SPATIAL DISTRIBUTION PATTERN OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) IN HAGÅTÑA, GUAM

BY

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Perfluoroalkyl substances (PFAS) have received attention as emerging contaminants of concern in drinking water resources. In 2014 and 2015, the U.S. Environmental Protection Agency (USEPA) examined, under the Third Unregulated Contaminant Monitoring Rule, six types of PFAS common in U.S. public water systems. Guam Waterworks Authority (GWA) subsequently examined 109 production wells for PFAS; six wells yielded one or more samples with PFAS concentrations above USEPA minimum reporting levels. Of particular concern to GWA was Well A-25 in Hagåtña, where concentrations of perfluorooctanesulfonic acid (PFOS) and perfluorohexanesulfonic acid (PFHxS) were detected at 410 and 140 ng/L, respectively.

To explore the incidence and types of PFAS in the Hagåtña area, we selected five potentially contaminated locations for sampling surface soil and sediment, as a first step toward characterizing the incidence of contamination and constraining potential or suspect sources. Multiple soil and sediment samples were collected from 22 sampling points across these five locations: 1) eight sampling points, half upstream and downstream, of a sewage lift station along the Chaot River; 2) four sampling points within 1,200 meters of Well A-25; 3) one sampling point amongst three Hagåtña River sites (upstream, midstream, and downstream) intersecting the Hagåtña Swamp; 4) one sampling point within four Hagåtña Heights ponding basins; and, 5)

three sampling points among two sites, upslope and downslope of the Fonte River. The Chaot River lift station has a history of episodes of wastewater discharges. Wastewater discharged into the Chaot River was routed downstream to a wetland near Well A-25. The Hagåtña Swamp also has a history of substantial illegal dumping. The Hagåtña Heights ponding basins receive large pulses of urban stormwater runoff during heavy storms. The Fonte River flows beneath a 1997 airliner crash site, where suspected aqueous fire-fighting foams were used.

Samples were tested at the University of Rhode for 34 different PFAS compounds. Analytical results were used to characterize PFAS spatial distribution in the Hagåtña area. Analyses from 21 of the 22 sampling points showed 1H,1H,2H,2H-perfluorooctane sulfonic acid (6:2 FTS) of >0.1 µg/kg. The highest time-average concentration of 6:2 FTS at any single sampling point was 3.7 µg/kg. Two points along the Chaot River showed 6:2 FTS from 14 to 16 µg/kg. Hexafluoropropylene oxide dimer acid (HFPO-DA, GenX), PFHxS, perfluorooctanoic acid (PFOA), and perfluorobutanoic acid (PFBA) were also found >0.1 µg/kg. Out of nine sampling points, a one-time high PFOS concentration (4.8 µg/kg) was found in a Hagåtña Heights ponding basin.

PFAS concentrations in three surface waters (Hagåtña Spring, Hagåtña Swamp, and Hagåtña Bridge) along the Hagåtña River were compared with the groundwater concentrations in Well A-25. Analyses results showed PFOS and PFHxS were highest amongst all PFAS. PFOS was concentrated in Well A-25 with 230 ng/L. PFHxS was concentrated in the Hagåtña Swamp with 85 ng/L. Fluorohexanesulphone amide (FHxSA) and perfluorobutane sulfonamide (FBSA), were also found in Well A-25 and the Hagåtña Swamp. Short-chain PFAS, with four to six carbons, in the water samples have relatively high solubility and are potential degradation products of other PFAS.

Keywords: *Guam, pfas, spatial distribution analysis, pfos, pfoa, fts, hfpo-da*

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11C-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid
4:2 FTS	1H,1H,2H,2H-perfluorohexane sulfonic acid
6:2 FTS	1H,1H,2H,2H-perfluorooctane sulfonic acid
8:2 FTS	1H,1H,2H,2H-perfluorodecane sulfonic acid
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
ADONA (NaDONA)	4,8-dioxa-3H-perfluorononanoic acid
AFFF	Aqueous Film-Forming Foams
DEM	Digital Elevation Model
ETFE	Ethylene tetrafluoroethylene
EtFOSA (NEtFOSA)	*N*-ethyl perfluorooctane sulfonamide
EtFOSAA (NEtFOSAA)	*N*-ethyl perfluorooctanesulfonamidoacetic acid
FBSA	perfluorobutane sulfonamide
FHxSA	fluorohexanesulphone amide
FOSA	perfluorooctanesulfonamide
FTCA	Fluorotelomer carboxylic acids
FTOH	Fluorotelomer alcohols
FTS	Fluorotelomer sulfonates
GWA	Guam Waterworks Authority
HFPO-DA (Gen X)	hexafluoropropylene oxide dimer acid
LC-MS/MS	Liquid Chromatography with Tandem Mass Spectrometry
LHAL	Lifetime Health Advisory Level
MeFBSE (NMeFBSE)	*N*-methyl perfluorobutane sulphonamidoethanol
MeFOSA (NMeFOSA)	*N*-methyl perfluorooctane sulfonamide
MeFOSAA (NMeFOSAA)	*N*-methyl perfluorooctanesulfonamidoacetic acid
MRL	Minimum Reporting Level
NFDHA	nonafluoro-3,6-dioxaheptanoic acid
NFMBA	perfluoro-4-methoxybutanoic acid
NGLA	Northern Guam Lens Aquifer
PASF (POSF)	Perfluoroalkane sulfonyl fluoride
PBT	Persistent Bioaccumulating Toxic
PFAA	Perfluoroalkyl acids
PFAS	Perfluoroalkyl and polyfluoroalkyl substances
PFBA	perfluorobutanoic acid
PFBS	perfluorobutanesulfonic acid
PFCA	Perfluoroalkyl carboxylic acids
PFDA	perfluorodecanoic acid
PFDoDA (PFDoA)	perfluorododecanoic acid
PFDS	perfluorodecanesulfonic acid
PFECA	Perfluoroether carboxylic acids
PFECHS	perfluoroethylcyclohexane sulfonate
PFEESA	perfluoro (2-ethoxyethane) sulfonic acid

PFHpA	perfluoroheptanoic acid
PFHpS	perfluoroheptanesulfonic acid
PFHxA	perfluorohexanoic acid
PFHxDA	Perfluorohexadecanoic acid
PFHxS	perfluorohexanesulfonic acid
PFMPA	perfluoro-3-methoxypropanoic acid
PFNA	perfluorononanoic acid
PFNS	perfluorononane sulfonic acid
PFOA	perfluorooctanoic acid
PFODA	Perfluorooctadecanoic acid
PFOS	perfluorooctanesulfonic acid
PFPeA	perfluoropentanoic acid
PFPeS	perfluoropentanesulfonic acid
PFSA	Perfluoroalkyl sulfonic acids
PFSAA	Perfluoroalkylsulfonamides or Perfluoroalkyl sulfonamido acetic acids
PTFE	Polytetrafluoroethylene
PFTeDA (PFTA)	perfluorotetradecanoic acid
PFTrDA	perfluorotridecanoic acid
PFUnDA (PFUnA)	perfluoroundecanoic acid
PVDF	Polyvinylidene fluoride
PWS	Public Water Systems
UCMR3	Third Unregulated Contaminant Monitoring Rule
UCMR5	Fifth Unregulated Contaminant Monitoring Rule
UOG	University of Guam
URI	University of Rhode Island
USEPA	U.S. Environmental Protection Agency
WERI	Water and Environmental Research Institute
WWTP	Wastewater Treatment Plant

1. INTRODUCTION

Perfluoroalkyl and polyfluoroalkyl substances (PFAS), commonly referred to as forever chemicals, are a family of sophisticatedly complex chemicals that recently received attention as emerging contaminants in water resources (Wang *et al.*, 2017). In the 1940s, 3M Company synthesized its first amphiphilic, fluorochemical compound having a charged head group and fully fluorinated tail group (3M, 1999; Interstate Technology Regulatory Council [ITRC], 2017). Between 1950 and 1970, 3M's production of perfluorooctanoic acid (PFOA), perfluorooctanesulfonic acid (PFOS), and several short- and long-chained fluorochemicals generated tons of wastes that were disposed at production sites or off-site facilities across the mainland. PFAS have been found in many household and industrial products, including fire-fighting foams, carpet stain removers, nonstick cookware, and food packaging. By 2002, 3M became the first company to completely phase out productions of both PFOA and PFOS (Minnesota Department of Health [MDH], 2012). In 2009, 3M competitor DuPont synthesized its first industry alternative, hexafluoropropylene oxide-dimer acid (HFPO-DA or Gen X), as a replacement for bioaccumulating compound PFOA; GenX has become the second highest PFAS (Wang *et al.*, 2019) detected today.

Since 2009, the intentional addition and use of PFAS chemicals in industrial and consumer products have brought about a new era of global environmental contamination. Landfill-disposed products, containing fluorochemical compounds, are one of several pathways in which PFAS are introduced into the environment (Prevedourous *et al.*, 2006; Wang *et al.*, 2011; Lang *et al.*, 2017; Lu *et al.*, 2017; California Environmental Protection Agency [CEPA], 2020). So much so that PFAS have been found in aquatic environments (freshwater, groundwater, marine water), including potable (i.e., tap water) (Abunada *et al.*, 2020) and nonpotable waters (treated and non-treated wastewater) (Lenka *et al.*, 2021); terrestrial environments, such as land, soil (Prevedourous *et al.*, 2006; Cai *et al.*, 2020), and landfills (Busch *et al.*, 2010); and in atmospheric conditions, such as air (Dixon-Anderson & Lohmann, 2018), rain, and snow (Abunada *et al.*, 2020). In fact, according to the Agency for Toxic Substances and Disease Registry (ATSDR, 2021) and Buck and others (2011), most of the U.S. population has some detectable level of PFAS in their bodies, and the same is said for other living organisms (Kjølholt *et al.*, 2015; Muir *et al.*, 2019; Feng *et al.*, 2021). PFAS have been linked to endocrine, reproductive, and developmental diseases and disorders, some of which include forms of cancers, tumors, lower sperm counts, ulcerative colitis, pregnancy-induced hypertension, preeclampsia, weakened immune systems, and increased cholesterol levels (ATSDR, 2021). Thus, the study and identification of fluorochemical-contaminated sites and their fate and transport in the environment are essential, especially in the U.S.

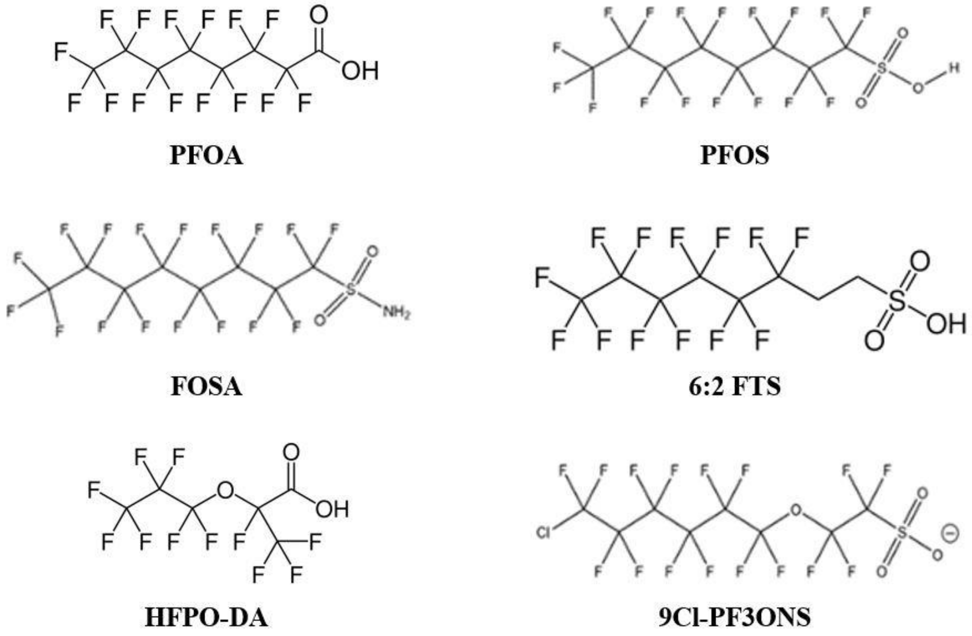
Recently, drinking water resources have been found to contain PFAS chemicals. The U.S. Environmental Protection Agency (USEPA) discovered six recurring fluorochemical compounds in more than 4,900 U.S. public water systems (PWS) between 2013 and 2015; these compounds were added to USEPA’s Third Unregulated Contaminant Monitoring Rule (UCMR3). The six PFAS under UCMR3 are PFOA, PFOS, perfluorononanoic acid (PFNA), perfluorohexanesulfonic acid (PFHxS), perfluoroheptanoic acid (PFHpA), and perfluorobutanesulfonic acid (PFBS) (USEPA, 2017; O’Connor, 2017). In 2016, USEPA published interim lifetime health advisory levels (LHAL) of both PFOS and PFOA at 70 ng/L. The LHAL benchmark also mandated cooperation, compliance, and proper remedial actions to be undertaken by public water agencies. By late 2021, USEPA announced the Fifth Unregulated Contaminant Monitoring Rule (UCMR5) will examine 29 PFAS in drinking water resources starting in 2023 (USEPA, 2021b). Under UCMR5, USEPA set the Minimum Reporting Levels (MRL) for covered PFAS between two and 20 ng/L, which were about five times lower than the MRLs of UCMR3 (10–90 ng/L). Due to increasing PFAS concerns, lower MCLs are crucial to addressing the impact and influence of these contaminants on the environment and human health.

1.1. Characteristics of PFAS

PFAS is a family of over 5,000 synthetic and highly fluorinated chemicals used in a variety of consumer and industrial products for their nonstick functional properties (Buck *et al.*, 2011; Blum *et al.*, 2015). Structurally, PFAS have hydrophilic functional head groups and hydrophobic tail groups. Tail groups are typically alkyl chains (expressed as C_nF_{2n+1}) made up completely of carbon and fluorine (expressed as C–F) bonds (i.e., perfluoroalkyl) or a combination of C–F and carbon-hydrogen (expressed as C–H) bonds (i.e., polyfluoroalkyl) (Buck *et al.*, 2011). Each fluorine atom increases molecular weight and bond strength between atoms and other molecules (3M, 1999; MDH, 2012; Karobe *et al.*, 2018; Lindstrom *et al.*, 2011; Rahman *et al.*, 2014) thereby also increasing stability, polarity, and resistivity to degradation at high temperatures (3M, 1999; Buck *et al.*, 2011; ITRC, 2020). As a result, the physiochemical properties of the tail groups inhibit PFAS from readily dissolving in water, hence, they are suspended between atmospheric and water interfaces. Despite this limitation, solubility still occurs and is afforded by the hydrophilic head group (Rahman *et al.*, 2014). Hydrophilic heads contain a charged (ionic; positive or negative) or uncharged (neutral) functional group with one or more nonmetal elements (e.g., oxygen, nitrogen, phosphorous, sulfur). Charged groups allow semi-volatile PFAS to sink from the surface into the water, making them soluble, mobile, and ubiquitous in the environment (ATSDR, 2009; MDH, 2012; ITRC, 2020; Ahrens, 2011; Rahman *et al.*, 2014).

Neutral groups are less soluble than ionic groups but tend to have higher volatility that contributes to atmospheric deposits of PFAS (Ahrens, 2011; Rahman *et al.*, 2014).

PFAS can be classified in up to five subgroups: 1) perfluoroalkyl carboxylic acids (PFCA); 2) perfluoroalkyl sulfonic acids (PFSA); 3) perfluoroalkylsulfonamide or perfluoroalkane sulfonamido acetic acids (PFSAA); 4) fluorotelomer sulfonates (FTS); and 5) other (alternative) PFAS, including halogenated (i.e., chloride), ether, hydroxyl, or cyclic groups. Representatives from each subgroup are shown in Figure 1. For PFOA and other members of the PFCA group, their main feature is a carboxyl (expressed as CO_2H) group. PFOS and others in the PFSA group have a sulfonyl (expressed as SO_2H) functional group. Perfluorooctanesulfonamide (FOSA), one of many sulfonamido substances, has a sulfonamide functional group in the form of an SO_2H bonded to an amine (expressed as NH_2). As for FTSs, 1H,1H,2H,2H-perfluorooctane sulfonic acid (6:2 FTS) includes an SO_2H and a methylene (expressed as CH_2). As for all other derivative PFAS, such as HFPO-DA and 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS), functional groups include an ether (expressed as C_2O or $R^{'}OR^{''}$) and CO_2H group, while the latter contains a $R^{'}OR^{''}$, SO_2H, and one C–H bond replaced with a carbon and halogen bond (e.g., chloride; expressed as C–Cl).



PFOA **PFOS**

FOSA **6:2 FTS**

HFPO-DA **9Cl-PF3ONS**

Figure 1. Molecular structures of representative PFAS.

The solubility of each PFAS varies depending on its molecular structure and charged or uncharged functional groups. Ding and Peijnenburg (2013) predicted water solubility factors for some PFCAs at 25°C. Solubilities of perfluorobutanoic acid (PFBA), PFOA, and PFNA were predicted at 447, 1.74, and 0.18 mg/L, respectively. PFNA, PFOA, and PFBA consist of nine, eight, and four carbon alkyl chains, respectively. In general, linear, short-chain PFAS with the same number of C–F bonds had relatively higher solubility. Other compounds such as 1H,1H,2H,2H-perfluorohexane sulfonic acid (4:2 FTS) and 6:2 FTS were predicted to have a solubility of 87.1 and 7.41 mg/L, respectively. In the PFSA group, PFOS (eight carbons) solubility had been predicted at 0.21 mg/L compared to the six-carbon member, PFHxS, predicted at 7.59 mg/L.

Table 1 shows thirty-four target PFAS and additional PFAS compounds included in UCMR5. MRL values established by USEPA are also shown in the table. As previously mentioned, UCMR3 included six PFAS for water monitoring but, starting in 2023, USEPA will expand its search for 29 PFAS per UCMR5.

Tables 2, 3, 4, 5, and 6 show the major products and environmental and health issues of each PFAS. The major sources of PFAS detected in the environment are aqueous film-forming foams (AFFF), surfactants, coating materials, paints, and food packing materials. PFOS and PFHxS were widely used as raw materials in AFFFs. PFOA, perfluorohexanoic acid (PFHxA), and fluorohexanesulphone amide (FHxSA) were also AFFFs components, but PFOA has been replaced with the industry alternative, HFPO-DA, since 2009. 6:2 FTS was also purposed as a replacement for PFOA. PFOA, PFBA, and HFPO-DA were used as a surfactant and processing aid in fluoropolymer manufacturing.

The main environmental and health issues related to PFAS exposure are 1) PFOA – carcinogen to animals; 2) PFOS – potential reduction of humoral immune response in children; 3) HFPO-DA – potential induction of tumors, and 4) 6:2 FTS – marine toxicity. Detailed environment and health information are also described in Tables 2 to 6.

Table 1. List of PFAS targeted in this research and MRLs per UCMR5.

Group	**Number**	**PFAS**	**MRL[a,c] (μg/L)**	**Target[b,d]**	**UCMR3[d]**	**UCMR5[d]**
PFCA	1	PFBA	0.005	X	-	X
	2	PFPeA	0.003	X	-	X
	3	PFHxA	0.003	X	-	X
	4	PFHpA	0.003	X	X	X
	5	PFOA	0.004	X	X	X
	6	PFNA	0.004	X	X	X
	7	PFDA	0.003	X	-	X
	8	PFUnDA	0.002	X	-	X
	9	PFDoDA	0.003	X	-	X
	10	PFTrDA	0.008	X	-	X
	11	PFTeDA	0.008	X	-	X
	12	PFHxDA	NI	X	-	-
	13	PFODA	NI	X	-	-
PFSA	14	PFBS	0.003	X	X	X
	15	PFPeS	0.004	X	X	X
	16	PFHxS	0.003	X	X	X
	17	PFHpS	0.003	X	X	X
	18	PFOS	0.004	X	X	X
	19	PFNS	NI	X	-	-
	20	PFDS	NI	X	-	-
PFSAA	21	FBSA	NI	X	-	-
	22	FHxSA	NI	X	-	-
	23	FOSA	NI	X	-	-
	24	MeFOSA	NI	X	-	-
	25	EtFOSA	NI	X	-	-
	26	MeFOSAA	0.006	X	-	X
	27	EtFOSAA	0.005	X	-	X
FTS	28	4:2 FTS	0.003	X	-	X
	29	6:2 FTS	0.005	X	-	X
	30	8:2 FTS	0.005	X	-	X
Other	31	HFPO-DA (Gen X)	0.005	X	-	X
	32	ADONA	0.003	X	-	X
	33	9Cl-PF3ONS	0.002	X	-	X
	34	PFECHS	NI	X	-	-
	35	11C-PF3OUdS	0.005	-	-	X
	36	NFDHA	0.02	-	-	X
	37	NFMBA	0.003	-	-	X
	38	PFEESA	0.003	-	-	X
	39	PFMPA	0.004	-	-	X

[a] Determined by USEPA (2021b); [b] total of 34 PFAS; [c] NI = no information; [d] “-” (dash) = not applicable; not listed. “X” = applicable; listed.

Table 2. Major products of each PFCA and their environmental and health issues.

PFAS	Major Products	Environmental and Health Issues
PFBA	Surfactant; non-stick and strain-resistant consumer products, fluoropolymer processing aid (Organization for Economic Cooperation and Development [OECD], 2013); an alternative to other long-chain PFCAs (Liu *et al.*, 2014; Kjølholt *et al.*, 2015)	Potential harm to marine organisms (Muir *et al.*, 2019); potential degradation product of fluorotelomer alcohols (FTOH) (Buck *et al.*, 2011) and potentially bioaccumulating in humans (Kjølholt *et al.*, 2015); potentially induce increased liver weight in rats (CEPA, 2020)
PFPeA	Surfactant; stain and grease-proof coatings on food packaging, couches, and carpets (CEPA, 2020)	Transformation product via breakdown of fluorotelomer carboxylic acids (FTCA) (Lee *et al.*, 2010)
PFHxA	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); PFOA replacement processing aid in fluoropolymer polymerizations (OECD, 2013); additive in paints, coatings, and surface treatments in buildings and construction (ITRC, 2020); AFFF component (Cortina & Korzeniowski, 2008; Smith *et al.*, 2016)	Persistent and potential degradation product from short-chain fluorotelomer-based surfactants (Stockholm Convention on Persistent Organic Pollutants [SCPOP], 2012)
PFHpA	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); PFOA replacement in chemical coatings, additives, and surface treatments of products, including food packaging, couches, carpets (CEPA, 2020; USEPA, 2021a)	Major transformation product in phosphate-depleted activated sludge containing 6:2 fluorotelomer phosphate monoester (monoPAP) and 6:2 fluorotelomer phosphate diester (diPAP) (Lee *et al.*, 2010; Wang *et al.*, 2011)
PFOA	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); processing aid (emulsifier) in fluoropolymer polymerizations (OECD, 2013; Lohmann *et al.*, 2020); AFFF surfactant (Bogdan, 2019)	Classified carcinogen to animals (Poulsen & Jensen, 2005); bioaccumulation and biomagnification in human serums (USEPA, 2016a), and potential reduction of humoral immune response in children (OECD, 2013); positive association with attention deficit hyperactivity disorder in children (CEPA, 2020)

Continued.

PFAS	Major Products	Environmental and Health Issues
PFNA	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); processing aid (emulsifier) in fluoropolymer polymerizations (Järnberg *et al.*, 2007; OECD, 2013; ITRC, 2020; Lohmann *et al.*, 2020); stain and grease-proof coatings on food packaging (CEPA, 2020)	Potential degradation product of FTOHs in the atmosphere and animals (Muir *et al.*, 2019); a listed persistent bioaccumulative and toxic (PBT) chemical (CEPA, 2020)
PFDA	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); processing aid (emulsifier) in fluoropolymer polymerizations (Järnberg *et al.*, 2007; OECD, 2013); aftermarket treatment for floor polishes (ITRC, 2020); treatment in food packaging (CEPA, 2020)	PBT chemical; Potential bioaccumulation contributor in female placentas and fetal exposures (CEPA, 2020)
PFUnDA	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); processing aid (emulsifier) in fluoropolymer polymerizations (Järnberg *et al.*, 2007; OECD, 2013); stain and grease-proof coatings on consumer products (Maruya *et al.*, 2014)	Potential degradation product in atmosphere and animals via breakdown of FTOHs or other fluorotelomer products (Zhang *et al.*, 2016; Muir *et al.*, 2019); persistent and bioaccumulative (Catherine *et al.*, 2019)
PFDoDA		Positive cytotoxicity of human placental cells (i.e., JEG-3) (Gorrochategui *et al.*, 2014); persistent and bioaccumulative (Catherine *et al.*, 2019); positive association of prenatal exposure increases the risk of childhood atopic dermatitis (CEPA, 2020)
PFTrDA		Persistent and bioaccumulative (Catherine *et al.*, 2019); potential degradation product of FTOHs in atmosphere and animals (Muir *et al.*, 2019)

Continued.

PFAS	Major Products	Environmental and Health Issues
PFTeDA	Surfactant (Buck *et al*., 2011; Schrenk *et al*., 2020); stain and grease-proof coatings on consumer products (Maruya *et al*., 2014; Catherine *et al*., 2019)	Persistent and bioaccumulative (Catherine *et al*., 2019; CEPA, 2020)
PFHxDA	Surfactant (Buck *et al*., 2011; Schrenk *et al*., 2020)	High bioaccumulation potential (Smith *et al*., 2016)
PFODA	Surfactant (Buck *et al*., 2011; Schrenk *et al*., 2020)	

Table 3. Major products of each PFSA and their environmental and health issues.

PFAS	Major Products	Environmental and Health Issues
PFBS	Surfactant (Buck *et al*., 2011; Schrenk *et al*., 2020); Polycarbonate flame retardant (OECD, 2013; ITRC, 2020); PFOS alternative (Bogdan, 2019)	Stable in the environment, and terminal degradation product of *N*-methyl perfluorobutane sulphonamidoethanol (NMeFBSE) and PFBS-based products (SCPOP, 2012); developmental toxicity in marine organisms (CEPA, 2020)
PFPeS	Surfactant (Buck *et al*., 2011); PFOS polymerization replacement compound for polytetrafluoroethylene (PTFE) and polyvinylidene fluoride (PVDF); esterification of perfluoroalkane sulfonyl fluoride (PASF or POSF) or FTOHs with phosphoric acid (ITRC, 2020)	Slight toxicity compared to PFBS (Kjølholt *et al*., 2015)

Continued.

PFAS	**Major Products**	**Environmental and Health Issues**
PFHxS	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); additive in hydraulic fluid (Järnberg *et al.*, 2007); potential carpet, nonstick cookware, and food packaging component (CEPA, 2020); associated with AFFFs (Moody *et al.*, 2003; Rotander *et al.*, 2015)	Relatively slow environmental degradation (Wang *et al.*, 2011); potential bioaccumulation and biomagnification in human serums as compared to PFOA and PFOS, and potential carcinogen (OECD, 2013; Smith *et al.*, 2016); environmentally persistent and stable and potential degradation product of other perfluorinated compounds (SCPOP, 2012)
PFHpS	Surfactant (Buck *et al.*, 2011; Schrenk *et al.*, 2020); additive in hydraulic fluid (Järnberg *et al.*, 2007)	Undetermined.
PFOS	3M's Scotchgard^{TM} and DuPont's Capstone; food (e.g., plates, food containers) and non-food (e.g., folding cartons, masking papers) contact applications and products; cleaning agents, waxes, polishes; additive in aircraft hydraulic oils; effective dispersant for radio-opaque ethylene tetrafluoroethylene (ETFE) copolymer layers (Bogdan, 2019); AFFF surfactant (Wang *et al.*, 2011); electroplating and mist suppressants (Wang *et al.*, 2013); photolithography and semiconductor applications and manufacturing (OECD, 2013; Bogdan, 2019)	Potential harm to marine environments but not acutely toxic (Wang *et al.*, 2013); chemically and biologically inert (Wang *et al.*, 2011); known to bioaccumulate in humans (Schulz *et al.*, 2020; MDH, 2012) and animals (Poulsen & Jensen, 2005), and potential reduction of humoral immune response in children (OECD, 2013)
PFNS	Surfactant (Buck *et al.*, 2011)	Potential bioaccumulation in animals (Schrenk *et al.*, 2020)
PFDS	Surfactant (Buck *et al.*, 2011)	Bioaccumulation in fish (ITRC, 2020)

Table 4. Major products of PFSAA and their environmental and health issues.

PFAS	Major Products	Environmental and Health Issues
FBSA	Polycarbonate flame retardant; surfactant for inks, paints, waxes, and solder pastes; fabric protectant for oil, water, and stain repellants (Bogdan, 2019); replacement compound for PFOS-related applications (Muir *et al.*, 2019)	PFBS precursor; rapid dealkylation in vitro in liver chromosomes (Muir *et al.*, 2019); bioaccumulation in marine organisms (CEPA, 2020)
FHxSA	Component in AFFFs (Houtz *et al.*, 2013b); raw material in PFSAA substances for surfactants and surface treatments (ITRC, 2020; Buck *et al*., 2011; Schrenk *et al*., 2020)	Potential biotransformation product of short-chain sulfonamide compounds (Houtz, 2013a)
FOSA	Raw material in PFSAA for surfactants and surface treatments (ITRC, 2020; Buck *et al*., 2011; Schrenk *et al*., 2020)	PFOS precursor (ITRC, 2020; Schulz *et al*., 2020); potential biotransformation product of accumulated *N*-ethyl perfluorooctane sulfonamide (EtFOSA or NEtFOSA) (Houtz, 2013a; Muir *et al*., 2019)
MeFOSA	Raw material for surfactants and surface protection products (ITRC, 2020)	PFOS precursor (Poulsen & Jensen, 2005); biota and environmental biotransformation product of other PFAS (Kim & Kannan, 2007)
EtFOSA	Raw material for surfactants and surface protection products (ITRC, 2020); surfactant and an active substance in insecticides (Bogdan, 2019; Järnberg *et al*., 2007) and biocides (ITRC, 2020)	Acute toxicity in small marine organisms (i.e., daphnids) (Poulsen & Jensen, 2005); PFOS precursor (ITRC, 2020)
NMeFOSAA	Raw material in surfactants and surface treatment products (Buck *et al*., 2011)	PFOS precursor (Zhang *et al*., 2016)
NEtFOSAA	Cleaning agents, floor, and auto polishes; U.S. banned surfactant and an active substance in pesticides (Bogdan, 2019); potential carpet, nonstick cookware, and food packaging component (CEPA, 2020)	Slight acute toxicity in small marine organisms (i.e., daphnids) (Poulsen & Jensen, 2005); PFOS precursor (Zhang *et al*., 2016)

Table 5. Major products of each FTS and their environmental and health issues.

PFAS	Major Products	Environmental and Health Issues
4:2 FTS	Surfactant; associated with AFFFs (Schultz *et al*., 2004; Muir *et al*., 2019)	PFCA precursor (Muir *et al*., 2019)
6:2 FTS	Surfactant, wetting agent, and mist suppressing agent (OECD, 2013; ITRC, 2020); AFFF surfactant and replacement for PFOA (Cortina & Korzeniowski, 2008; Wang *et al*., 2011; SCPOP, 2012; Wang *et al*., 2013; Schultz *et al*., 2004); a replacement for long-chain PFSAs (Swedish Chemicals Agency [SCA], 2015); PFOS alternative (Hoke *et al*., 2015)	Potential traces of PFOA (Cortina & Korzeniowski, 2008); PFCA (Muir *et al*., 2019; Houtz *et al*., 2013b) and PFSA precursor (Lindstrom *et al*., 2011); relatively slow environmental aerobic degradation (Wang *et al*., 2011); acute and repeated-dose mammalian and aquatic toxicity (SCPOP, 2012); degrades into perfluoropentanoic acid (PFPeA) and PFHxA (Zhang *et al*., 2016; Hoke *et al*., 2015)
8:2 FTS	Surfactant; AFFF fuel repellant and film former (OECD, 2013)	Aerobic degradation contributes to PFBA and other fluorinated PFAS (Wang *et al*., 2011); potential contributor to endocrine effects in vitro and in vivo (OECD, 2013); PFCA precursor (Muir *et al*., 2019)

Table 6. Major products of other (alternative) PFAS and their environmental and health issues.

PFAS	Major Products	Environmental and Health Issues
HFPO-DA	Surfactant; PFOA replacement for fluoropolymer and perfluoropolyether manufacturing (ITRC, 2020)	Bioaccumulate in biota; high potential to induce tumors or increased liver weight in rats (Lohmann *et al.*, 2020; ITRC, 2022); higher cytotoxicity in human liver cells as compared to PFOA or PFOS (CEPA, 2020); not easily broken down in the environment (ITRC, 2020)
ADONA		Not easily broken down in the environment (ITRC, 2020); mild skin irritant in rats and severe eye irritant in rabbits (CEPA, 2020)
9Cl-PF3ONS	PFOS replacement in chrome plating industries and mist and fume suppressants (Liu *et al.*, 2017; CEPA, 2020; ITRC, 2020; USEPA, 2021a)	Not readily biodegraded by wastewater treatment plants (WWTP) (Wang *et al.*, 2013); potential developmental toxicity and bioaccumulation in land and marine organisms (Liu *et al.*, 2017); potential placenta transfer product to a fetus; higher cytotoxicity in human liver cells as compared to PFOA or PFOS (CEPA, 2020)
PFECHS	Additive in aircraft hydraulic oils (Bogdan, 2019; Muir *et al.*, 2019; Schulz *et al.*, 2020)	Accumulates in the liver, kidney, blood, and bladder (Wang *et al.*, 2016)

1.2. PFAS Occurrence in Guam

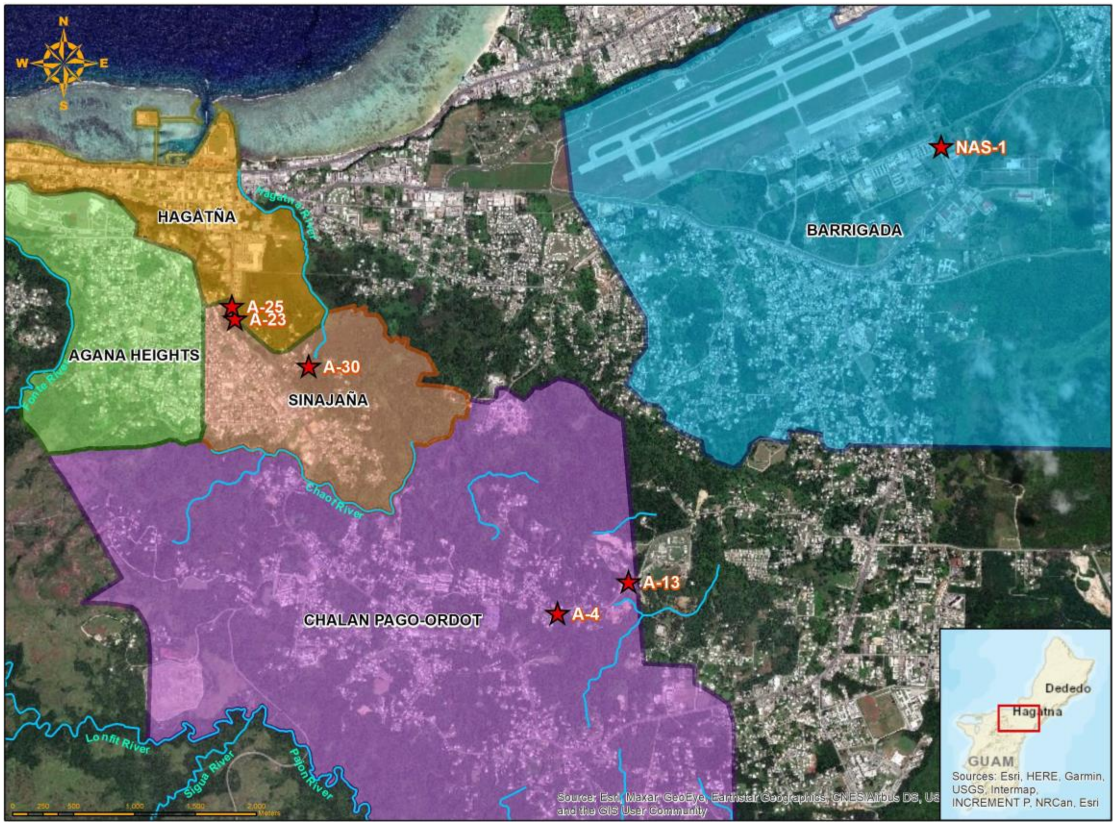
PFAS monitoring of Guam's drinking water production wells began in March 2015. Overall, six out of 109 wells examined yielded detection levels above the MRL (10–90 ng/L) for at least one of the six PFAS listed under UCMR3. PFOS was the most frequently detected compound in five of six wells contaminated with PFAS. Surprisingly, PFOA was not detected in any of the examined wells. These preliminary data indicate approximately 5% of Guam’s drinking water wells are contaminated. Moreover, PFOS exceedances of 70 ng/L LHAL were noted in 2.4% of Guam’s wells.

The six PFAS-contaminated wells of Guam are scattered across three areas: a) Tiyan; b) Chalan-Pago Ordot; and c) Hagåtña (Table 7 and Figure 2). Wells NAS-1, A-23, and A-25 are contaminated with PFOS over 70 ng/L, and A-23 and A-25 are currently offline. NAS-1 is in the former Naval Air Station where AFFFs (a major source of PFAS) were potentially used (Meyer, 2022). Wells A-23 and A-25 are in the capital of Hagåtña; PFOS sources in the area are currently unknown. In the case of Wells A-4 and A-13, Ordot Dump is approximately three miles from these wells and is suspected to be a primary PFAS source in the watershed, however, no further validation or research has been explored.

Table 7. Summary data of preliminary PFAS analyses in Guam (UCMR3).

Location	Production Well ID	PFAS (ng/L)[a,c]			Sample Date
		PFOS	PFHxS[b]	PFHpA[b]	
Tiyan	NAS-1	67	44	-	3/15/2015
		110	68	14	9/27/2015
Chalan-Pago Ordot	A-13	41	-	-	3/22/2015
	A-4	-	32	-	3/22/2015
		-	51	-	9/27/2015
Hagåtña	A-23	88	31	-	5/10/2015
		160	51	-	9/27/2015
	A-25	220	71	-	3/25/2015
		410	140	-	9/27/2015
	A-30	-	30	-	3/22/2015
		66	51	-	9/27/2015

[a] PFOA, PFBS, and PFNA were not detected. [b] perfluorohexanesulfonic acid (PFHxS), perfluoroheptanoic acid (PFHpA). [c] "-" (dash) = unquantified; below detection limit.



Source: Esri, Maxar, GeoEye, Earthstar Geographics, CNES/Airbus DS, US
and the GIS User Community

Sources: Esri, HERE, Garmin, USGS, Intermap, INCREMENT P, NRCan, Esri

Figure 2. Estimated area coverages and locations of Guam Waterworks Authority (GWA) groundwater production wells (red stars).

Before 2015, PFOS had not been identified nor traced to any potentially contaminated site in Hagåtña. Thus, Denton and others (2018) conducted the preliminary PFOS monitoring research of Well A-25 to understand the seasonal concentration pattern of the contaminant. PFOS data were collected from June 2017 to January 2018, along with GWA’s three data sets from 2015 to 2016, and were analyzed with cumulative precipitations recorded at 30, 60, 90, and 120 days before each sampling. A second-order polynomial regression analysis based on a 90-day cumulative rainfall featured the best correlation coefficient for PFOS-rainfall data plots. Based on the findings by Denton and others (2018), variations in the duration and intensity of rainfall events during the preceding month seemed to have the greatest influence on this estimate, indicating a PFAS point source exists within some distance from Well A-25.

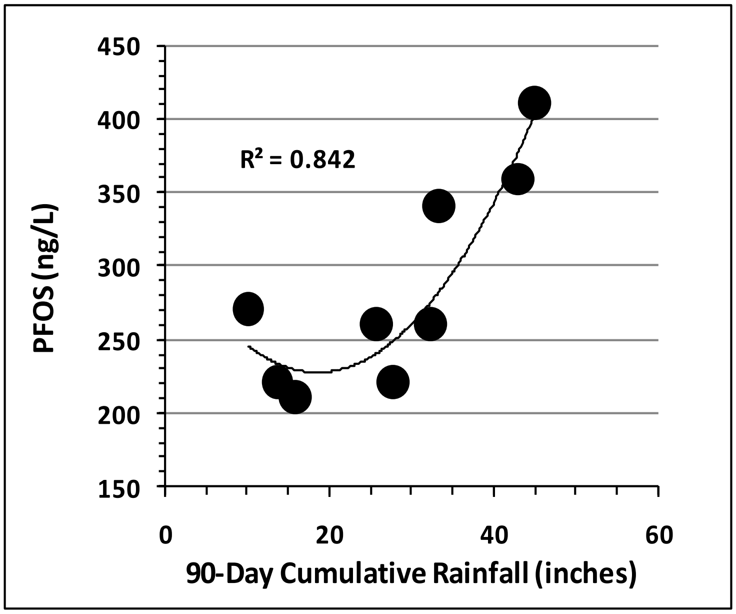


Figure 3. PFOS levels in Well A-25 vs. 90-day cumulative rainfall before each sampling event (Denton *et al.*, 2018).

1.3. Potential PFAS Contamination Locations

In the Hagåtña area, the five potentially contaminated locations were 1) near Well A-25, 2) Chaot River, 3) Hagåtña Swamp, 4) Hagåtña Heights ponding basins, and 5) Fonte River.

At the location near Well A-25, Wells A-23 and A-25 were installed along Highway 4 and were approximately 200 to 300 ft from the Hagåtña Swamp, respectively. Both wells are downhill from some upland residences, businesses, gas stations, and a condominium. Well A-23 is less than 100 ft from Well A-25 and is closest to the Hagåtña Shopping Mall and Guam Auto Spot car retail/parking lot. Well A-25 was stationed at a lower cliff side of the Hagåtña Heights village; it sits along the Pago-Adelup fault. Surface water and groundwater flow in the Hagåtña area generally flows from east to west and discharges into the Hagåtña Bay. Overland flow from upland areas is transported downhill in surface drainages and collected at two nearby stormwater drains. One stormwater drain was located next to Hagåtña McDonald's restaurant on Highway

7B. A second stormwater drain, which seemed to be connected to the drain on Highway 7B, was found closest to a nearby wetland area.

At the Chaot River location, a wastewater pipeline was installed along the Chaot River path. Chaot River water flows east underneath two man-made bridges, and next to several residential houses. It was reported that raw wastes had leaked and discharged into Chaot River for numerous years (Wiles & Ritter, 1993). A defective wastewater pump station next to the Chaot River was reportedly upgraded sometime in 2006 (GWA, 2006; Denton *et al.*, 2018). Along the Chaot River path, household waste (i.e., trash bags and beverage bottles) and an appliance were observed inside or outside of the river. The surface water flow of the Chaot River is funneled through two concrete drainage channels. A drainage channel was downriver from the bridges, and the other was approximately 200 ft downstream of the first channel; they both seemed to be maintained and cleaned regularly. At certain sections of Chaot River, where natural channels had formed, household wastes were floating and collecting at these downstream river bends. More than 200 ft downstream of Chaot River, a few homes were built and viewed along the cliff line of an upland area in Chalan Pago-Ordot. More than 300 ft downriver from the pump station, bridges, and drainage channels, sediments became less and less visible. Eventually, the river path was concealed by muddier, wetland soils, signifying a convergence with a nearby wetland, Hagåtña Swamp.

Along a midpoint section of the Hagåtña River, Hagåtña Swamp was a second location closest to Wells A-23 and A-25. The swamp was located next to the Hagåtña Shopping Mall and the Guam Auto Spot car retail/parking lot. It was reported that household trash had been illegally dumped at Hagåtña Swamp for numerous years (Wiles & Ritter, 1993). Today, household wastes, trash, and an appliance were observed within the swamp. Surface water in Hagåtña

Swamp may not fully drain or discharge. To some extent, water in the swamp can infiltrate the topsoils but at a much slower rate than, for instance, limestone, because of the oversaturated underlying soil layers. Water can also be discharged downriver through a narrow channel that is part of the Hagåtña River. This channel crosses through a man-made dirt road where electric power poles have been installed. While some drainage and discharge may occur, additional load from rainfall events can increase the swamp's water level and, at times, cause flooding in the surrounding areas. Closest to the Guam Auto Spot retail/parking lot, a stormwater drain had been built at the southwest end of Hagåtña Swamp. The length of the drain could not be determined due to foliage overgrowth, but it appeared to be connected to another drain across Highway 7B.

The Hagåtña Swamp also receives surface water flow from two sources, Chaot River and Hagåtña Spring. As previously mentioned, raw wastes were discharged into Chaot River for numerous years. The latter water source comes from a natural spring (Hagåtña Spring), downhill of Highway 4, that flows into the Hagåtña River. Hagåtña Spring is upstream from Hagåtña Swamp and is also nearest to Wells A-23 and A-25. It was also located downslope from residential homes, a groundwater pump station, and a high-voltage electrical transmission tower. Before 1970, Hagåtña Spring was used as a potable water resource, however, recurring coliform contamination resulted in its shutdown (Denton *et al.*, 2018). Foliage overgrowth was observed at the spring, along with evidence of household wastes floating and collecting at the spring's discharge point after a man-made culvert. Water from Hagåtña Spring flows west through the Hagåtña River and underneath one or more man-made bridges.

Several bridges have been built overhead of the Hagåtña River. One of the bridges downstream of Hagåtña Spring and Hagåtña Swamp was located west of the Hagåtña Shopping Mall, swimming pool, and district mayor's office on Highway 33. The Hagåtña bridge, which had a stormwater drain installed to it, receives surface runoff from upland villages (Mongmong-Toto-Maite), where a laundromat, a convenience store, condominiums, and several banks were built. Water from the upland villages will mix with the Hagåtña River water and travel west towards the Hagåtña Bay shore.

A third location upland from Hagåtña Swamp, Hagåtña Spring, and Well A-25 was the Hagåtña Heights ponding basins. The Hagåtña Heights village, which slopes west towards Hagåtña Heights, is mainly a residential area. Most ponding basins in Guam are enclosed with fences and positioned downslope from heavily populated areas. Surface runoff flowing from high to low elevations is transported and collected in ponding basins, which act as main stormwater drainage systems. Water collected in ponding basins will evaporate over time, but it also can infiltrate through topsoils and, if present, groundwater injection wells that transport surface water to the groundwater table. Ponding basins can also collect debris (e.g., household wastes and trash) and water-soluble chemicals and contaminants. Household wastes and debris were observed in nearly all ponding basins surrounded by residential homes.

A Hagåtña Heights Ponding Basin, approximately 1,000 ft upland from Well A-25, was closest to a community center, gym, church, and fire station. Closest to another village (Sinajaña), a ponding basin was located near a gym and an elementary school. A third ponding basin with five groundwater injection wells was nearest to a baseball field. At the west side of Tutuhan, a ponding basin nearest to the Naval Hospital Guam was directly behind a few residential homes and was not enclosed with a fence. Most residential homes in Hagåtña Heights were connected to upgraded sewer lines, however, a portion (30%) of them still relies on septic systems (GWA, 2006; Denton *et al.*, 2018).

On the underside of Nimitz Hill, the Fonte River flows from east to west toward Adelup Bay. It also flows at lower elevations from a tourist attraction (Fonte Dam), a high school, and a nearby military base. In an upland area of Nimitz Hill, Korean Air Flight 801 crashed into the terrain on August 6, 1997. The local fire squad used fire extinguishing liquids and materials to control and suppress the fires, even hours after the impact (Denton *et al.*, 2018); a monument was built as a tribute to this event. Alongside Nimitz Hill, Fonte River also flows about 2,000 ft (or 600 m) downslope from the crash area. It can also receive surface water and runoff from surrounding areas closest to populated villages, including Hagåtña Heights. Water from various sources will mix with those in the Fonte River and flow downstream through several man-made bridges. At one of these bridges, the natural-occurring channel had excess sedimentation possibly a result of soil erosion from substantial water flow in the Fonte River. At this same bridge, it was also located downslope from a residential area. Surface runoff was directed to, collected in, and mixed with the eastward water in the Fonte River.

Table 8. A brief history of potential PFAS contamination locations in the Hagåtña area.

Potential Contamination Locations	Main issue
Near Well A-25	• Located near a car retail parking/storage lot and shopping mall. • Stormwater drains connected to Hagåtña Swamp.
Chaot River	• Raw wastes were discharged into Chaot River until 2006. • Chaot River flows next to the GWA Wastewater Pump Station that converges with Hagåtña Swamp.
Hagåtña Swamp	• Chronic illegal dumping since WWII. • While the swamp itself rises several feet above mean sea level in places, the water table within it rarely varies by more than a few inches above this value except during extreme wet weather conditions. • Hydrological gradient in the vicinity of Well A-25 runs from west to east which precludes the possibility of swamp water infiltrating the aquifer at this point.
Hagåtña Heights ponding basins	• Mainly a residential area uphill from Hagåtña. • Hagåtña Heights stands at a height of approximately 200 ft and gently slopes to the west. • While most houses are connected to sewer lines, about 30% of homes in Hagåtña Heights are dependent on septic systems. • The area is also prone to flooding during wet weather conditions. • Four ponding basins are connected to the underlying aquifer.
Fonte River	• Korean Air Flight 801 crashed into Nimitz Hill on August 6, 1997. • Fires reportedly burned for at least eight hours after impact. • Fonte River flows along a valley next to the Nimitz Hill area, where Korean Air Flight 801 crashed. • Distance between the Korean Air crash area and the source of the Fonte River is approximately 2,000 ft.

PFAS can be found in water because their hydrophilic head groups are highly polar. Most detections in aquatic environments are associated with human activities. PFAS detected in the environment have been linked to both direct (or point) sources, e.g., chemical spills, solid wastes, and wastewater leakages, and indirect (or non-point) sources, e.g., landfill leachate. In other cases, though, PFAS can also occur from biodegradation and biotransformation. Long-chain perfluoroalkyl substances, for instance, can degrade into short-chain perfluoroalkyl substances (Kjølholt *et al.*, 2015). In the case of polyfluoroalkyl substances, which have multiple C–F and C–H bonds, they can biotransform into other short- and long-chain PFCAs and PFSAs (Lindstrom *et al.*, 2011; SCA, 2015).

PFAS in the environment can absorb and accumulate in soils to potentially create contamination plumes. Since PFAS are highly soluble, they too can be transported by water and dispersed away from direct or indirect sources. As such, fluorochemical compounds can be carried down to the groundwater supply through soil infiltration (Jocson *et al.*, 2002; Figure 2). Thus, identifying PFAS contamination in surface soil is a top priority in revealing PFAS detections in water resources.

1.4. Objectives

The groundwater samples from Hagåtña production Well A-25 showed elevated concentrations of PFOS, as opposed to other wells in Guam. Concerning potential sources of PFAS, five potentially contaminated locations in Hagåtña were selected for further study and investigation. The selected locations were 1) near Well A-25, 2) Chaot River, 3) Hagåtña Swamp; 4) Hagåtña Heights ponding basins, and 5) Fonte River. The objective of this study was to evaluate the PFAS spatial distribution in the Hagåtña area. The following hypotheses were proposed:

H_1: Relatively elevated concentrations of PFOS will be detected in surface soils at all sampling points near Well A-25 and Hagåtña Swamp.

H_2: Historically contaminated locations (i.e., dumping and leaking) and locations of high human activities (i.e., drains, basins, and swamps) will detect relatively elevated concentrations of UCMR3 PFAS and other unregulated PFAS compounds.

H_3: Long-chain PFAS (nine or more carbons) may potentially be degrading into other short-chain, e.g., PFHxS, or long-chain, e.g., PFOS, PFAS. Thus, surface soils will detect 1) relatively elevated concentrations of three or more UCMR3 PFAS and 2) other unregulated PFAS compounds.

A total of 22 soil or sediment sampling points were collected from the Hagåtña area and tested for 34 different PFAS compounds. Concentration levels and spatial patterns were evaluated based on each sample's analytical data (e.g., concentrations and detections) of each sample and at each location. In addition, water samples collected from Well A-25 and within several locations of the Hagåtña area were compared with similar soil data. Thus, the contamination status of the potentially contaminated PFAS areas was evaluated herein.

2. METHODS

2.1. Sampling

The literature review revealed the Hagåtña area had been exposed to one or more PFAS sources. As stated previously, the five potentially contaminated locations were 1) near Well A-25, 2) Chaot River, 3) Hagåtña Swamp, 4) Hagåtña Heights ponding basins, and 5) Fonte River. Topographical features in the Hagåtña area, including elevations and contours, were mapped using a two-meter scale digital elevation model (DEM). Figure 4 shows the five potential PFAS contamination locations in a topographical map. Groundwater flow maps and resources of the Northern Guam Lens Aquifer (NGLA) were also used as references in the selection of sampling points.

On October 4, 2018, a site survey was conducted, and a total of 22 sampling points were selected. Sampling procedures for soil and water samples were adopted from the established guidelines by the Architecture, Engineering, Construction, Operations, and Management (AECOM, 2017). Figure 5 shows the selected sampling points at each potential PFAS contamination location. Approximately 100 g of soil and sediment were collected at each sampling point. Samples were placed into 12 x 12 in aluminum foil sheets and sealed within quart-size Ziplock bags. Sample data, including geographical coordinates, were recorded. In addition to soil/sediment samples, one groundwater sample from Well A-25 and three surface water samples along the Hagåtña River were collected as grab samples. Collected soil, sediment, and water samples were stored in a freezer (-10°C or below) until they were shipped for analysis.

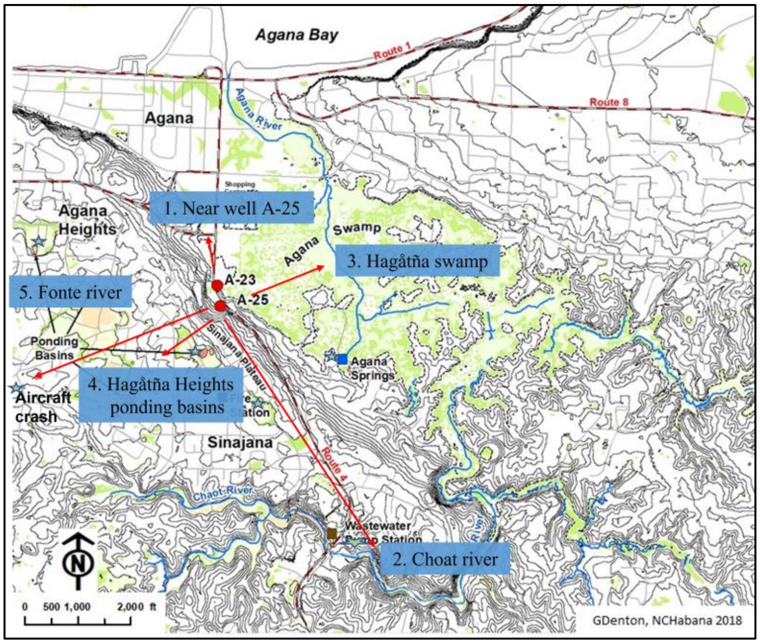


Figure 4. Five potential PFAS contamination locations.

Figure 5. Selected sampling points at each potential PFAS contamination location.

2.1.1. Near Well A-25

Figure 6 shows four sampling points W1, 2, 3, and 4 near Well A-25. A soil sample at W1 was collected close to an abandoned house at the same elevation of the Hagåtña Swamp. W1 is downslope from Well A-23 and the Guam Auto Spot car retail/parking lot in Hagåtña. Along Highway 4, a soil sample at W2 was collected in the downhill valley area of Well A-25. Next to Hagåtña McDonald's, a soil sample at W3 was collected within the stormwater drain on Highway 7B. A soil sample was also collected at the stormwater drain of W4, which was closest to the Hagåtña Swamp and the car retail/parking lot.

W1

W2

W3

W4

Figure 6. Four sampling points (W1, 2, 3, and 4) near Well A-25.

2.1.2. Chaot River

At Chaot River, four sampling points C1, 2, 3, and 4 were located near a GWA Wastewater Pump Station below the bridge on Highway 4 (Fig. 7). At C1, a sediment sample was collected upland of the pump station. The sediment sample at C3 was collected within the

Chaot River near a wastewater pipeline. A soil sample at C4 was collected at a sewage manhole; the soil sample was dark (blackish) in color and sludge-like. The C4 soil sample was collected upstream of the Chaot River. The C2 sediment sample was used as a control to compare PFAS levels between the upstream and the downstream samples.


C1
C2
C3
C4

Figure 7. Four sampling points (C1, 2, 3, and 4) were located near a GWA Wastewater Pump Station below the bridge on Highway 4.

Figure 8 shows four river sediment samples at C5, 6, 7, and 8 that were collected downstream in Chaot River. Sampling points C5 and C6 were collected within Chaot River and were downstream from the pump station below Highway 4. At C5, a sediment sample was collected downstream from another bridge and nearby residential homes. Sampling point C6 was further downstream in Chaot River and also close to residential homes. At C6, a sediment sample was collected within the river downstream from the pump station and two bridges. Sampling points C7 and C8 were located approximately 300 ft and 1,500 ft downriver from the pump station, bridges, and residential homes. At C7 and C8, downstream river samples appeared muddier and swamp-like.

Figure 8. Four river sediment samples (C5, 6, 7, and 8) were collected downstream in Chaot River.

2.1.3. Hagåtña Swamp

At Hagåtña Swamp, three sampling points (S1, 2, and 3) were collected along the Hagåtña River (Fig. 9). Sampling point S1 was located at the discharge point of the Hagåtña Spring. The spring was located downhill from a groundwater well station. Sampling point S2 was on the west side of Hagåtña Swamp. It was collected along a dirt road where electric power poles had been installed. It was also located next to the Hagåtña Shopping Mall. Sampling point S3 was collected downstream beneath a bridge along the Hagåtña River. The bridge was also located next to the shopping mall and a swimming pool. A stormwater drain was connected to the bridge and, at the time, received substantial water flow from the upland village. Sufficient surface water was observed at each Hagåtña Swamp location to allow surface water samples to be collected at three sampling points, to compare with the soil data. A groundwater sample from Well A-25 was used as a reference for surface water detections. Additionally, a laboratory reagent water blank was obtained from the University of Guam (UOG) Water and Environmental Research Institute's (WERI) Water Quality Laboratory.

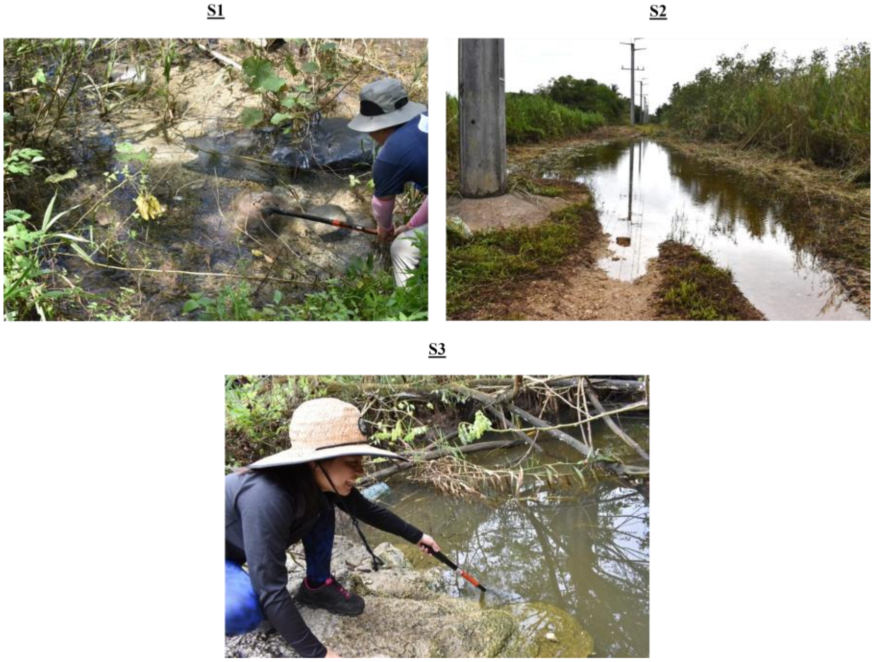


Figure 9. Three sampling points of the Hagåtña Swamp (S1, 2, and 3) along Hagåtña River.

2.1.4. Hagåtña Heights Ponding Basins

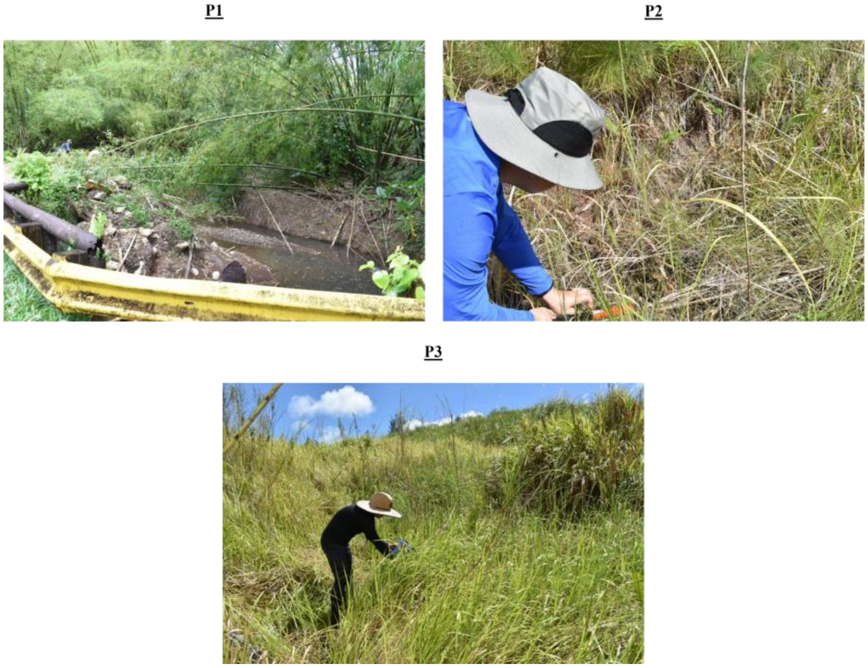
Four ponding basins at Hagåtña Heights (A1, 2, 3, and 4) were selected as sampling points (Fig. 10). At A1, a soil sample was collected at the end of the ponding basin's drain channel; vast foliage overgrowth was observed in the basin. The ponding basin was downgradient from a church, a community center, a fire station, and surrounding residential homes. The A1 ponding basin was about 1,000 ft southeast of Well A-25; it was a point of interest based on groundwater flow in the area. The ponding basin at sampling point A2 was closest to Sinajaña. At A2, a soil sample was collected at the base of the drainage channel; it was also located downhill from residential homes. The basin at sampling point A3 contained five groundwater injection wells and was surrounded by residential homes, an elementary school, and a community baseball field. At A3, a sediment sample was collected at the base of a drainage channel. In the basin at A4, nearest to the Naval Hospital Guam, a soil sample was collected within the ponding basin behind a few homes.


A1
A2
A3
A4

Figure 10. Four sampling points (A1, 2, 3, and 4) of Hagåtña Heights ponding basins.

2.1.5. Fonte River

The Fonte River location included two sampling points at the crash area and one sampling point downstream in the Fonte River (Fig. 11). At P1, a sediment sample was collected beneath a bridge downstream of the Fonte River. Sampling point P2 was collected at Nimitz Hill where the crash occurred. A second soil sample (P3) was collected approximately 30 ft from P2.



P1

P2

P3

Figure 11. Three sampling points (P1, 2, and 3) at the Fonte River.

2.2. PFAS Analysis

Soil, sediment, and water samples were shipped to Dr. Rainer Lohmann's Laboratory at the University of Rhode Island (URI) for analysis of thirty-four PFAS. Approximately 50 g of each soil/sediment sample was re-packed into a shipping container according to the U.S. Department of Agriculture requirements. Water samples were sealed and included in the shipping container as well.

URI prepared soil and sediment samples by drying, crushing, and sieving each one before solid phase extractions. Soil, sediment, and water samples were extracted three times, with Oasis® WAX (Waters) cartridges, for one hour using 400 mM ammonium acetate in methanol; extracts were centrifuged at 5,000 rpm. Concentrated extracts were cleaned using a 250 mg EnviCarb column before evaporating at 35°C with nitrogen.

A total of 34 PFAS were analyzed by URI using liquid chromatography with a tandem mass spectrometry (LC-MS/MS) detector (EPA method 537). The LC-MS/MS analysis was performed using an LC equipped with BEH® C18 column (1.7µm, 2.1mm X 100mm; WATERS) coupled to an MS/MS (AB Sciex 4500 QTRAP) operating in negative mode. URI prepared and tested calibration standards of each PFAS compound. Recovery spiked mass-labeled standards were also prepared and tested by URI.

3. RESULTS

3.1. Degree of contamination

3.1.1. Detection per individual PFAS

Figure 12 shows average concentrations of 17 PFAS detected above 0.1 µg/kg in 22 soil/sediment samples. The prevalent compound in 95% of samples was 6:2 FTS with a summation average of 3.7 µg/kg. HFPO-DA (or GenX), a PFOA replacement compound synthesized by DuPont in 2009 (Wang *et al.*, 2019), was ranked the second highest and most prevalent compound amongst 22 samples. From the PFCA group, PFNA was the third-highest compound with a summation average of 1.7 µg/kg. PFNA was also highest amongst other highly detected PFCA members, PFOA and PFBA. The final ranked compound detected in all samples was PFHxS, which had the highest average detection compared to PFOS and other members of the PFSA group.

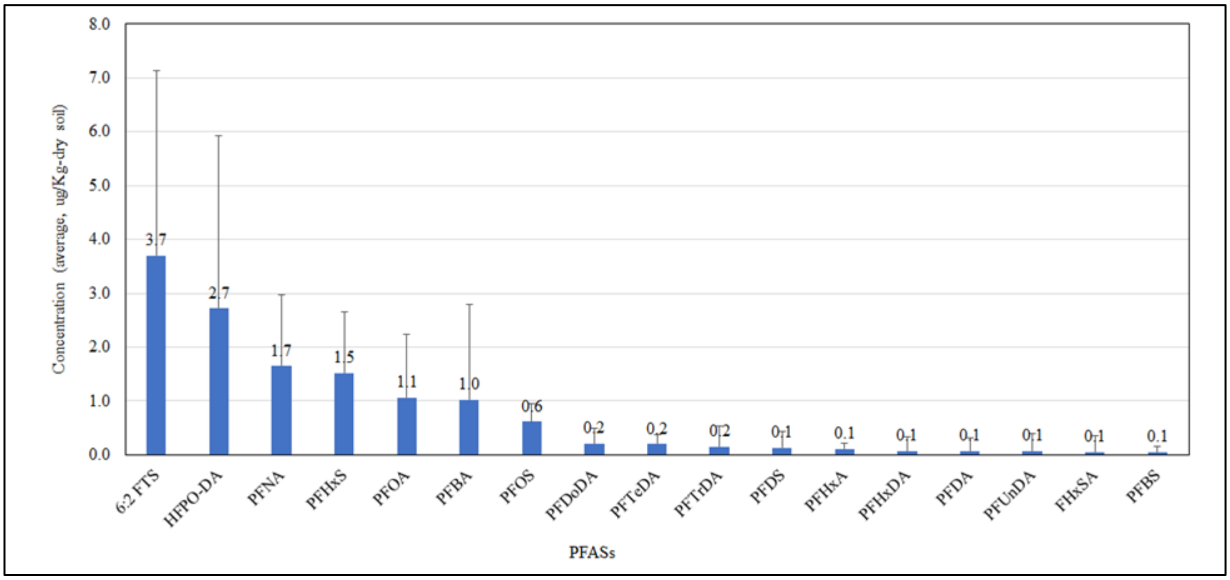


Figure 12. Average concentrations of 17 PFAS detected above 0.1 µg/kg in 22 soil/sediment samples.

FTSs have been widely used as PFOA replacement compounds in polymer and electroplating industries. A subgroup member, 6:2 FTS, was also detected in AFFF (Schultz *et al*., 2004; Wang *et al*., 2011; SCPOP, 2012; Lu *et al*., 2017). In marine environments, 6:2 FTS does not bioaccumulate (Cortina & Korzeniowski, 2008; Hoke *et al*., 2015; Lu *et al*., 2017) despite its low biodegradability (Lu *et al*., 2017). FTSs are intermediate biotransformation products that can potentially release and deposit other PFAS in the environment. 6:2 FTS biotransformation and breakdown can then 1) release new-found PFAS in the environment or 2) combine with existing PFAS and add to observed detections in the environment (Wang *et al.*, 2011). In both instances, compounds derived from 6:2 FTS can accumulate over time. In light of recent alternative options, HFPO-DA replaced legacy PFOA in various industries and products, including food packaging, non-stick coatings, paints, and AFFFs. HFPO-DA has been detected in various environments (i.e., soil, water) (Wang *et al.*, 2019) and fish (Feng *et al.*, 2021).

In groundwater production Wells A-23 and A-25, PFOS and PFHxS detected approximately 88 to 410 ng/L and 31 to 140 ng/L, respectively. In the soil samples, relatively elevated detections of PFHxS suggested a potential point source may be present in the Hagåtña area. Also, in the soils, somewhat low PFOS levels suggest detections possibly came from a nearby point source. However, it could also suggest that PFOS may have entered into the water-soluble phase. As opposed to PFSA detections in soils, PFNA and PFOA, which were highest in their subgroup, were nearly twice as high as PFOS. Interestingly, PFNA and PFOA were undetected in groundwater samples from Wells A-23 and A-25 during Denton and others' (2018) study. Other UCMR3 compounds detected in the soil samples were PFHpA and PFBS, however, their averages were below 0.1 µg/kg; both compounds were also undetected in the two groundwater wells.

Figure 13 presents 33 PFAS quantified in 22 soil samples. The dominant compounds in all samples were HFPO-DA, PFHxS, and PFOA. HFPO-DA, as mentioned previously, is currently an alternative to the documented bioaccumulating compound PFOA (Wang *et al.*, 2019). PFHxS is a main component of AFFFs (Moody *et al.*, 2003; Rotander *et al.*, 2015) and, like PFOS, it too has a potential to bioaccumulate after being introduced into the environment (MDH, 2012). PFOS-based AFFFs were historically manufactured in the U.S. by 3M Company (ITRC, 2020). Long-chain PFAS, including PFOS, can break down into short-chain PFSAs, including PFHxS (Poulsen & Jensen, 2005; ITRC, 2022). PFHxS has been revealed with limited degradation characteristics or uncleared degradation behavior in the environment (Zhong *et al.*, 2022). A relatively high detection of PFHxS suggests preferential binding and absorption onto soils (Schulz *et al.*, 2020).

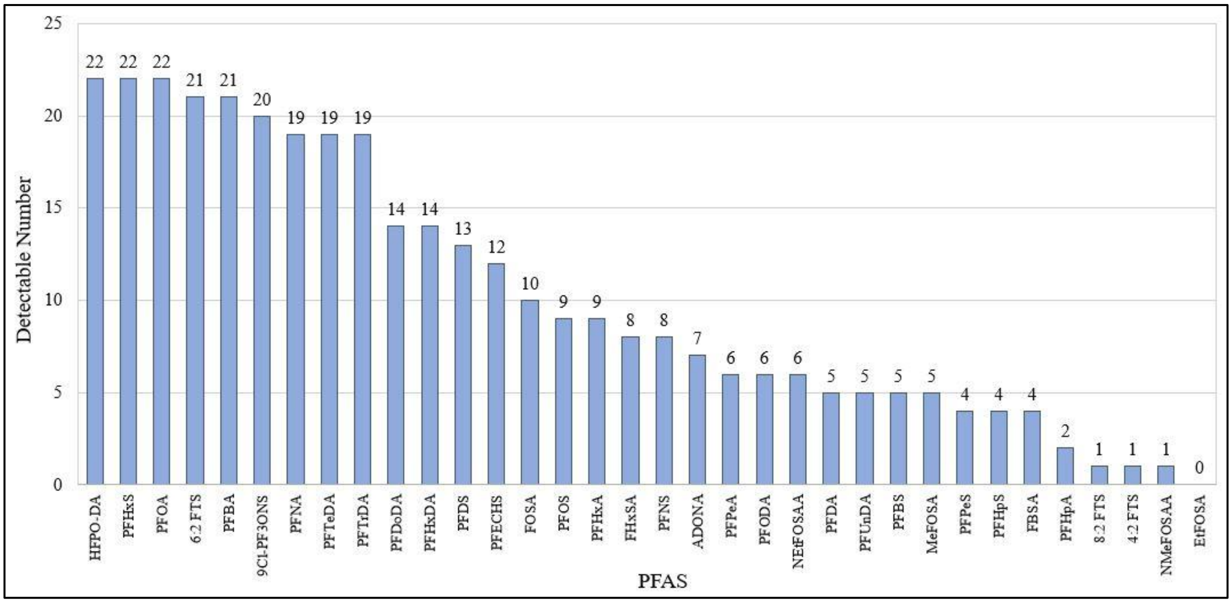


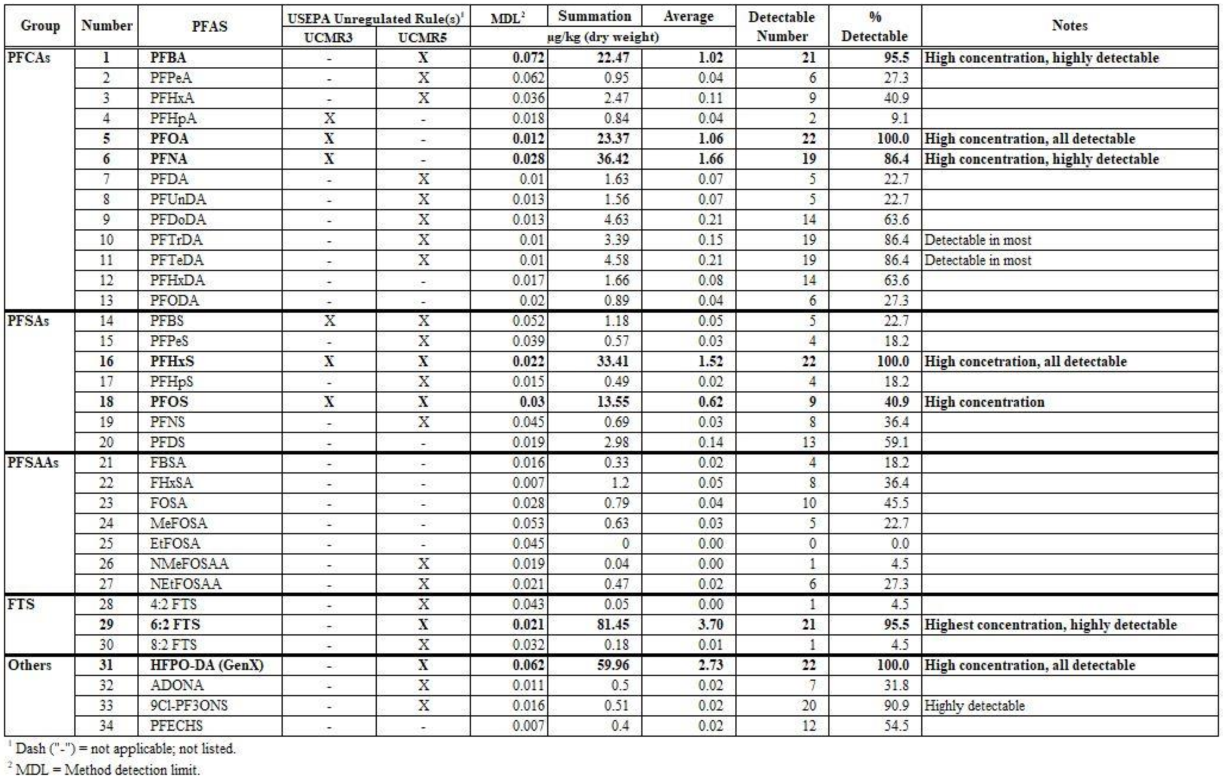
Figure 13. 33 PFAS quantified in 22 soil samples.

Other detectable PFAS in 95% of soils were 6:2 FTS and PFBA. As previously mentioned, the eight-carbon fluorotelomer, 6:2 FTS, can potentially contribute to PFAS accumulation in the environment. PFBA, having the shortest, four-carbon chain of its group, is one of several replacement alternatives for long-chain PFCAs (Liu *et al.*, 2014; Kjølholt *et al.*, 2015). PFBA has been identified as a breakdown product of other PFAS (MDH, 2022). In terms of biodegradability under normal environmental conditions, PFBA does not readily degrade in soil or water (Kjølholt *et al.*, 2015). Direct sources of PFAS particularly in Guam may be: 1) disposal and deterioration of imported goods and products containing PFAS, 2) outflows or leakages from wastewater systems, and 3) leachate seepages from landfills. Historically, illegal dumping of industrial and consumer wastes (i.e., household trash, appliances, and tires) has been apparent in Hagåtña. PFAS-containing products disposed of in the area can potentially leach contaminants into the environment (Lang *et al.*, 2017) and restart the breakdown cycle of those PFAS to their final degraded compounds, which typically includes short-chain compounds like PFBA (CEPA, 2020).

3.1.2. Contamination levels per detected groups

Table 9 shows a summary of soil analysis results for 34 PFAS and their detectable numbers sorted by groups. Figure 14 shows the summary of FTS concentrations measured at 22 sampling points. The highest total (Σ) PFAS (81.45 μg/kg) concentration in 21 samples originated from the FTS subgroup, namely, 6:2 FTS. FTSs have been used as alternative compounds for short- and long-chained PFCAs and PFSAs (Lindstrom *et al.*, 2011; SCA, 2015). PFOA and PFOS were used as ingredients and components in AFFFs (Houtz *et al.*, 2013b; Muir *et al.*, 2019). Recent changes to the AFFF industry brought about the substitution of legacy PFAS (PFOA and PFOS) with safer, non-toxic alternatives, such as FTSs (Cortina & Korzeniowski, 2008).

Table 9. Summary of soil analysis results for 34 PFAS and their detectable numbers sorted by groups.



Group	Number	PFAS	USEPA Unregulated Rule(s)[1]		MDL[2]	Summation	Average	Detectable Number	% Detectable	Notes
			UCMR3	UCMR5	µg/kg (dry weight)					
PFCAs	**1**	**PFBA**	-	**X**	**0.072**	**22.47**	**1.02**	**21**	**95.5**	**High concentration, highly detectable**
	2	PFPeA	-	X	0.062	0.95	0.04	6	27.3	
	3	PFHxA	-	X	0.036	2.47	0.11	9	40.9	
	4	PFHpA	X	-	0.018	0.84	0.04	2	9.1	
	5	**PFOA**	**X**	-	**0.012**	**23.37**	**1.06**	**22**	**100.0**	**High concentration, all detectable**
	6	**PFNA**	**X**	-	**0.028**	**36.42**	**1.66**	**19**	**86.4**	**High concentration, highly detectable**
	7	PFDA	-	X	0.01	1.63	0.07	5	22.7	
	8	PFUnDA	-	X	0.013	1.56	0.07	5	22.7	
	9	PFDoDA	-	X	0.013	4.63	0.21	14	63.6	
	10	PFTrDA	-	X	0.01	3.39	0.15	19	86.4	Detectable in most
	11	PFTeDA	-	X	0.01	4.58	0.21	19	86.4	Detectable in most
	12	PFHxDA	-	-	0.017	1.66	0.08	14	63.6	
	13	PFODA	-	-	0.02	0.89	0.04	6	27.3	
PFSAs	14	PFBS	X	X	0.052	1.18	0.05	5	22.7	
	15	PFPeS	-	X	0.039	0.57	0.03	4	18.2	
	16	**PFHxS**	**X**	**X**	**0.022**	**33.41**	**1.52**	**22**	**100.0**	**High concetration, all detectable**
	17	PFHpS	-	X	0.015	0.49	0.02	4	18.2	
	18	**PFOS**	**X**	**X**	**0.03**	**13.55**	**0.62**	**9**	**40.9**	**High concentration**
	19	PFNS	-	X	0.045	0.69	0.03	8	36.4	
	20	PFDS	-	-	0.019	2.98	0.14	13	59.1	
PFSAAs	21	FBSA	-	-	0.016	0.33	0.02	4	18.2	
	22	FHxSA	-	-	0.007	1.2	0.05	8	36.4	
	23	FOSA	-	-	0.028	0.79	0.04	10	45.5	
	24	MeFOSA	-	-	0.053	0.63	0.03	5	22.7	
	25	EtFOSA	-	-	0.045	0	0.00	0	0.0	
	26	NMeFOSAA	-	X	0.019	0.04	0.00	1	4.5	
	27	NEtFOSAA	-	X	0.021	0.47	0.02	6	27.3	
FTS	28	4:2 FTS	-	X	0.043	0.05	0.00	1	4.5	
	29	**6:2 FTS**	-	**X**	**0.021**	**81.45**	**3.70**	**21**	**95.5**	**Highest concentration, highly detectable**
	30	8:2 FTS	-	X	0.032	0.18	0.01	1	4.5	
Others	**31**	**HFPO-DA (GenX)**	-	**X**	**0.062**	**59.96**	**2.73**	**22**	**100.0**	**High concentration, all detectable**
	32	ADONA	-	X	0.011	0.5	0.02	7	31.8	
	33	9Cl-PF3ONS	-	X	0.016	0.51	0.02	20	90.9	Highly detectable
	34	PFECHS	-	-	0.007	0.4	0.02	12	54.5	

[1] Dash ("-") = not applicable; not listed.

[2] MDL = Method detection limit.

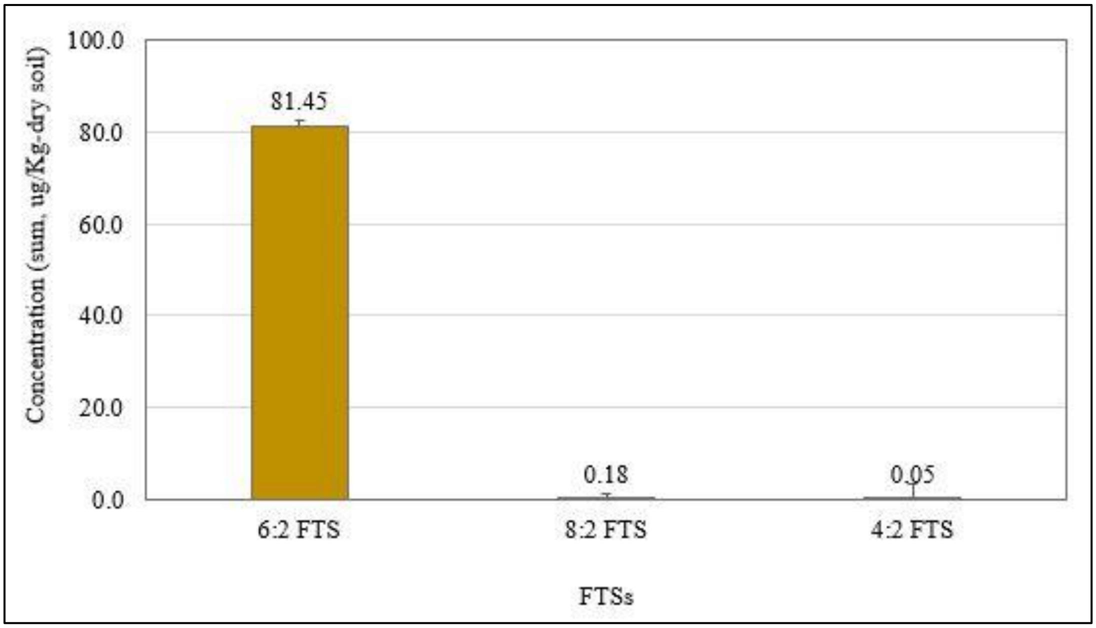


Figure 14. Summary of FTS concentrations measured at 22 sampling points.

In the soil samples, high detection of 6:2 FTS suggested that major uses and releases of AFFFs occurred in Hagåtña. However, it was also suggested that 6:2 FTS may arise from the degrading of other PFAS, particularly, long-chain compounds (Hoke *et al.*, 2015). Under aerobic conditions, FTS in soils were suspected of degrading further to PFCAs (four to six carbons) (Wang *et al.*, 2011), making them potential fluorochemical contributors to soil and groundwater contamination. As mentioned previously, FTSs do not bioaccumulate, but global detections of these compounds resulted in their inclusion in UCMR5 (USEPA, 2021b) and European chemical listings (CEPA, 2020). Discharged wastewater (e.g., influent and effluent) was also found to contain FTS. Field and Seow (2003) proposed dilution and transportation of FTSs in rivers may result in lower hydrophobicity as opposed to other similar PFAS. In other words, water solubility may be the preferential pathway for FTSs.

Other highly prevalent compounds discovered in more than 95% of samples were derived from the PFCA (Figure 15) and PFSA (Figure 16) subgroups. Amongst PFCAs shown in Figure 14, three compounds with concentrations ranging from 22 to 36 µg/kg were detected in decreasing order from longest to shortest carbon chains (PFNA (nine carbons) > PFOA (eight carbons) > PFBA (four carbons)). PFNA and PFOA, like PFOS (eight carbons) and PFHxS (6 carbons) (Figure 16), were mostly used as surfactants for consumer products because of their low surface water tension (Anadón *et al*., 2017). PFNA and PFOA were identified as persistent, bioaccumulating compounds in humans and animals (Poulsen & Jensen, 2005; USEPA, 2016a; CEPA, 2020; Panieri *et al.*, 2022). A direct source of PFNA detections has been credited to human activities, particularly, heavy uses in fluoropolymer productions (Prevedourous *et al.*, 2006). However, there has never been a record of any fluoropolymer manufacturing facilities in Guam that could contribute to direct PFNA detections. Therefore, PFNA concentrations are likely linked to exposure to the latter for extended periods. Schultz and others (2006), though, also suggested that PFNAs originate from WWTPs; treated activated sludge produces PFNAs through aerobic degradation of precursors. In this case, WWTPs become potential point sources of PFNA.

PFBA, as mentioned before, was a replacement compound used for long-chained PFCAs, including PFNA and PFOA. Studies showed PFBA was also a potential bioaccumulating compound in humans and animals (Kjølholt *et al.*, 2015; Muir *et al.*, 2019). Its presence in the environment has been linked to the final degradation of other PFAS compounds (Houtz *et al.*, 2013b; Kjølholt *et al.*, 2015; Muir *et al.*, 2019), including FTSs (Buck *et al.*, 2011; Wang *et al.*, 2011). PFBA is one of three short-chain (four carbons) constituents monitored under UCMR5. Noteworthy PFCAs also detected in the samples were perfluorododecanoic acid (PFDoDA), perfluorotridecanoic acid (PFTrDA), and perfluorotetradecanoic acid (PFTeDA). Concentrations (<5 µg/kg) of each compound were lower than their forerunners. Nevertheless, PFTrDA and PFTeDA concentrations have been positively correlated with detections of PFDoDA (Catherine *et al.*, 2019).

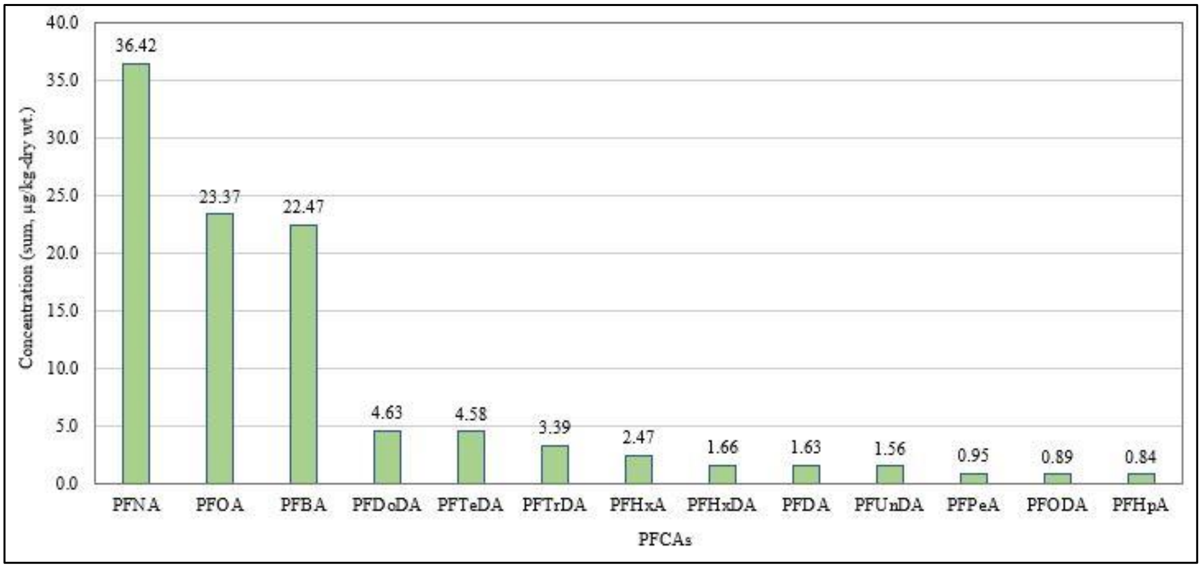


Figure 15. Summary of PFCA concentrations measured at 22 sampling points.

Figure 16 shows the summary of PFSA concentrations measured at 22 sampling points. PFHxS (Σ = 33.41 µg/kg) was the most prevalent and highly detected compound surpassing PFOS in all samples. As stated before, PFHxS had been used as a component in AFFFs. It also was used as a surfactant for industrial (i.e., carpets) and consumer products (i.e., nonstick cookware, and food packaging) in place of legacy PFOS. Thus, the high detection of PFHxS is potentially attributed to its use as a PFOS alternative. Interestingly, though, PFHxS and PFOS can also occur from the biotransformation of other short- (six carbons) and long-chain (eight carbons) PFAS and precursors (SCPOP, 2012; Dauchy *et al.*, 2019). Having detected concentrations of both PFHxS and PFOS suggests both compounds have high affinities for soil absorption (Dauchy *et al.*, 2019; Mussabek *et al.*, 2019; Schulz *et al.*, 2020). In sediments, especially when particulate organic matter was present, PFHxS and PFOS showed increasing concentrations (Mussabek *et al.*, 2019). In wastewater treatment processes, where organic matter can also be found, PFOS and PFHxS were minimally removed during processing (raw influent to final effluent). Sediment-water partition coefficients for both compounds increased due to carbon chain lengths (Schultz *et al*., 2006; Panieri *et al*., 2022); partition coefficients had not been further explored in this study.

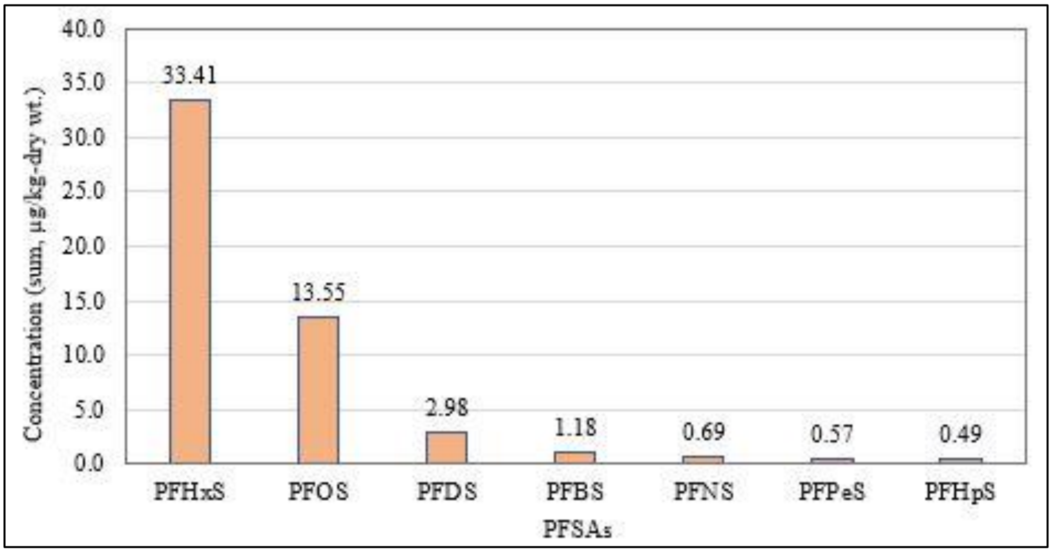


Figure 16. Summary of PFSA concentrations measured at 22 sampling points.

Figure 17 shows the summary of other (alternative) concentrations measured at 22 sampling points. In the alternative subgroup, the short-chain compound, HFPO-DA, had the highest concentration at 59.96 µg/kg. HFPO-DA, as mentioned previously, is one of several PFOA alternatives in industrial and consumer applications. It is highly stable and does not break down easily in the environment (ITRC, 2020), making it an effective replacement for PFOA. However, HFPO-DA is also considered to be persistent, mobile (Lohmann *et al*., 2020), and ineffectively removed from drinking water resources (Bao *et al*., 2018; Wang *et al*., 2019). Another alternative, yet specific to PFOS, 9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS or 6:2Cl-PFESA), was found in 90% of soils. Despite having significantly lower detections than HFPO-DA, 9Cl-PF3ONS was consistently detected between 0.02 and 0.03 µg/kg. Wang and others (2019) suggested detections were potentially a result of PFAS industrial processes, however, 9Cl-PF3ONS had been found in discharged sludge from a treatment plant, evidence that the compound had not been removed nor degraded by routine treatment processes (Wang *et al.*, 2013). A second PFOA alternative detected in less than half of the soils was 4,8-dioxa-3H-perfluorononanoic acid (ADONA). ADONA is stable and, like HFPO-DA, does not easily break down in the environment (ITRC, 2020). Wang and others (2013) found detections of ADONA in discharged effluent wastes. Global environmental detections of three alternative compounds (HFPO-DA, 9Cl-PF3ONS, and ADONA) resulted in their inclusion in UCMR5.

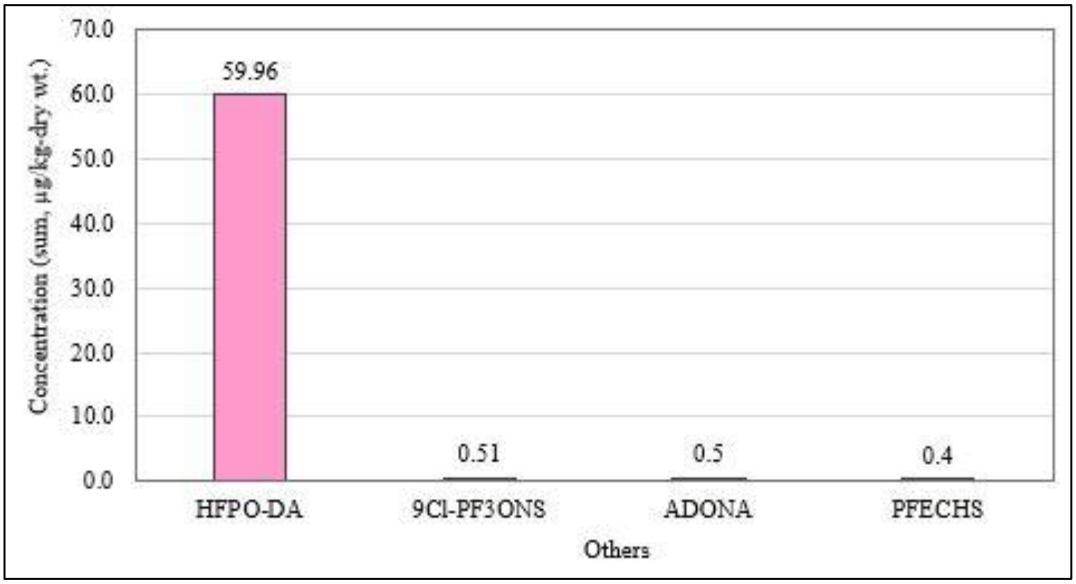


Figure 17. Summary of other (alternative) concentrations measured at 22 sampling points.

Figure 18 shows the summary of PFSAA concentrations measured at 22 sampling points. Compared with the subgroups previously mentioned, PFSAA detections were significantly lower than all the others. The highest concentration detected in seven constituents was 1.2 µg/kg from FHxSA. PFSAAs, though, were utilized as raw materials for surfactants and surface protection products (ITRC, 2020; Buck *et al.*, 2011; Schrenk *et al.*, 2020). PFSAAs were segregated into smaller subgroups (Table 9) based on chemical structures and corresponding amide functional groups that bind protons and other groups (e.g., methyl, ethyl, or acetic acid) to a nitrogen atom. FHxSA, a component previously linked to legacy AFFFs (Houtz, 2013a), has been associated with the biotransformation of precursor compounds produced by 3M Company (Houtz *et al.*, 2013b). Other highly detected PFSAAs were FOSA and *N*-methyl perfluorooctane sulfonamide (MeFOSA). Although soil concentrations were significantly lower than their predecessors, these volatile PFAS potentially contribute more to atmospheric depositions (Panieri *et al.*, 2022), which may be a typical pathway for all members of this subgroup. Out of 34 PFAS, EtFOSA was undetected in all soil samples.

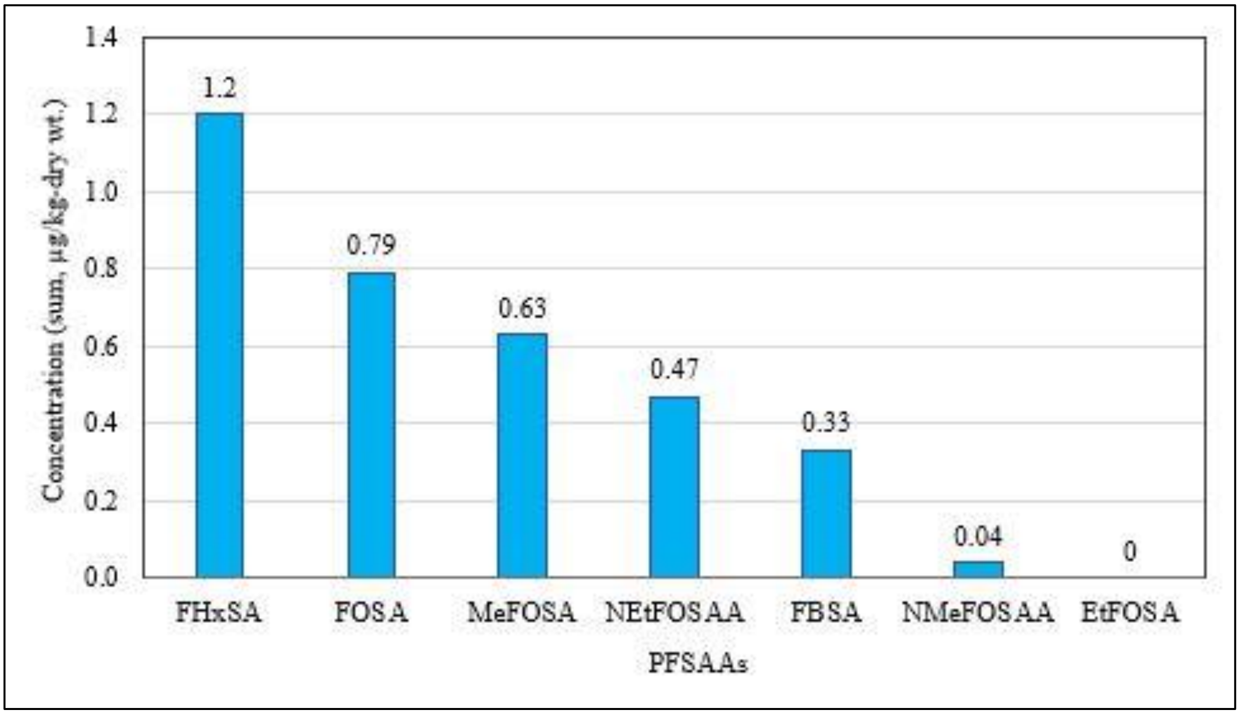


Figure 18. Summary of PFSAA concentrations measured at 22 sampling points.

3.2. Spatial distribution

3.2.1. Collective distribution amongst 22 sampling points

Figure 19 shows the summary concentrations of 33 PFAS in 22 sampling points. Soils and sediments were collected at 22 points located upstream (uphill), downstream (downhill), or at a known PFAS contamination location. Five locations suspected of potential PFAS contaminations were 1) near Well A-25 (W1 to W4), 2) Hagåtña Heights ponding basins (A1 to A4), 3) Hagåtña Swamp (S1 to S3), 4) Chaot River (C1 to C8), and 5) Fonte River (P1 to P3).

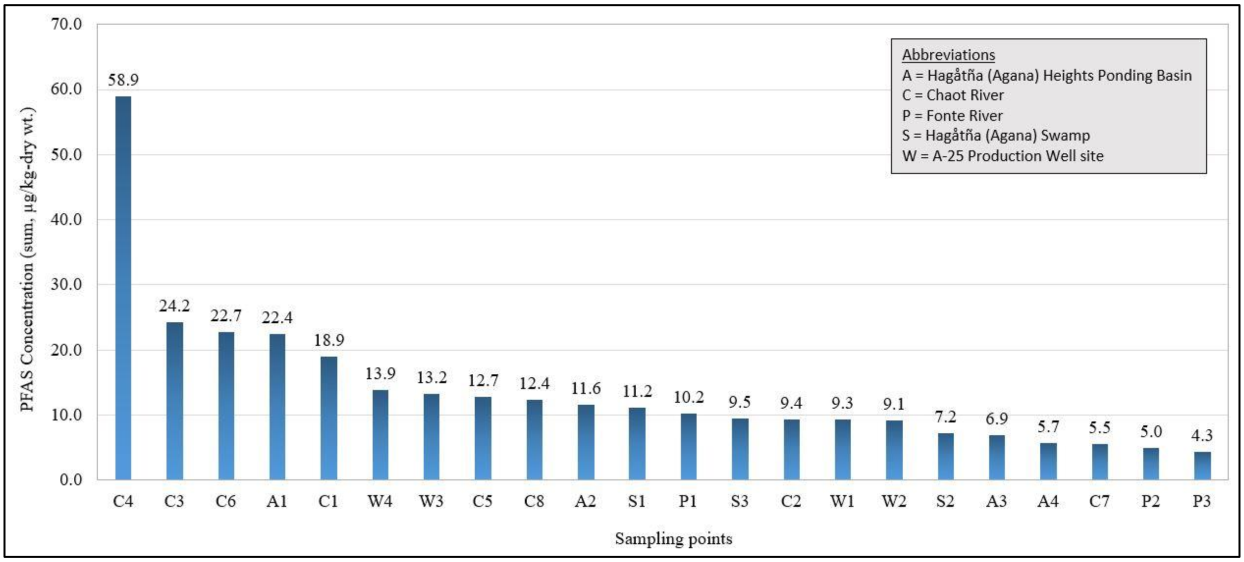


Figure 19. Summary concentrations of 33 PFAS in 22 sampling points.

The highest summation was observed at sampling point C4 from Chaot River. Points C1, C3, and C4 to C6 were among the top eight contaminated points and were collected upstream from an old GWA Wastewater Pump Station along Highway 4 in Chalan Pago-Ordot. As mentioned in the Methods section, a sediment sample at C2 was collected upstream in Chaot River. Thus, it was presumed to be unaffected by nearby contamination sources. At points C1 and C4, topsoil samples were collected next to a sewage manhole; topsoil collected from C1 and C4 appeared muddy and sludge-like. Sampling points C3, C5, and C6 were exhumed downriver nearest the pump station where a mix of soil and sediment was collected. At C6, a household appliance was found along the river channel further downstream from the station. Upriver samples were at least one and a half times higher than background levels observed in C2 whereas levels in C4 were six times higher than the control.

The conduits of wastewater treatment facilities are the main route of contaminant conveyance; thus, they are regarded as a potential PFAS contamination source (Tavasoli *et al.*, 2021). Chaot River is not a designated wastewater discharging point. Yet, wastewater from a defective pump station discharged mass volumes of raw waste into the Chaot River for more than a decade (Wiles & Ritter, 1993; Denton *et al.*, 2018). The discharging source was upgraded thereafter (GWA, 2006; Denton *et al.*, 2018) but Chaot River had already been susceptible to years of exposure. Elevated PFAS levels at Chaot River then are presumed to be a direct result of wastewater contamination. Two additional samples from Chaot River, C7 and C8, were collected downstream from the station. At both sampling points, black trash bags, beverage containers, and other consumer products were observed in the river. The summation level, however, at sampling point C8 (12.4 µg/kg) was comparable to the level at C5 (12.7 µg/kg). Sampling point C8 was collected at an endpoint of Chaot River; relatively low concentrations were observed as compared to other upriver samples. Based on supporting data and evidence, historic and current PFAS impact has been apparent and evident at Chaot River.

At the Hagåtña Heights Ponding Basin sampling point A1, it had the fourth-highest detection of PFAS (22.4 µg/kg). The soil sample from A1 was collected from a ponding basin located 1,000 ft southwest of the Pago-Adelup fault and uphill from Well A-25. The area of the basin was approximately 15,000 ft^2 with 20 ft in depth; in fact, a handful of basins in the village had similar areas and depths as the A1 ponding basin. Ponding basins are the main stormwater drainage systems for substantial volumes of surface and runoff water (Habana *et al.*, 2013) in Hagåtña Heights. They are also considerable collection points for debris and water-soluble chemicals and contaminants. Collected waters tend to evaporate over time, but infiltration through the topsoil and groundwater injection wells can also occur (Habana *et al.*, 2013), and, with favorable rainfall events, infiltrated water can reach the aquifer's vadose (recharge) zone.

Groundwater in the Hagåtña Heights area flows west along the gradient of Well A-25. Ponding basins along the gradient of Well A-25 can considerably influence the groundwater quality in the area. Three other basin points (A2, A3, A4) in Hagåtña Heights were sampled further from Well A-25. A2 was located approximately 1,600 ft southwest of A1; it had half the total concentration of its predecessor. The remaining two basins (A3 and A4), which were further southeast from A1, showed relatively low PFAS levels ranging from 5.7 to 6.9 µg/kg.

Figure 20A shows a valley near Well A-25 sampling point W1. Figure 20B shows a burnt and charred trash bin next to sampling point W4. Sampling points W3 and W4 showed relatively elevated PFAS levels of 13.2 and 13.9 µg/kg, respectively. W3 was sampled from a stormwater drain along Highway 7B next to McDonald's while W4 was from a drain near the Guam Auto Spot retail/parking lot. The drain at W3 receives mainly runoff water from upland residential homes and business establishments in Hagåtña Heights. W4 was collected along the southeast end of the Hagåtña Swamp, and, during field sampling, researchers observed a burnt trash bin less than 30 ft from the channel. At W1 and W2, samples were about 100 ft next to Well A-25 and 200 ft from Well A-23, respectively. Soil samples from both sampling points did not exhibit significant PFAS detections. In this case, Wells A-23 and A-25 may not have been contaminated by surface sources in the area. Nonetheless, PFOS levels in the groundwater supply were possibly migrating from a nearby contaminated source, where underground waters contained PFOS and other compounds, along the gradient of Well A-25.

Figure 20. A) Valley near Well A-25 sampling point W1. B) Burnt and charred trash bin next to sampling point W4.

A historical site, Hagåtña Spring, was also sampled in this study. Records indicated significant coliform contamination had been detected in the spring water pre-1970s (U.S. Army Engineer District [USAED], 1977); to this day it is no longer a viable water resource. Sampling point S1 was collected at the spring's discharging point, where evidence of household trash had been collected or dumped. Water from the spring flows eastward towards Hagåtña Bay but not before passing through a nearby wetland, Hagåtña Swamp, and four constructed bridges. Sampling point S2 then was collected at Hagåtña Swamp along an access road on the southeast end. Illegal dumping of household appliances and wastes has occurred at the swamp. After the swamp, spring water flows downstream under a bridge at Highway 33. A stormwater drain was installed on the bridge, and, during heavy rainfall events, water from the upland village mixed with the water from Hagåtña Spring and Hagåtña Swamp. PFAS detections at these points ranged from 7.2 to 11.2 µg/kg and were comparable to background levels observed in the control. Although observations were considerably low, consistent water flow can dilute PFAS concentrations at each of these points.

Further south of Hagåtña, a sediment sample (P1) (10.2 µg/kg) from Fonte River and two soils (P2 and P3) from an airplane crash site in Nimitz Hill were included in this study. PFAS levels in P2 (5.0 µg/kg) and P3 (4.3 µg/kg) were twice as low as the control but even lower among all other samples. The PFAS-contaminated locations in the Hagåtña area were Chaot River (C1 to C6), next to the wastewater pump station area, and the Hagåtña Heights Ponding Basin (A1).

3.2.2. Distribution of top seven ranked PFAS at sampling points

Figure 21 shows the top seven detected PFAS per sampling point. More detailed graphs were shown in Figures 22, 23, 24, 25, and 26. At Chaot River sampling points C1 to C8, the dominant PFAS observed in each sample was 6:2 FTS. 6:2 FTS was most concentrated in sampling point C6 collected downriver of the pump station, manhole, and residential homes. 6:2 FTS was also detected in C1 to C5 upriver from C6, however, it was undetected in C7 and C8 further downstream. Upstream detections of 6:2 FTS suggest preferential binding to sediments and soils where raw wastes had been discharged; the upriver channel upstream was both shallow and narrow. Downstream from C6, the Chaot River channel was deeper and wider. Low levels of 6:2 FTS at C7 and C8 suggest 1) potential contaminant re-suspension from sediments and soils into the water column during sample collection due to lower hydrophobicity (Field & Seow, 2003), or 2) contaminant dilution (Field & Seow, 2003) from a deeper channel with constant water flow and transport.

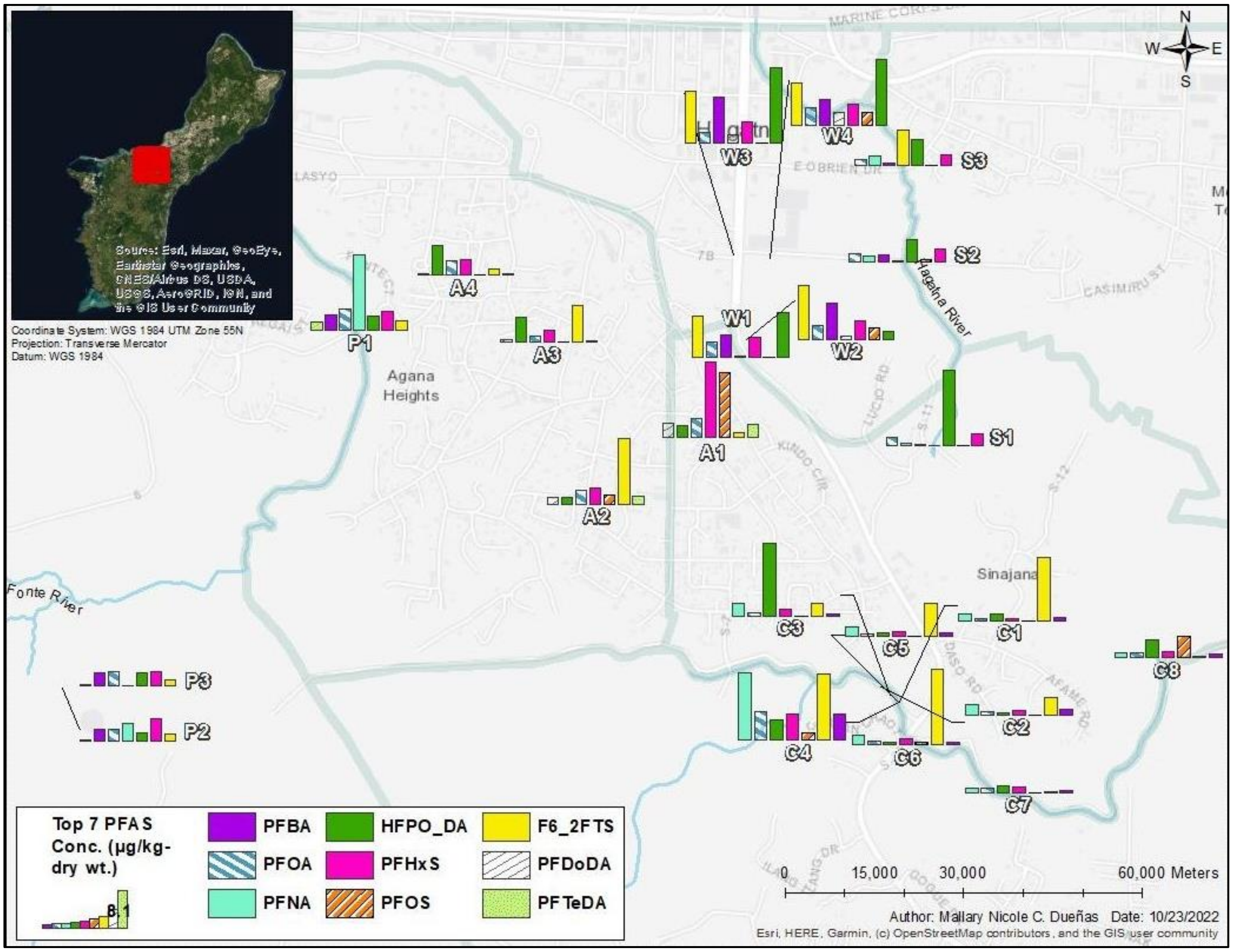


Figure 21. Top seven PFAS detected per sampling point.

At the Hagåtña Swamp, the highest-ranked compound was HFPO-DA, which was mostly concentrated in sampling point S1 at Hagåtña Spring. The spring was previously subjected to fecal contamination rendering it no longer viable as a water source. HFPO-DA detections may be a result of contamination from consumer products collected in the spring. The spring's main inflow channel is blocked by foliage overgrowth; however, remnants of household trash were found in and around the spring. HFPO-DA is not easily removed in water resources, thus, elevated detections in this area are potentially a result of human activities. It is also excessively applied to consumer and industrial products in place of legacy PFOA.

The highest-ranked compound near Well A-25 was also HFPO-DA. It was detected in sampling point W3 next to a restaurant and downhill from residential homes and businesses. As previously mentioned, HFPO-DA is not easily removed from the water, and it has been linked to human activities. At W1, HFPO-DA was detected highest along with 6:2 FTS. 6:2 FTS was consistently detected between 2 to 3 µg/kg amongst all sampling points. It is presumed that these detections may be a result of non-point source transport of the contaminant from high to low elevations.

At the Hagåtña Heights ponding basins, the highest-ranked compound was PFHxS, and it was most concentrated at sampling point A1. As previously mentioned, Well A-25 groundwater detected about 31 to 140 ng/L of PFHxS in 2018. The A1 ponding basin was centralized around residential homes, a community center, and a church. PFHxS has been linked to AFFFs, and the A1 ponding basin was stationed nearly 200 ft downslope from a fire station. The Well A-25 groundwater sample also detected about 88 to 410 ng/L of PFOS, another known compound in AFFFs. Both PFHxS and PFOS have high affinities for soil absorption. Based on groundwater detections and, perhaps, under certain conditions, they also have a higher affinity for water solubility. While PFHxS and PFOS detections point to AFFF, especially fluorine-based foams, both could have also occurred from the environmental breakdown of other long-chain PFAS. 6:2 FTS has been known to biotransform into other short- and long-chain PFAS, which may include PFHxS and PFOS. Interestingly, four out of seven compounds increased in concentrations from Hagåtña Heights ponding basins A4 to A2; A2 resides at a lower elevation from A4. HFPO-DA, on the other hand, was concentrated in A4 but least in A2.

At the Fonte River location, the highest-ranked compound was PFNA; it was detected at the downstream Fonte River bridge (P1). Sludge, an identifier of potential wastewater discharge and a point source of PFNA, was not observed at sampling point P1. PFNA detections then suggest a vast influence of human activities within the surrounding areas. PFCAs, including PFNA, are typically used as surfactants and surface treatments in consumer and industrial products unlike PFSAs, i.e., PFOS, that have been used in airplane hydraulic fluids. Sampling points P1 to P3 detected PFHxS, but not PFOS.

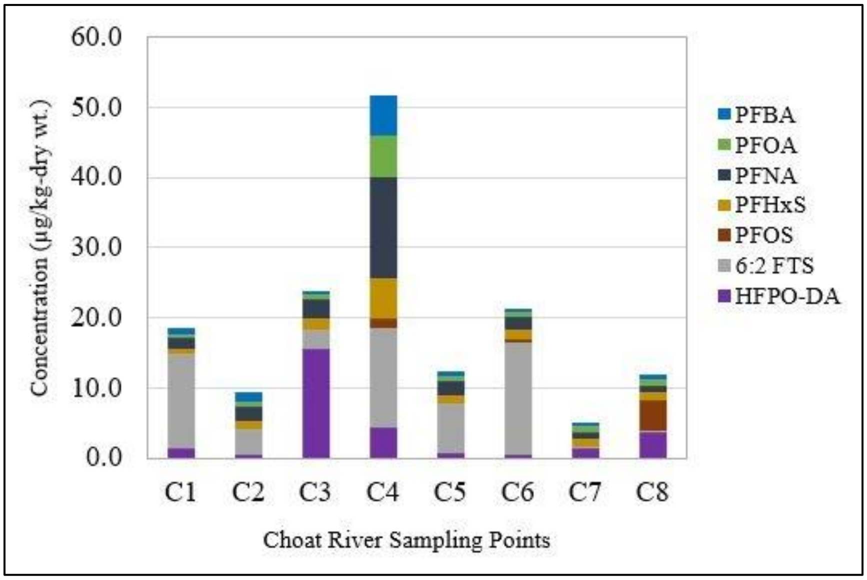


Figure 22. Top seven summation concentrations at Chaot River sampling points.

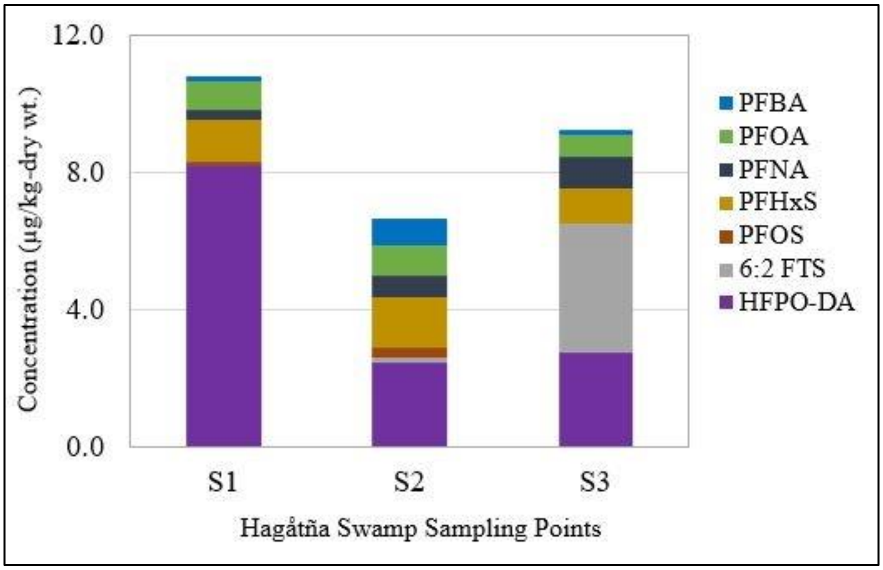


Figure 23. Top seven summation concentrations at Hagåtña Swamp sampling points.

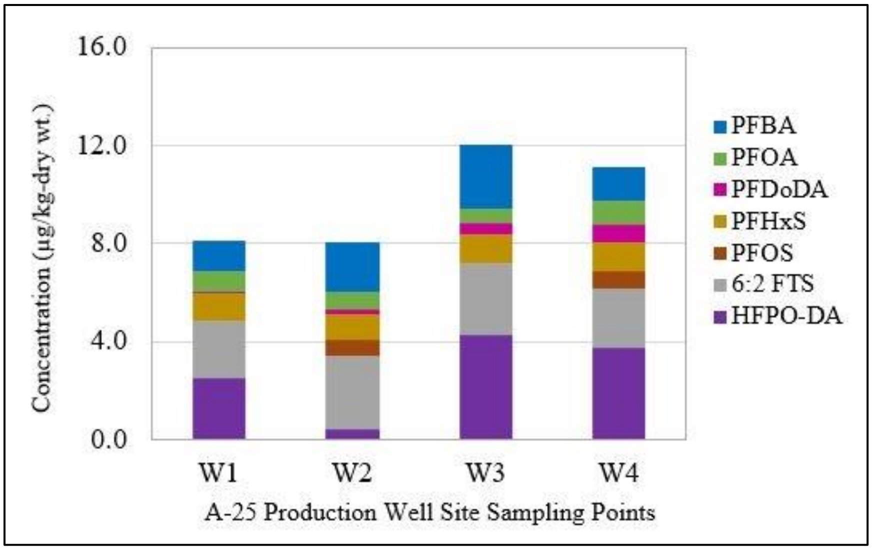


Figure 244. Top seven summation concentrations near Well A-25 sampling points.

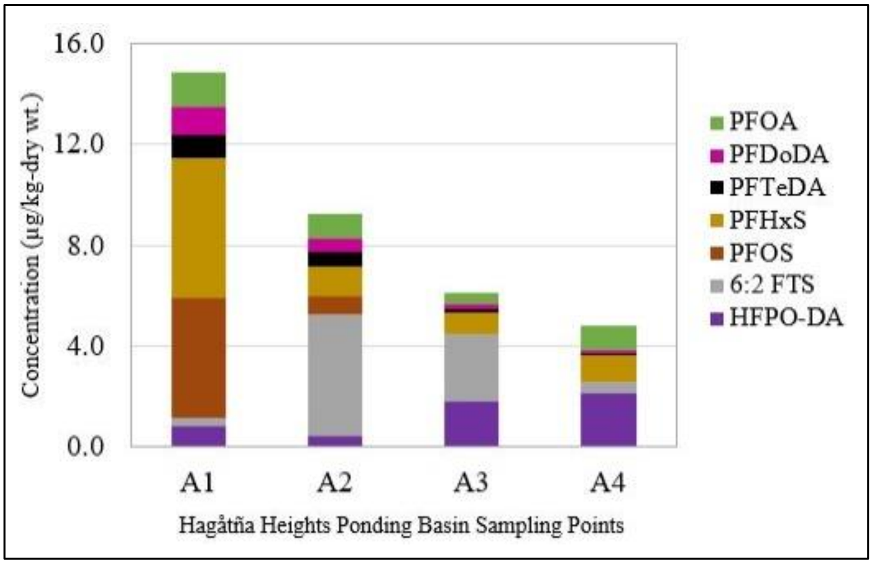


Figure 25. Top seven summation concentrations at Hagåtña Heights ponding basins sampling points.

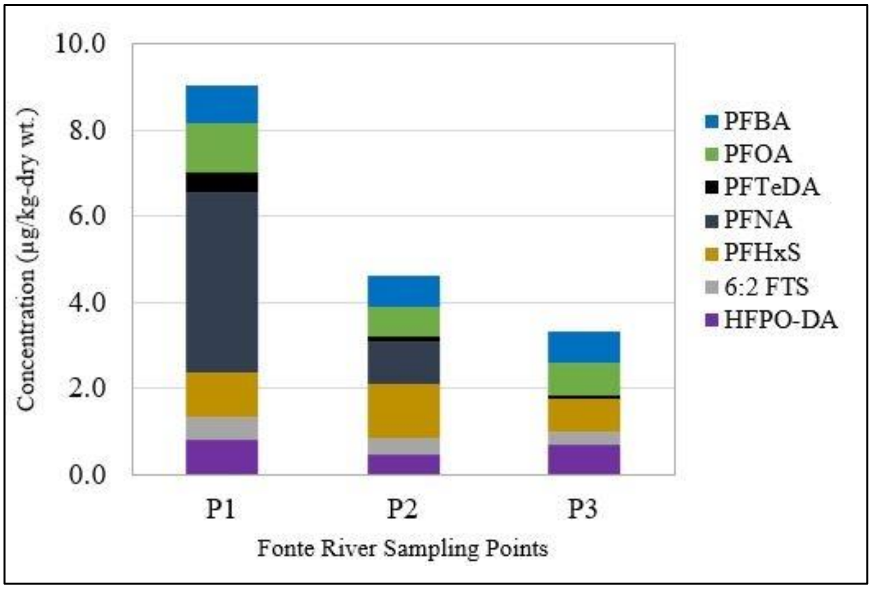


Figure 26. Top seven summation concentrations at Fonte River sampling points.

3.2.3. Collective distribution amongst four water sampling points

Figure 27 shows PFAS detections in four water sampling points (W1, S1, S2, and S3). A sample from Well A-25 was used as a reference for the study. Similarly, to Denton and others' (2018) study, PFOS and PFHxS continued to impact Well A-25. A contributor of PFOS and PFHxS is presumed to be caused by an up-draw of contaminated groundwater from a nearby impacted source. Unfortunately, no other groundwater samples were collected to verify PFOS or PFHxS levels elsewhere. Nonetheless, surface water samples from the Hagåtña River were collected and tested. PFOS and PFHxS were the most concentrated compounds detected in the water samples found in Hagåtña Swamp sampling points S1 to S3. PFOS and PFHxS are considerably high water-soluble PFAS (Panieri *et al.*, 2022) that may potentially infiltrate through surface waters and non-point sources, e.g., basins.

In several studies conducted in China, Panieri and others (2022) wrote that HFPO-DA was notably prominent in water samples as opposed to long-chain compounds. In our water samples, we observed an opposite trend was observed in that HFPO-DA was undetected despite its prevalence in numerous soil sampling points. Other notable compounds undetected in the water samples were PFNA and 9C1-PF3ONS. Additionally, eight PFAS that were either undetected or detected at low levels (<0.1 µg/kg-dry soil) were detectable in the water samples. These compounds were FHxSA, PFBS, PFHpA, 4:2 FTS, FBSA, PFHxA, PFPeA, perfluoropentanesulfonic acid (PFPeS), and perfluoroheptanesulfonic acid (PFHpS). The compounds with lower carbon numbers (FHxSA, FBSA, PFHxA, 4:2 FTS, and PFBS) were presumed to have relatively high solubility.

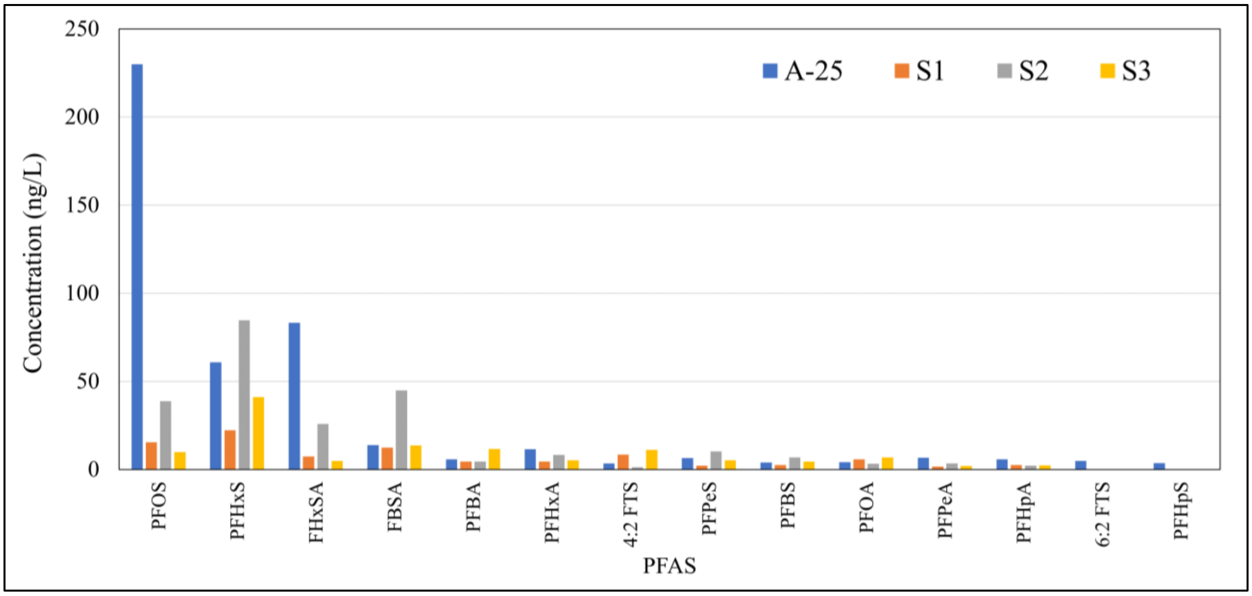


Figure 27. PFAS detections in four water sampling points (W1, S1, S2, and S3).

4. CONCLUSION

Imported goods and products are essential to the livelihood of Guam's people. Products treated, coated, or made with fluorochemicals can potentially release PFAS into the environment. Before 2000, PFAS had not been researched, known, or detected in any environmental samples in Guam. Samplings by USEPA and Denton and others (2018) between 2013 and 2015 brought attention to these chemicals and their presence in several groundwater production wells. This study then focused on the spatial distribution of 34 PFAS in the Hagåtña area, surrounding the two contaminated Wells A-23 and A-25. Relationships between PFAS were evaluated using soil/sediment and water concentrations.

In Hagåtña, 22 soil/sediment samples revealed all locations (Chaot River, near Well A-25, Hagåtña Swamp, Hagåtña Heights ponding basins, and Fonte River) had been exposed to 33 PFAS contaminants. Out of 33 detected compounds, 6:2 FTS, HFPO-DA, PFOA, PFNA, PFBA, and PFHxS were highly and frequently detected.

6:2 FTS is a known PFCA precursor (Houtz *et al.*, 2013b; Muir *et al.*, 2019) and PFSA precursor compound (Lindstrom *et al.*, 2011). In nearly all samples, the PFOA alternative, 6:2 FTS had relatively elevated detections at Chaot River. Past releases of wastewater in the area likely exposed the nearby soils, sediments, and water to this contaminant. Yet, 6:2 FTS, used as surfactants in AFFFs and other products, may also originate from the breaking down of other PFAS (Hoke *et al.*, 2015). 6:2 FTS can also degrade to short-chain compounds (Wang *et al.*, 2011) thereby contributing to other PFAS concentrations. 6:2 FTS was also detected near Well A-25, and detections were relatively similar amongst all sampling points. Detections at each point were presumed to be a result of human activities from upland sources.

Another PFOA alternative detected in all samples was HFPO-DA. Liu and others (2017) reported that potential developmental toxicities may be associated with exposure to HFPO-DA. Liu and others (2017) also reported that HFPO-DA can bioaccumulate in aquatic and land animals. HFPO-DA was highest in sediment next to the wastewater pipeline and a sewage manhole at Chaot River. Wastewater released in the area suggested HFPO-DA may not have been removed from water sources (Bao *et al.*, 2018; Wang *et al.*, 2019) and perhaps even WWTP processes. HFPO-DA is also persistent (Lohmann *et al.*, 2020) and has a high affinity for water (Panieri *et al.*, 2022); this supports both sediment detections collected at the Chaot River and the Hagåtña Swamp's upriver location (Hagåtña Spring). HFPO-DA has been – and continues to be – used as surfactants and processing aids in products. Products containing HFPO-DA and other PFAS can potentially break down and release fluorochemical contaminants into the environment (Lang *et al.*, 2017). Household wastes dumped and collected at the spring's discharge point may have leached, or continue to leach, this contaminant into the river. HFPO-DA was also detected as relatively high in a soil sample near Well A-25. As mentioned previously, the likely source of pollution had been linked to human activities.

Despite being replaced by alternatives such as HFPO-DA and 6:2 FTS, PFOA was found in all samples and detected most at Chaot River. USEPA (2016a) reported that PFOA bioaccumulates and biomagnifies in humans, therefore, this compound is no longer produced on the mainland. PFAS- and PFOA-based products, including AFFFs, can potentially leach PFOA and its precursors into the environment. Sources about AFFFs used and released in Hagåtña were not found. Thus, PFOA detections at this site were perhaps a result of exposure to a point source of contamination (i.e., raw wastes).

For the Fonte River locations, PFNA had the highest concentration at the sampling point downriver at a bridge. PFNA has been listed as a Persistent Bioaccumulative Toxic (PBT) chemical (CEPA, 2020). It was also known to be a degradation product of FTOHs in animals (Muir *et al.*, 2019). Water-soluble PFAS from the crash were presumed to have been carried downhill into Fonte River. In this case, elevated levels of PFSAs (Table 3; Fig. 16), used in airplane hydraulic fluids, were expected. Downstream sediment collected near a man-made bridge detected PFCAs instead. Exposures to consumer products and even human activities (Prevedourous *et al*., 2006), including runoff water collected from surrounding residential areas, likely deposited PFNA at this river. PFNA was also detected at Chaot River, next to a wastewater pump station, which was particularly contaminated with PFAS. Muddy, sludge-like soil collected near a sewage manhole detected elevated levels of PFNA, indicating point source contamination (Schultz *et al.*, 2006) occurred at Chaot River.

Amongst the PFSAs, PFHxS, a persistent and bioaccumulating compound in humans (OECD, 2013) and the environment (SCPOP, 2012), had similar detections at two locations, Chaot River and Hagåtña Heights ponding basins. At Chaot River, detections of this potential carcinogenic compound (OECD, 2013) were linked to a direct source of discharged wastes. At Hagåtña Heights Ponding Basin uphill from Well A-25, it had the highest PFHxS levels compared to other sampled basins. The basin serves as the main stormwater drainage for surrounding residences, community facilities (i.e., gym, community center, church), and the district's fire station. PFHxS, a surfactant alternative to PFOS, has been used as a component in AFFFs. A point source was presumed to be a likely contributor to PFHxS at this site. Yet, like 6:2 FTS, PFHxS and PFOS can also form from the breaking down of other PFAS and precursors (SCPOP, 2012; Dauchy *et al.*, 2019).

Like PFHxS, PFOS can also bioaccumulate and persist in the environment (SCPOP, 2012), animals (Poulsen & Jensen, 2005), and humans (MDH, 2012; OECD, 2013; USEPA, 2016e; Schulz *et al.*, 2020). PFOS was also highest at the Hagåtña Heights Ponding Basin uphill from Well A-25. Stormwater drainages, including basins, around populated areas, detected a variety of PFAS compounds, suggesting human activities were the main pollution sources. PFOS was considerably elevated in the soils, but detections were confined to certain areas rather than dispersed like other PFAS. The Hagåtña Heights Ponding Basin then is potentially contaminated by a point source.

A final prevalent compound in nearly all samples was PFBA. PFBA has been used as an alternative for long-chain, bioaccumulating PFCAs. Muir and others (2019) and CEPA (2020) reported that exposure to PFBA caused potential harm to animals. It was also reported that PFBA can bioaccumulate in humans (Kjølholt *et al.*, 2015). PFBA was highest near the pump station at Chaot River. At Chaot River, although detections may point to its uses as a PFCA replacement and discharged wastewater, PFBA can also form from the degrading of other PFAS, including FTSs (Buck *et al.*, 2011; Wang *et al.*, 2011). At Chaot River, PFBA appeared to be positively correlated with a highly detected compound, 6:2 FTS. Samples upstream and closest to the station showed relatively elevated levels as opposed to levels observed downstream. In this case, PFAS degradations may be occurring at the Chaot River location as a result of point source exposure.

In this study, one reference groundwater sample from Well A-25 and three surface water samples from the Hagåtña River were also analyzed. Highly detected compounds were PFOS, PFHxS, FHxSA, and FBSA, respectively. PFOS and PFHxS, highly detected in the study by Denton and others (2018), continued to be prevalent in the water samples. Both compounds have an affinity for soil absorption (Dauchy *et al*., 2019; Mussabek *et al*., 2019; Schulz *et al.*, 2020), yet the water data suggests an even higher preference for solubility. Elevated detections of PFOS and PFHxS at high and low elevations from Wells A-23 and A-25 raise major concerns about the impact on water quality in the Hagåtña area.

Other highly detected compounds in the water were PFSAA compounds, FHxSA and FBSA. PFSAA compounds are biotransformation products of other PFAS (Kim & Kannan, 2007; Houtz, 2013a). They can also contribute to PFOS concentrations as precursor compounds (ITRC, 2020; Schulz *et al.*, 2020). PFSAAs were suggested to be atmospheric PFAS contributors (Panieri *et al.*, 2022). Relatively low detections in soils/sediments support this theory as PFAS are known to move between water and atmospheric interfaces (Ahrens, 2011; Rahman *et al.*, 2014). HFPO-DA and PFNA were undetected in the water, despite their prevalence in soils/sediments. PFOA was detected in the water, but its impact was negligible. Surprisingly, eight PFAS, which were detected at exceptionally low levels in the soils/sediments, were quite detectable in water; these compounds had lower carbon chains that suggest a preference for water solubility.

This study, albeit investigative and foundational, has several limitations that require further research support. Definitive evidence supporting point (i.e., AFFFs) or non-point (i.e., ponding basins) sources, respectively, were neither found nor further studied. Evidence supporting PFAS degradation and biotransformation was not explored in depth as well. Researchers should consider collectively focusing on these subjects as their data may complement one another. In this project, the focus was only on surface soil and sediment sampling at each sampling point. It would be beneficial to collect core samples below the surface as PFAS can accumulate and create potential contamination plumes near point (direct) PFAS sources. The final limitation of this project is that a minimal quantity of samples was collected, and was also unduplicated. Only a total of 22 soil/sediment and four water samples were collected, and they were collected from broad areas within the study area. In this case, researchers should focus on two locations, one study and another as a control, and conduct duplicated soil, sediment, or water samplings over extended periods (e.g., two or three years). Duplicated data can provide a better understanding of PFAS concentrations over time and during Guam’s everchanging weather conditions, especially precipitation events.

Overall, surface soil/sediment and water sampling are the key first steps in understanding the spatial PFAS distributions near the contaminated wells (A-23 and A-25) in Hagåtña and other areas in Guam. Amongst the five potentially contaminated sites in Guam, four of them have been linked to exposures to human activities (i.e., dumping) and products (i.e., consumer and industrial). Since Guam relies heavily on imported goods and products from the mainland, it is essential to understand the impact, influence, and persistence of PFAS chemicals on Guam’s environment and its inhabitants. A great deal of research support is also needed to determine whether PFAS degradations are occurring at each of the potentially contaminated locations in Guam. This information can help researchers delineate whether PFAS detections are from past or current direct sources.

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APPENDIX – SAMPLING INFORMATION

Sampling Point		Sample Type	Location	GPS Coordinates	
Site	ID			Latitude	Longitude
Chaot River	**C1**	Sediment	Next to sewage manhole	13.45590415	144.7589028
	C2	Sediment	Chaot River (control sample)	13.45540546	144.7595236
	C3	Sediment	Chaot River	13.45565681	144.7592267
	C4	Soil	Sewage manhole	13.45540546	144.7595236
	C5	Sediment	Chaot River downstream near a pump station	13.45541333	144.7595163
	C6	Sediment	Chaot River downstream from a pump station	13.45408771	144.7604181
	C7	Sediment	Chaot River downstream river bend	13.452581	144.7634481
	C8	Sediment	Chaot River downstream near Hagåtña Swamp	13.45693659	144.7682491
Near Well A-25	**W1**	Sediment	Well A-25 valley	13.46635448	144.7542608
	W2	Sediment	Hagåtña Swamp next to Well A-23	13.46692963	144.7544471
	W3	Soil	Hagåtña McDonald’s stormwater drain	13.46961822	144.7539995
	W4	Soil	Hagåtña Swamp next to car storage/parking lot drain	13.46950246	144.7551832
Hagåtña Swamp	**S1**	Sediment	Hagåtña Spring	13.46358685	144.7606015
	S2	Sediment	Hagåtña Swamp	13.46941981	144.7593395
	S3	Sediment	Hagåtña Bridge	13.47251808	144.7594958
Hagåtña Heights ponding basins	**A1**	Soil	Ponding basin near Well A-25	13.46378875	144.7533231
	A2	Soil	Ponding basin – Sinajaña	13.46163679	144.7495969
	A3	Sediment	Ponding basin – Hagåtña Heights (groundwater injection wells)	13.46677723	144.7480376
	A4	Soil	Ponding basin – Tutuhan	13.46889827	144.7453033
Fonte River	**P1**	Soil	Fonte River bridge	13.4671008	144.7418632
	P2	Soil	Korean Air Crash site 1	13.45562939	144.7323251
	P3	Soil	Korean Air Crash site 2	13.455708	144.7324813