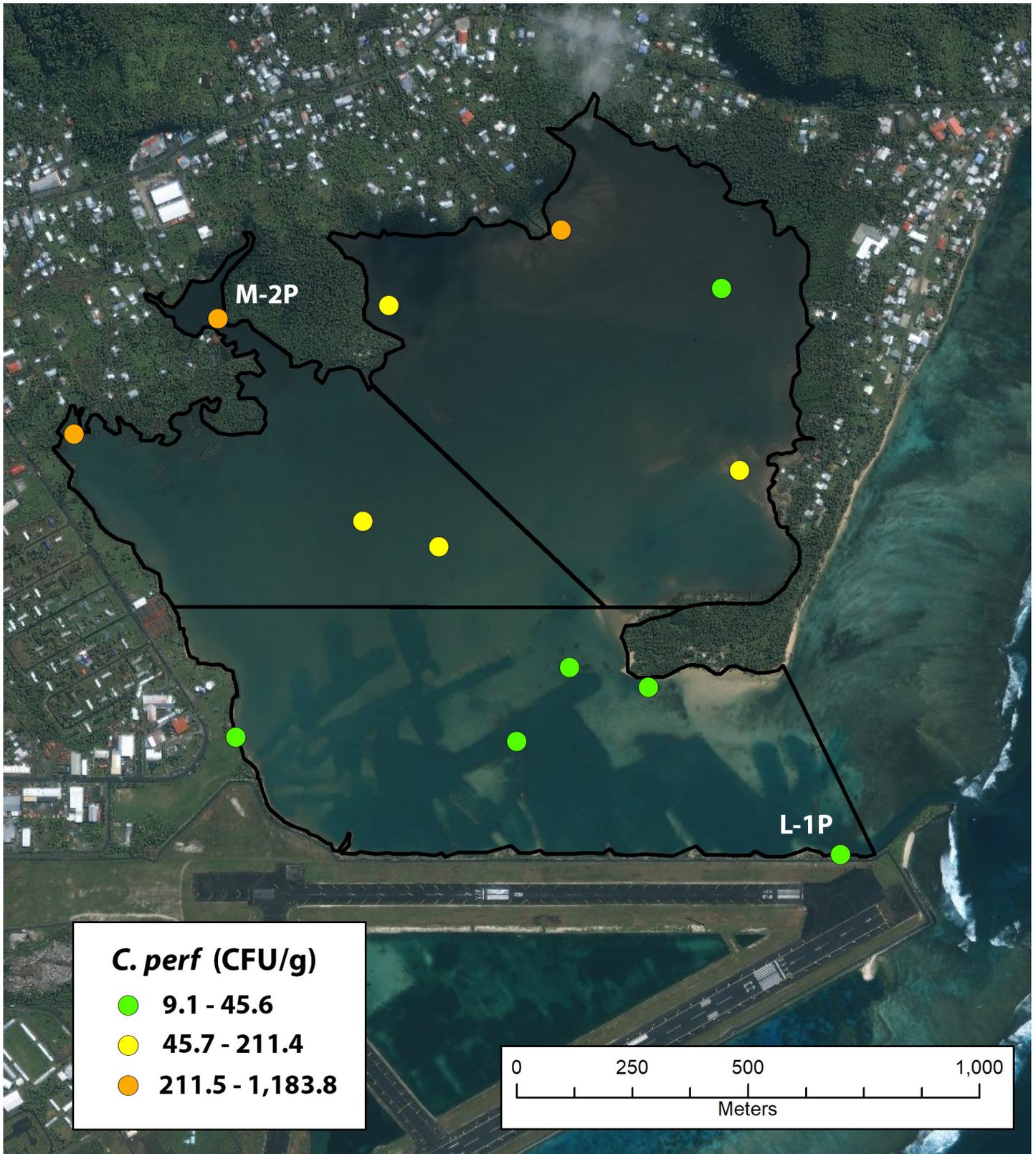


Figure 15. Measured colony forming units (CFU) per gram of *Clastridium perfringens* (*C. perf*) in sediment from the Nu'uuli Pala Lagoon.

A bivariate analysis of *C. perfringens* versus distance from freshwater inputs shows a significant correlation ($F > 0.0152$, $\alpha=0.05$), but the adjusted R-square value is low at 0.3765. This points toward non-point source (i.e. stream runoff, ground water) based sources of potential human and animal waste entering the Pala Lagoon.

Comparison with other data.

There was no statistically significant difference in *C. perfringens* concentrations between this study and that conducted by Whitall and Holst (2015) in the Faga'alu watershed and bay. The maximum measured amount of *C. perfringens* from Faga'alu was higher than that measured in the Pala Lagoon (1,722 CFU/g) though that concentration was measured from the watershed of Faga'alu and not from the bay. The maximum concentration in the Faga'alu bay was 639 CFU/g (Whitall and Holst, 2015) located at their site NB14, or North Bay 14. This is comparable to the amount of *C. perfringens* measured in sediment in the Pala Lagoon at site M-4P (located at the north end of Lions Park) and at site U-3P (the farthest northeast site sampled), but well below the 1,183.8 CFU/g measured at site M-2P (just south of the Laufou shopping center).

The amount of *C. perfringens* present in the Pala Lagoon seems to be elevated as compared to national level sediment data collected by NOAA's NS&T program with the highest concentration of *C. perfringens* found in the Pala Lagoon is in the top 10% of all measured *C. perfringens* samples. That said, these overall values for the Pala Lagoon appear to be typical for what has been measured in other coral coastal environments (Whitall et al., 2014; Whitall and Holst, 2015).

CONCLUSIONS

Overall, concentrations of organic contaminants in sediment from the Nu'uuli Pala Lagoon are low as compared to other studies conducted by NOAA's National Status and Trends (NS&T) Program, and are similar to those measured in the Faga'alu watershed and bay. The only organic contaminants measured in the Pala Lagoon that exceeded any known guidelines or thresholds was for total DDT, and then only the Effects Range Low (ERL). That said, there are currently no established guidelines for the flame retardant class of chemicals that comprise PBDEs. PBDEs in the Pala Lagoon appear to be elevated as compared to other relatively lower population coastal US areas (see above results section for PBDEs). While PBDE levels in the Pala Lagoon are not high compared to some of the most highly contaminated areas, such as Busan Bay, South Korea, concentrations of PBDEs in sediments in the Pala Lagoon are comparable to levels measured in other heavily populated areas in the continental US, such as the San Francisco Bay area. The fact that the relatively low population of the Nu'uuli Pala Lagoon watershed are generating similar levels of PDBEs as those of much more highly populated areas could be an area of potential concern. As PBDEs are often associated with flame retardants in furniture and other household goods, the reduction of bulk trash and other marine debris to the Pala Lagoon could potentially help mitigate future loading of PBDEs to the marine environment. Because of the persistent and ubiquitous nature of PBDEs, any management actions may take decades to have measureable effects on sediment concentrations of PBDEs in the Pala Lagoon.

The levels of multi residue pesticides, human use pharmaceuticals, and perfluorinated compounds appear to be low. Because many of the 299 chemicals measured as part of this analysis are polar, adhesion to the relatively low organic carbon containing sediments in the Pala Lagoon may not be occurring at appreciable levels. The fact that any of these organic contaminants are measureable in the sediment of the Pala Lagoon points toward the potential presence of additional organic contaminants at lower levels that may not be detectable in sediment. A potential follow up study of either the dissolved phase of marine waters or of aquatic organisms, such as fish or invertebrates, could provide further context and information on the presence or absence, and relative abundance of these chemicals that do not tend to accumulate in sediment. Levels of legacy organic contaminants, including PAHs, PCBs, and DDT, appear to be low and not currently a concern for the Pala Lagoon. A qualitative pattern that appeared across almost all contaminants for this study, including both organic and inorganic compounds, was higher levels of contaminants at or near the sources of freshwater to the Pala Lagoon. Site M-4P, located near the north end of Lions Park, represented 60% of all maximum values measured in the Pala Lagoon (Figure 16). No maximum values for any measured chemical contaminant occurred in the Lower stratum, with all but five of the maximum measured concentrations occurring in the Middle stratum (sites M-4P and M-2P).

The concentrations of trace and major elements had a number of exceedances of established guidelines (NOAA's Effects Range Low (ERL) and Effects Range Median (ERM)), including arsenic, chromium, copper, nickel, and zinc. Nickel was

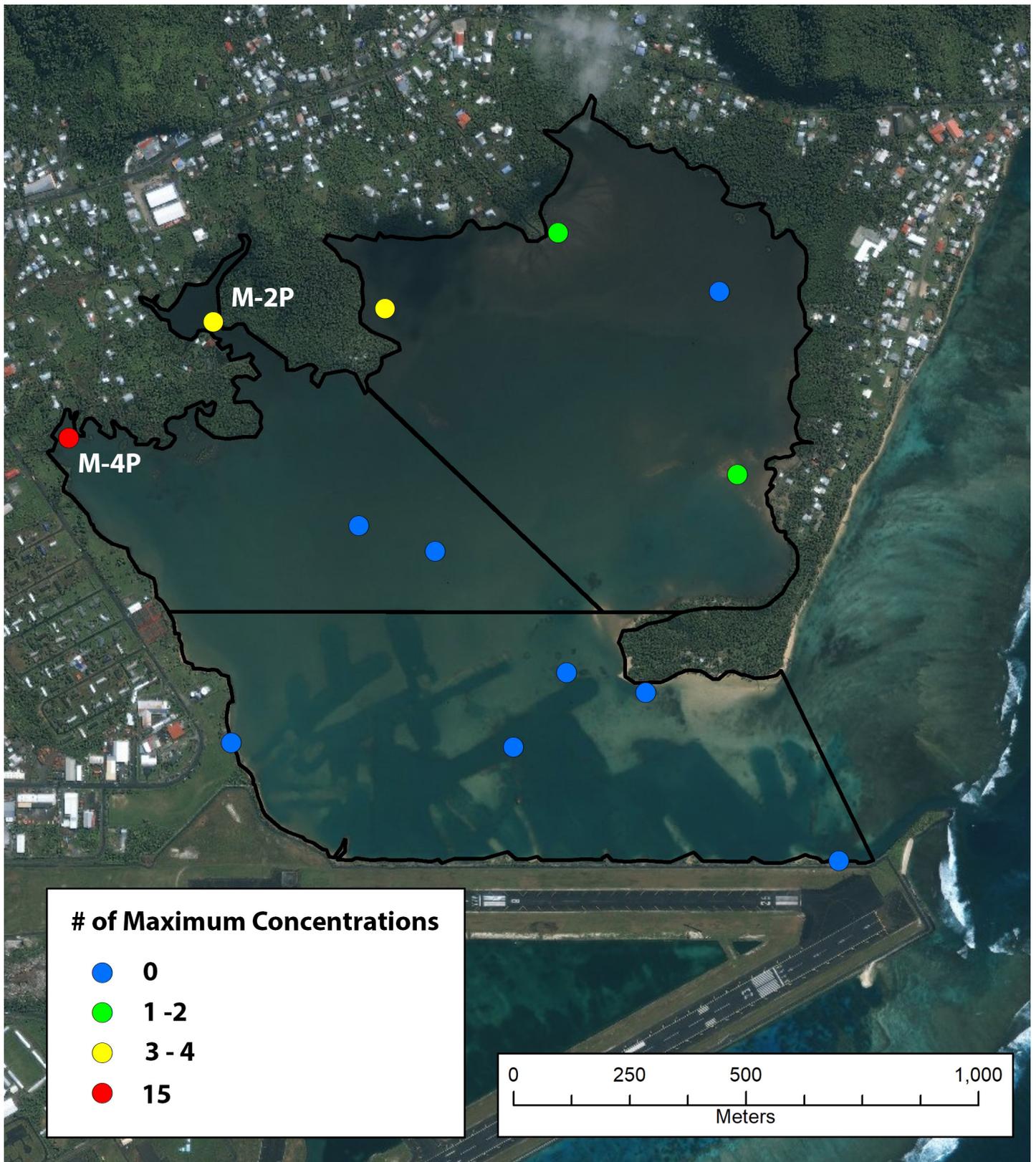


Figure 16. Map depicting the number of maximum concentrations measured in sediments for all chemical contaminant classes in the Nu'uuli Pala Lagoon.

the only metal that exceeded both the ERL and ERM, pointing towards potential adverse impacts to the Pala Lagoon. Of the above mentioned metals, both nickel and arsenic concentrations were not primarily driven by erosion (as compared to any crustal element concentration; Table 9). Trace and major element concentrations in the Lower stratum for the Pala Lagoon were all low with the exception of site L-1P, located at the northeast side of the end of the Pago Pago airport runway, adjacent to the mouth of the Pala Lagoon. The location of L-1P makes it one of the most likely sites to be well-flushed by tidal action and therefore contaminant loads would be expected to be at or near the lowest measured in the Pala Lagoon. While this held true for organic contaminants and most metals, for chromium, nickel, and lead, measured concentrations at site L-1P were the highest for the Lower stratum and even exceeded many of the concentrations from throughout the Pala Lagoon (chromium = 7th highest concentration, nickel = 5th highest concentration, and lead = 4th highest concentration). The concentration of nickel in sediment from site L-1P also exceeded the ERL, indicating potential adverse impacts to benthic organisms at the site. While there could be a number of possible drivers of the elevated levels of these select metals at L-1P, one potential contributor could be the improper disposal of lead-acid batteries along the southern shoreline of the Pala Lagoon. Chromium, nickel, and lead are all common components of lead-acid batteries. During field collections for this study, the authors observed a large number of batteries, in various states of decomposition, along the airport runway fence line and near the sample location for L-1P. Discussions with locals brought to light that the strip of shoreline north of the airport is a popular spot for night fishing activities, and that the source of the batteries may be fishermen improperly discarding flashlight batteries into or adjacent to the marine environment.

Both this study and that of Whittall and Holst (2015) found relatively elevated levels of trace and major metals. Based on comparisons with crustal metals, such as aluminum and iron, it appears that although many of these metals are elevated, much of these measured concentrations may be attributed to naturally high rates of erosion. For example, zinc concentrations in the Pala Lagoon exceeded the ERL at four locations while concentrations in Faga'alu exceeded the ERL at five locations and the ERM at one, but for both waterbodies, zinc concentrations were very highly correlated to aluminum with adjusted R-square values of 0.96 for each. This high level of correlation points toward these elevated concentrations occurring naturally.

Data from this study provide a baseline characterization of the Pala Lagoon that can be used by local managers to both inform their decision making process and to detect changes, both positive and negative, that may occur over time. Additional research may be warranted to better understand the sources and effects of some of the potentially problematic pollutants measured, including arsenic, chromium, nickel, zinc, and PBDEs. Further studies to quantify contaminants in the biota of the Pala Lagoon could also be of interest. All data generated by this study are available at doi:10.25921/d4gh-p564.

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