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# Tracking Perfluoroalkyl Substances From Wastewater Influent to its Accumulation in Vegetables and Forage Grass

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# TRACKING PERFLUOROALKYL SUBSTANCES FROM WASTEWATER INFLUENT TO ITS ACCUMULATION IN VEGETABLES AND FORAGE GRASS

by

Simon Kozik

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of

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# ABSTRACT

Tracking Perfluoroalkyl Substances from Wastewater Influent to its Accumulation in Vegetables and Forage Grass

by

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Utah State University, 2023

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Per and poly-fluorinated alkyl substances (PFAS) are a class of compounds that are persistent in the environment. The PFAS measured in this study are the sulfonic and carboxylic acid PFAS from C4 to C10. PFAS concentrations were tracked from the influent of the wastewater treatment plant to the effluent, to the biosolids, to reclaimed water used for irrigation, to the soil, and finally into the vegetables and forage grass grown on this reclaimed water and biosolids. PFAS were found in wastewater, which can be used after treatment for irrigation water, and in the biosolids that can be used as a soil amendment. When wastewater was used for either of these purposes it increased the level of PFAS in vegetables and forage grass. Similar to other studies, some PFAS compounds were found to increase in concentration between the influent and effluent but decreased in concentration as it mixed with surface water. At the levels of PFAS measured in garden vegetables sampled in this study their consumption exceeded the Rfd of 1.5x10-9 and 7.9x10-9 mg/kg body weight-day for Perfluorooctanoic acid and Perfluorooctanesulfonic acid, respectively. PFAS were even found in background soil and plant samples irrigated with culinary or surface water. This may be due to PFAS in rainwater, which yielded a similar soil loading to that estimated from irrigation with reclaimed wastewater. In addition, biological concentration factors were estimated.

The use of biosolids as a soil amendment was also found to increase the level of PFAS in forage grass compared to forage grass grown without the use of biosolid amendments. The use of biosolids in garden soils is not recommended due to elevated levels of PFAS in produce from home gardens that already occur from exposure to rainwater and the use of treated wastewater for irrigation.

To help direct further research on dangerous PFAS compounds, general trends for the accumulation of PFAS in soil based upon physical properties were reported. Physical properties of PFAS are correlated with the accumulation in various media, but to tease that out of data a linear mixed effect model had to be used.

(148 Pages)

# PUBLIC ABSTRACT

Tracking Perfluoroalkyl Substances from Wastewater Influent to its Accumulation in Vegetables and Forage Grass

Simon Kozik

Per and poly-fluorinated alkyl substances (PFAS) are a class of chemicals that are persistent in the environment. PFAS was found in wastewater, which can be used after wastewater treatment for irrigation water, and in the biosolids that can be mixed with soils to provide nutrients and generally improve soil quality. This study found when wastewater was used it increases the level of PFAS in vegetables and forage grass. PFAS concentrations were tracked from the influent of the wastewater treatment plant to the effluent, to the irrigation water, to the soil, and finally into the vegetable and forage grass grown on this treated wastewater and biosolids. Similar to other studies, some PFAS compounds were found to increase in concentration after wastewater treatment but dropped in concentration as it mixed with surface water. Similar levels of PFAS were found in rainwater and treated wastewater irrigation spigots. The level of PFAS measured in vegetables even grown in background soils without exposure to treated wastewater or biosolids were high enough that their consumption would exceed the safe exposure levels for Perfluorooctanoic acid and Perfluorooctanesulfonic acid. This is suspected to be because of the PFAS concentrations that these background soils are exposed to rainwater. The use of biosolids in soil was also found to increase the level of PFAS in forage grass when compared to forage grass grown without the use of biosolids so the use of biosolids on home gardens using treated wastewater for irrigation is not recommended. Physical properties of PFAS are strongly correlated with the accumulation in various media, but to tease that out of data a linear mixed effect model had to be used.

(148 Pages)

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# 1 Introduction

Per and poly-fluorinated alkyl substances (PFAS) are a class of compounds that are persistent in the environment. PFAS compounds have carbon chains with fluorine attached to the available carbon bonds. The high electronegativity of fluorine gives PFAS useful properties compared to other hydrocarbons which include being water and grease repellent as well as acting as a surfactant and fire retardant. These compounds generally have functional groups attached to a carbon in the chain which gives a PFAS compound its specific properties. The two largest industrially used functional groups are carboxylic acids and sulfonic acids. PFAS are sparingly water soluble, and their solubility decreases as the number of carbons in a chain increases (Rayne et al. 2009). They also have low vapor pressures that decrease as the number of carbons increase (Rayne et al. 2009).

Moreover, PFAS tend to be recalcitrant because of their thermodynamic stability. They have been shown to accumulate in surface water and groundwater, wastewater (Hu et al. 2016), landfills (Lang et al. 2016), and around areas, such as airports, where Aqueous Film-Forming Foam (AFFF) has been or is being used to extinguish fire fueled by flammable liquids (Hu et al. 2016). PFAS compounds, specially PFOA and PFOS, have been shown or are suspected to cause cancer in humans (Barry et al. 2013), and ongoing studies by the United States Environmental Protection Agency (EPA) are showing that they may serve as endocrine disrupters (To and Dawley, 2019). In response to this concern, the producers of PFAS have signed an agreement with the EPA to reduce the amount of perfluorooctanoic acid (PFOA) and perfluorosulfonic acid (PFOS) used in industrial applications. These two compounds are the eight carbon (C8) perfluorinated substances that were the subject of litigation by the EPA in 2008 against 3M and DuPont in 2013 (OECD 2018). However, because PFAS compounds are stable and the litigated

agreement to reduce PFAS applies only to C8 PFAS compounds, contamination by other PFAS compounds remains prevalent, and shorter chain PFAS compounds are still being released to the environment.

The advisory level recommended by the EPA for PFAS in drinking water is 0.004 ng/L of PFOA or PFOS or the combination of the two (EPA 2022). However, this level only accounts for the concentrations of the two most prevalent PFAS compounds. Recently an additional 5,000 PFAS compounds are being litigated. The low level of this advisory also presents an analytical challenge. Lab equipment often contains PFAS and may be a source of contamination during PFAS analysis (Shoemaker et al. 2009). The USEPA has also proposed chronic oral reference doses (RfD) of 0.0003, 0.0000000015, and 0.0000000079 mg/kg body weight-day for Perfluorbutanesulfonic acid (PFBS), PFOA, and PFOS respectively (USEPA 2022, 2021).

Additionally, only drinking water has a set advisory level, despite other pathways through which humans are exposed to PFAS including indoor air, rainwater, and the use of treated wastewater for irrigation (Shane et al. 2020, Zhou et al. 2019). In addition, consumer goods, such as non-stick cookware, water- and stain-resistant clothing and other fabrics, and food packaging (Kim et al. 2007; Cai et al. 2012; Kotthoff et al. 2015) contribute to human exposure to PFAS and environmental PFAS concentrations. The highest potential for exposure in the United States to PFAS contamination is where PFAS compounds were produced or used for manufacturing and as fire retardants (Hu et al. 2016). Low levels of contamination in water have been shown to bioaccumulate in plants and can move up aquatic and terrestrial trophic levels (Penland et al. 2020). PFAS can also accumulate in fruits, cereals and vegetables which may then be consumed by humans (Felizeter et al. 2012, 2014). Few studies have been conducted on the presence of PFAS in vegetables in areas where contamination is likely low and the contributors to the contamination will largely be from consumer goods (USEPA 2019) or on pathways that

involve the reuse of material such as contaminated and reclaimed water from municipal wastewater treatment plants (WWTPs). Despite the lack of studies, the reuse of water and the use of biosolid fertilizer may magnify PFAS contamination by reintroducing it to a population (Stoiber et al. 2020).

A small mechanical WWTP in Utah was chosen to investigate the levels of uptake of PFAS in vegetables from treated wastewater used as irrigation water in home gardens and some agricultural fields during the summer irrigation season. Biosolids from the treatment plant have also been used as a soil amendment for growing forage crops. The WWTP has been producing reclaimed wastewater as a secondary source of irrigation water for more than 10 years. Effluent from the treatment plant contains low but detectable levels of PFAS as this wastewater is from households which are expected to have lower concentrations of PFAS than many industrial wastewaters.

However, even with a reduction in the use of PFAS in industries, any reclaimed water used in the irrigation of homegrown plants can contain low levels of PFAS as is seen in municipal WWTPs without large industrial inputs (Coggan et al. 2019). It has also been seen that WWTPs are generally ineffective at removing PFAS from the influent wastewater (Chen et al. 2018). This is particularly concerning as wastewater from the WWTP evaluated in this study is used to irrigate some residential gardens and the biosolids produced are land applied as a fertilizer for forage crops. As indicated above, edible plants can take up PFAS from irrigation water and contaminated soil (Stahl et al. 2009, Felizeter et al. 2012), and this accumulation in plants could potentially increase the risk of PFAS released from WWTPs, even when effluent PFAS concentrations are below the current drinking advisory water limit.

## 1.1 Research Questions

The purpose of this study was to determine the concentration of PFAS compounds associated with the irrigation of edible plants and the fertilization of forage grass with PFAS-containing municipal wastewater products and the relative risk posed to individuals exposed to these PFAS compounds through reuse water and garden vegetable consumption. The PFAS studied were C4-C10 carboxylic and sulfonic acids. The following questions directed this study to identify the risks associated with the use of reclaimed water for home garden irrigation and the use of biosolids as a fertilizer and soil conditioner in forage hay grass fields.

Question 1: Does the use of reclaimed water (treated wastewater treatment plant effluent) for home garden irrigation increase the concentration of PFAS in garden soils and edible fruits and vegetables compared to the use of other sources of irrigation water?

Question 2: Based on the USEPA RfD do fruits and vegetables from home gardens irrigated with reclaimed water represent a significant risk of PFAS exposure to residents through their ingestion?

Question 3: Does amending soils with biosolids increase the concentration of PFAS in the soils and forage grass grown in those fields compared to forage grass and soils in fields with no biosolids amendment?

Question 4: What properties of individual PFAS compounds correlate with the concentration of PFAS in soils, forage grass and the edible portions of fruits and vegetables irrigated with reclaimed water or grown in biosolids amended fields?

## 1.2 Hypotheses and Objectives

Hypothesis 1: The soil and vegetables irrigated with reclaimed water will have higher PFAS concentrations than soil and vegetables in home gardens irrigated with other water sources.

Objective 1: Determine the distribution and concentration of PFAS compounds through the wastewater treatment plant, focusing on their final composition and concentration in wastewater treatment plant effluent that is the reclaimed water used for garden and field irrigation. Compare the PFAS concentrations in soils and edible portions of fruits and vegetables in gardens irrigated with reclaimed water with those from gardens irrigated with other water sources.

Hypothesis 2: Ingestion of fruits and vegetables from gardens irrigated with reclaimed water exceed the USEPA RfD for PFAS intake.

Objective 2: Compare the concentrations of PFAS found in the fruits and vegetables harvested from gardens irrigated with reclaimed water to the USEPA RfD for a 70 kg adult consuming 336 g of vegetables a day (for example, for PFOA and PFOS this RfD concentration would be 0.313 ng/kg vegetable and 1.65 ng/kg vegetable, respectively).

Hypothesis 3: Biosolid amended soil and forage grass contain higher PFAS concentrations than soil and in forage grass not amended with biosolids.

Objective 3: Determine the distribution and concentration of PFAS compounds removed from the wastewater treatment plant and concentrating in the process biosolids that are used for

forage crop field amendment. Compare the PFAS concentrations in soils and forage grass in biosolids amended fields with those using conventional fertilizer.

Hypothesis 4: PFAS concentrations measured in soils and the edible portion of fruits and vegetables, and forage grass will be correlated with various chemical properties of the PFAS compounds such as water solubility, chain length, octanol-water partition coefficient ($K_{ow}$), and acid dissociation constant (pKa).

Objective 4: Explore the relationships among properties of PFAS compounds ($K_{ow}$, organic carbon partition coefficient ($K_{oc}$), air-water partition coefficient ($K_{aw}$), solubility, Henry's Law constant $K_H$, molecular weight, chain length, pKa) and PFAS concentrations observed in the edible portion of fruit and vegetables irrigated with and without reclaimed water and forage grass exposed to wastewater biosolids to determine which properties are able to reliably predict PFAS bioconcentration factors in the harvested plant material.

# 2 Literature Review

## 2.1 Occurrence and Distribution of PFAS Compounds

PFAS compounds have been found in the serum of approximately 95% of the population tested in the U.S. between 1999 and 2008 by the Centers for Disease Control (Kato et al. 2011) and as such, exposure from sources other than drinking water has increasingly become of interest. In addition to drinking water, sources of PFAS have been linked primarily to firefighting with AFFF used at airports, manufacturing and industrial sites, wastewater plants (Hu et al. 2016), and landfill leachate (Lang et al. 2016). However, the prevalence of PFAS in humans, air media (Kim et al. 2007), and background samples (Brusseau et al. 2020) implies that there are

significant non-point and background sources of PFAS that affect much of the population. Examining PFAS pathways that impact communities away from highly contaminated sites is important to determine what possible pathways may impact the levels of PFAS in the general population. Consumer goods are the obvious culprit for much of the PFAS contamination found in the general populace, and the lifecycle of those consumer goods must be considered. In consumer products, PFAS compounds have been found to be released from non-stick cookware, clothing (Kotthoff et al. 2015), and even food packaging such as microwave popcorn bags (Zabaleta et al. 2017; Straková et al. 2021). Consumer goods, such as these, are the obvious culprit for much of the PFAS contamination found in the general populace, and the lifecycle of those consumer goods must be considered. The contamination of wastewater and landfills has been attributed to the release of PFAS from discarded consumer goods (Stoiber et al. 2020; Lang et al. 2016).

Experimental data for PFAS compounds are sparse with most of the compounds only having computational data to estimate their properties (Kim et al. 2009). This is largely because the properties of PFAS compounds, like $K_{ow}$, are difficult to determine using conventional methods. A summary of selected properties is presented in Table 1. $K_{aw}$ and Henry's law represent the same property; however, they were measured by two different labs and have two different values and patterns from each laboratory, so each was kept in Table 1. The sulfonic acids are expected to have pKas that are below 0 (Rayne and Forest, 2009) and therefore are always deprotonated at environmental conditions. This means their pKas are of less interest in predicting their distribution and fate in the environment.

*Table 1: Available literature values for physical and chemical properties of select PFAS compounds.*

| *Compound name and acronym* | *Carbon Chain Length* | *Molecular Weight* | *Log $K_{oc}$* | *pKa Experimental* | *pKa Calculated* | *Henry's Law (Pa $m^3$ $mol^{-1}$)* | *Log Solubility log(mol/L)* | *Log $K_{AW}$* | *Log $K_{OW}$* |
|---|---|---|---|---|---|---|---|---|---|
| *Perfluorobutanoate (PFBA)* | 4 | 214.0 | 1.9[a] | 0.4[b] | | 1.24[e] | **0.44**[f] | **-1.77**[f] | **2.51**[f] |
| *Perfluoropentanoate (PFPeA)* | 5 | 264.1 | 1.4[a] | 0.4[b] | **-0.1**[d] | 1.5[e] | **-0.56**[f] | **-1.16**[f] | **3.24**[f] |
| *Perfluorohexanoate (PFHxA)* | 6 | 314.1 | 1.3[a] | 0.7[b] | **-0.16**[d] | 0.928[e] | **-1.62**[f] | **-0.47**[f] | **3.91**[f] |
| *Perfluorooctanoate (PFOA)* | 8 | 414.1 | 1.89[a] | 3.8[c] | **-0.2**[d] | 0.362[e] | **-3.74**[f] | **0.91**[f] | **5.24**[f] |
| *Perfluorononanoate (PFNA)* | 9 | 464.1 | 2.36[a] | | **-0.21**[d] | | **-4.81**[f] | **1.61**[f] | **5.92**[f] |
| *Perfluorobutane sulfonate (PFBS)* | 4 | 300.1 | 1.79[a] | | **0.14**[d] | | **-0.94**[f] | **-0.6**[f] | **3.78**[f] |
| *Perfluorooctane sulfonate (PFOS)* | 8 | 500.1 | 2.8[a] | | **0.14**[d] | | **-5.19**[f] | **2.18**[f] | **6.46**[f] |

Experimental data for a property is listed if reported for five or more compounds from a single study, otherwise the modeled value is reported. Modeled values are in bold and are taken from the protonated species of a compound. Values for perfluorpentasulfonic acid (PFPeS), perfluornonan esulfonic acid (PFNS), and perfluorheptasulfonic acid (PFHpS) were not reported by any of the studies. a: Guelfo and Higgins (2013), b: Moroi et al. (2001), c: Burns et al. (2008), d: Steinle-Darling and Reinhardt (2008), e: Kwan (2001), f: Kim et al. (2015).

## 2.2 PFAS Toxicity

The toxicity of PFOA and PFOS is well documented in the literature analyzed for this study, and health science experts indicate PFOA has been shown to cause cancer in humans (Barry et al. 2013), resulting in PFOA and PFOS being included under the USEPAs Unregulated Contaminant Monitoring Rule (UCMR) 3. The number of PFAS compounds of concern has expanded under UCMR 5 to include all alkyl carboxylic acids between four and 13 carbons, and alkyl sulfonic acids between four and eight carbons. A lawsuit in Ohio found Dupont Chemical responsible for PFAS contamination due to their manufacturing of PFOA prompted the founding of the C8 Scientific Panel that has also concluded that PFOA exposure is a probable cause of thyroid diseases, ulcerative colitis, and high cholesterol (NTP 2019). The relationships between other PFAS compounds and toxicity have been evaluated through animal studies done by the USEPA which include effects on the immune system (Frawley et al. 2018), mitochondrial function (Wallace et al. 2013), and thyroid function (To and Dawley 2019). There is little research reported, however, on the toxicity of PFAS through non-drinking water exposures, although PFAS is known to be found in edible plants, fish, and recreational water sources (Pendland et al. 2020). In rats, dermal exposure of PFOA has been shown to change organ size (Shane et al. 2020). Lower carbon chain PFAS are observed to be less toxic at similar doses than the large PFAS compounds (To and Dawley 2019), nonetheless, this finding may be because the smaller compounds have a higher water solubility, allowing them to be more easily excreted by an exposed individual. There is also a concern for PFAS accumulation in humans over time as the half-life for PFOA and PFOS in humans is 3.8 to 5 years (Olsen et al. 2007).

## 2.3 Behavior of PFAS in the Environment

The movement of PFAS within and among environmental media is largely a function of their physicochemical properties. Experimental research on the physicochemical properties of PFAS is limited

(Rayne and Forest 2009; Kim et al. 2014; ITRC 2020); however, computational models have been used to explain the trends in these physicochemical properties among PFAS compounds (Rayne and Forest 2009) and computational model values have been found to compare favorably to some experimental results (Rayne and Forest 2009). The two most studied PFAS groups are sulfonic and carboxylic acids (Rayne and Forest 2009). The most pertinent behavioral difference between these two classifications is that sulfonic acids tend to adsorb to solids more readily than carboxylic acids, which have a higher water solubility (Wang et al. 2011; Deng et al. 2013; Feng et al. 2017), although both sulfonic and carboxylic acids have oleophilic tails and hydrophilic heads (Rayne and Forest 2009). PFAS compounds also increase in water solubility and vapor pressure as chain length decreases (Rayne and Forest 2009; Feng et al. 2017). The pKa values for sulfonic acids range from -5 to -9, while for carboxylic acids they range from 0.8 to 3.8 which means, in most naturally occurring environments, both PFAS compound classes are deprotonated and charged (Rayne and Forest 2009). The octanol-water partition coefficient has been hard to experimentally calculate due to the surfactant nature of the carboxylic and sulfonic PFAS compounds, so a similar partition coefficient using cyclic voltammetry with an n-octanol membrane has been found. These are the numbers reported in Table 1 (Jing et al. 2009; de Vogt et al. 2012; Kim et al. 2014).

Individual compounds of a specific class can also vary in behavior as isomeric forms can affect their distribution and mobility. Many of these PFAS compounds have isomers and these isomers exist in different ratios depending on the manufacturing process. Electrochemical fluorination (ECF) was largely used before 2001 by 3M (Benskin et al. 2012) produces both telomerized PFAS with a functional group on a terminal carbon and branched PFAS with the functional group located in a position other than on a terminal carbon (Rayne and Forest 2009) indicating that legacy contamination will show both types of isomers (Schulz et al. 2020). ECF does produce more linear isomers than branched isomers, but post-2001, most PFAS have been produced by telomerization which only produces linear isomers. The

branched isomers have been shown to have a lower affinity for soils and sediments than the telomerized isomers (Schulz et al. 2020). The difference between isomer mobility also affects a compound's half-life and its toxicity (Sharpe et al. 2010).

Some PFAS precursors are known to break down and form smaller carboxylic PFAS products (Houtz et al. 2016). Therefore, the distribution of PFAS compounds can change in a relatively new contamination source over time whereas old contamination should remain stable in composition because PFAS half-lives of the terminal compounds are so long (Schulz et al. 2020). Carboxylic acid concentrations increase from influent to effluent in WWTPs and this has been attributed to the degradation of precursor PFAS compounds in these facilities (Coggan et al. 2019).

## 2.4 Biomagnification and Pathways

Drinking water is the only media for which there is a PFAS advisory level in the US. However, non-carcinogenic references doses, which can be applied to different media, have been set for PFOA and PFOS at 0.0015 and 0. 0079 ng/kg BW/day, respectively, as well as for PFBS at 300 ng/kg BW/day (USEPA 2022a). In addition, a draft toxicology chronic reference dose has been released for review for PFBA (perfluorobutanoic acid) at 1,000 ng/kg BW/day (USEPA 2021). There have been other pathways of PFAS exposure proposed including ingestion of food and dust (Hollander et al. 2010), inhalation (Poothong et al. 2020), as well as dermal exposure (Shane et al. 2020). The ingestion pathway can be exacerbated by biomagnification through trophic levels (Pendland et al. 2020; Brambilla et al. 2015) or the land application of biosolids or irrigation water containing PFAS (Sepulvado et al. 2011; Scher et al. 2018). Brown et al. (2020) modeled PFAS intake and hazard index for the most common PFAS compounds. Their model presents a way to calculate the risk factors for the levels of PFAS in irrigation water; irrigation water that contained 70 ng/L PFOS consistently had a hazard quotient above 1. Hazard quotients are taken as additive so a mixture of PFOA, PFOS, PFPeA (Perfluoropentanoic acid), and PFHxA

(Perfluorohexanoic acid) would have a hazard quotient above 1 with a summed concentration ≥ 70 ng/L (Brown et al. 2020).

Food that is stored in packaging containing PFAS, including takeout food from restaurants, may also be a contributor to PFAS ingestion (Susmann et al. 2019). However, the reduction in the use of PFAS in industry does appear to reduce the amount of PFAS leaching from food packaging (Monge Brenes et al. 2019). Inhalation is mostly associated with indoor air (Ericson et al. 2012; Poothong et al. 2020), although there has been some research that shows atmospheric transport is possible (Kim et al. 2007). Dermal exposure has been proposed as a pathway, but limited toxicology studies have been conducted addressing dermal exposure with pure compounds (Shane et al. 2020).

Tefera et al. (2022) examined the vegetables, fruits, and eggs grown near firefighting stations in Australia. Their study reported a large range of PFAS compounds and higher concentrations than found in other studies looking at PFAS ingestion through vegetables. With a concentration of 236,000 ng/L of all measured PFAS in ground water, the vegetables had a maximum of 128,000 ng/kg. These are very high concentrations, and the water onsite is highly contaminated. However, unlike PFAS from wastewater treatment plants this PFAS is from firefighting and therefore has a different distribution of PFAS. Tefera et al. (2022) largely saw PFHxS in the irrigation water used at these sites.

A study conducted on industrially processed vegetables in Europe (Piva et al. 2023) showed that vegetables that were bought from stores in various states of preparation did have detectable PFAS in them. This PFAS was found in the highest concentrations in the salads and leafy greens. The concentrations were far below the Tefera et al. (2022) study with concentrations up to 400 ng/kg. They did however see that PFOA was commonly found in vegetables and that PFBA was found in the highest concentrations of the PFAS compounds they identified.

A way to calculate if a plant is bioaccumulating PFAS from the soil is to calculate a Biological Accumulation Factor (BAF), or the ratio of PFAS in the soil to PFAS in a plant's tissue. This was done by Xu et al. (2022), who applied it retroactively to previous studies of PFAS in plants. The BAFs calculated for many of these plants and compounds vary widely, sometimes by several orders of magnitude. This would indicate that the absorption of PFAS into a plant is highly dependent on conditions when PFAS is being absorbed. In hydroponic studies, like that of Gu et al. (2023), the concentration factor seems to be more consistent.

## 2.5 Wastewater and Sources in Irrigation Water

PFAS can be transmitted through wastewater treatment plants, which can lead to PFAS contamination in surface or irrigation water. A variety of wastewater treatment effluents have been tested for PFAS. Although the concentrations in municipal wastewaters remain low, when industries are known to release PFAS discharge to a municipal plant, the concentrations increase 10 to 100 times (Loganathan et al. 2007; Houtz et al. 2016; Coggan et al. 2019). Conversely, a study performed by Nugyen et al. (2022) to determine population demographic relationships and possible point sources of PFAS in wastewater found that apart from military bases, landfills, and airports there was a low correlation between industries contributing to a wastewater influent and subsequent influent PFAS concentrations. A study found that wastewater treatment plants in Australia release PFAS compounds at concentrations anywhere between less than the limit of detection (0.01-0.1 ng/L) to a maximum of 520 ng/L (Coggan et al. 2019) when all the averages for the 21 PFAS compounds are summed. The only demographic correlation found was increased PFOS concentrations in communities with older populations (Coggan et al. 2019). This demonstrates an ongoing issue with PFAS research in wastewater, the need to determine point sources from residential and commercial waste streams. WWTPs from a

variety of geographic areas and populations served show similar PFAS concentrations in their effluent, implying that population demographics do not affect the release of PFAS to wastewater (Table 2). The PFAS concentrations from municipal sources are low compared to the approximately 2,000 ng/L sum of all measured PFAS concentrations from industrial wastewaters recorded in the San Francisco Bay area (Houtz et al. 2016). The increased levels of industrial wastewaters examined by Houtz et al. (2016) were attributed to waste from AFFF from airport and military sites in the area. Wastewater sites tested in Kentucky and Georgia are more similar in population size, 15,000 and 110,000, to the WWTPs in the area studied and both have greater PFOA concentrations (Loganathan et al. 2007) than that found in local WWTPs by the Utah Water Research Laboratory (UWRL; Table 2).

Australia has instituted more stringent PFAS levels than the United States in aquatic systems of 0.23 ng/L of PFOS, compared to the United States which had 70 ng/L of PFOS as a standard until 2022 (USEPA 2016) when a new draft proposal dropped them to 0.02 ng/L (USEPA 2022). Many PFAS producers have moved to China in response to increasing concern about PFAS contamination elsewhere in the world. Consequently, the wastewater concentrations in China (Table 2) are far higher than anywhere else in the world (Wang et al. 2020). The wastewater from a treatment plant in Assamara, Jordan makes up a considerable amount of the flow of the Zarqa River which is then used to drip irrigate mint, lettuce, and alfalfa (Shigei et al. 2020). PFAS was found in the soils irrigated with the Zarqa River water. PFAS was not found in mint or alfalfa irrigated with this river water. Analysis could not be performed on lettuce due to analytical interferences. The soil for both the mint and lettuce crops showed concentrations for the substances found in the river, which were PFDA and PFOA. However, PFOS and PFHxS were also found in the soil. This is one reason why solely testing the water being irrigated will not provide an accurate view of PFAS levels via the irrigation exposure route. There is the potential that something undetected in water will concentrate and become detectable in soil and vegetables. The values found in the Jordan plant's wastewater are lower than those found in the WWTP

sampled in Utah. Since the Utah WWTP slowly mixes their effluent with surface waters throughout the irrigation season, the expectation is that the initial irrigation water will contain higher levels of PFAS earlier in the season for all locations except those adjacent to the WWTP.

*Table 2: Summary of various wastewater effluents, in ng/L, that have been reported in other studies, as well as the concentrations found in the Utah WWTP prior to this study using a different method of extraction*.*

| *Compound Acronym* | *2009 San Francisco Bay, USA** | *2014 San Francisco Bay, USA** | *Kentucky, USA*** | *Georgia, USA*** | *Australia (Mean value) **** | *China (Mean value) †* | *As-samra, Jordan ††* | *UT, USA* |
|---|---|---|---|---|---|---|---|---|
| *PFBA* | 7.4± 4.7 | 16 ± 5.8 | Not reported | Not reported | 13±33 | 87.14±74.80 | Not detected | 1.88 ± 0.37 |
| *PFPeA* | 6.7 ± 7.5 | 12 ± 11 | Not reported | Not reported | 5.3±8.8 | 40.46±54.13 | 6.4-6.8 | 32.21 ± 3.86 |
| *PFBS* | 6.0 ± 6.5 | 2.7 ± 1.5 | Not reported | Not reported | 4.0±4.9 | 74.59±101.62 | Not detected | 9.43 ± 0.61 |
| *PFHxA* | 17 ± 4.0 | 26 ± 5.1 | Not reported | Not reported | 21±17 | 66.04±59.47 | None detected | 20.98 ± 1.92 |
| *PFHpA* | 5.3 ± 1.2 | 4.4 ± 2.2 | Not reported | Not reported | 6.1±5.1 | 39.62±41.31 | Not detected | 1.21 ± 0.09 |
| *PFOA* | 32 ± 30 | 21 ± 13 | 122 | 52 | 19±19 | 481.70±677.31 | 7.0-8.7 | 4.14 ± 0.83 |
| *PFNA* | 12 ± 5.6 | 8.4 ± 3.6 | 2.4 | 9.3 | 0.92±1.1 | 14.40±10.71 | 0.8-0.9 | <0.19 |
| *PFOS* | 24 ± 32 | 13 ± 4.4 | 13 | 9.3 | 15±24 | 50.57±106.93 | Not detected | <0.35 |

* 95% confidence intervals reported by the studies. Samples from UT are reported below an MRL if more than 50% of the samples are below that limit. *Houtz et al. (2016); **Loganathan et al. (2007); *** Coggan et al. (2019); † Wang et al. (2020); ††Shigei et al. (2020).

## 2.6 PFAS in Biosolids

Biosolids that are mixed with agricultural soil to improve soil quality and provide nutrients to growing crops are another product from WWTPs that can contribute to PFAS exposure/contamination. Blaine et al. (2013) examined PFAS uptake from biosolid-amended soils by plants in laboratory and field scale studies. The crops grown in soils amended with municipal biosolids showed an increase in PFAS over control soils in the laboratory scale study. The soils within the field scale study did not show an

increase in the small chain carboxylic PFAS compounds with an increase in the application of biosolids. However, the field scale study plants did show an accumulation of PFBA and PFPeA, even at an application of 0.5 times the agronomic rate for tomato plants. This study was conducted with a 1.5 ng/g quantification limit and more sensitive experiments will need to be done to confirm these results and build on them. Blaine et al. (2013) compared their transpiration stream concentration factor (TSCFs) to hydroponic studies and found their TSCF values were higher than hydroponic studies for PFBA and PFBS indicating that biosolids may increase the transport of some PFAS into plants. Blaine et al. (2013) also confirmed that the $K_{oc}$ is effective at predicting the mobility of PFAS in soil. Considering only PFOA and PFOS the highest concentrations found in biosolids were PFOS (Brusseau et al. 2020). PFOS also has a higher log $K_{oc}$ than many of the other compounds at approximately 3.34, while PFOA's log $K_{oc}$ is approximately 2.31. This means that although PFOS is commonly found in biosolids, it may stay in the soil if there is enough soil organic matter to retain it. Analysis of PFAS compounds with soil depth also shows that the compounds with lower $K_{oc}$ values, which are those with smaller carbon chains, leach further than those with higher $K_{oc}$ values (Sepulvado et al. 2011). Smaller chain PFAS compounds are also expected to accumulate in plants as they move into the plants through transpiration pathways. In a study done by Johnson (2022), no groundwater transport was detected away from biosolid amended fields, and the highest concentrations of PFOA and PFOS were found in the soils closest to the surface, even in fields that had been continually amended with biosolids since the 1990s. This suggests that decreases in PFAS concentrations that occur over time in biosolid amended fields are likely due to degradation or absorption and uptake by plants in the amended fields not due to leaching to groundwater.

## 2.7 Plant Physiology

Specific plant physiology has not been studied extensively regarding the uptake of PFAS. There have been studies that examined where plants accumulate PFAS based on plant anatomy (Felizeter et al. 2014) and fruit lipid and protein fractions (Wen et al. 2016). None of the plants gathered in this study are the same as those analyzed in Wen et al. (2016), however, if the conclusions for these studies are representative of the plants collected from the study area, they should follow a similar trend with the majority of PFAS compounds concentrating more readily in the roots, shoots, and fruits with higher lipid and protein fractions. For example, there should be higher accumulation in zucchini relative to cucumbers (USDA 2021) even though their anatomy is similar because the protein and lipid fractions are higher in zucchini than in cucumbers.

Hydroponic studies show that plants can concentrate long chain and sulfonic PFAS compounds within roots, but those compounds are not concentrated in the foliage or stems (Felizeter et al. 2014). The greatest total concentration of PFAS compounds in plants is normally found in roots, followed by the stems, and then leaves (Felizeter et al. 2014) with less in storage tissues such as grain, fruit, and tubers (Stahl et al. 2009). Low molecular weight carboxylic acids are the PFAS that seem to accumulate most readily in leaves and fruits, while larger carboxylic acids accumulate in roots, and sulfonic acids accumulate in roots more readily than carboxylic acids (Felizeter et al. 2014). The greatest accumulation of smaller chain PFAS compounds occurs in the leaves for all plants; however, dramatic differences have been reported in where PFAS accumulates in cabbage, zucchini, and tomato leaves (Felizeter et al. 2014).

Furthermore, the soil parameters that affect the uptake of PFAS into plants are the presence of organic matter, salinity, cation exchange capacity (CEC), and temperature. There are few data points demonstrating the specific mechanisms that cause these parameters to affect the uptake of PFAS from soils. In Zhao et al. (2011), the phytotoxicity of PFOS and PFOA to Bok Choy (*Brassica chinensis*)

decreased in relation to increasing soil organic matter and cation exchange capacity. Both organic matter and cation exchange capacity increase PFAS affinity to sediments. Zhao et al. (2011) did not include a mechanistic explanation of why cation exchange capacity is a major factor in the adsorption of PFAS to soils; however, in Ebrahimi et al. (2021), divalent cation bridging was found to be a major mechanism by which PFAS adsorbed to soils. With an increase in CEC, the number of potential sites for cation bridging to occur increases. However, greater salinity and temperature have both been observed to increase the uptake of PFAS from the soil into wheat plants (Zhao et al. 2016a). These increases in salinity and temperature increase transpiration and therefore are directly related to the amount of water that a plant uses.

The most common PFAS compounds found to accumulate in plants grown in soils are carboxylic acids (Blaine et al. 2013). Lettuce grown in soils show an order of magnitude difference between the uptake of sulfonic acids and carboxylic acids, with carboxylic acids being taken up more readily (Blaine et al. 2013). However, lettuce grown hydroponically shows a similar concentration factor between sulfonic and carboxylic acids, with sulfonic acids being concentrated slightly higher (Felizeter et al. 2014). The difference between the PFAS concentrations of hydroponically and soil grown plants is the interaction of PFAS with the soil before it can be taken up into the plants. This finding is potentially due to the differences in water solubility and adsorption to soils between the two PFAS groups.

PFAS moves into a plant through the uptake of water, and because sulfonic acids are more likely to adsorb into the surrounding soil, carboxylic acids should preferentially be available for plant uptake. The uptake through water was observed when an uptake study was conducted with contaminated drinking water that demonstrated that the amount of PFBA (a C4 carboxylic acid) in fruits and vegetables correlated more with the amount of water that was being used to water plants than with the soil concentrations (Scher et al. 2018). A similar study, looking at groundwater contaminant effects on PFAS in home gardens, was conducted in Fuxin, China. This study reported much higher levels of PFAS in

the contaminated groundwater than is found in the Utah WWTP effluent. This study showed that there is a geospatial component to PFAS spread in groundwater as the primary PFAS compound in the plume changed from PFBS to PFOA. This was attributed to PFBS's preferential adsorption to soils as the contaminant plume migrated (Bao et al. 2019). This resulted in the plume's primary component changing to the less preferentially adsorbed PFAS, like PFOA. The study also showed that PFBA is the compound that had a higher daily intake from eating vegetables than just drinking the water that was used to irrigate the vegetables (Bao et al. 2019).

Moreover, a treatment plant in Kampala, Uganda has also examined the addition of wastewater effluent to the Nakivubo channel, which is then used to irrigate agricultural sites within a wetland around Lake Victoria (Dalahmeh et al. 2018). They see low level PFAS release from the treatment plant, about 5.6 to 9.1 ng/L for all species summed together, and slightly higher concentrations in the channel itself from 8.5 to 12 ng/L (Dalahmeh et al. 2018). The plants irrigated with this water do show PFAS at detectable levels in the edible portions of maize, sugarcane, and soybeans (Dalahmeh et al. 2018). The sugarcane stem absorbed the most PFAS at 380 ng/kg and maize cobs absorbed the least at 160 ng/kg. Studies done on the movement of PFAS into vegetables have shown high concentration factors from the soils into the plant material, resulting in concentrations within the µg/kg to mg/kg range in potatoes (Stahl et al. 2009). There has also been PFAS at the level of ng/kg found in commercially available foods in Europe (Herzke et al. 2013).

## 2.8 Air Deposition of PFAS

PFAS have been observed in rainwater samples in Northern Germany (Dreyer et al. 2010), Japan (Taniyasu et al. 2013), and the Maltese Islands (Sammut et al. 2017). In all these instances, PFAS concentrations for an individual sample were below 10 ng/L (Dreyer et al. 2010; Taniyasu et al. 2013; Sammut et al. 2017). However, the distribution of PFAS compounds varied greatly in these locations,

with some samples including all carboxylic PFAS compounds between four and 10 carbons, some that contained telomerized compounds, and some that contained PFOS (Dreyer et al. 2010; Taniyasu et al. 2013; Sammut et al. 2017). The absorption of PFAS in the respiratory system has been found to be limited (Wen et al. 2016), but rainfall has been found to contain PFAS in several studies in the United States. PFAS has been found in Ohio, Indiana, and Wyoming as reported by Pike et al. (2021). The range of PFAS with more than three carbons was found to be 0.002 to 290 ng/L, and both the minimum and maximum concentrations observed were PFBA. Pfotenhauer et al. (2022) and Olney et al. (2023) examined PFAS levels in air deposition as well in Wisconsin and Massachusetts. Both studies found far lower PFAS in rainwater but did not gather their own samples. Instead they analyzed samples from the National Air Deposition Program and had sites further east than Pike et al. (2021).

## 2.9 Factor Affecting Sorption/Desorption and Mobility of PFAS

Salinity, pH, cation concentration, and organic carbon have been found to affect the adsorption of PFAS to various sorbents, as well as the surface properties of the sorbents themselves (Higgins and Luthy 2005; Bräunig et al. 2019; Li et al. 2019). The difference in the soil adsorption coefficient ($K_d$) between sulfonic and carboxylic acids was demonstrated through the comparison of PFOA, PFOS, and PFOSA (Perfluorooctanesulfonamide) adsorption studies (Li et al. 2019). PFOA and PFOS are both C8 perfluoroalkyl substances, the only difference is their functional group. PFOS shows a significantly greater affinity for sediments compared to PFOA (Ahrens et al. 2014) and this affinity is seen in field measurements of suspended particles in river sediments that are high in PFOS concentrations, while PFOA is largely found in the dissolved phase (Ahrens et al. 2009).

Another consideration for PFAS adsorption is pH. The $pK_a$s for sulfonic acids are between -9 and -5, while the $pK_a$s for carboxylic acids are between 0.8 and 3.8 (Rayne and Forest 2009). These low values indicate that PFAS will remain deprotonated in alkaline buffered soil systems, like those common

throughout Utah. An increase in organic carbon has also been shown to cause a significant increase in PFAS sorption to soil because hydrophobic attraction is the primary mechanism by which PFAS are sorbed (Higgins and Luthy 2006). The effect cations have on the sorption of PFAS compounds was found to be related to a reduction in surface charge through the comparison of the effects of sodium and calcium ion concentrations. Sodium has a minimal effect on the adsorption of PFDA and PFOS compared to calcium concentration, which has a far greater effect on adsorption (Higgins and Luthy 2006). Despite these observations, no single property has been identified to reliably predict the adsorption of all PFAS to soil surfaces.

The effect of cations was also reported by Ebrahimi et al. (2021) with the adsorption of PFAS onto sludge and biosolids. Higher concentrations of ammonium and calcium corresponded to higher partition coefficients. The largest increase in partition coefficients was associated with the divalent cations, and as such Ebrahimi et al. (2021) attributed the increase to divalent cation bridging between the surfaces and the anionic heads of PFAS compounds. Depending on the functional group and PFAS size, adsorption may be affected differently by pH, organic carbon and cation concentration (Higgins and Luthy 2005). Larger PFAS and sulfonics sorption is increased more by the organic carbon content than smaller PFAS compounds, but this may be because the sulfonic functional group itself is larger than the carboxylic group (Higgins and Luthy 2005). Smaller PFAS sorption is affected more by the cations in the solution, with higher concentrations of divalent cations increasing the sorption of PFAS to sediments (Higgins and Luthy 2005).

Surface properties can also affect the adsorption of PFAS. As discussed above, the relationship that pH has on the sorption of PFAS is related to the change in surface composition and charge occurring in response to pH changes. There have been studies that compare more specific surface compositions to PFAS sorption. Li et al. (2019) divided total organic carbon in soil into saccharides, humic acid, fulvic acid and proteins to examine the effect of each of these characteristics on PFAS sorption. The existence of

proteins in soil was the most dominant influence on PFAS sorption. This may be because the hydrophilic and hydrophobic interactions between proteins and the PFAS are far stronger than those with humic and fulvic material (Li et al. 2019). Other studies have shown that humic and fulvic coated materials do sorb PFAS (Li et al. 2018), but PFAS is not readily associated with that material in the soil (Zhao et al. 2016). Navarro (2020) came to a similar conclusion when testing the desorption of PFAS in estuarine sediments. He found that organic carbon content was the dominant factor affecting PFAS adsorption to sediments rather than the salinity of the desorbing solution. Navarro (2020) commented that this finding was contradictory to an earlier study by Higgins and Luthy (2006) and attributed this difference to the possibility that differences in the type of organic material might be more important than total organic content in affecting the magnitude of PFAS sorption to soils.

# 3 Materials and Methods

All sampling containers for water samples were made of HPDE rinsed with methanol to minimize contamination. Solids were collected into ziplock bags (which were tested for PFAS contamination and found to be PFAS free) and mixed before being dried. Trip blanks showed no contamination. All chemicals used for extraction and analysis were HPLC grade or higher. Method and instrument blanks had no contamination even after the septas were pierced several times.

## 3.1 Sampling Sites

### 3.1.1 Wastewater Treatment Plant

The WWTP chosen for this study primarily serves a residential area, as well as some business and handles an average flow of approximately 1.0 MGD. The membrane bioreactor WWTP uses two

anoxic basins and four aeration tanks to remove nitrogen (Utah DEQ 2018), with the addition of alum and T-floc for phosphorus removal and to help prevent membrane fouling. The plant produces approximately 18 tons of waste activated sludge (WAS) per year that is dewatered in a belt press and dried in sand drying beds and windrow piles before being used on adjacent fields as a soil amendment. The effluent is UV disinfected prior to discharge from the plant (Utah DEQ 2018). The wastewater from this plant is treated for phosphorous removal and discharged into an irrigation ditch during non-irrigation season and treated without phosphorous removal and discharged to a pressurized secondary irrigation system during the summer when it is used for irrigating crops, lawns, and home gardens.

To track PFAS from influent to the irrigation water, influent and effluent samples were gathered in triplicate using 250 mL HDPE bottles. The influent was collected after preliminary treatment while the effluent was collected from a spigot after the UV disinfection step on three sampling dates in 2021 (June 16$^{th}$, July 21$^{st}$, and September 25$^{th}$) during the study. These samples were then placed in a cooler with a trip blank filled with double distilled water (DDW) from the UWRL and were cooled with ice packs to ≤ 4°C during transit. The samples were then stored at 4°C until processing and analysis. Aqueous samples were extracted within a month of their collection and the extracts were analyzed within a month of extraction in accordance with the holding times from USEPA Method 533 (USEPA 2019).

Water from the secondary distribution system was sampled in triplicate at six spigots along the main line that fills a reservoir at the beginning of the irrigation season. This reservoir is mixed with surface water throughout the irrigation season (Figure 1). The mixture of reclaimed water and surface water results in the variable dilution of PFAS compounds in the secondary distribution system at locations away from the WWTP over the course of the irrigation season. Measurements at the initial pressurizing of the system in April, with secondary water, were made to determine the impact of the secondary water on distribution system PFAS concentrations at the initiation of irrigation water use. Soil

was collected at the beginning of the growing season from gardens that were previously sampled in 2018. This provided a comparison between the two sampling times and allowed a comparison between the current PFAS concentrations in the irrigation water and those concentrations found in the irrigated soils.

### 3.1.2 Spigots

Spigots along the main secondary water distribution line between the WWTP and the irrigation reservoir were sampled in triplicate in June, July, and September of 2021 at seven different sites when soil and vegetable samples were sampled for a given garden location. Spigot were allowed to run for 5 seconds before sampling occurred. These were collected in 250 mL HDPE bottles and transferred back to the UWRL chilled. These sampling locations reflect the sites used in a 2018 project sampling for Pharmaceutical and Personal Care Products (PPCPs) (Figure 1).


*Figure 1: Map of mainline spigots and garden spigots.*

### 3.1.3 Gardens

Garden vegetables were harvested from five home gardens in July and September of 2021, including three gardens irrigated with reclaimed water (Gardens 1, 2 and 3; Figure 1) and two control gardens (Table 3). One control garden was a home garden irrigated with potable city water while the other uses surface water. All sites used a drip irrigation system and grew a variety of vegetables (Table 3).

Vegetable samples harvested from the gardens were transported to the UWRL, cubed and then frozen at -70°C. Once frozen, they were freeze dried and ground in a mortar and pestle for extraction.

Soil samples were freeze dried and ground in a mortar and pestle in the same way the as the vegetables. The soil samples were then sifted through a 2 mm size to remove rocks.

*Table 3: Location number, plant variety and irrigation type for the home gardens sampled in this study (locations marked on Figure 1). The control garden locations are marked in Figure 1 and 3.*

| *Garden number* | *Plants* | *Irrigation type* |
|---|---|---|
| *1* | zucchini, potato, butternut squash, soybean, tomato, carrot | secondary water drip |
| *2* | zucchini, lettuce, peppers, tomato | secondary water, drip |
| *3* | tomato, kale, beet, onion, garlic, carrot, pepper | secondary water, drip |
| *Control 1 (Background Garden Figure 3)* | lettuce, chard, peppers, tomato, potato, spinach, zucchini, lemon cucumber, green bean | culinary tap water, drip |
| *Control 2 (Background Garden Figure 1)* | zucchini, tomato, cucumber, onion, green beans | surface water, sprinkler |

### 3.1.4 Forage Crop Fields

Biosolids produced at the WWTP have been applied to soils surrounding the WWTP at a rate of 7 tons per acre on a 3-year rotational basis for 17 years. The fields are used to grow grass and other forage crops. Two fields that recently received biosolids were chosen for this study. The eastern most field had biosolids applied in the Fall of 2020, while the western most field had biosolids applied in the Fall of 2019. These fields are irrigated with surface water. There were also control fields which have never had commercial fertilizer or biosolids application on them. These fields are used for sheep forage and lie about 2 miles east from the biosolids amended fields.

Each field was split into approximately 2.5-acre sections and five random sections were chosen to sample both soil and vegetation. This was done in triplicate, so the resulting samples are three

different composites from five locations. The samples from each group of random locations were combined within a single Ziploc bag, one for soil and one for vegetation. The soil was sampled approximately 0-2 inches below the surface and hay was collected from the same area. Collection at biosolids amended sites occurred in July of 2021 after the first crop cutting, while soil and vegetation sample collection at the control fields occurred in October 2021 after a fourth crop cutting.

Ziploc bags containing the samples were transported back to the UWRL in a cooler with ice and were then frozen at -70°C to be freeze dried. Once freeze dried, the vegetation samples were ground and homogenized in a mortar and pestle. The soil samples were sieved through a 2 mm sieve to ensure any rocks were removed.

### 3.1.5 Rainwater Capture

Rainwater was collected from four AgWeather sites (a system of weather arrays throughout Utah), UWRL and a house that was previously used to gather vegetables in 2018. Rainwater was collected using a 22" stainless steel bowl with a hole drilled into the bottom (Figure 2). The bowl was covered in a plastic sheet, which was pulled through the hole and a 3/8" inner diameter tube was inserted to keep the sheet taut allowing easier drainage into 1 L HDPE bottles that were used to store the collected rainwater. The rain samplers were placed at the sites (Figure 3) before a storm that was predicted to have sufficient rain, about 0.17 inches, to sample and then samples were gathered and transported after the storm. T.W. Daniels Experimental Forest (TWDEF) was used as a background site. TWDEF was used because it sits over 1,000 m higher than Logan, UT when most of the other rainwater samples were taken. TWDEF also sits next to the Wasatch national forest which means it's also away from an industry where PFAS may have been produced.

*Figure 2: Rainwater capture apparatus. Within the bucket is a 1L polypropylene bottle to capture the rainwater.*

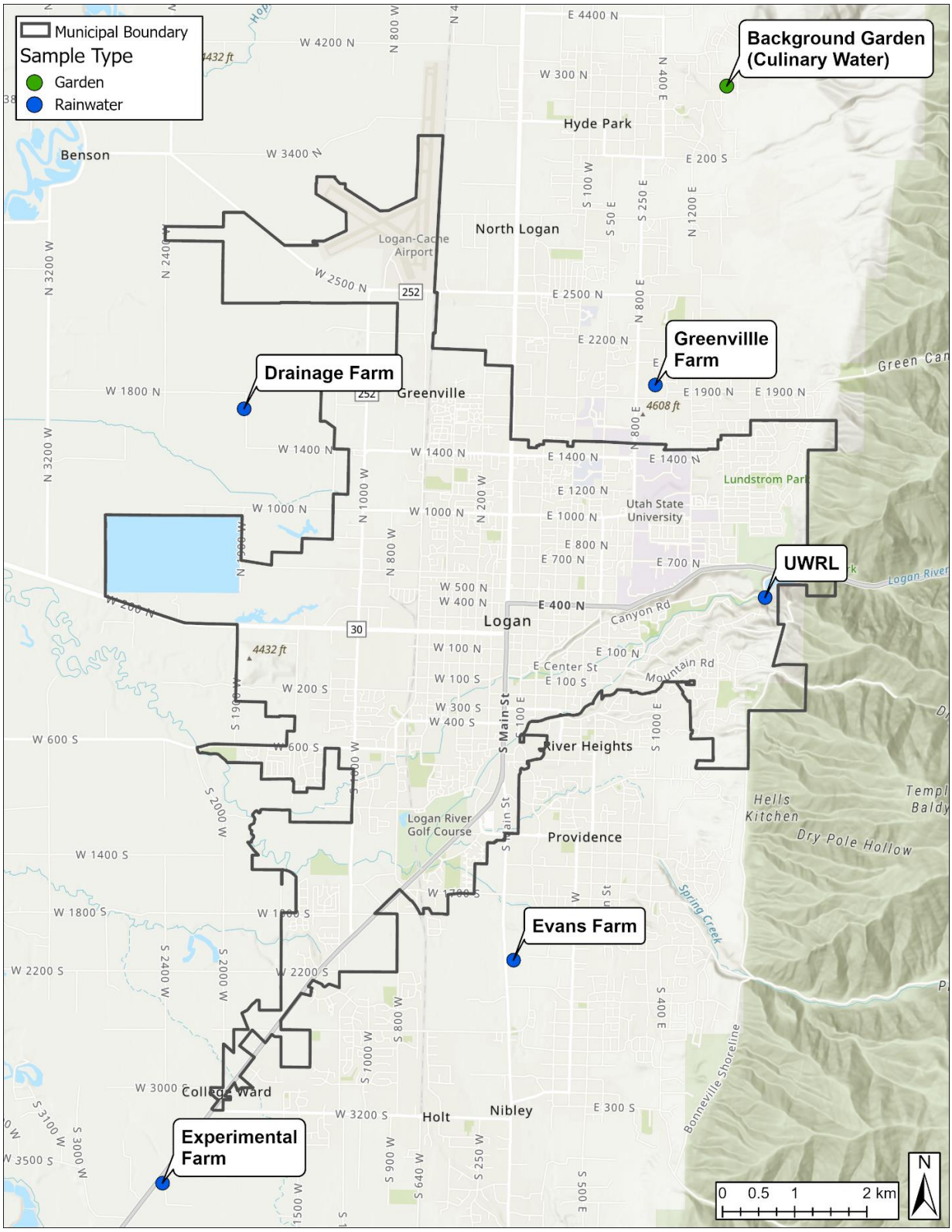


*Figure 3: Rainwater capture and culinary water background locations surrounding Logan, Utah.*

The collected water was split among four 250 mL HDPE bottles. Although USEPA Methods 533 and 537 advise against sub-sampling like this, the 1 L HDPE bottles were rinsed with 40 mL of methanol, which was then split and transferred equally to the four 250 mL bottles. This reduced the number of set-ups that needed to be assembled and dissembled for every rain event, maximizing the number of capture sites. The methanol rinse step was adapted from the USEPA Method 533 and 537 extraction process to capture any PFAS that may have been adsorbed to the bottles' surface. Moreover, the addition of less than 2% methanol into an aqueous phase has been shown to not affect the recoveries of PFAS based on the preliminary experiments, testing the methods spike recovery. Transferring the 1 L sample into four equal subsamples, rinsing the 1 L bottle with methanol to extract any adsorbed PFAS, and then equally distributing that rinse among those four subsamples is meant to avoid the adsorption issues advised about in USEPA method 533, while maintaining the sample collection system and the 250 mL sample volume for extraction.

## 3.2 Analytical Methods

### 3.2.1 Sample Preparation and Extraction

#### *3.2.1.1 Weak Anion Exchange Process (WAX cartridges) Cartridges*

Water's Oasis WAX for PFAS (6cc 150 mg 30µm Oasis Wax cartridge for PFAS analysis) cartridges were used to extract PFAS from aqueous and solid samples after extraction. These cartridges have been successfully used to extract PFAS from laboratory control samples, are shown to not contaminate samples, and are able to clean the matrix of samples extracted from vegetable and fruit samples enough to pass quality control.

#### *3.2.1.2 Cleaning*

The cartridges from the manufacturer have passed their preliminary QC check for PFAS contamination, however 10 mL of Optima grade methanol with 2% Ammonium hydroxide was flushed through each cartridge prior to use in this study to clean any residual PFAS out of them. Transfer lines were wiped with a methanol-soaked paper towel and had 1 mL of methanol drawn through them to clean out any contamination or residual water prior to use. The line system (Figure 4a) was switched to a reservoir system (Figure 4b) after the line system was found to be contaminated (Figure 4). This reservoir system was washed with soap and water, dried, then rinsed with methanol, and dried again.

#### *3.2.1.3 Conditioning*

The cartridges were conditioned by flushing 5 mL of methanol and then 5 mL of DDW through each cartridge. The cartridge reservoirs were then filled with DDW to keep the resin wet while sample loading began. This DDW was then flushed through the cartridge during the initial sample loading.

#### *3.2.2.1 Loading Aqueous Samples*

Aqueous samples were acidified with acetic acid to a pH below 3. Sample acidification is key, due to the resin needing to be protonated to extract PFAS. These samples were then spiked with 10 µL of 20,000 ng/L surrogate standard. These samples were never subsampled, so the entire sample was used, therefore ~270 mL of sample was placed into the 250 mL HDPE sample bottles. The 270 mL was a more consistent volume when samples are poured into the bottles a PVC pipe duct taped to a liter HDPE bottle. The samples were also weighed before extraction to monitor the change in volume.

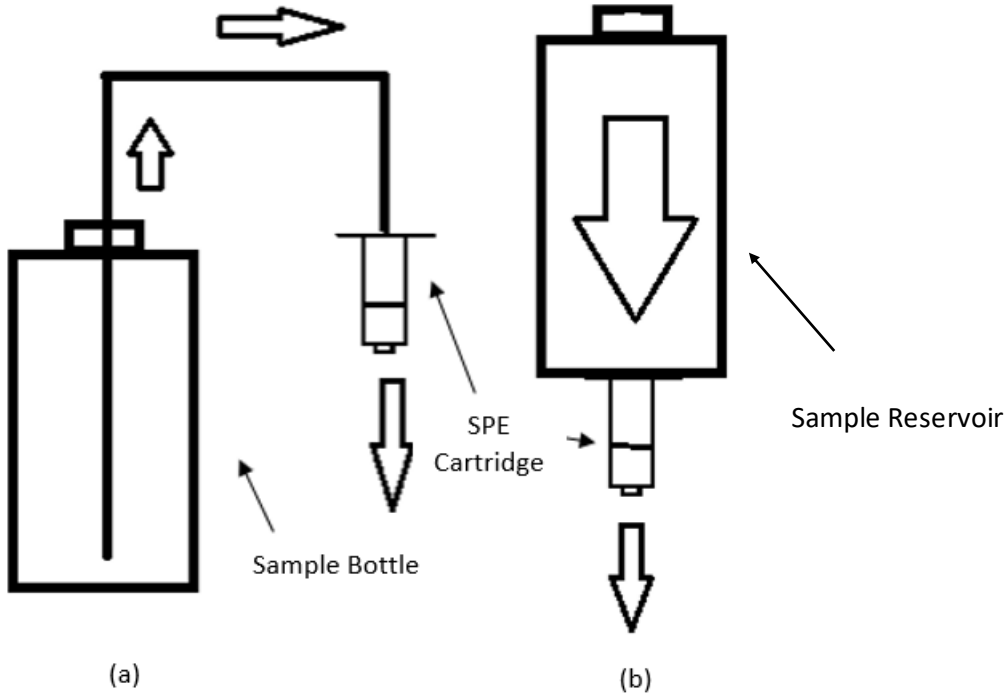


*Figure 4: Diagram of the line (a) and reservoir (b) systems used for the WAX SPE. The thick arrows represent the sample flow direction.*

#### *3.2.1.4 Flushing*

After the sample was transferred, the cartridge was dried under 15 in Hg of vacuum for 3 minutes, then the sample bottle was rinsed with 10 mL of the acetate buffer, which was drawn through the lines and cartridge under 5 in Hg of vacuum. The samples were then dried again under 15 in Hg of vacuum for 5 minutes.

#### *3.2.1.5 Drying Cartridge and Elution*

When the cartridge was dry, 15 mL polypropylene centrifuge tubes were placed under the cartridges, and the sample bottles were thoroughly rinsed with 5 mL of the 2% ammonium hydroxide in methanol solution and then poured into the corresponding sample's cartridge reservoir. Approximately 2 mL of this solution was drawn through the cartridge at 5 in Hg of vacuum and collected in the centrifuge tubes. The flow path was closed, and the methanol was allowed to sit within the cartridge bed for 3 minutes. While the methanol was soaking the cartridge bed, another 5 mL of methanol and ammonium hydroxide were used to wash the sample bottles and were then poured into the cartridge reservoir. The vacuum was disconnected, and the flow paths were opened all the way to let the elution solvent flow under gravity into the 15 mL centrifuge tubes.

### 3.2.2 Turbovap

The drying tubes were washed with soap and water, rinsed with methanol, and dried before the samples were poured from the centrifuge tubes into the Caliper ZA7516 Turbovap tubes. The eluted sample was poured from the 15 mL centrifuge tubes into Caliper drying tubes and placed into a Caliper Life Sciences Turbovap 2 to be dried under gentle nitrogen flow. The bath temperature was set to 60°C and the pressure was set to 0.9 bar. When the sample was reconstituted, it was transferred back into the centrifuge tubes that were used to collect the WAX elution.

### 3.2.3 Extraction of Solid Samples

#### *3.2.3.1 Freeze drying*

Vegetable and soil samples were placed in plastic Ziploc bags and frozen. These plastic bags have been tested using equipment blanks and have been found not to leach any of the PFAS in the standards. These samples were freeze dried for 7 days at -40 °C and below 0.280 mbar of pressure in a Labconco Freezone 4.5.

#### *3.2.3.2 Surrogate Spiking and Weight*

After the samples were dried, 1.00 g of each sample was transferred to a 15 mL polypropylene centrifuge tube. The dry sample was then spiked with 10 µL of a 20,000 ng/L surrogate standard and left to dry.

#### *3.2.3.3 Extraction*

Seven mL of methanol were added to the 15 mL polypropylene tubes and the tubes were sonicated in a Kendal ultrasonic cleaner (model HB-S-49DHT) for 30 minutes. After sonication, the samples were centrifuged for 20 minutes at 3,000 g to compact the solids into the bottom of the centrifuge tube. After the liquid was decanted, the solids left behind were then vortexed with another 3 mL of methanol, and then centrifuged and decanted like the previous 7 mL.

#### *3.2.3.4 WAX Cartridge*

The methanol with extracted PFAS in it in the centrifuge tubes was decanted into 250 mL of DDW that was acidified to below pH of 3 with acetic acid. The tubes used for sonication were disposed of. This sample was then loaded onto the WAX cartridges using the same aqueous procedures described above and new centrifuge tubes were used to gather the sample, without additional spiking.

#### *3.2.3.5 Drying*

After a sample was extracted with the WAX cartridge, it was dried in a Turbovap Drier until completely dried. The centrifuge tubes used to gather the mixture of methanol and ammonium hydroxide were left uncapped to dry after the methanol was added to the Turbovap tubes. When the sample was completely dried, the Turbovap tube was removed and allowed to cool. One mL of methanol was then placed into the Turbovap tube and swirled. The methanol was then transferred from the Turbovap tube back to the centrifuge tube that was used to collect the sample extract from the WAX extraction.

### 3.2.4 Characterization of Soils

The organic carbon, pH, EC, and texture of the soils were analyzed by the USU Analytical Laboratory. The organic carbon was calculated using a Walkley-Black test. The pH and EC were evaluated using a saturation paste, while soil texture was determined using a hydrometer method.

### 3.2.5 Chemicals

#### *3.2.5.1 Standards and surrogates*

A PFAS standard was purchased from Wellington Laboratory (product code PFAC-24PAR). This standard contained 2.0 µg/mL of $C_4$-$C_{14}$ Perfluoroalkyl carboxylic acids; $C_4$-$C_{10}$ Perfluoroalkyl Sulfonic Acids; perfluorooctane sulfonamide; N-ethyl perfluorooctane sulfonamide; N-methyl perfluorooctane sulfonamide; 4:2 Fluorotelomer sulfonic acid; 6:2 Fluorotelomer sulfonic acid; and 8:2 Fluorotelomer sulfonic acid. The sulfonic acids were in their salt form and concentrations of the salt anion were used to calculate the standard concentrations. Both linear and branched isomers were included for the $C_6$ and $C_8$ sulfonic acids. This standard was diluted into several 20,000 ng/L working standard solutions for easier storage and further dilution.

A surrogate standard, a mass labeled PFAS compound, was purchased from Wellington Laboratories (product code MPFAC-24ES). This standard contains 1 µg/mL of 19 of the 24 compounds mentioned for the non-mass labeled standards. The compounds that were not present in the surrogate mixture were $C_{13}$ carboxylic acid, and $C_5$, $C_7$, $C_9$, and $c_{10}$ sulfonic acids. This stock standard was also diluted into several 20,000 ng/L working standard solutions. These mass-labeled compounds were used for isotope dilution to track and correct for the recovery of analytes during the extraction and analysis procedures.

#### *3.2.5.2 Acetate Buffer*

Acetate buffer was made by adding 410 g of sodium acetate to 200 mL of DDW. This solution was then pH adjusted using sodium hydroxide and acetic acid to a pH of between 3.9 and 4.1.

#### *3.2.5.3 Ammonium acetate*

Ammonium acetate was used in the mobile phase and in pH stabilization and to increase the ionic strength of samples. Laboratory grade ammonium acetate was used and stored in an airtight container as ammonium acetate is hygroscopic.

#### *3.2.5.4 2% Ammonium Hydroxide in Methanol*

The elution of the WAX cartridges requires a mixture of 2% ammonium hydroxide in methanol. This was made by adding 2 mL of approximately 29% ammonium hydroxide to 98 mL of Optima methanol. This mixture was made the same day as the elution procedure and capped with parafilm.

### 3.2.6 Quality control

#### *3.2.6.1 Standard Curve*

The standard curve for each non-mass labeled compound had nine standard levels at 25, 50, 200, 600, 1,000, 2,000, 5,000, 10,000, and 20,000 ng/L. Each standard was spiked with 200 ng/L of surrogates, and the standard curve was generated. The curves were acceptable when the $R^2$ was greater

than 0.999 and all the back calculated standard concentrations were within ±20% of the expected value, except for the lowest standard which had an acceptance criterion expanded to ±50%. Only one standard could be excluded, or the standard failed this check.

#### *3.2.6.2 Reagent Blanks and CCVs*

The calculated value for the continuing calibration verification (CCVs) samples were run immediately after the standard curve and as the penultimate injection of each run. CCVs were also run after every 10 samples and at the end of a run]. These CCVs had an acceptance criterion of ±20% of the prepared value. Samples were only considered valid if all the CCVs before and the closest CCV after passed this criterion. These CCVs were prepared separately from the standard curve by someone different than who prepared the standard curve.

Reagent blanks were run after every CCV to ensure that there was no carryover or contamination between samples. These reagent blanks had to be below 1/3 of the method reporting limit (MRL) value to pass QC.

#### *3.2.6.3 LCS and Methods Blanks*

Before any method was used, laboratory control samples (LCS) that were spiked at the same concentration as the CCVs and run through the same procedure as the Samples. All had to return a value ±30% of the original spike value to pass QC. A method blank was also run through the entire procedure to make sure that there was no contamination source from the procedure. These method blanks had the same acceptance criteria as the reagent blanks.

#### *3.2.6.4 Spike Recoveries*

Duplicate matrix spikes were run for every unique matrix in a run, and another set of duplicate matrix spikes was run for every additional set of 10 samples of that matrix. These matrix spikes had an acceptance criterion of ±30% of the spiked value. These spikes were used to calculate the relative percent difference (RPD) of duplicate spiked samples.

#### *3.2.6.5 Isotope Recoveries*

Isotope recovery was calculated for every sample in a run. The surrogate recovery criterion was ±50% and any sample that fell outside of this was re-run or re-extracted if needed and possible. The recovery was calculated by calculating a one-point calibration curve using the average response of the surrogate in the standards and CCVs. The mass labelled compounds use the same acronym as their non-mass labelled surrogate, but with the additional of an M#. The # indicates the number of carbons that has been exchanged with $C^{13}$.

#### *3.2.6.6 Trip Blank*

A sample of DDW was prepared and brought to the field with every aqueous sampling group. This sample had the same acceptance criteria as the reagent blanks, and if it fell outside that criteria the samples were re-collected.

### *3.2.8 Mass Spectroscopy and Chromatography*

An Agilent triple quad 6490 was used for mass spectroscopy. The multiple reaction monitoring (MRM) used to quantify the compounds are from USEPA Method 537.1 (USEPA 2018).

#### *3.2.8.1 Autosampler Volume*

Three hundred µL conical polypropylene autosampler vials were used to hold samples prior to injection in the mass spectrometer. The samples were transferred by pouring and approximately 200 µL were transferred.

#### *3.2.8.2 Reduction in Contaminating Parts*

Agilent suggests the replacement of LCMS parts that may contaminate the samples after they have been injected into the mass spectrometer. This was not done, however, reagent blanks and the baselines of the runs were monitored and compared to previous MRL studies to ensure that no contamination from the instrumentation was affecting the results.

#### *3.2.8.3 Delay Column*

An additional chromatography column was attached before the autosampler compartment in the liquid chromatography stack. This was done to remove any contamination that may have come from before the sampling port of the liquid chromatograph by pushing the retention time of the contamination beyond the window used to record data. The column used in this study was a 3.5-µm Zorbax Eclipse Plus C18 and reduced the maximum pressure allowed in the system to 400 bar.

#### *3.2.8.4 Analytical Column*

The Chromatography column used was a 1.8-µm Zorbax Eclipse Plus C18 column. It was heated to 50°C to avoid increasing the pressure beyond 400 bar which is allowable for the analytical column but not the delay column.

#### *3.2.8.5 Mobile Phases*

The mobile phases for chromatography were 20 mM ammonium acetate and methanol. The ammonium acetate serves as a weak buffer and as a signal booster. The ammonium acetate was prepared the same day as the first sample for a run and was prepared in methanol and DDW rinsed glassware. Failure to correctly rinse the glassware can cause an increase in the baseline for perfluoropentanoic acid (PFPeA). Optima LC/MS grade methanol was found to increase the baseline for perfluorobutanoic acid (PFBA). Honeywell Chromosolv methanol was found to have a lower baseline, so the PFBA had a similar MRL to the other Perfluorocarboxylic acid (PFCA) compounds and was the organic mobile phase of choice in this study.

## 3.3 Statistical and Modeling Methods

### 3.3.1 Statistical Methods

R studio running R v4.2.1 was used for all statistical analysis including post hoc tests, Box-Cox transformations, and correlations. Box-Cox transformations were run on all data using the lambda with

the lowest residual. ANOVAs were then used to determine statistical differences between treatments ($p<0.05$) including sampling date as a blocking factor if sampling dates were not the same. If there was a significant difference, a Tukey Honest Significant Different or a Dunnett's post-hoc test was performed to further identify treatments associated with significant differences ($p<0.05$).

### 3.3.2 Modeling Methods

To test the hypothesis that the majority of PFAS is being removed from the amended soils through the absorption into hay and then subsequent harvesting of that hay, a mass balance of the removal of PFAS from soil through grass harvesting was conducted. This mass balance was calculated assuming an annual hay harvest averages 3.3 tons per acre (USDA 2021) in Utah and an average spreading rate of 5.5 tons/ac of WWTP biosolids every 3 years (WWPT manager, personal communication, September 2022). Assuming three cuttings per year, the Hay mass removed at each harvest event was 1.1 ton/ac. This value may be an overestimation as some fields may be harvested four to five times per year. However, the total annual harvest remains 3.3 ton/ac indicating that the estimate of total PFAS removal from soils is relatively similar regardless of the number of cuttings.

Because the uptake of PFAS into the grass is driven by the concentration in the soil creating an exponentially decreasing compound removal rate (Dettenmaier et al. 2009), as PFAS concentrations decrease in the soil, less PFAS is removed via plant uptake over time. To adjust to this non-linearity, the compound hay:soil partition coefficients for each PFAS compound of New and Old Soil (Equation 1) were averaged and used to estimate the soil concentrations over time. The dilution equation was used to convert the measured concentration of PFAS in soil to the mass of soil (Equation 2), where the concentration of PFAS in the soil is sampled 3 months after application. Finally, the new PFAS

concentration in the soils following the cutting and removal of Hay was calculated (Equation 3). This calculation is recursive and was used to calculate the soil PFAS concentration for the nineth cutting to compare with concentrations observed in the field samples collected from the site with biosolids applied 3 years prior to sample collection.

$$Hay: Soil\ Partition\ Coefficient = \frac{Conc.\ in\ Hay}{Conc.\ in\ Soil} \quad (1)$$

$$Mass\ of\ Soil = \frac{1}{n}\sum_{n}^{i} \frac{Conc.of\ Biosolids * Mass\ of\ Biosolids_i}{Conc.\ of\ Soil_i} \quad (2)$$

$$Conc_i \begin{cases} = Conc_{New\ Field}\ For\ i = 1 \\ = \frac{(Conc_{i-1} * Mass\ Soil - (Conc_{i-1} * ratio * Mass\ of\ Grass))}{Mass\ Soil} \quad For\ i \geq 2 \end{cases} \quad (3)$$

A generalized linear mixed-effects model (GLMM) was used to explore the effect of the physical properties of the compounds on the accumulation of PFAS in media with time and vegetable included as random intercepts to account for variation introduced by these variables (lme4 version 1.1; Bates et al. 2023). GLMMs are linear models that can take the input of several variable as both slopes and intercepts. They are often used when a system is too complex to be described by a simple two-dimensional model. In this study, GLMMs were used due to the models needing to take into account vegetable, irrigation concentration, biosolid concentration, garden, or sampling date to create robust estimates of the correlation between physical properties and measured concentrations in a matrix. The GLMMs used in this study only used random intercept values for these additional factors with the assumption that the rate of a PFAS uptake only changes with a PFAS's physical properties.

# 4 Results and Discussion

## 4.1 Quality Assurance Results

The mixture of PFAS standards supplied by Wellington Laboratories contains a total of 24 PFAS compounds. Eight of the PFAS (PFBA, PFPeA, PFBS, PFHxA, PFOA, PFNA, PFOS) consistently passed all QC criteria and are the focus of this study. Other PFAS were excluded from further consideration due to failing various QC protocol .

Spike recovery for the aqueous samples passed all QC (Table 4), . The matrix spike acceptance criteria are ±30%. Soil samples also passed QC and matrix spikes. The vegetable samples, however, failed some of their matrix spikes. The vegetable samples failing matrix spikes were excluded from further data analysis, and all samples that were included in data analysis had passing matrix spikes for both the analytical compounds (Table 4) and the mass-labelled compounds (Table 5). Similar matrices have been combined for ease of reading. The Squash category in Tables 4 and 5 contains zucchini, cucumber, and butternut, while the greens category contains spinach, lettuce, kale, and chard (Table 4). This was not how the data were handled for statistical purposes and is only for the ease of presenting spike recoveries. The excluded samples were PFBA in garlic; PFPeA in squash, onions, and hay; PFOA in beets, garlic, and greens; PFNA (perfluorononanoic acid) in garlic; and PFOS in garlic (Table 4).

*Table 4: Matrix recoveries for each analytical compound used.*

| *Matrix* | *PFBA* | *PFPeA* | *PFBS* | *PFHxA* | *PFHpA* | *PFOA* | *PFNA* | *PFOS* |
|---|---|---|---|---|---|---|---|---|
| *Rain* | *103%* | *87%* | *107%* | *86%* | *94%* | *95%* | *124%* | *93%* |
| *Spigots/Effluent* | *103%* | 104% | 114% | 100% | 102% | 104% | 108% | 100% |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| *Influent* | *93%* | 76% | 103% | 101% | 108% | 112% | 99% | 106% |
| *Soil* | *101%* | 104% | 119% | 112% | 112% | 114% | 115% | 100% |
| *Squash* | *87%* | 53% | 93% | 86% | 94% | 101% | 98% | 103% |
| *Bean* | *88%* | 97% | 122% | 101% | 92% | 95% | 118% | 113% |
| *Carrot* | *99%* | 102% | 118% | 110% | 107% | 80% | 110% | 114% |
| *Tomato* | *110%* | 78% | 118% | 83% | 71% | 81% | 83% | 73% |
| *Onion* | *95%* | -14% | 106% | 91% | 100% | 89% | 119% | 102% |
| *Beet* | *95%* | 102% | 121% | 115% | 112% | -294% | 104% | 73% |
| *Garlic* | *60%* | 115% | 114% | 106% | 90% | 440% | 61% | 64% |
| *Greens* | *103%* | 72% | 115% | 78% | 97% | 26% | 127% | 108% |
| *Hay* | *95%* | 60% | 86% | 91% | 87% | 88% | 91% | 109% |

The spike recovery in the isotope labeled compounds was also considered as an interference with the isotope which could cause an over correction of the final concentration (Table 5). So those compounds that failed the ±30% matrix spike acceptance criteria were removed from the data analysis and those that passed had their analytical concentrations adjusted according to USEPA Method 533. M5PFPeA for onions and hay failed matrix recovery (Table 5). These compounds are evaluated for use in data analysis individually, however.

PFPeA posed the most issues with matrix interferences due to glassware that was not properly cleaned, increasing the background signal for that MRM. This was solved with all glassware being cleaned with methanol before use, even after being rinsed thoroughly with water. These were purely instrument interferences that were not seen in the isotope dilution results. Hay did show extraction issues, with poor recoveries for both the mass labeled and analytical compound PFPeA. Hay was extracted at half the mass of the rest of the vegetation to avoid extraction issues. The full data set of matrix spikes is included in the appendix (Table A2). If both spike duplicates failed for a single run, the

data for that run were not used in further data analysis. If only one of the two duplicates failed, then the data were annotated but still used in further data analysis.

*Table 5: Mass labelled matrix recoveries for each mass labelled compound used.*

| *Matrix* | *M4PFBA* | *M5PFPeA* | *M3PFBS* | *M5PFHxA* | *M4PFHpA* | *M8PFOA* | *M9PFNA* | *M8PFOS* |
|---|---|---|---|---|---|---|---|---|
| *Rain* | 94% | 94% | 95% | 92% | 105% | 98% | 101% | 96% |
| *Spigots* | 105% | 104% | 103% | 102% | 100% | 98% | 90% | 80% |
| *Influent* | 104% | 120% | 113% | 92% | 91% | 95% | 109% | 103% |
| *Soil* | 90% | 95% | 95% | 99% | 92% | 91% | 99% | 87% |
| *Squash* | 100% | 87% | 91% | 95% | 94% | 94% | 99% | 99% |
| *Bean* | 112% | 87% | 93% | 95% | 107% | 93% | 112% | 124% |
| *Carrot* | 113% | 122% | 89% | 107% | 99% | 111% | 110% | 98% |
| *Tomato* | 101% | 79% | 100% | 87% | 79% | 92% | 88% | 102% |
| *Onion* | 99% | 8% | 95% | 80% | 92% | 100% | 111% | 105% |
| *Beet* | 105% | 113% | 106% | 112% | 101% | 83% | 116% | 113% |
| *Garlic* | 104% | 102% | 112% | 102% | 99% | 90% | 100% | 107% |
| *Greens* | 97% | 70% | 94% | 80% | 89% | 93% | 105% | 105% |
| *Hay* | 103% | 48% | 106% | 83% | 93% | 107% | 113% | 125% |

## 4.2 PFAS Compound Transformation through the WWTP

ANOVA results evaluating influent and effluent concentrations over the three sampling events (6/16, 7/21, and 9/25/21) indicated that concentrations of both influent and effluent streams were statistically different ($p<0.05$) as of function of time of sampling (Table 6). Interestingly, Thompson et al. (2010) did not find this to be the case in their survey of WWTP influent PFAS in Queensland, Australia.

Removal efficiencies were calculated for each sampling event for each compound and are shown in Table 7 for carboxylic acids and Table 8 for sulfonic acids.

*Table 6:Influent and effluent concentrations* ± a 95% confidence interval per *sampling date. Letters Tukey Honest Significance Difference for each compound by date.*

| | *Sampling Date* | *PFBA* | *PFPeA* | *PFHxA* | *PFOA* | *PFNA* | *PFBS* | *PFOS* |
|---|---|---|---|---|---|---|---|---|
| *Influent* | 6/16/21 | 1.40 *±0.02* A | 89.3 *±18.3* B | 3.57 *±0.10* B | 0.0803 *±0.0219* B | 0.217 *±0.003* B | 0.457 *±0.006* AB | 0.217 *±0.010* C |
| | 7/21/21 | 0.0200 *±0.0031* C | 228 *±0.8* A | 8.16 *±0.60* A | 13.7 *±1.1* A | 1.27 *±0.08* A | 0.947 *±0.068* A | 1.33 *±0.049* B |
| | 9/25/21 | 0.150 *±0.013* B | 214 *±2* AB | 7.59 *±0.01* A | 12.2 *±0.8* A | 1.09 *±0.01* A | 0.360 *±0.021* B | 2.54 *±0.10* A |
| *Effluent* | 6/16/21 | 5.33 *±0.05* a | 17.4 *±0.051* a | 21.7 *±0.2* a | 7.32 *±0.06* b | 0.17 *±0.01* b | 2.94 *±0.05* b | 0.610 *±0.008* b |
| | 7/21/21 | 2.44 *±0.11* c | 20.0 *±0.12* a | 15.0 *±0.2* b | 10.3 *±0.2* a | 0.43 *±0.02* a | 6.14 *±0.21* a | 2.74 *±0.19* a |
| | 9/25/21 | 4.03 *±0.04* b | 14.4 *±0.1* b | 9.96 *±0.26* c | 7.55 *±0.25* b | 0.31 *±0.01* a | 4.85 *±0.13* a | 1.80 *±0.06* a |

*Table 7: Percent of carboxylic acid PFAS removed from influent to effluent per sampling date. PFOA did not show a significant change between influent and effluent therefore no removal efficiency was calculated.*

| *Sampling Date* | *PFBA* | *PFPeA* | *PFHxA* | *PFOA* | *PFNA* |
|---|---|---|---|---|---|
| *6/16/21* | -280% | 80.5% | -509% | NA | 21.5% |
| *7/21/21* | -12,100% | 91.2% | -84.3 | NA | 66.0% |
| *9/25/21* | -2,590 | 93.2% | -31.2% | NA | 71.6% |
| *Mean* | -4,990% | 88.4% | -208% | NA | 53.1% |
| *95% Confidence interval* | 1,140% | 1.2% | 47% | NA | 5.0% |

*Table 8: Percent of sulfonic acid PFAS removed from influent to effluent per sampling date.*

| *Sampling Date* | *PFBS* | *PFOS* |
|---|---|---|
| *6/16/21* | -543% | -182% |
| *7/21/21* | -549% | -106% |
| *9/25/21* | -1247% | 29.2% |
| *Mean* | -780% | -85.9% |
| *95% Confidence interval* | 73% | 19.4% |

The wastewater effluent had detectable levels of PFAS (Figure 5), and the number of these compounds increased in concentration through the WWTP. The concentration change through the WWTP was significant. The concentrations increasing through the WWTP was consistent with findings from Coggan et al. (2009) who observed that PFAS compound concentrations, especially carboxylic acids, increase during treatment. This pattern is often attributed to the oxidation and transformation of larger PFAS compounds into smaller chained PFAS degradation products. The oxidation of precursor compounds typically increases the concentration of only carboxylic acids (Houtz et al. 2016), therefore this process does not explain the significant increases in concentration of PFBS and PFOS observed in the wastewater samples. In more complex matrices Rehnstam et al. (2023) attempted the TOP assay on treated leachate and found that larger chain carboxylic acids did not degrade significantly. Rehnstam et al. (2023) do mention larger PFAS breaking down into carboxylic and sulfonic acid PFAS, but the paper they cite about this is related to the improved matrix cleaning methods in food. The remaining compounds, PFPeA and PFNA did show variable but significant removal from the water after treatment

of 884% ± 1.2% and 53.1% ± 5.0%, (Tables 7 ; Figures 5). Thompson et al. (2011) saw high removal efficiencies through a reverse osmosis membrane, but in an ultrafiltration system the concentration of PFHxA, PFOA, and PFNA, increased. Pan et al. (2016) saw removal efficiencies for C4 through C10 carboxylic and sulfonic acids between -9.3% to 56% in an MBR plant. Concentration changes, both positive and negative observed in the wastewater at this WWTP were generally more extreme than those reported by Pan et al. (2016).

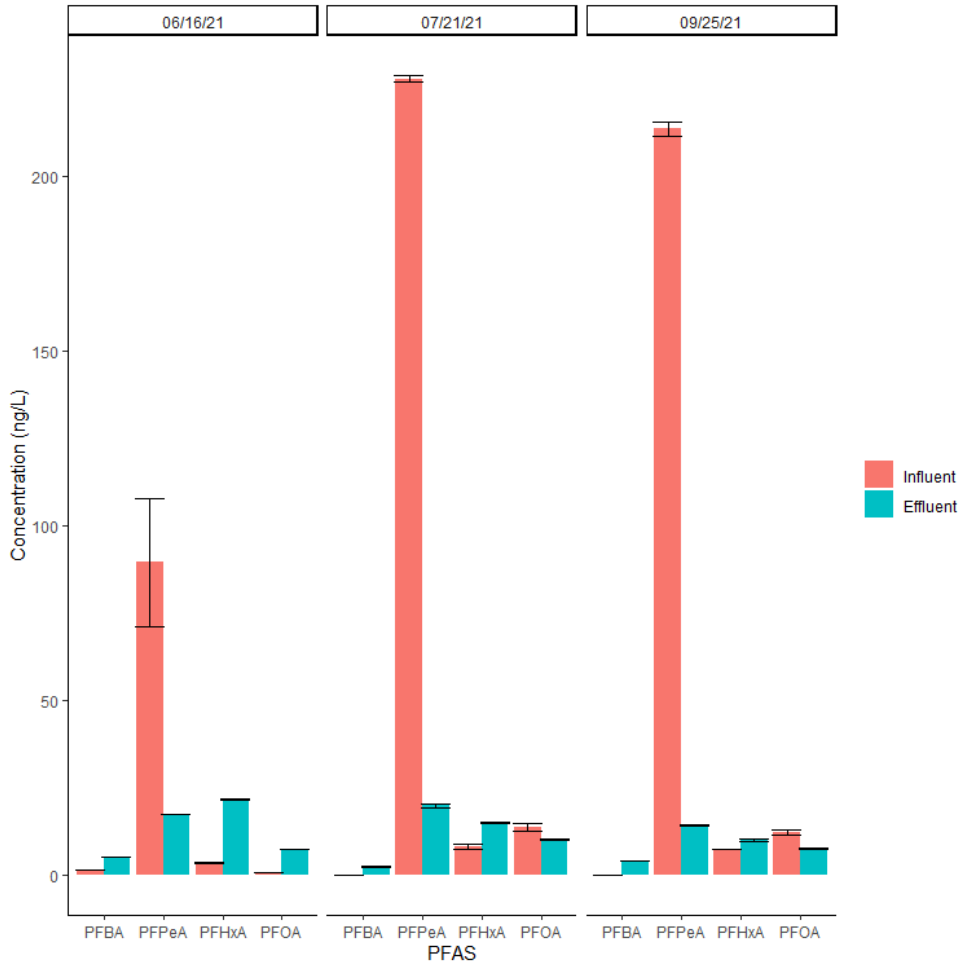


*Figure 5: Change in PFAS carboxylic concentrations through the WWTP. Error bars indicate 95% confidence intervals of all the replicates.*

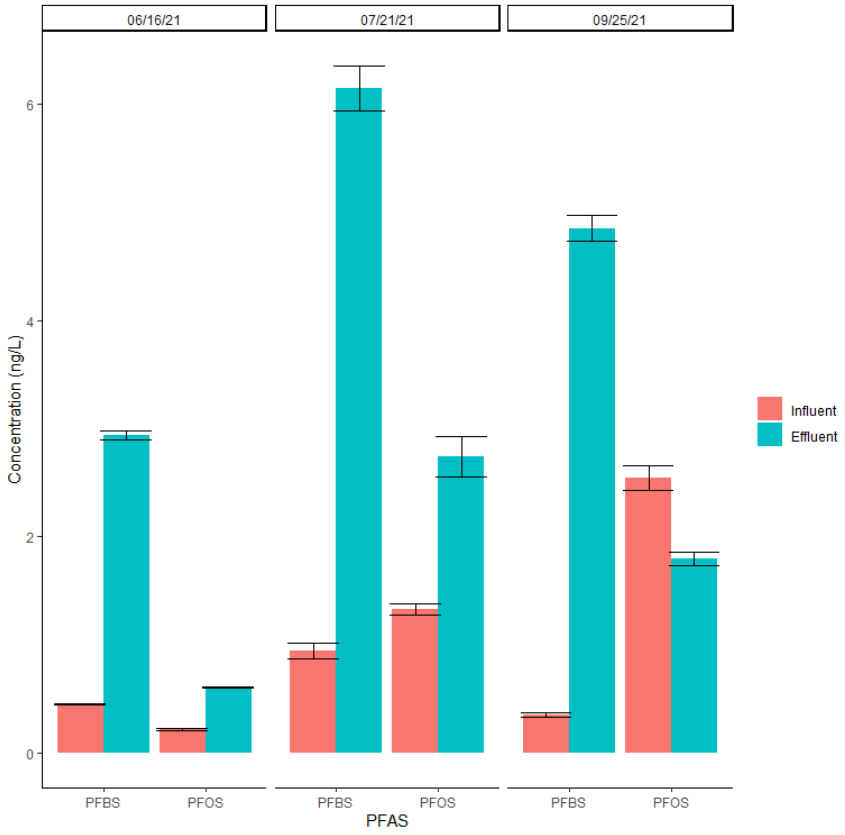


*Figure 6: % Change in PFAS sulfonic concentrations through the WWTP. Error bars indicate 95% confidence intervals of all replicates.*

Compared to effluent concentrations found in northern UT wastewater treatment plants reported in 2020 by the UWRL, similar concentrations were observed using this method from the sampling events reported in this study. These values are compared in Table 9.

*Table 9: Comparison of previous WWTP effluent concentration to effluent concentrations in this study in the same region as the study.*

| *Compound Acronym* | *2020* | June, 2021 | *July, 2021* | *September, 2021* |
|---|---|---|---|---|
| *PFBA* | 1.88 ± 0.37 | 5.33 ± 0.794 | 2.44 ± 0.646 | 4.03 ± 0.197 |
| *PFPeA* | 32.21 ± 3.86 | 17.4 ± 0.794 | 20.0 ± 3 | 14.4 ± 0.624 |
| *PFBS* | 9.43 ± 0.61 | 2.94 ± 0.246 | 6.14 ± 1.14 | 4.85 ± 0.671 |

| | | | | |
|---|---|---|---|---|
| *PFHxA* | 20.98 ± 1.92 | 21.7 ± 0.907 | 15.0 ± 1.15 | 9.96 ± 1.45 |
| *PFOA* | 4.14 ± 0.83 | 7.32 ± 0.333 | 10.3 ± 1.22 | 7.55 ± 1.36 |
| *PFNA* | <0.19 | 0.17 ± 0.0500 | 0.43 ± 0.0889 | 0.310 ± 0.0458 |
| *PFOS* | <0.35 | 0.61 ± 0.0458 | 2.74 ± 1.05 | 1.8 ± 0.344 |

## 4.3 Removal of PFAS via Solids Wasting

Using PFAS concentration in the influent, effluent, and biosolids allows an annual mass balance to be conducted for the WWTP. These mass balance calculations can be carried out by assuming that an average of 1.05 MGD of influent enters the treatment plant throughout the year and that 1.05 MGD of effluent leaves the plant as well. From this 1.05 MGD, an average of 140 tons of biosolids are produced annually by the WWTP. These assumptions are from the Year 2022, which is the closest year that accurate data are available (A. Prichett, personal communication, 2023). If PFAS are not degrading but are being removed through adsorption to the biosolids, the mass balance calculations should show the mass of PFAS compounds removed from the wastewater equal the mass of PFAS compounds associated with wasted biosolids. Biosolids concentration results shown in Tables 10 and 11 were used to complete these mass balance calculations.

*Table 10: Carboxylic acid PFAS compound concentrations in biosolids generated from the WWTP.*

| *Compound* | *PFBA* | *PFPeA* | *PFHxA* | *PFOA* | *PFNA* |
|---|---|---|---|---|---|
| *Mean (ng/kg)* | 2,030 | 2,120 | 2,740 | 11,900 | 2,450 |
| *95% Confidence interval* | 483 | 724 | 578 | 2,340 | 723 |

*Table 11: Sulfonic acid PFAS compound concentrations found in biosolids generated from the WWTP.*

| *Compound* | *PFBS* | *PFOS* |
|---|---|---|
| *Mean (ng/kg)* | 1,539 | 27,900 |
| *95% Confidence interval* | 298 | 4,690 |

Equation 4 was used to determine the mass of each PFAS compound removed from the wastewater on an annual basis. The average annual influent concentrations and average annual % removals were used in these calculations.

$$\text{Mass PFAS removed}, \frac{\text{g}}{\text{yr}} =$$

$$\text{Q (MGD)} \left(\text{Influent concentration}, \frac{\text{ng}}{\text{L}}\right) (\%\ \text{Removal}) \left(\frac{1\ \text{g}}{10^9\ \text{ng}}\right) \left(\frac{3.74\ \text{L}}{1\ \text{Gal}}\right) \left(365\ \frac{\text{d}}{\text{yr}}\right) 10^6 \quad (4)$$

Equation 5 was used to estimate the mass of PFAS associated with the biosolids wasted from the WWTP on an annual basis. An average biosolid concentration was used to calculate the PFAS mass in the solids.

$$\text{Mass PFAS removed with biosolids}, \frac{\text{g}}{\text{yr}} =$$

$$\left(\text{Biosolids}, \frac{\text{T}}{\text{yr}}\right) \left(907.2\ \frac{\text{kg}}{\text{T}}\right) \left(\text{Biosolids concentration}, \frac{\text{ng}}{\text{kg}}\right) \left(\frac{1\ \text{g}}{10^9\ \text{ng}}\right) \quad (5)$$

Results are shown in Tables 12 and 13 as the percent of annual PFAS mass removal from the wastewater associated with WAS. Less than 5% of total mass removal from the wastewater can be attributed to removal associated with the wasting of biosolids for PFPeA suggesting that transformation is taking place for this compound within the WWTP. For PFNA significantly more compound mass is associated with the wasted biosolids than was removed from the liquid, suggesting that other compound transformation resulting in PFNA production and subsequent sorption to biomass took place within the system.

There is no clear pattern of biosolids-associated removal relating to the physical and chemical properties of these PFAS compounds. As stated before, under highly oxidative conditions carboxylic acids can be generated by the degradation of larger and more complex PFAS compounds (Houtz and Sedlak 2012). Sulfonic acids are not usually formed from this in situ oxidative degradation of larger PFAS compounds (Houtz and Sedlak 2012). There are compounds that can form sulfonic acids through oxidation, however the compounds that have been found in influent are normally the variety that form carboxylic acid degradation products (Houtz et al. 2016). When any PFAS compounds break down, they form lower molecular weight degradation products (Houtz and Sedlak 2012). Therefore, the conventional ideas that PFAS is being removed through the removal of solids or that PFAS is being oxidized to form small compounds, do not explain the results observed in this study.

A complication to these data could be the extraction procedure used for the influent. The influent, which has more solids than the effluent, and could have solid associated PFAS that were not extracted as efficiently as the liquid. Unless a sample is completely dried, the water in the samples prevents the extraction of PFAS off a surface, leading to the standard procedure of completely drying solid samples before they are extracted (Lange et al. 2021). When the solids in a liquid sample are not extracted with the same procedure as the biosolids, a reduction in extraction efficiency can result in an under estimation of the incoming mass of PFAS associated with these solids.

*Table 12: Percent removal through biosolids wasting for carboxylic acid PFAS compounds that are significantly removed through the treatment plant. With the annual mass of PFAS in each stream.*

| | *PFBA* | *PFPeA* | *PFHxA* | *PFNA* |
|---|---|---|---|---|
| *Removal from Water (g/yr)* | -4.9 | 231 | -13.4 | 0.62 |
| *Removal with Biosolids(g/yr)* | 0.26 | 0.27 | 0.35 | 0.31 |
| *Percent removal through biosolids wasting* | -0.72% | 0.12% | -1.90% | 243% |
| *% of influent in biosolids* | 33.9 | 0.11 | 3.72 | 24.9 |

*Table 13: Percent removal through biosolids wasting for sulfonic acids PFAS compounds that are removed through the treatment plant. With the annual mass of PFAS in each stream.*

| | *PFBS* | *PFOS* |
|---|---|---|
| *Removal from Water (g/yr)* | -6.27 | 0 |
| *Removal with Biosolids(g/yr)* | 0.20 | 3.54 |
| *% of influent in biosolids* | | |
| *Percent removal through biosolids wasting* | -3.12% | -1.11% |

## 4.4 Secondary Distribution Line PFAS Concentrations

A Tukey HSD test showed that the concentrations of all PFAS compounds except for PFOA in Spigots 1 through 4 were distinguishable from Spigots 5 through 7 and the reservoir (Figures 7 and 8). The concentrations of PFOA in Spigots 1 through 4 were indistinguishable from wastewater effluent and PFOA in Spigots 6 and 7 were indistinguishable from concentrations measured in the surface water reservoir. Spigot 5's PFOA concentration was found to be an outlier using JMP's robust outlier test statistic with a mean concentration measured during the study of 148 ± 16.3 ng/L. The high concentration of PFOA in Spigot 5 may come from the fixtures used at this spigot, although there is no clear indication of where this high PFOA contamination might be coming from. If the WWTP was the sole

source of PFAS contamination and the mixing of reclaimed water and surface water originating from the opposite end of the distribution system occurred within the line, PFAS concentrations would be expected to decrease as the distance from the WWTP increases. This was partially consistent with the findings that Spigots 1 through 4 had higher PFAS concentrations than Spigots 6 and 7. However, a clear gradient of decreasing PFAS was not observed.

The garden irrigation spigots and mainline spigots are both fed by the main water line, however garden taps had lower PFAS concentrations than the main that feeds them (Figures 9, 10, and 11). All compounds were detected across the irrigation line, except PFPeS was not detected in Spigot 6, Spigot 7, and the reservoir samples. Garden 3 is irrigated by Spigot 5 and is the only garden that draws directly from the main line. Secondary delivery lines may experience fluctuations in water flow, and these fluctuations may result in different ratios of reclaimed and surface water being blended in the line, each with different PFAS concentrations. The amount of reclaimed water and the amount of surface water in the laterals and main lines vary and this could create a situation where the laterals to the garden spigots have trapped surface water while the reclaimed water with PFAS contamination has filled the main line on either side of the line feeding the garden spigots. More extensive and frequent sampling of spigots and monitoring of water usage across the entire system would be needed to determine how the different water sources and accompanying PFAS contamination are distributed across the system. PFAS concentrations in the respective spigot water applied to the home gardens were used for subsequent analysis of the distribution of PFAS in irrigation water and garden plant and soil samples.

When comparing samples with a Tukey Honest Significant Difference test ($p<0.05$), Every compound is different from the control in at least one spigot (Figure 12). Every compound, except PFPeS, had the highest concentration at Spigot 5 in Garden 3. Garden 3/Spigot 5 had five of six PFOA samples that were identified as outliers relative to the mean PFOA concentrations at the other spigots. JMP's outlier test does not account for replicates, it tests a single value against the existing range of all

samples of that type. This can cause a sample that was consistently high to be considered an outlier. These exceptionally high PFOA measurements did not have a clear cause since samples that were on either side of Garden 3 had lower PFOA concentration; however, the consistency of the high concentrations indicates that the PFOA concentration is indeed high. Spigot 5 was the only spigot to not follow the gradient presented by the other mainline spigots.

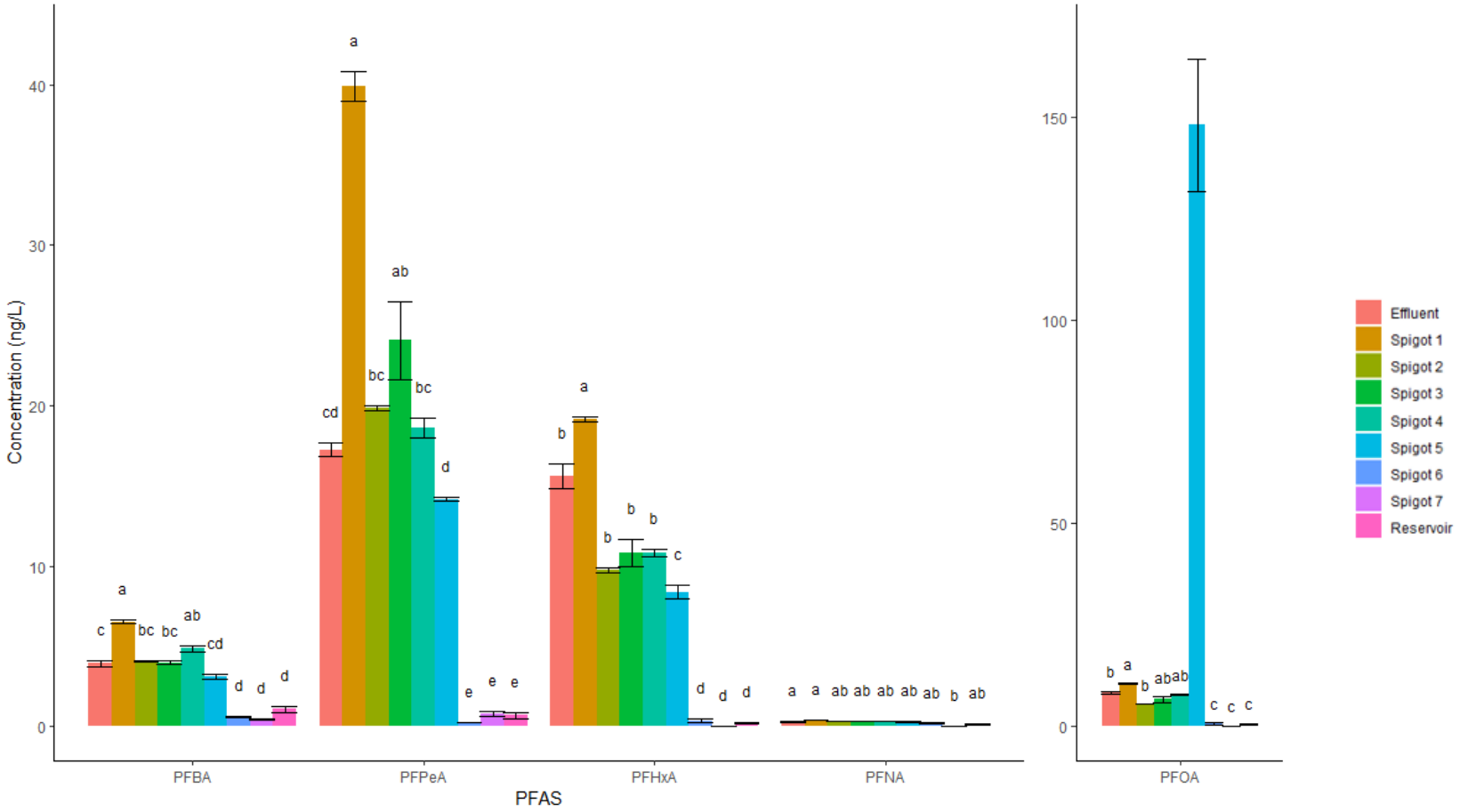


*Figure 7: Detectable PFAS concentrations with all sampling dates combined in the secondary water irrigation line starting from the effluent of the WWTP to the surface water reservoir. Spigots were sampled along the main distribution line. Error bars are 95% confidence intervals. The same letters indicate locations not significantly different for a specific compound in a Tukey HSD ($p<0.05$). Blocked by sampling date and sample spigot.*

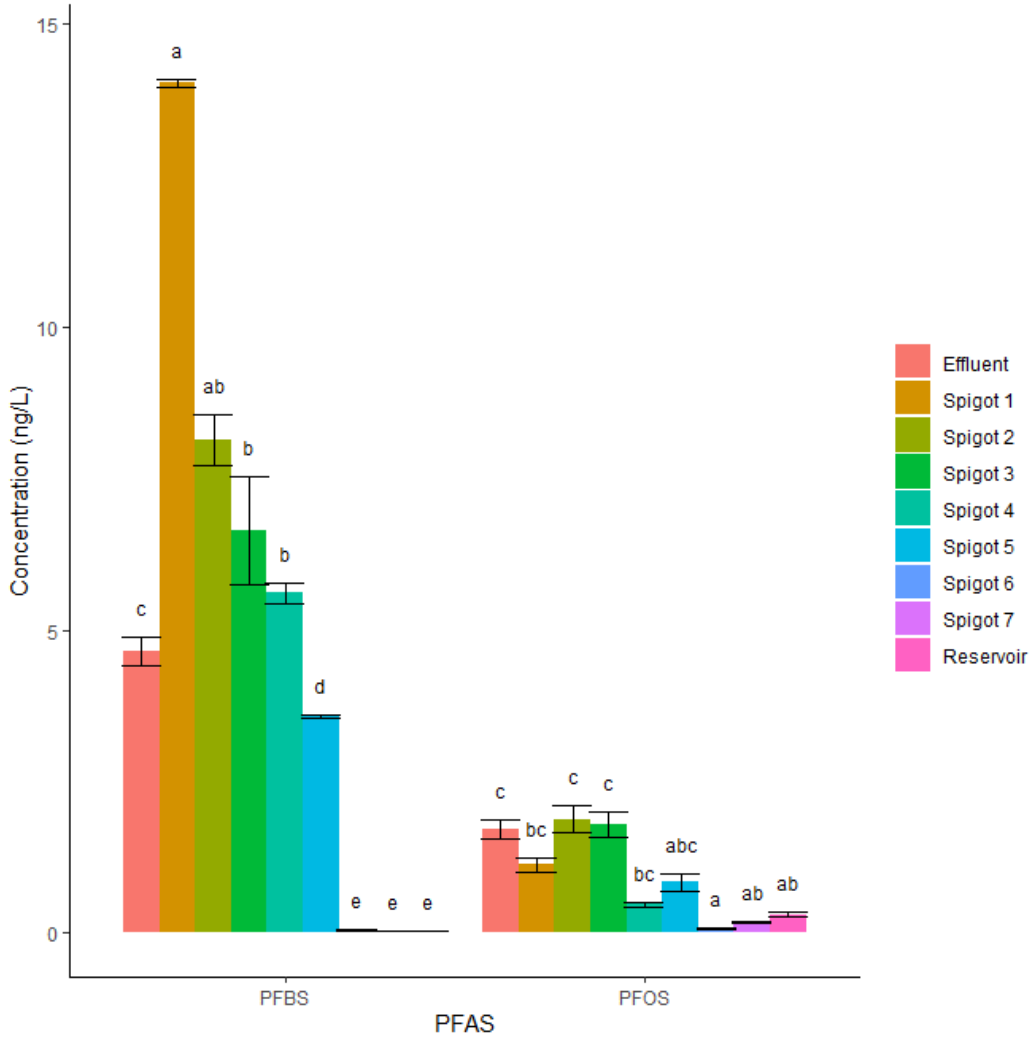


*Figure 8: Detectable PFAS concentrations in the secondary water irrigation line starting from the effluent of the WWTP to the surface water reservoir. Spigots were sampled along the main distribution line. Error bars are 95% confidence intervals. The same letters indicate locations not significantly different for a specific compound in a Tukey HSD ($p<0.05$) test.*

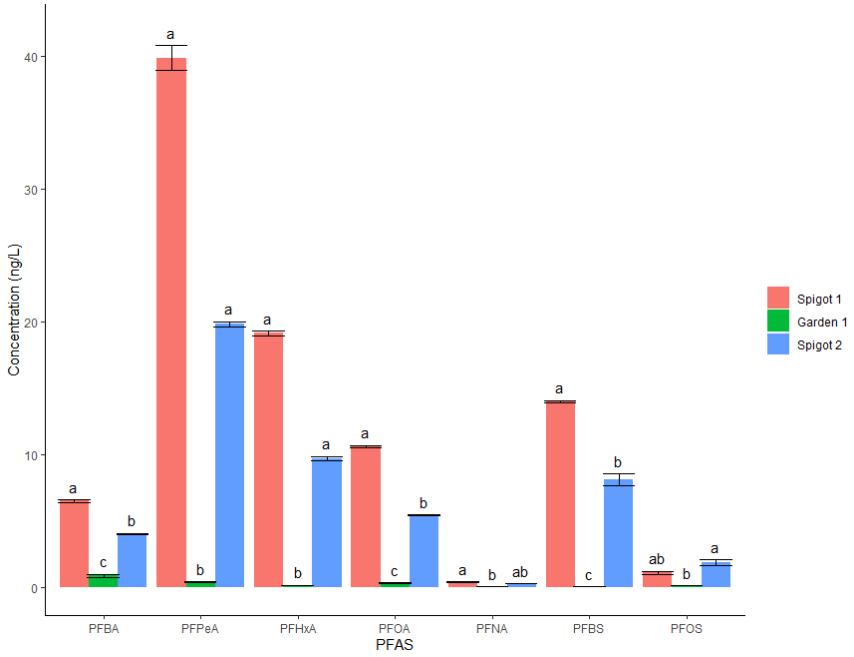


*Figure 9: Detectable PFAS concentrations from garden taps and the main line spigots on either side of the lines leading to the Garden 1 irrigation system*. Error bars indicate 95% confidence intervals.

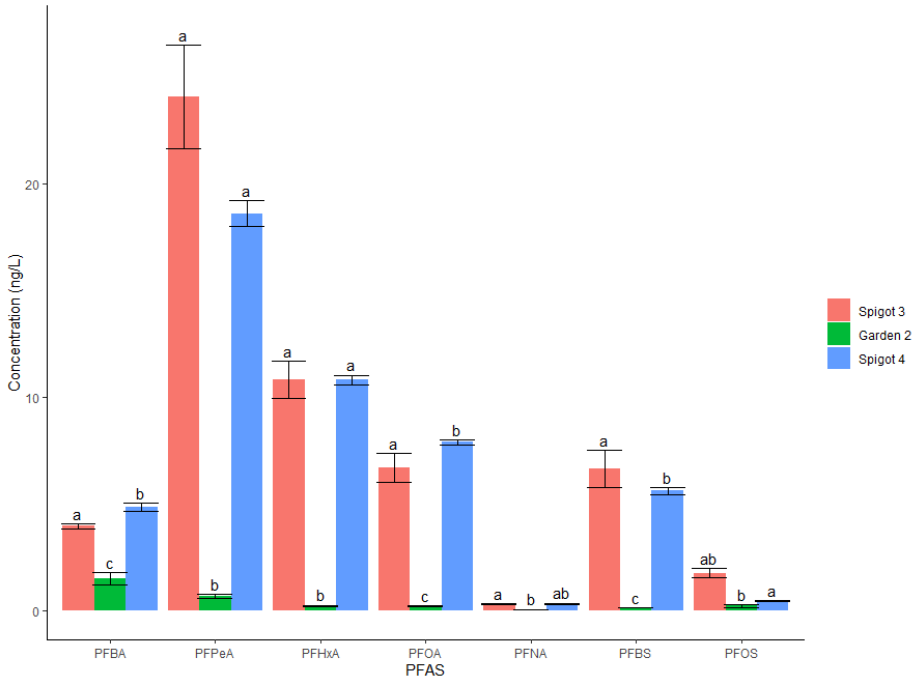


*Figure 10: Detectable PFAS concentrations from garden taps and the main line spigots on either side of the lines leading to the Garden 2 irrigation system. Error bars indicate 95% confidence intervals.*

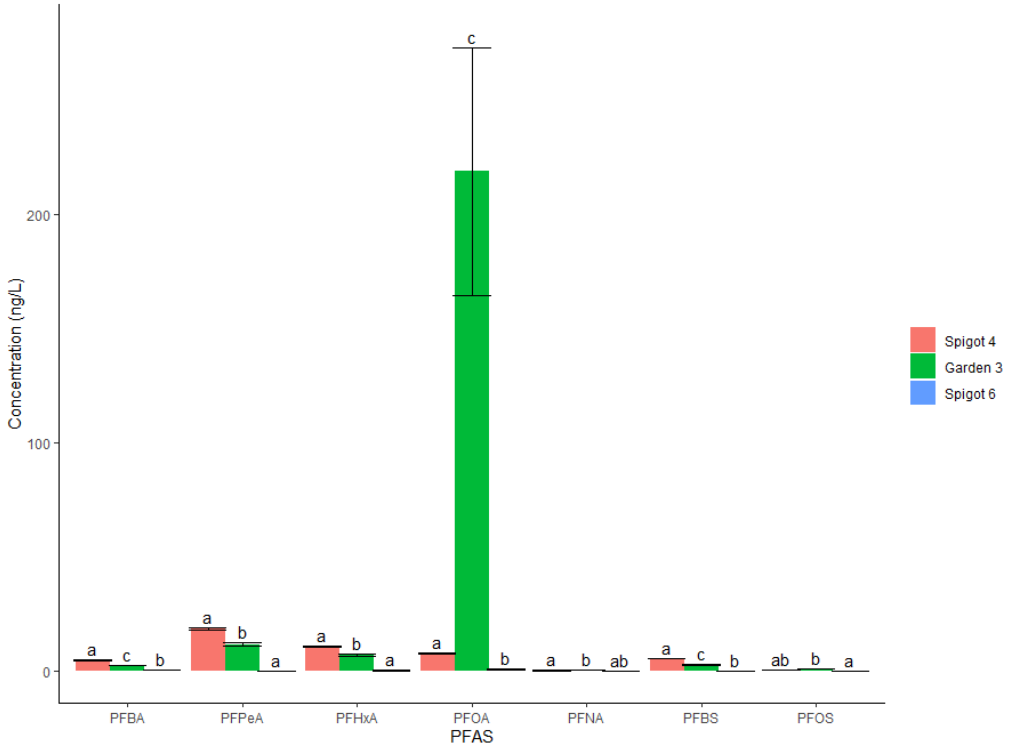


*Figure 11: Detectable PFAS concentrations from garden taps and the main line spigots on either side of the lines leading to the Garden 3 irrigation system. Error bars indicate 95% confidence intervals.*

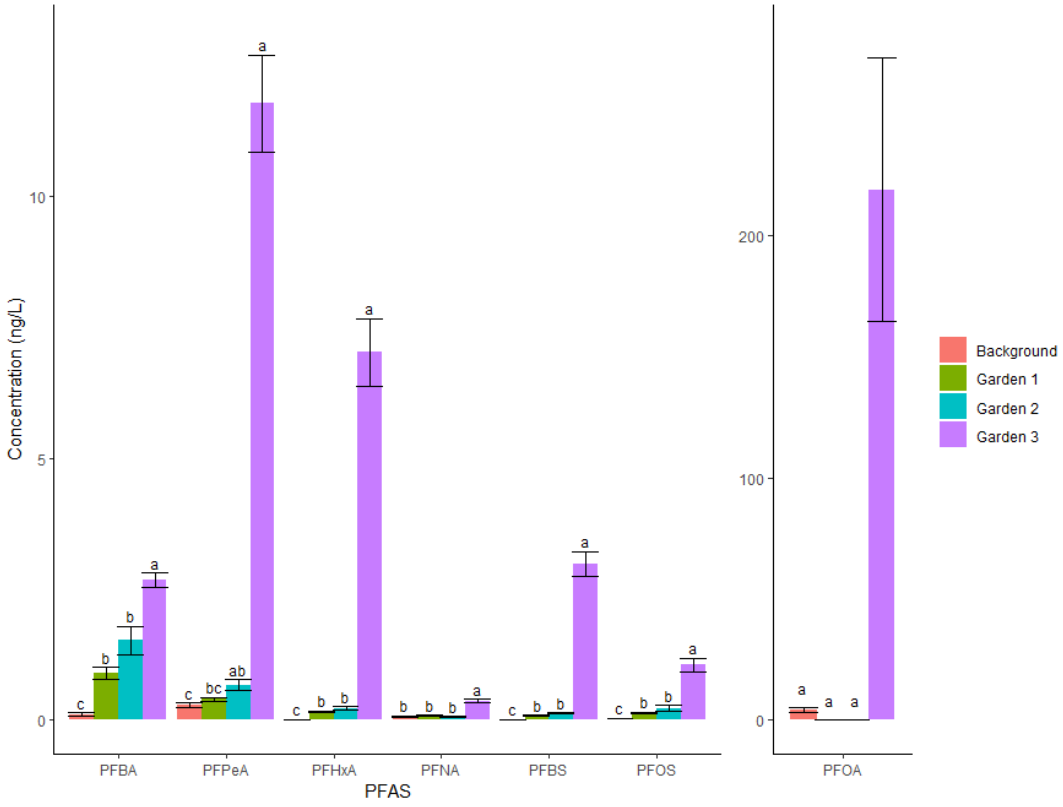


*Figure 12: Detectable PFAS concentrations in spigot samples from three Gardens and the Background Garden sites. Error bars indicate 95% confidence intervals. Letter indicates a significant difference based on a Tukey Honest Significant Difference ($p<0.05$) test.*

## 4.5 PFAS Contribution from Rain

Additional sources of PFAS could be from atmospheric deposition. Rainwater was collected to determine if PFAS was being deposited from atmospheric deposition. This study excluded dry deposition due to a lack of a method to collect and test dry deposition for PFAS. Sampling occurred for rainfall events greater than 0.43 cm to generate enough sample to analyze. PFAS was found in rainwater collected throughout the area. This could be indicative that wet, and possibly dry deposition, results in PFAS being deposited on gardens in the area. Two of the later rainwater samples collected at the end of July and beginning of August had exceptionally high PFOA concentrations, this may be due to forest fire

smoke that was around the study area at the time (Figure 13). During the other rainfall events, the PFOA concentrations were present at the same magnitude as concentrations of PFBA (Figure 13). The two PFAS compounds with the highest concentrations in rain at every sampling event were PFOA and PFBA, which were also the most common PFAS compounds to appear in soil and vegetable samples, the data for which are presented below. Pike et al. (2021) also found that PFBA was the most prevalent PFAS in the rainwater they tested, excluding trifluoric acid, which was not tested in this study. Pike et al. (2021) found similar carboxylic acids PFPeA, PFHxA, and PFOA concentrations to those observed in this study, with the exception of the last event which have exceptionally high concentrations of PFOA as discussed above (Figure 13). The sampling at TW Daniels experimental forest (TWDEF) was meant to compare wet deposition in the valley to wet deposition at an elevation high enough to isolate local sources from upper atmosphere deposition, however smoke from forest fires likely causing the elevated PFOA concentrations observed in these samples makes that comparison difficult.

Compared to literature data in the Unites States, Pike et al. (2021) reported similar values to the non-smoke rain events seen in this study. Pfotenhauer et al. (2022) and Olney et al. (2023) both found lower concentrations in rainwater. These differences may be due to geographic differences between the areas sampled, Pike et al. (2021) and this study are both further west than the other two study locations. A review of rainfall across China, US, and Europe found events in China were generally higher, however these sites are also more urban which also were found to have more PFAS in the rainfall events. More evidence from different geographical locations is needed to track the pathway of PFAS across the world through wet deposition. However, the studies find that carboxylic acids are most commonly found in rainfall and PFBA, PFHxA, or PFOA are the most common of those (Pike et al. 2021, Cousins et al. 2022, Pfotenhauer et al. 2022, Olney et al. 2023).

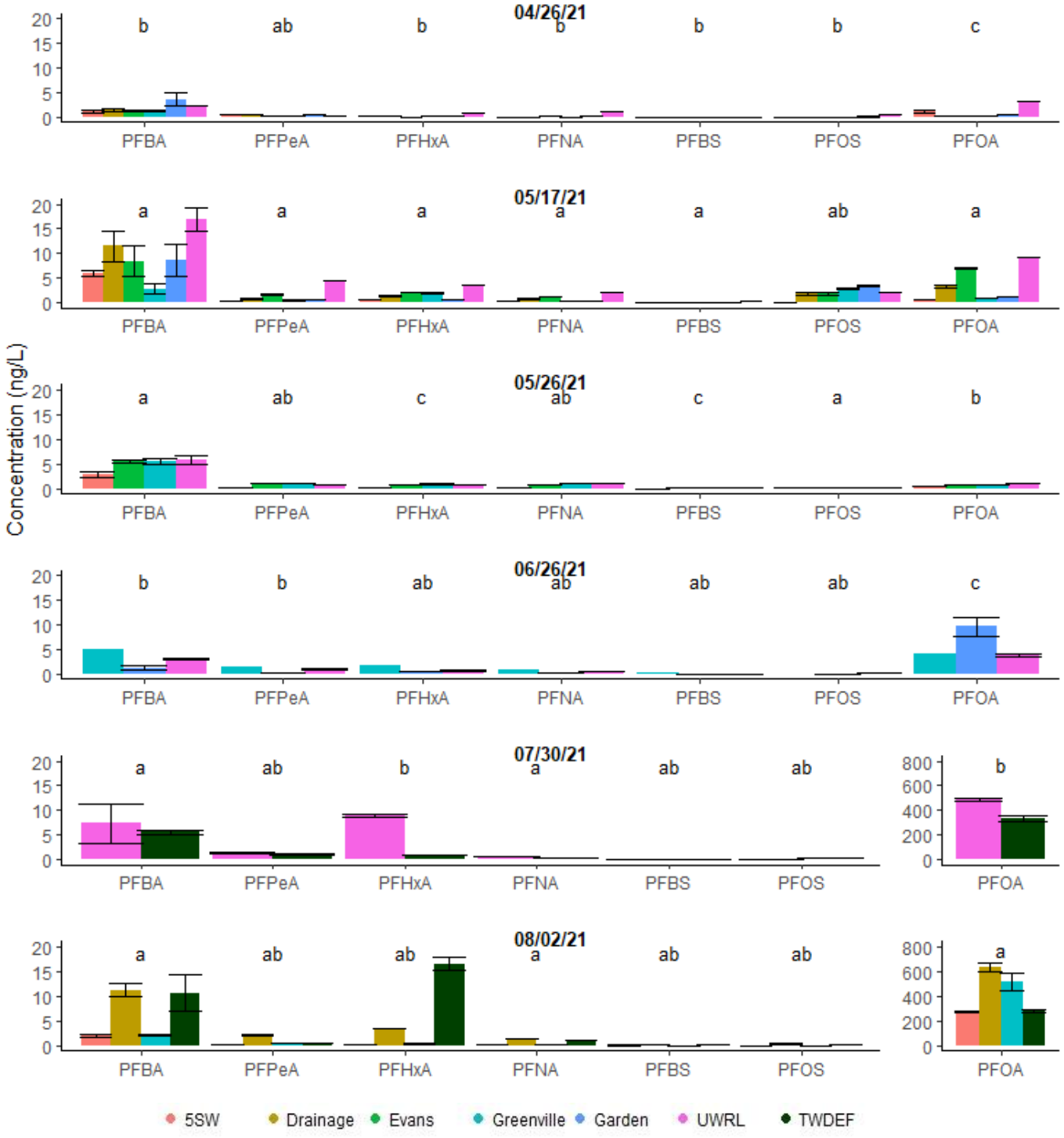


*Figure 13: Concentrations of PFAS in rainwater sampled over the summer of 2021. Error bars indicate 95% confidence intervals from subsamples. PFOA has been separated for 7/30/21 and 8/02/21 because of sampling results that are substantially higher for PFOA in July and August. Tukey groups are measuring the difference between each event with a compound blocked by location. Events that are missing sites did not have enough rainwater to collect the minimum of two samples.*

The application rate of the water to these gardens was not measured. However, USU produces water consumption data for the area, utilizing average rainfall and known plant requirements, this is known as the Consumptive Use of Water Estimates (Hill et al. 2011). The Consumptive Use of Water Estimates give an average rainfall of 8.87 (Hill et al. 2011) during the irrigation season. These data are from 2008-2011. In addition, an estimate of the amount of water used by plants is provided and includes the use by onions, potatoes, and garden vegetables more generally as 25.37 in, 18.53 in, and 17.93 in, respectively (Hill et al. 2011). This irrigation is assumed over the months of April through September, which is also when the secondary irrigation system in Hyrum is pressurized with treated wastewater. Although this is an approximation, it provides a means of estimating the relative loading between irrigation water and rainwater.

PFAS loading rates from the rainwater and irrigation water were estimated using the total rainfall number (8.87 in) during the growing season from Hill et al. (2011), the consumptive water use for onion (25.37 in) as the worst case for irrigation water, and the respective average of all events for PFAS concentrations in the rainfall and secondary irrigation water presented above. When a Tukey HSD test was run using rain, and each garden spigot carboxylic acid loadings in the rain was equivalent to either background or one of the garden spigots (Figure 14). When the Tukey test was run for the sulfonic acids, the rain loading for these compounds generally were equivalent to the background garden site and lower than the spigots (Figure 14).

The rain loading should be applied to all gardens, increasing the total loading of PFAS to each garden equally. The spigot loading is in addition to the loading from rain being applied to a garden (Figure 14). Dry deposition containing PFAS compounds could also be a source of PFAS in these gardens, however quantitation of dry deposition was beyond the scope of this study. The assumption in Figure 14

is that the gardens are only irrigating to meet the consumptive needs of the plants. It could easily be the case that residents are overwatering their gardens causing the loading from irrigation water to exceed that of rain. Ultimately, rain is a contributing factor to PFAS loading to local gardens and should be considered of the same magnitude as loading from irrigation water, particularly for the carboxylic acid PFAS compounds.

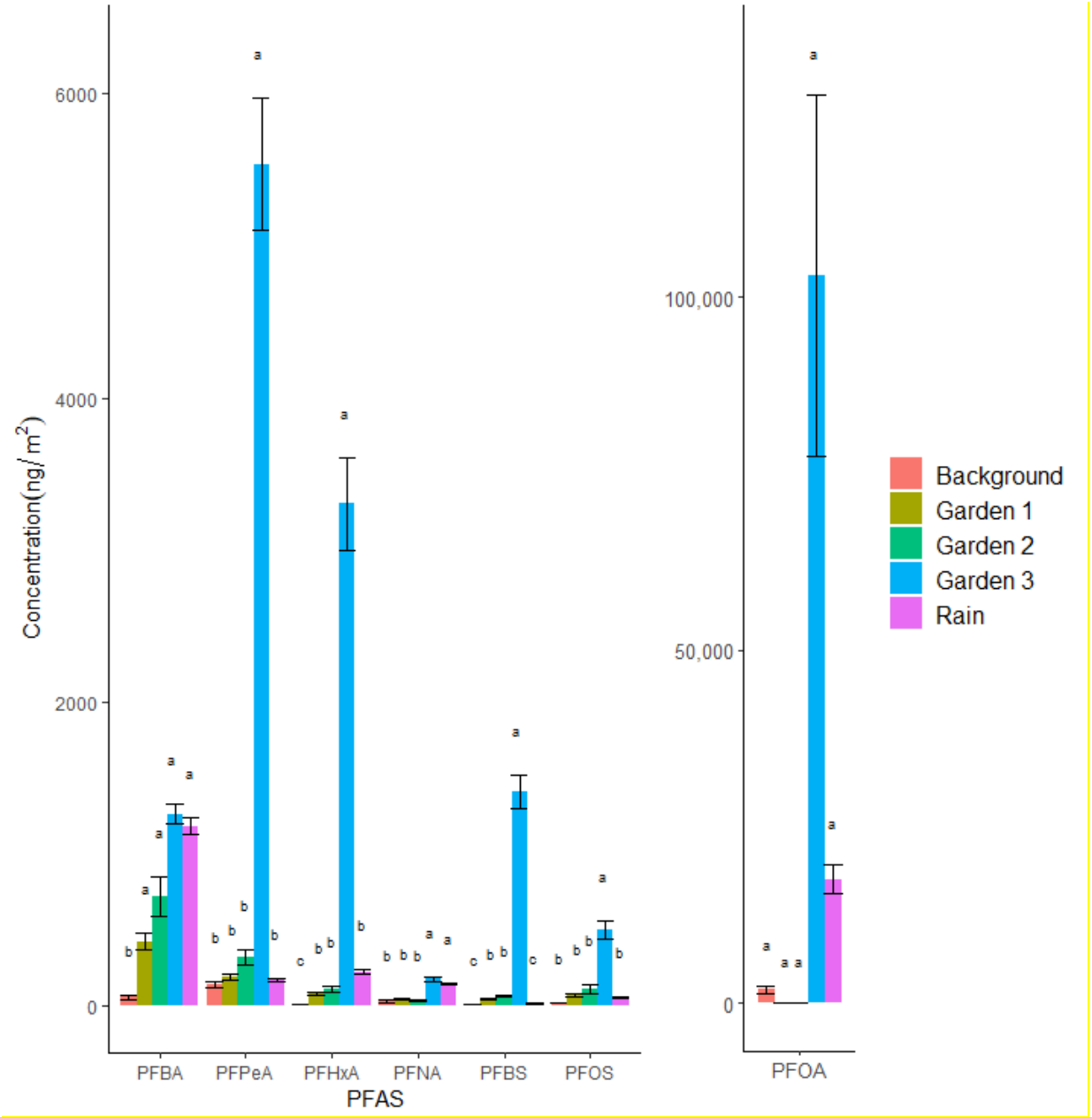


*Figure 14: Annual PFAS loading from Garden spigots and Rain per square meter of irrigation, using the plant consumption data from Hill et al. (2011), with Tukey HSD groups.*

## 4.6 Effect of Sampling Time on PFAS Concentration

Vegetable samples were collected at different sampling events. This was due to the availability of vegetables in each garden when sampling took place. Vegetables were generally collected when they would be edible and sometimes, they were too immature in July to harvest. Therefore, they were harvested in September. The difference in sampling date was included in the linear models used to run the ANOVAs and post-hoc tests. This creates a more robust test for the difference in PFAS in vegetables by accounting for the difference that is created by sampling date. This does also create Tukey groups that are non-intuitive when only one of the variables is viewed. The significance in sampling date is found in Table 14. PFBA and PFOA had the most significantly different sampling events.

When all garden soils were aggregated, all compounds were significantly different. The significance of these differences were not explored because of the lack of data. Including them in the ANOVA models was only to make sure that the sampling times were accounted for.

*Table 14: Table of the significantly different dates of sampling. Y stands for Yes; it was significant. N stands for No; it was not significant. NA is not applicable because compounds were not run for that matrix.*

| | *PFBA* | *PFPeA* | *PFHxA* | *PFOA* | *PFNA* | *PFBS* | *PFOS* |
|---|---|---|---|---|---|---|---|
| *Garden 1 Soil* | Y | N | N | Y | N | N | N |
| *Garden 2 Soil* | Y | Y | N | Y | N | Y | N |
| *Garden 3 Soil* | N | N | N | Y | Y | N | N |
| *All Garden Soils* | Y | Y | Y | Y | Y | Y | Y |
| *Garden 1 Vegetables* | Y | N | Y | Y | NA | Y | N |
| *Garden 2 Vegetables* | N | Y | N | Y | NA | N | Y |
| *Garden 3 Vegetables* | Y | Y | Y | Y | NA | Y | N |

## 4.7 PFAS Concentrations in Garden Soils

The gardens irrigated with secondary water, hereafter call the Gardens, had soil PFAS concentrations that were significantly higher than background soils for every compound except PFBS, PFOA, and PFOS based on a Tukey's HSD test (Figure 15). This indicates that some PFAS are accumulating in soils through irrigation by secondary water. However, PFBA, PFOA, and PFOS are at equal and greater concentrations in the background soil compared to the irrigated systems. This supports the observation that rainwater is an additional source of loading to soils in Cache Valley for these three compounds. None of the background gardens used commercial fertilizer or compost, however one control garden was composed of fill from an agricultural field.

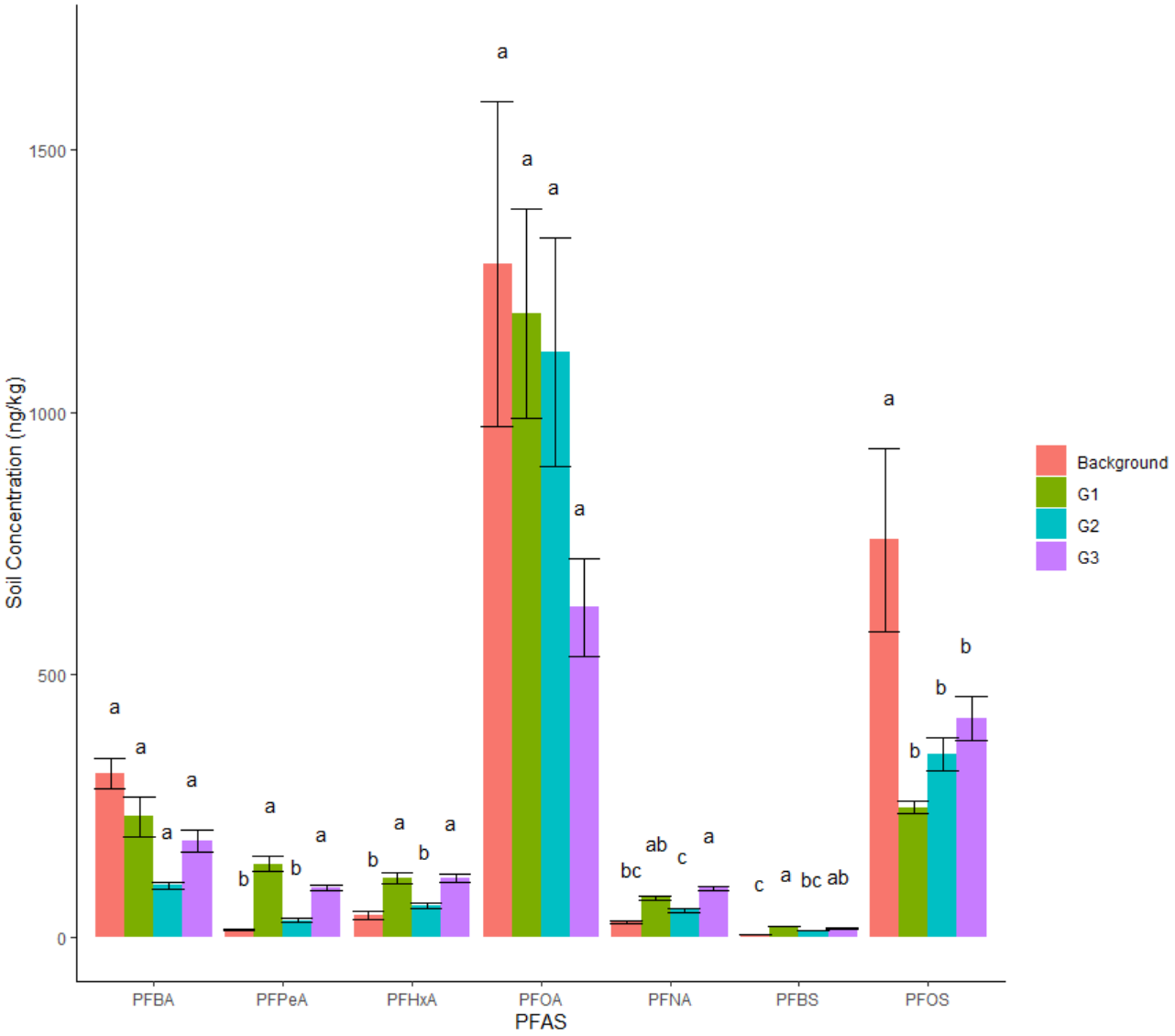


*Figure 15: PFAS concentrations in soil samples from three Gardens and two Background Garden sites. Error bars indicate 95% confidence intervals. Letters Indicates a significant difference from the mean of the Background Garden soils based on a Tukey HSD test p<0.05.*

Soil PFAS concentrations associated with specific vegetable types are shown in Figures 16, 17, and 18. For most compounds the soil concentration was no dependent on vegetable type being grown in a specific garden. The Tukey groups in Figures 16-18 include the sampling times as a covariate with

vegetable. PFOA had the greatest concentrations in all three garden soils, while concentrations of PFBS was present at the lowest concentrations. These compounds were also low in rainwater, but in irrigation water PFBS was found to have similar concentrations to other compounds. The variability in sampling times and the existing variation in soil samples makes it difficult to draw conclusions about which vegetables may remove PFAS most effectively from the soils. Interestingly, a soil sample unassociated with a vegetable collected from Garden 2 (Sample "None") had greater concentrations of PFHxA, PFNA, PFBS, and PFOS than at least one of the surrounding soils (Figure 15). This suggests the removal of PFAS through the growth and harvesting of vegetation, keeping in mind the area chosen without vegetables would not intentionally get more water than those with vegetables. Brusseau et al. (2020) agglomerated data from several studies to compare background soil PFAS concentrations from background sites. Some of their reported background numbers were much higher than the ones found in this study with PFOA concentrations they report ranging from 10 ng/kg to 123,000 ng/kg. When specifically looking at samples they report for the United States, their concentrations ranged from 3,000 to 33,000, putting the PFAS concentrations found in background soil in this study well below the background found in the Brusseau et al. (2020) study.

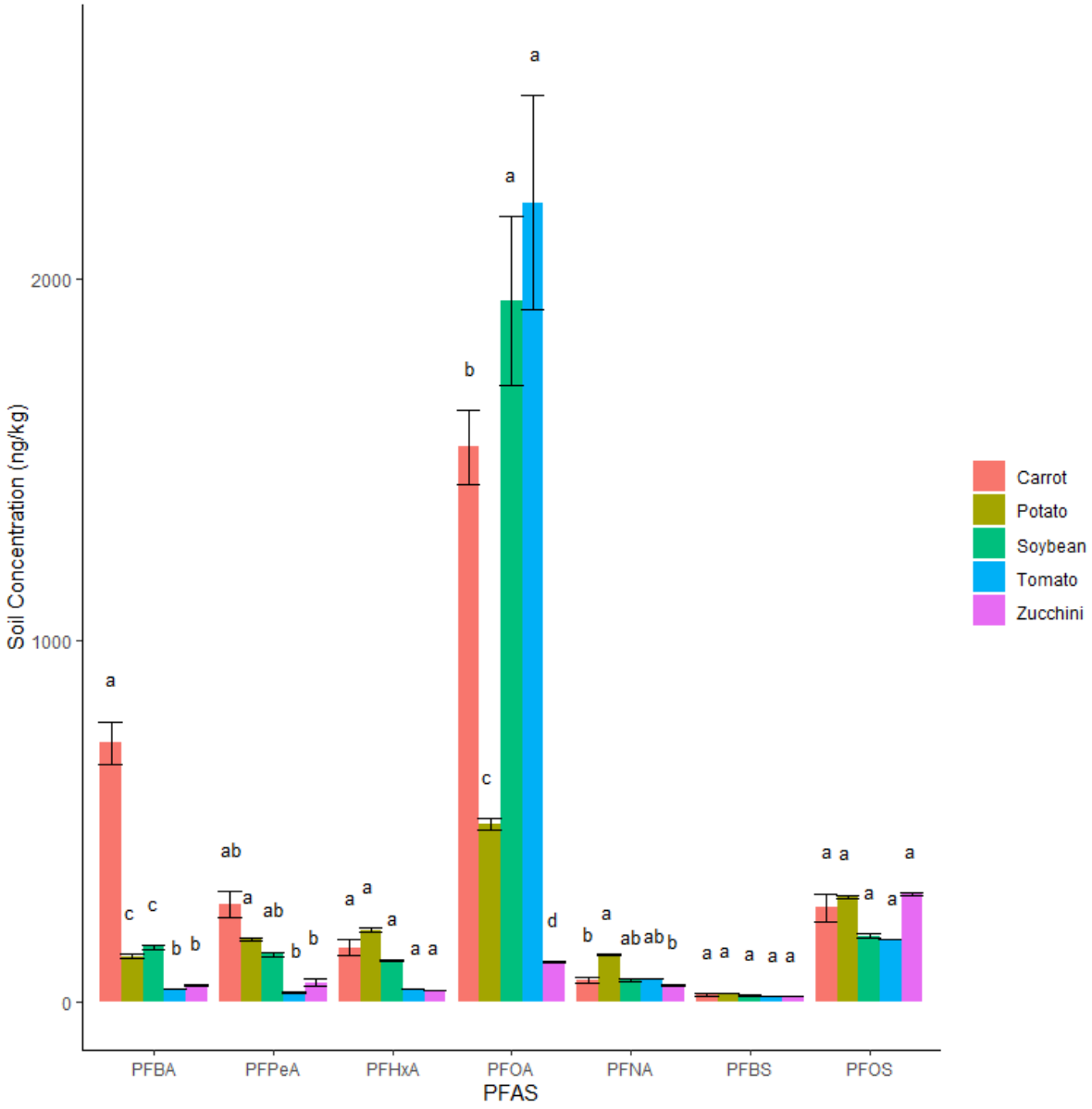


*Figure 16: Detectable concentrations of PFAS in Garden 1 soils associated with vegetables grown there. Letters are Tukey's HSD groups ($p<0.05$).*

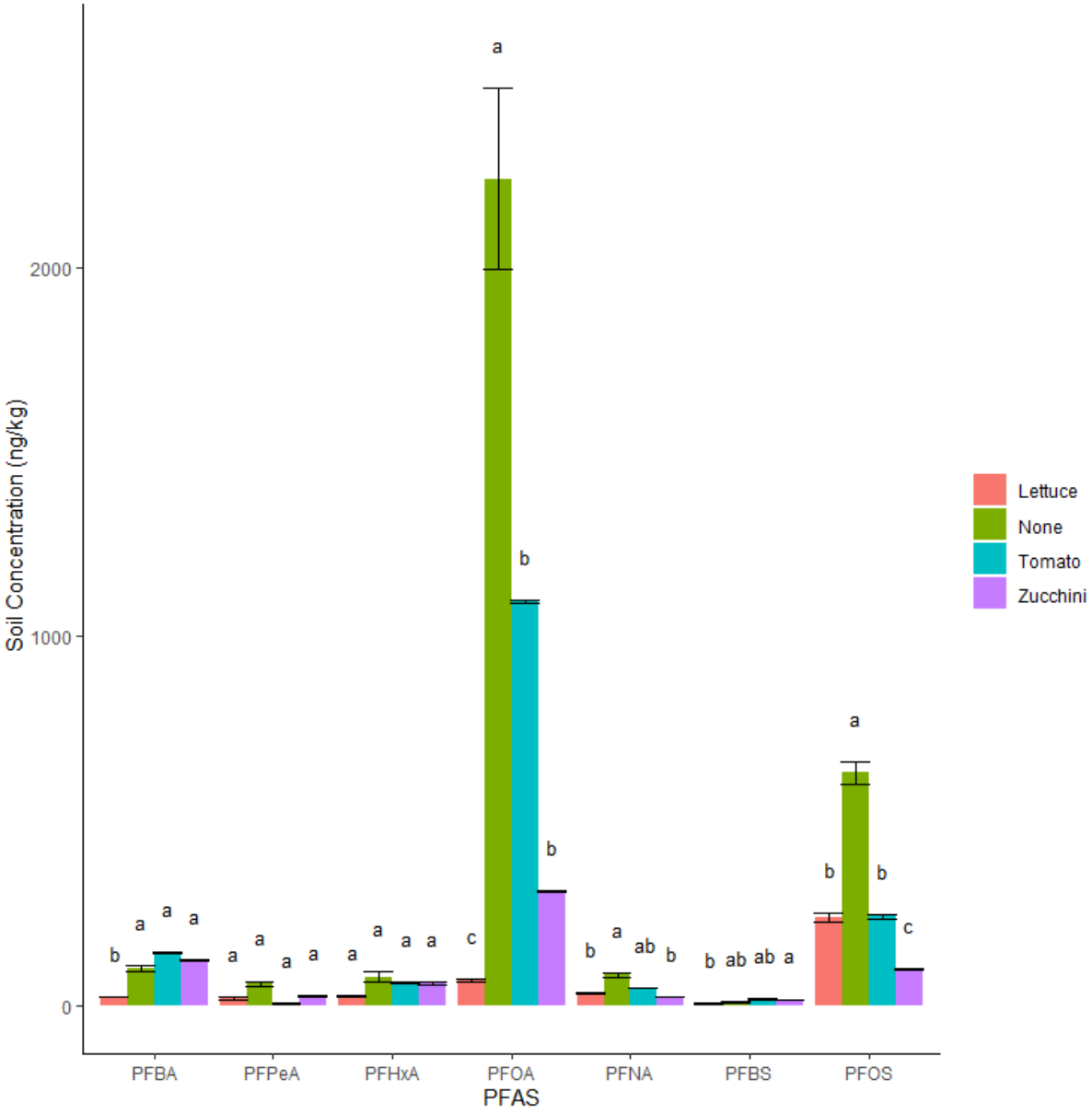


*Figure 17: Detectable concentrations of PFAS in Garden 2 soils associated with vegetables grown there. Letters are Tukey's HSD groups ($p<0.05$).*

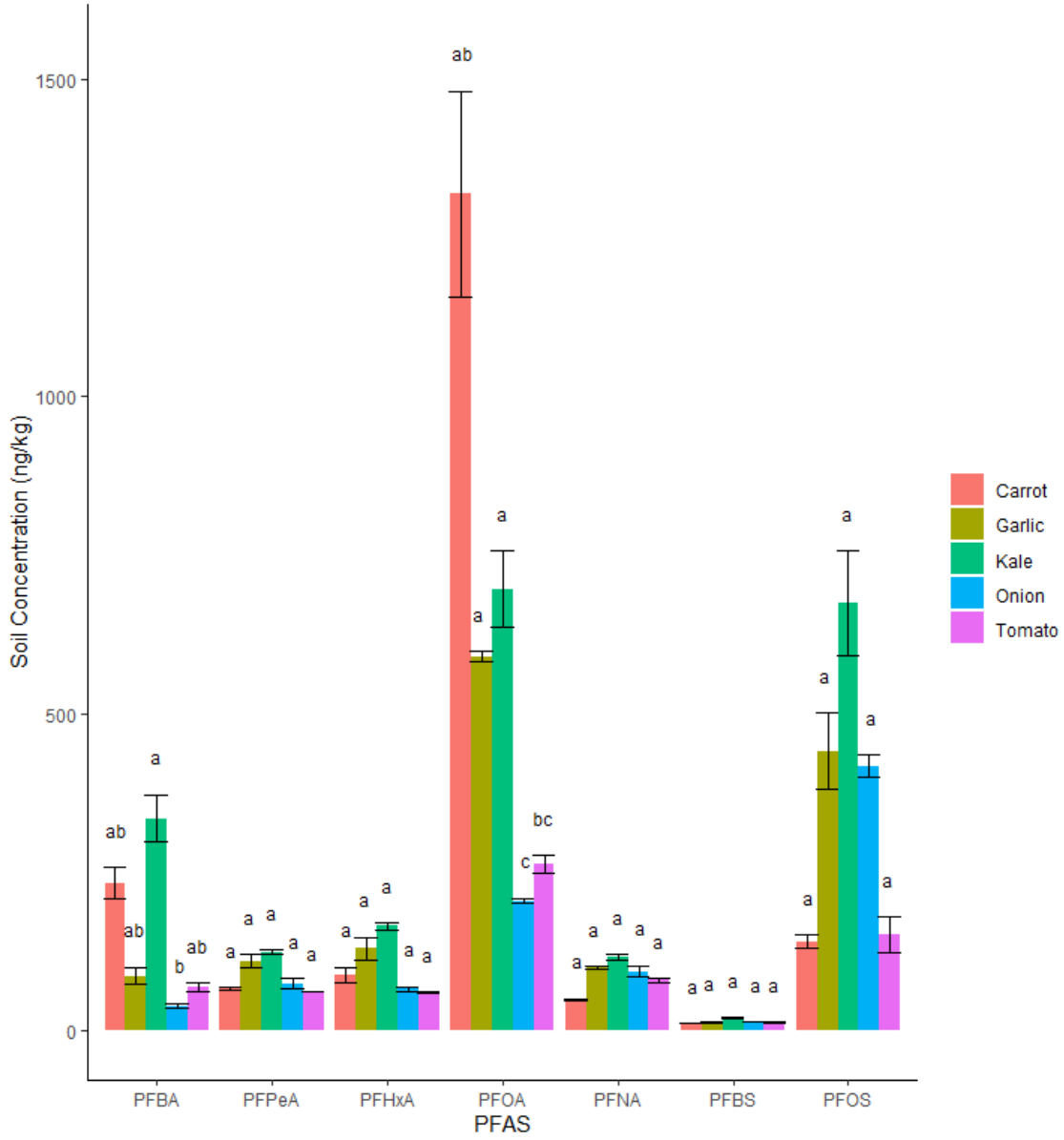


*Figure 18: Detectable concentrations of PFAS in Garden 3 soils associated with vegetables grown there. Letters are Tukey's HSD groups (p<0.05).*

## 4.8 PFAS Concentrations in Gardens with like Vegetables

Vegetable samples represented in both the Background and secondary water irrigated Gardens showed few significant differences in PFAS concentrations. Zucchini showed a significantly greater PFOA concentration in Garden 1 than zucchini from the Background Gardens, while carrots showed a

significantly greater PFOA concentration in Garden 3 than in carrots from the Background Gardens (Figure 19). Tomatoes showed significantly higher PFBA and PFPeA concentrations in both Gardens 2 and 3, and significantly higher concentration of PFHxA in Garden 3 compared to the Background Gardens, while tomatoes from Garden 1 showed significantly lower concentrations than in the Background Gardens for PFBA (Figure 19). Garden 3, which had the highest PFOA concentration in irrigation water, had greater concentrations of PFOA in harvested carrots and tomatoes than the Background Gardens. However, in Garden 3 only carrots had a significant difference from the Background Garden for PFOA in a Dunnett's Test ($p<0.05$) as shown in Figure 19.

Of all the PFAS compounds, sulfonic acids PFBS and PFOS were present in the lowest concentrations in vegetables and were not significantly different in the secondary water irrigated Gardens and Background Gardens based on a Dunnett's Test. This is consistent with the low concentrations of PFHpA, which did not have sufficient detectable data to be shown on Figure 7, and PFOS in the reclaimed irrigation water, but contradictory to the much higher concentration of PFBS detected in the irrigation water (Figure 7 and 8). In addition, these results are inconsistent with PFOS being present at the second highest PFAS concentration in soils (Figures 16-18). The low concentrations of PFBS and PFOS in vegetables analyzed in this study supports the results of Stahl et al. (2009) and Felizeter et al. (2014) that indicate that sulfonic acids are not readily taken up by plants.

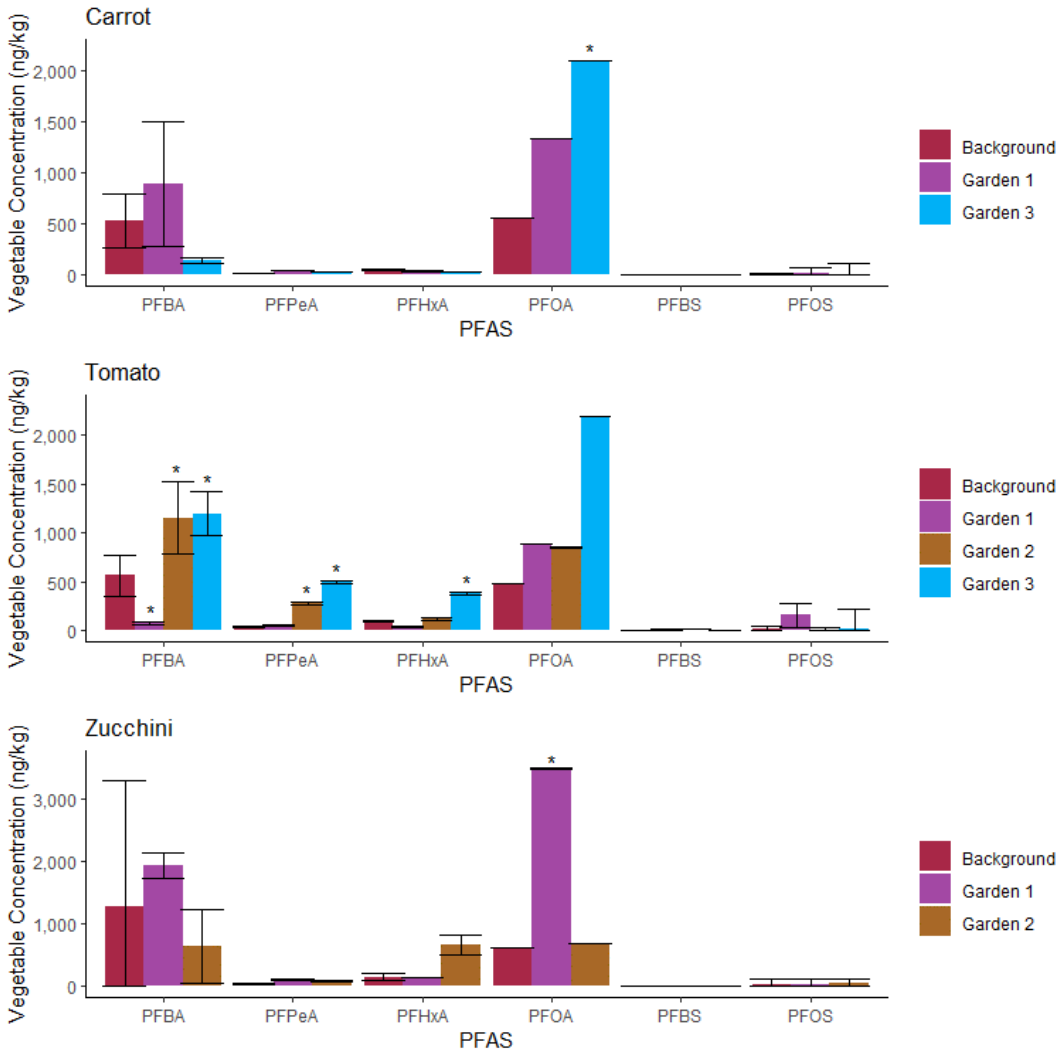


*Figure 19: Detectable PFAS concentrations in vegetables collected from reclaimed water irrigated gardens and background gardens. Error bars indicate 95% confidence intervals. * Indicates a significant difference from the mean of the control garden concentrations using a Dunnett's test P<0.05.*

The first objective of this study was to examine the concentrations of PFAS in reclaimed wastewater and compare the PFAS concentrations of soils and vegetables in home gardens irrigated with reclaimed water with those from gardens irrigated with other water sources. It was hypothesized that the soil and vegetables irrigated with reclaimed water would have higher PFAS concentrations than soil and vegetables in home gardens irrigated with other water sources. Four of the nine PFAS

compounds quantified in all media in this study (PFHxA, PFNA, PFOA, PFBS) were present in greater concentrations in Garden soils irrigated with secondary effluent compared to Background Garden soils (Figure 15). Build-up of PFOA was consistent across all Garden soils while greater concentrations of other compounds were found in only one or two of the three Garden soils analyzed.

In vegetable samples, only PFOA was consistently found above Background Garden levels in all comparable vegetables (carrot, tomato, zucchini), although tomato was not found to be significantly higher. Accumulation of PFBA and PFPeA in two of the three Gardens, and PFHxA in one Garden were also observed in tomatoes where they were grown. These results suggest that PFAS accumulation in soil and plants is highly compound and plant specific. Overall, it appears that irrigating with PFAS-contaminated reclaimed water contributes only slightly to increases in PFAS concentrations in vegetables as only six of the 49 possible plant-compound-garden combinations had statistically greater concentrations in Gardens relative to the Background Gardens (Figure 19).

The lack of vegetable, soil, and water samples that were collected at the same time as the Background vegetables make a direct connection between the secondary water irrigation and PFAS contamination in vegetables difficult to track and identify, especially when the uptake by vegetables is variable. More frequent sampling at Garden taps could capture the temporal variation of PFAS concentrations present in reclaimed water and therefore provide a more accurate measure of the PFAS compounds applied to home gardens throughout the growing season. It is possible that PFAS may build up in soils during the early part of the season when a larger proportion of the irrigation water at gardens near the wastewater treatment facility is reclaimed wastewater which has higher PFAS concentrations. Then, the built-up PFAS may be washed out during the second half of the irrigation season as the demand for water outpaces the reclaimed water produced by the WWTP and surface water with lower PFAS concentrations is used to supplement irrigation water demands. In addition, the rate of PFAS

uptake may differ across a plant's life cycle, and thus variation in the PFAS concentrations within the vegetables could result if the plant receives higher or lower doses of PFAS at different growth stages.

## 4.9 Assessment of Risk from PFAS Accumulation in Vegetables

Some PFAS compounds do appear to accumulate in vegetables from home gardens, however the source(s) of these PFAS compounds remains unclear as PFAS was present in wastewater, surface water, rainwater, and irrigation water samples. The highest concentrations were observed in Garden 3 vegetables (Figures 20-22) which also had the highest concentration of PFAS in its spigot samples. The greatest concentrations of PFAS, and particularly PFOA, were observed in leafy vegetables, including kale and lettuce (Figures 21 and 22). The greater accumulation of PFAS in leafy greens is consistent with the results of Felizeter et al. (2014) who found that more short-chain carboxylic acids were found in the leaves of plants relative to other plant compartments. Vegetables that develop underground do not show the same trend as tomatoes and squash that develop above ground, with below ground vegetables showing higher PFOA concentrations. Tomatoes and squash show relatively low concentrations of all PFAS compounds, but a larger variety of compounds are present within these vegetables (Figure 20-22) than those that grow below ground. Literature values from European vegetables have found lower concentrations in store bought and industrially processed vegetables finding up to 400 ng/kg (Piva et al. 2023). Piva et al. (2023) reported that PFBA had the highest concentrations in vegetables while PFOA, which was found to have the highest concentrations in this study, was found at concentrations similar to PFHxA, and PFPeA in the store bought and industrially processed vegetables they analyzed. Another study carried out at an Australian AFFF contaminated field found higher concentrations, ~38,000 ng/kg, than this study, and reported the highest concentrations were PFOS (Tefera et al. 2023). PFOS was found at low concentrations in vegetables analyzed in this

study (Figure 19) as was reported by Piva et al. (2023). Tefera et al. (2023) did not detect any PFOA in the vegetable samples they analyzed.

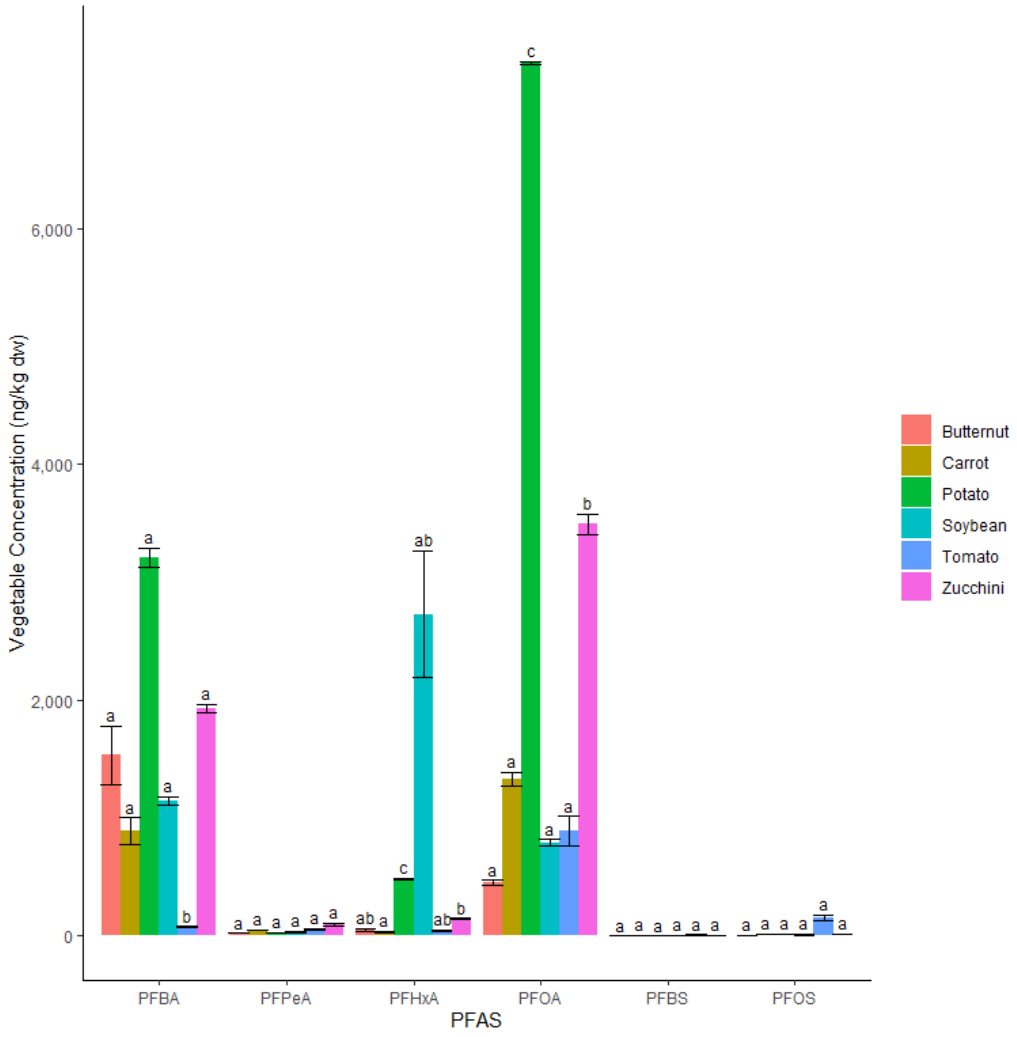


*Figure 20: PFAS concentrations for vegetables, on a dry weight basis from Garden 1. Letters are the groups from a Tukey's HSD test ($p<0.05$) within a compound blocked by sampling date and vegetable.*

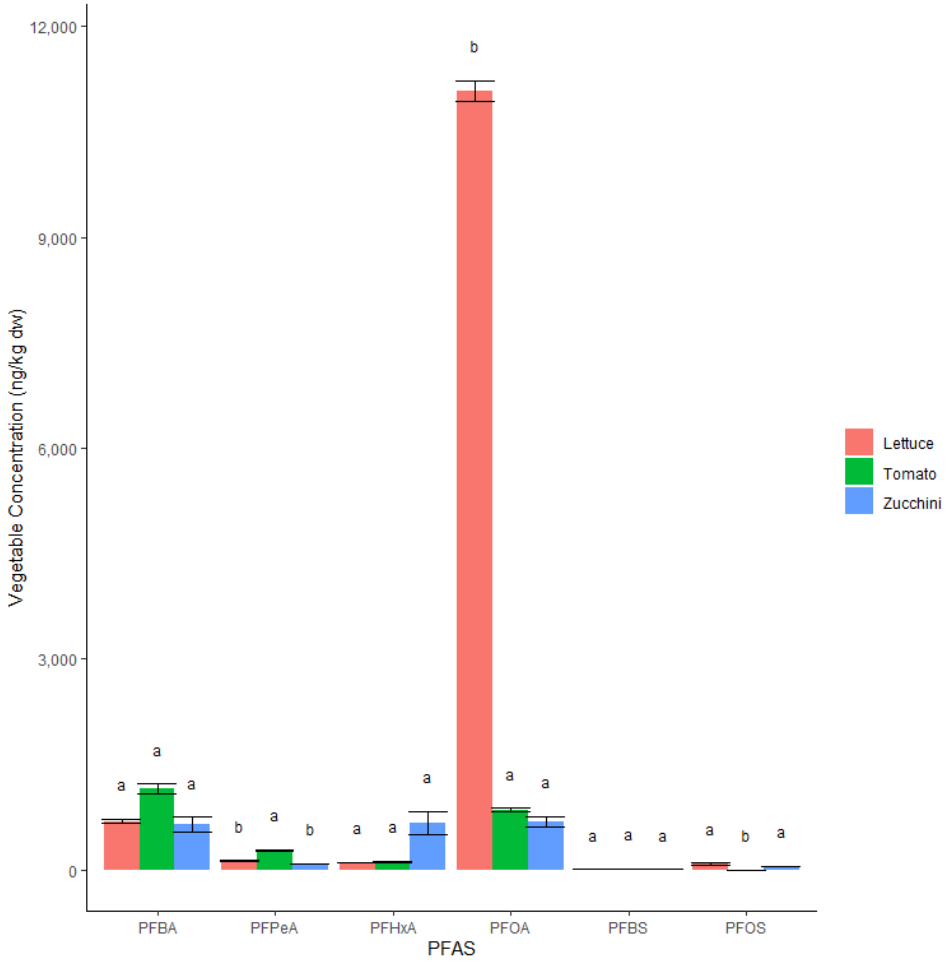


*Figure 21: PFAS concentrations for vegetables, on a dry weight basis from Garden 2. Letters are the groups from a Tukey's HSD test ($p<0.05$) within a compound blocked by date and vegetable.*

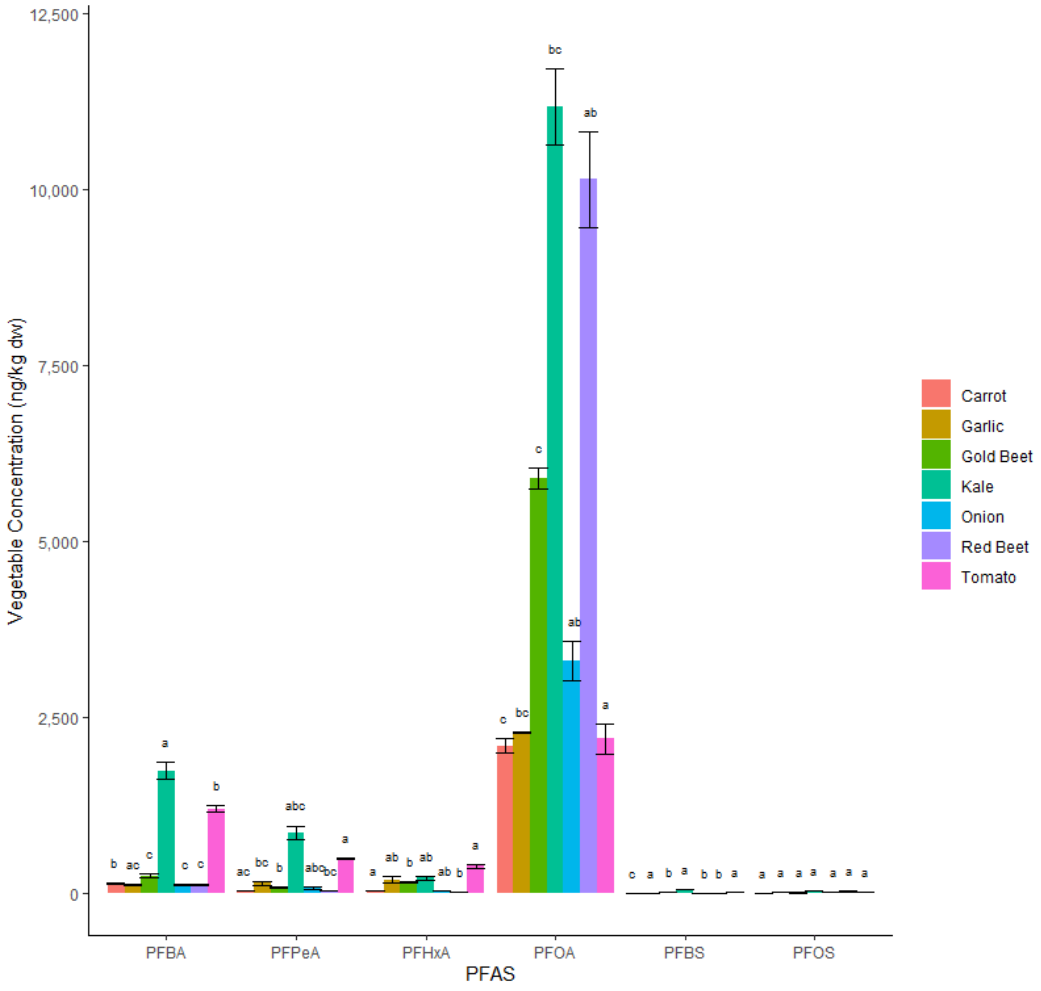


*Figure 22: PFAS concentrations, on a dry weight basis, for vegetables from Garden 3. Letters are the groups from a Tukey's HSD test ($p<0.05$) within a compound blocked by date and vegetable.*

## 4.12 Toxicity measurement of PFAS in vegetables

The USEPA PFOA toxicology limit of 0.313 ng PFOA/kg wet weight vegetables consumed per day was found by multiplying the Rfd ($1.5 \times 10^{-9}$ mg/kg$_{bw}$/day), average human body weight (70 kg), and the average daily consumption rate of vegetables (336 wet weight g/d) (USEPA 2022). Every sample had PFOA above the 0.313 ng/kg body weight limit, except for those samples that failed for quality control issues (Figure 23). The highest concentrations were in lettuce, potatoes, kale, and beets. Even when

adjusting for the potential that 96% of the weight of plants like lettuce (USDA 2002) is water, the limits would be 7.83 ng/kg and all detected PFOA was beyond that level as well.

Another interesting finding is that even in the background gardens PFOA exceeds the toxicity limit (Figure 23), which are unconnected to the reclaimed water. PFOS would have a detection limit of 1.65 ng/kg, which would indicate that every detection of PFOS also exceeds that toxicology limit. This was also found in every vegetable sample tested. With PFOA and PFOS at levels above these toxicology limits in both the secondary irrigated Gardens and the Background Gardens, removing PFAS from water system would not be enough to reduce PFAS compounds below the toxicology limit. The subchronic RfDs for PFOA and PFOS are the same as their chronic RfDs due to their effect at the developmental stages in humans. This indicates that all measured vegetable concentrations also exceeded the toxicity limits for these compounds based on the sub chronic RfDs, suggesting that even less than life-time consumption of these vegetables may have adverse health effects to the humans consuming them.

PFBS has a chronic RfD of 300 ng/kg/d and a sub chronic RfD of 3,000 ng/kg/d (USEPA 2022). These RfDs yield chronic and sub chronic toxicology limits for this study of 62,500 ng/kg, and 625,000 ng/kg, respectively. No measured concentrations exceed these limits for PFBS.

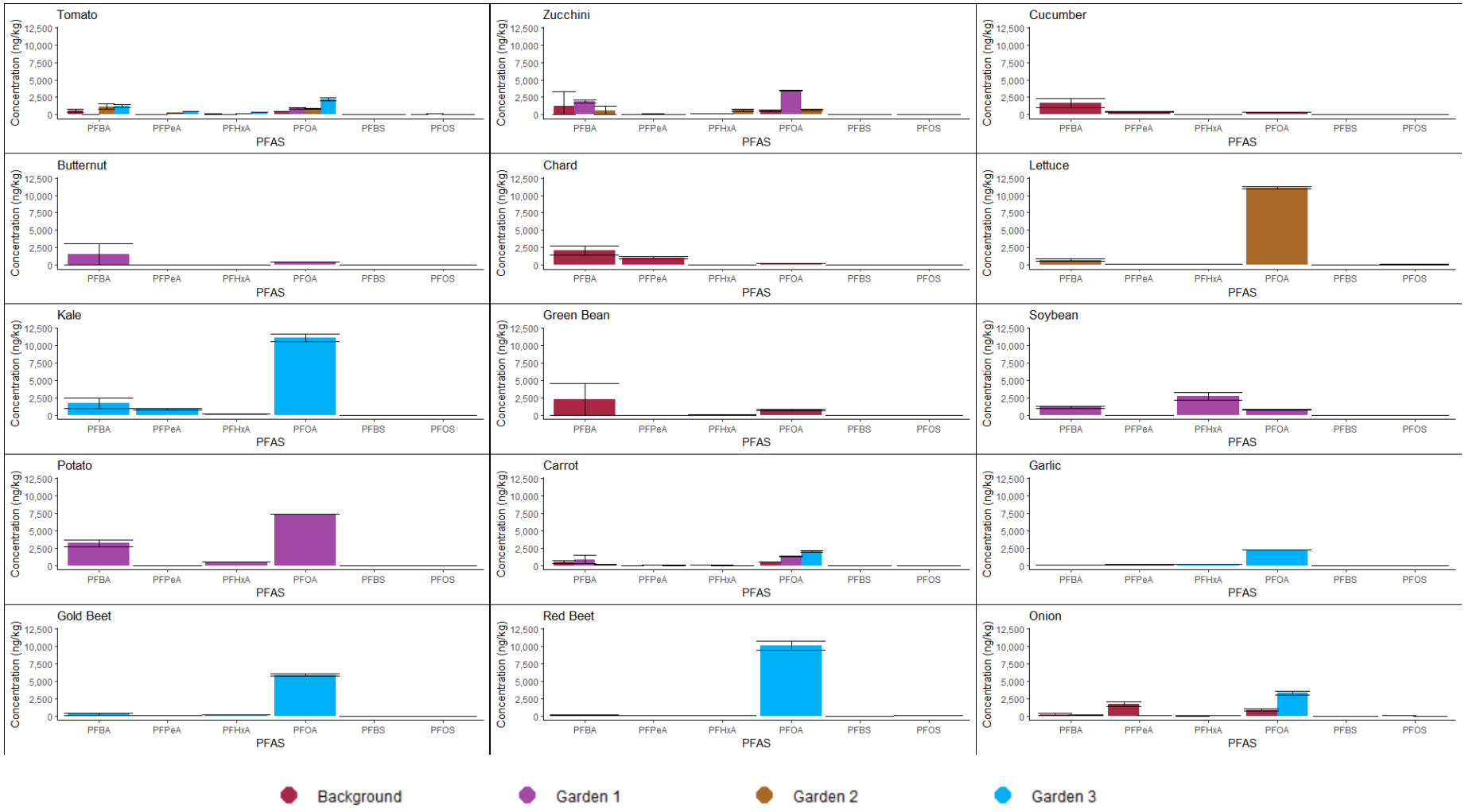


*Figure 23: PFAS Concentrations found in vegetables irrigated with reclaimed wastewater and the control gardens. Sampling was done on the available vegetables and therefore a variety of vegetables were sampled. No data were removed due to being an outlier to produce these graphs. All confidence intervals were made with at least n=3 except Garden 1's Butternut and Potato; as well as Garden 3's Garlic, which had n=2.*

## 4.13 Biosolids PFAS Contribution to PFAS in Forage Grass and Soil

The amended soils, which were amended 6 months (New Field Soil), and 3 years (Old Field Soil) prior to sampling, had significantly greater concentrations relative to the Background Hayfield (Figure 24) for most PFAS compounds analyzed in the study. The general trend of the samples was as expected with the non amaneded soils having the lowest concentration then Old Field Soil having the lowest PFAS concentration of the amended field soils, then the newly amended field soil, with the biosolids themselves having the highest concentration. This trend would imply that the PFAS applied with the biosolids is being either broken down, washed out, or extracted by the grass over time. PFBA did not show a significant difference between the background soil and the newly amended field soils. PFBA in the Old Field Soil was at a statistically lower concentration than the Background Soil based on a Dunnetts test, suggesting other loading routes for PFBA such as rainwater.The Tukey analysis for significant differences between the amended and unamended soils was run without including biosolids as the concentrations of biosolids is much greater than corresponding concentrations in the soils and increases the variance of the test.

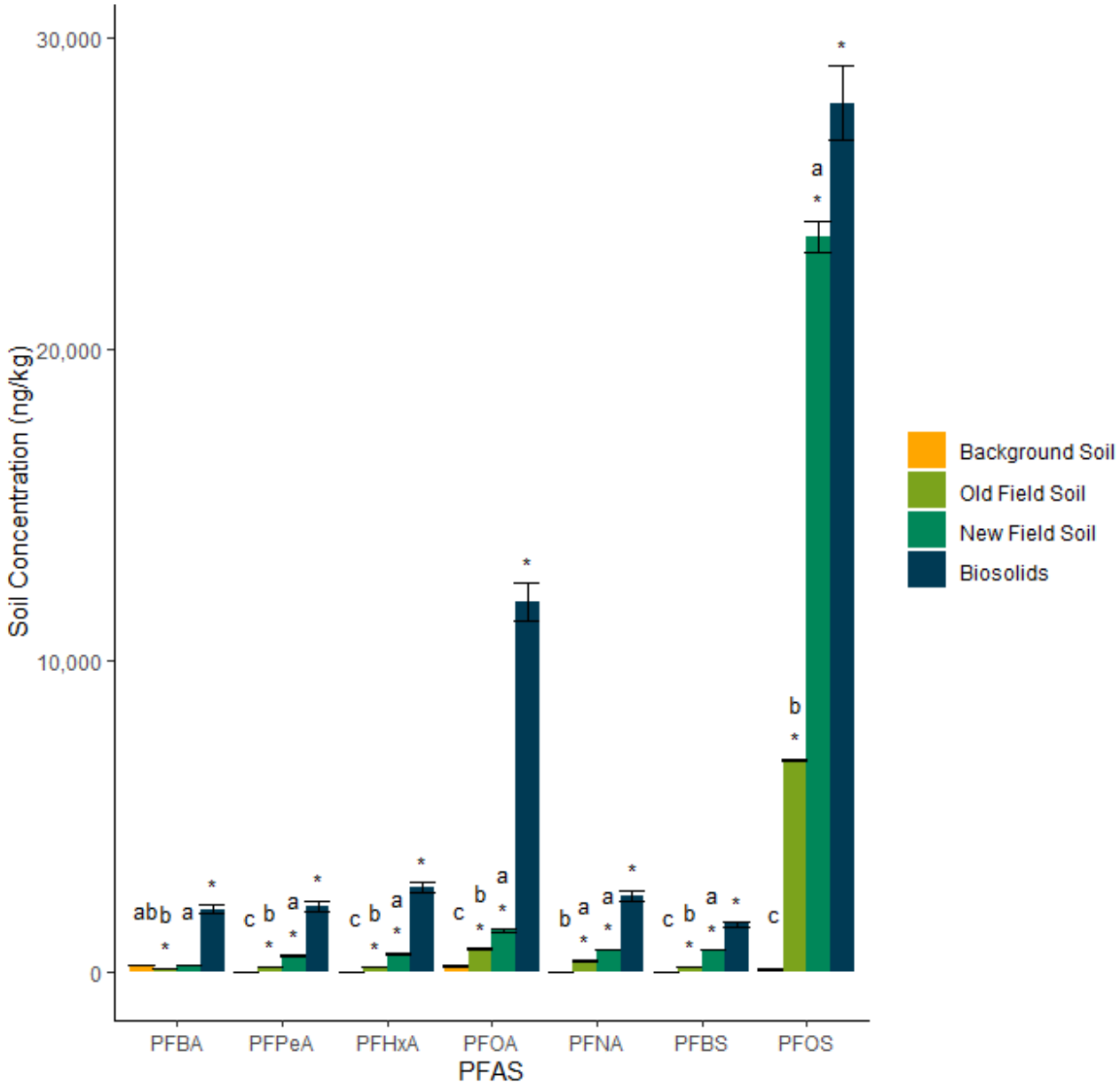


*Figure 24: Concentrations of PFAS compounds found in background hayfield, biosolid amended field soils, and the biosolids. Error bars indicate 95% confidence intervals. * Indicates a significant difference from the mean of the control field Dunnett's test P<0.05. Letters are Tukey HSD groups.*

A similar trend was seen in the grasses that was seen in the soils. The newly amended field had grass concentrations that were highest in PFAS, while the older field had lower PFAS grass

concentrations (Figure 25). Nearly all detectable levels of PFAS in the newly amended soil grasses were found to be significantly higher with only PFNA and PFDA not found to have significantly greater concentrations in the newly amended fields than the Control samples ($p<0.5$). PFBA, PFHxA, PFOA, and PFBS concentrations were found to be statistically greater in the Old Field than the Control suggesting that these compounds persist in soils for at least 3 years.

The forage grass and soil of the Control Field had high concentrations of PFBA and PFOA and is consistent with the high concentrations of these compounds in rainwater, soils and garden vegetables, showing that these compounds are the most likely to contaminate these systems regardless of their introduction through biosolids and reclaimed water. The persistence of these two compounds in the fields may be, in part, due to their presence in rainwater which would provide a continuous source of wet deposition. This wet deposition is still less than the loading through the biosolid application, however, as the control hay fields are still statistically lower than the fields amended with biosolids. The compounds remaining in the biosolid-amended soil after 6 months were mostly recalcitrant compounds that are either long chained or sulfonic acids with high affinities for soils and low uptake rates in the hay.

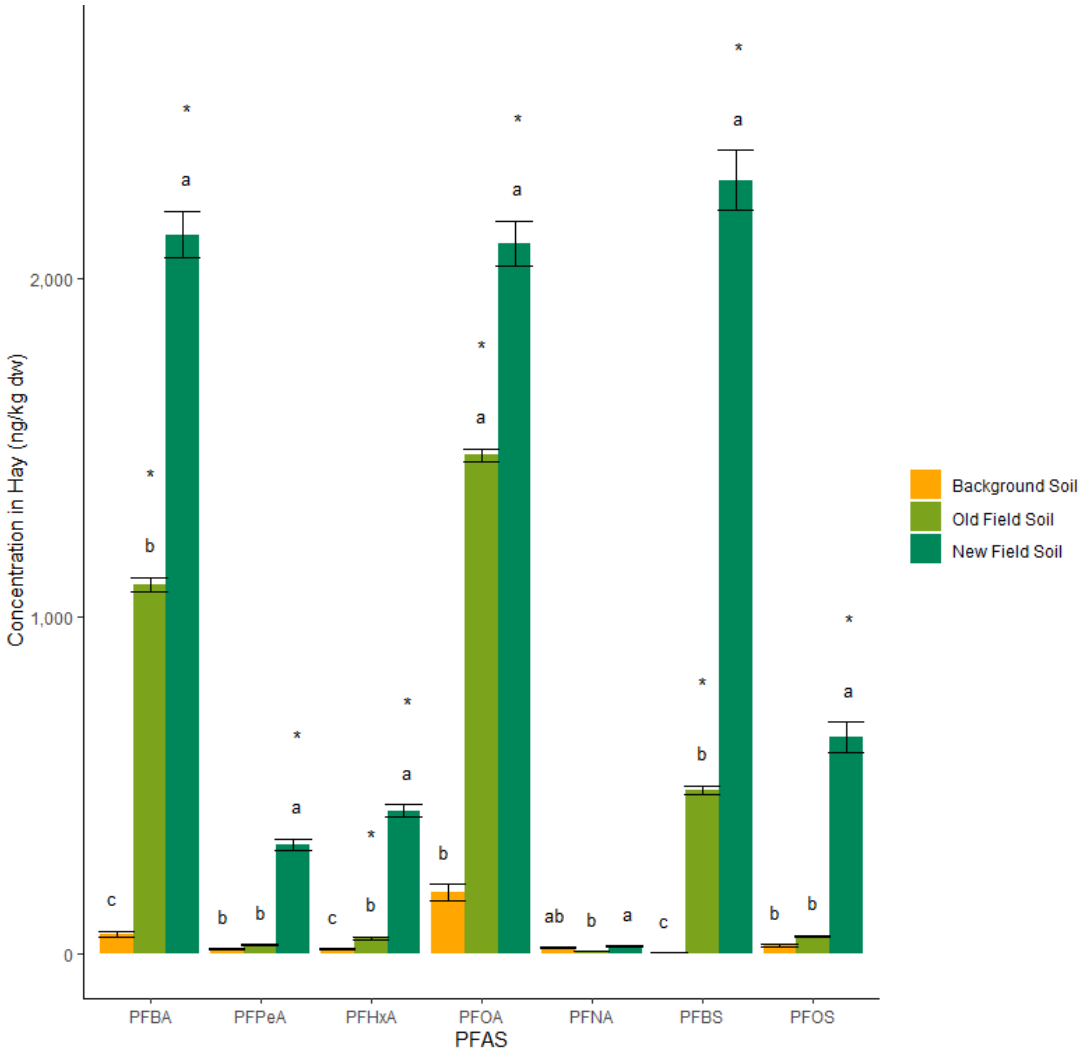


*Figure 25: Concentrations of PFAS compounds found in control grass and biosolid amended field grasses. Error bars indicate 95% confidence intervals. * Indicates a significant difference from the mean of the control grass sample Dunnett's test P<0.05. Letters are Tukey HSD groups.*

## 4.14 Removal of PFAS from Harvesting Grass

The removal of PFAS from the soil through harvesting grass accounts for most of the loss of PFAS observed in the amended soils over time. Figure 26 shows the measured concentrations in the Old Field Soil are all less than the estimated concentration from the mass balance calculations using Equations 1 through 3 that account for compound uptake and removal from the soil through hay harvesting. There is general agreement between the measured and predicted soil concentrations, ranging between 0.29 and 4.81 and averaging 2.46 times the measured values for all compounds except

PFOS. PFOS showed the worst correlation with the measured soil concentration after nine cuttings, this could be because PFOS has the highest affinity for soil and therefore has the least removal through the harvesting of grass. The similarity between the estimated and measured concentrations would indicate that the primary removal of PFAS out of the soil happens with the harvesting of grass. The observed PFAS concentrations are likely less than the estimated concentrations because PFAS can be washed out of the soil during rain and irrigation events and the calculation procedure does not account for this loss pathway. Conversely, the greater actual concentration of PFBA than estimated is likely because this compound is found in most rainwater and would therefore be loaded into rather than washed out of the soil during rain events.

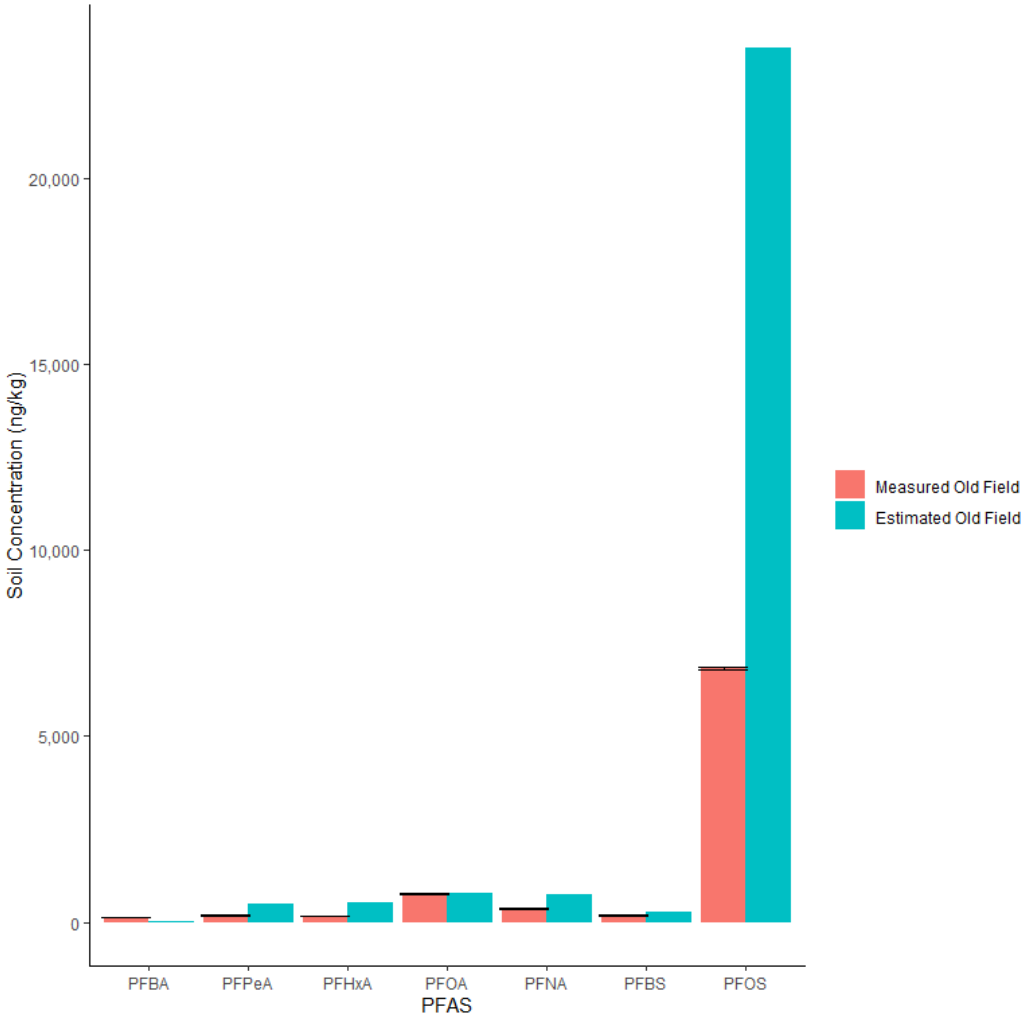


*Figure 26: Measured Old Field and estimated concentration of Soil PFAS Concentrations in biosolid amended field soils. Estimations were generated using Equation 3. Error bars are 95% confidence intervals.*

Equation 3 can be used to calculate how many years until the concentrations in the amended soils are less than or equal to than the background soil. These results can be found in Table 15. Most compounds will take over two decades to return to the background concentrations, even with active removal through the harvesting of plants. PFBA, PFOA, and PFHpA are the exceptions because they

already exist in high background concentrations and are removed in large quantities during each harvest.

*Table 15: Calculated years until newly amended soils will reach background PFAS Concentrations exclusively through removal by plants.*

| | *PFBA* | *PFPeA* | *PFHxA* | *PFOA* | *PFNA* | *PFBS* | *PFOS* |
|---|---|---|---|---|---|---|---|
| *Years until amended soil reaches background levels* | 0.67 | 134 | 87.3 | 0.33 | 28.6 | 24.3 | 1,310 |

## 4.15 Bioaccumulation Factor

Bioaccumulation Factor (BAF) is the ratio of the PFAS concentration in the edible portion of the plants and the soils or the ratio between the edible portion of the plant and the irrigation water. Figure 27 shows the average ratio of vegetables to their soil. There are not many patterns that arise with these BAFs. Most of the BAFs, 47 of 70, are greater than 1 which would imply that on a dry weight basis plants are accumulating PFAS at greater concentrations than in the soils. All the BAFs for PFOS are below one and seven of the ten PFBS BAFs are also below 1`. This would indicate that sulfonic acids have a greater affinity for soil than for being taken up into the edible portions of plants. Xu et al. (2022) found BAFs that generally followed this trend of sulfonic acids having lower BAFs than carboxylic acids. They also found that the greatest BAFs were in stems, leaves, and roots which is similar to the findings in this study with higher BAFs being found in leafy greens, and modified stem and root vegetables.

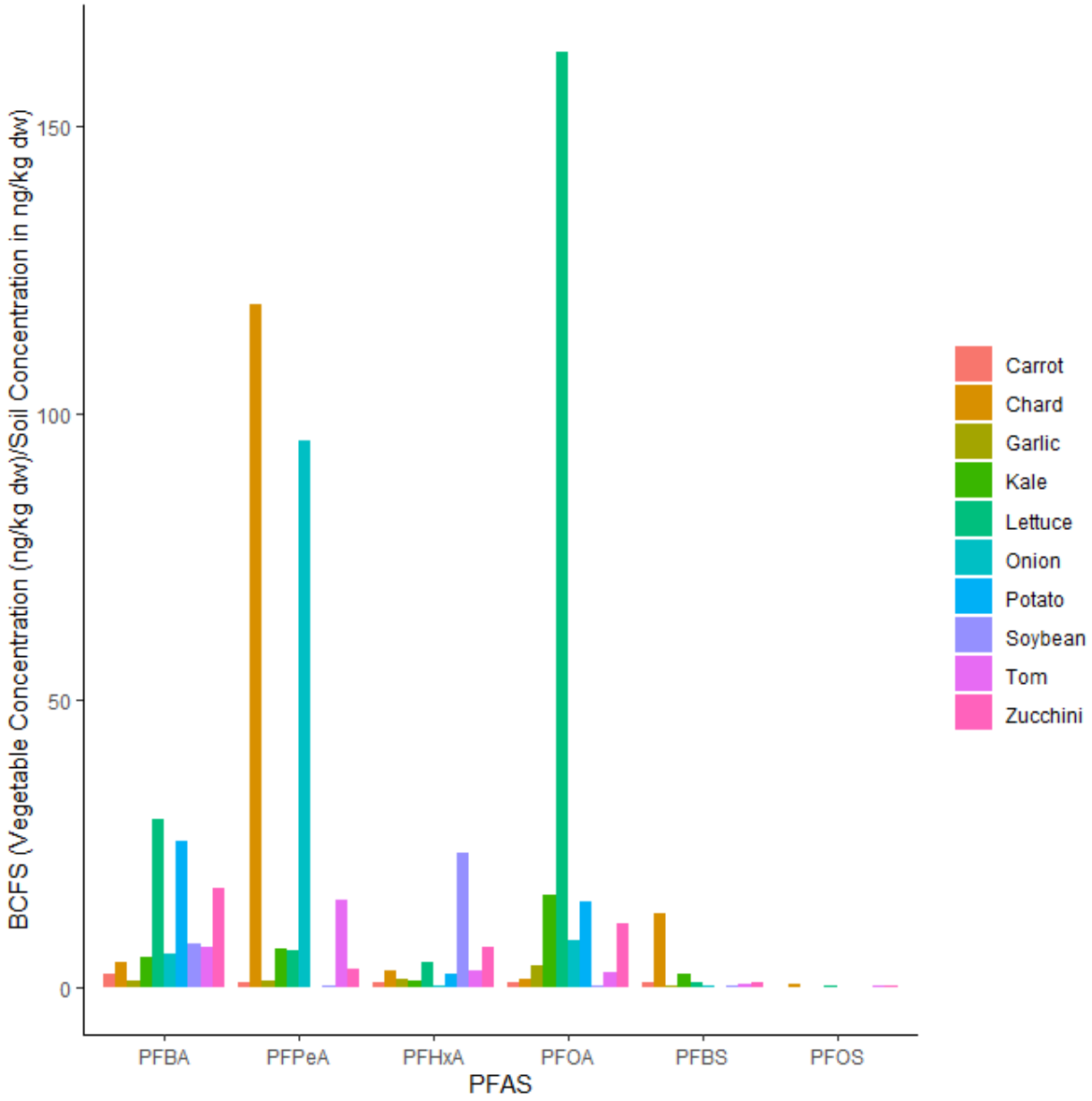


*Figure 27: BAFs for every vegetable with associated soil. If multiple gardens had the same vegetable that BAF was averaged.*

## 4.16 Correlation to Physical Properties

The physical properties of each PFAS compound from Table 1 cannot be directly compared to the accumulation of PFAS in soils and vegetation without complication from the additional variables of

garden, associated vegetable, irrigation concentration, biosolid, and sampling date. A GLMM can give an estimation of the effects that a variable has on the dependent variable, which in this case is the accumulated concentration of PFAS. It also provides the probability that the independent variable, which in this case is physical properties, influences the dependent variable by calculating the standard error for the dependent variables slope and creating a probability (p-value) that the slope is zero. The coefficients in the linear model, beta values, and the p-values for the significance of those models are summarized in Table 17 for each of the independent variables tested. The tests were run on the vegetable concentration, soil concentration, hay concentration, and the biosolids amended soil concentration.

The beta values are scaled so they are directly comparable to each other, i.e., a greater beta value would indicate a greater change associated with that property. The greatest beta values in the vegetables and soils were from $pK_a$ experimental and $pK_a$ Calculated, indicating that the smallest change in $pK_a$ results in the largest change in concentrations of PFAS in vegetables and irrigated soil. $pK_a$ is related to the bonding strength of the weak acid group, which is where much of the PFAS to matrix binding takes place. For soil amended with biosolids the great significant beta value is from $K_{oc}$ which is a property often associated with the partition coefficient in soil. The beta coefficients and p-values vary for the rest of properties. These conflating variables are not considered by the GLMM model. The GLMM assumes compounds have the same prevalence in all sources of PFAS. It also assumes that the PFAS comes solely from irrigation water.

*Table 16: The beta and p-values for the GLMM models generated for the concentrations of PFAS with the physical properties of those PFAS in compounds.*

| Physical Property | N | Beta | p-value | Physical Property | N | Beta | p-value |
|---|---|---|---|---|---|---|---|
| ***Vegetable Concentration*** | | | | **Hay Concentration** | | | |
| *Carbon Chain Length* | 540 | 377 | 0.093 | *Carbon Chain Length* | 63 | -56 | 0.8 |
| *Molecular Weight* | 540 | 145 | 0.5 | *Molecular Weight* | 63 | -98 | 0.6 |
| $K_{oc}$ | 540 | 33 | 0.9 | $K_{oc}$ | 63 | 15 | >0.9 |
| $pK_a$ *experimental* | 270 | 986 | <0.001 | $pK_a$ *experimental* | 36 | 333 | 0.3 |
| $pK_a$ *Calculated* | 472 | -610 | 0.019 | $pK_a$ *Calculated* | 54 | -108 | 0.6 |
| *Henry's Law Constant* | 270 | -846 | 0.002 | *Henry's Law Constant* | 36 | -299 | 0.3 |
| *Solubility* | 540 | -193 | 0.4 | *Solubility* | 63 | 90 | 0.6 |
| *Log* $K_{aw}$ | 540 | 137 | 0.5 | *Log* $K_{aw}$ | 63 | -96 | 0.6 |
| *Log* $K_{ow}$ | 540 | 129 | 0.6 | *Log* $K_{ow}$ | 63 | -102 | 0.6 |
| ***Soil Concentration*** | | | | **Biosolid Amended Soil Concentration** | | | |
| *Carbon Chain Length* | 543 | 106 | 0.033 | *Carbon Chain Length* | 150 | 2,005 | 0.1 |
| *Molecular Weight* | 543 | 66 | 0.2 | *Molecular Weight* | 150 | 2,336 | 0.022 |
| $K_{oc}$ | 543 | 24 | 0.7 | $K_{oc}$ | 150 | 2,764 | <0.001 |
| $pK_a$ *experimental* | 313 | 385 | <0.001 | $pK_a$ *experimental* | 75 | 238 | <0.001 |
| $pK_a$ *Calculated* | 463 | -141 | 0.028 | $pK_a$ *Calculated* | 135 | 1,529 | 0.4 |
| *Henry's Law Constant* | 391 | -355 | <0.001 | *Henry's Law Constant* | 75 | -208 | 0.006 |
| *Solubility* | 543 | -44 | 0.14 | *Solubility* | 150 | 2,324 | 0.025 |
| *Log* $K_{aw}$ | 543 | 64 | 0.2 | *Log* $K_{aw}$ | 150 | 2,366 | 0.019 |
| *Log* $K_{ow}$ | 543 | 64 | 0.2 | *Log* $K_{ow}$ | 150 | 2,334 | 0.023 |

# 5 Conclusion

The removal of PFAS through the WWTP reflected the patterns observed in previous literature. The majority of PFAS compounds were not removed through the wastewater treatment process and some increased in concentration in the effluent. A difference from the literature seen in this study is the effective removal of PFPeA, which occurred in all three sampling events. To explain this increase in PFAS from influent to effluent other studies have proposed the transformation of PFAS, however Houtz and Sedlak (2012) when performing their TOP assay only observed the creation of carboxylic acids. Although there are PFAS compounds that can form sulfonic acids when oxidized, the commonly identified precursors like those used in Houtz and Sedlak result in carboxylic acids. Little work has been done applying the TOP assay or other methods of PFAS oxidation to WWTP influent which would create a more direct link between the rise in the carboxylic and sulfonic acids found in WWTP effluent. Houtz and Sedlak (2012) found a distribution of smaller chain compounds were formed from the oxidation of precursors and these distributions are what allowed for back calculation of what pre-cursors may have been in the original mixture. The rise in PFHxA and PFBA, with a decrease in PFPeA observed in this study does not relate to the pattern seen by the Houtz and Sedlak (2012) TOP analysis. The wastewater treatment system, however, is possibly more complicated than a simple oxidation process, including preferential removal of compounds like PFPeA, and methodological issues in quantifying PFAS in influent. The methodological issues may arise from the use of WAX or Divinylbenzene cartridges to clean and concentrate influent wastewater as is used in USEPA Methods 533 and 537 that did not quantify the PFAS associated with the suspended solid portion of the sample

effectively, leading to lower measured values for the influent. A methodological issue like this could also affect the mass balance using influent, effluent, and biosolids since the suspended solids in the influent are under measured while the solid concentrations in the biosolids are correctly measured.

With the persistence of PFAS compounds throughout the wastewater treatment process, PFAS was released into the secondary irrigation line. As distance from the WWTP plant increased, the PFAS concentrations decreased as it mixed with PFAS free surface water. However, the garden spigots which were sampled more often than the mainline spigots varied in concentration between sampling times. This could be due to fluctuating rates of usage of households through the lines during the summer. This pattern was contradicted by extremely high concentrations of PFOA at Spigot 5 in five of six samples taken from it. This dramatic increase does not have a clear cause because the water lines to the house were the same as everywhere else in the secondary distribution system. However, tracking the path of PFOA would require a large amount of sampling through the secondary water system which was beyond the scope of this study.

The PFAS concentrations in soil samples did not follow the pattern assumed by hypothesis 1. The background soil sample PFAS concentrations were greater than or equal to the secondary water irrigated soils, depending on the compound measured. The compounds that were indistinguishable or significantly higher in the background soil than irrigation water likely have other sources that deposit PFAS into the soils. Some compounds did have lower background concentrations than the irrigated soils indicating irrigation is the primary source of deposition of these compounds.

The PFAS deposited on soils can be incorporated in the vegetables grown in those contaminated gardens. The concentrations of PFAS in the soil or in the water did not match the expected concentrations of PFAS in the vegetables. This could be because some compounds are preferentially sorbed to soil while others are taken up in vegetables and neither property is connected to which

compounds are most likely to appear in wastewater. Accumulation of compounds in soils from below detection limit values in reclaimed water could also explain the lack of correlation between water, soil, and plant concentrations. PFAS uptake by vegetables was statistically greater for compounds with shorter carbon chain length and the carboxylic acid PFAS compounds than the longer carbon chain or sulfonic acid PFAS compounds.

Only some compounds had greater concentrations in vegetables grown in reclaimed water irrigated gardens than observed in background samples. However, all the vegetables that passed QC contained PFOA and PFOS concentrations greater than the RfD set by the USEPA. The data were inconclusive regarding a correlation between the PFAS concentrations in the garden spigots and in the vegetables. Regardless of the source of PFAS applied to the gardens, all vegetables showed high levels of PFAS indicating that vegetables are a vector of PFAS ingestion.

The use of biosolids as soil amendment is another potential vector of PFAS exposure. The soils amended with biosolids had greater PFAS concentrations than the secondary water irrigated soils. PFAS was also present in forage grasses grown in biosolid amended soils. Calculations for the time for PFAS to be reduced to background levels in biosolid-treated soils indicates that concentrations will continue to increase within the soil as more biosolids are reapplied to the fields for all compounds except PFBA, PFHpA, and PFOA. This indicates that using phytoremediation is not a promising method to remove PFAS from contaminated soils.

In summary, PFAS entering a WWTP are not readily removed. PFAS entering the plant result in PFAS in both the liquid effluent and the biosolids. If that effluent is reused as a source of irrigation it may result in an increase in PFAS in garden vegetables, however the base level of PFAS in garden vegetables is already above the Rfd for PFOA and is therefore dangerous for ingestion. This base level may be due to the deposition of PFAS from the atmosphere as PFAS was found in detectable concentrations in

rainwater. Meanwhile the use of biosolids as a soil amendment has increased the PFAS in the soil and that results in an increase in the vegetation grown there, in this study it was pasture hay.

# 6 Engineering Significance

The removal of PFAS from the environment is a difficult task as there are no single point sources for PFAS. In this study, PFAS was detected in rain. With PFAS being detected in rain, the deposition of PFAS can occur anywhere it rains. Some studies have found that their rainwater does not contain detectable levels of PFAS (Pfotenhauer et al. (2022) and Olney et al. (2023) ) so the deposition of PFAS in rain does not always occur, and further study is needed to decipher the patterns of PFAS wet deposition. PFAS existing in rainwater makes completely removing PFAS from the environment infeasible, however, monitoring and eliminating PFAS from areas where it may be a vector for human exposure may reduce the amount of PFAS present in the populace. One of the sources of human exposure could be vegetables that are irrigated with water that contain even “low” levels of PFAS, vegetables that receive contaminated rain, and vegetables that may be grown in biosolids amended soils. The lowest PFAS concentrations in vegetables and wastewater were compounds that already had low concentrations in the wastewater influent or effluent, meaning the reduction of PFAS use in all goods would be the most effective way to reduce PFAS in a populace.

The ability to track and quantify PFAS in all vectors is becoming increasingly important. The prevalence of some PFAS compounds in environmental samples and laboratory equipment reduces the effectiveness of conventional sampling techniques. For compounds that are of most interest it is especially difficult to free all solvents and equipment of these compounds. The use of sample cleaning and sample concentrating techniques such as SPE, which was used in this study, needs to be expanded to handle more difficult matrices such as soil, vegetables, and waste streams. Moreover, the improvement of methods to quantify compounds like the telomer sulfonates and the perfluorooctane amines, both of which were not reported in this study due to instrumentation issues, are needed to expand the range of PFAS compounds that can be quantified.

In conclusion, the use of wastewater as a source of irrigation poses a risk of increasing the ingestion of PFAS by people who eat the vegetables grown with this water source. The use of reclaimed water for irrigation increases the amount of PFAS in vegetables above those resulting from exposure to rainwater. On top of this, the USEPA has PFOA and PFOS sub-chronic doses at the same level as their chronic RfD, meaning the ingestion of any of the vegetables tested in this study over less than a lifetime could result in adverse effects to human health. Ingesting PFAS from vegetables above the pending USEPA chronic RfDs for PFOA, and PFOS is already a possibility in the Cache Valley even without the use of reused water for irrigation as Figure 23 shows. The Background Gardens also had detectable, and sometimes high levels of PFAS, especially PFBA, due to natural exposure to rainwater in the area. These issues are exacerbated by the difficulty of measuring PFAS concentrations in environmental media. The use of biosolids as a soil amendment poses a more indirect problem for humans because humans generally are not directly eating what is being grown in the amended soil. Because of the slow rate at which some of the PFAS are removed from soil (Table 14), however, the repeated application of biosolids could result in ever increasing levels of PFAS in soil and therefore in the hay grown there. The potential risk to humans is from ingestion of animal products generated from those fields and will

increase with increased biosolids applications over time. The use of biosolids for amendment to home gardens is particularly problematic and not recommended as even without exposure to the high PFAS concentrations in the biosolids, PFAS accumulation in the garden vegetables appears to be at undesirable levels.

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# Appendix

## QC

| | |
|---|---|
| Low sensentivity (initial evaluation) | 8:2 FTS |
| | 6:2 FTS |
| | PFOSA |
| | N-MeFOSAA |
| | EtFOSAA |
| Poor chromatography | 4:2 FTS |
| | C11-C14 carboxylic acids |
| Poor matrix recovery | PFHpS |
| | PFDS |
| Lack of detectable concentrations | PFPeS |
| | PFHpA |
| | PFDA |

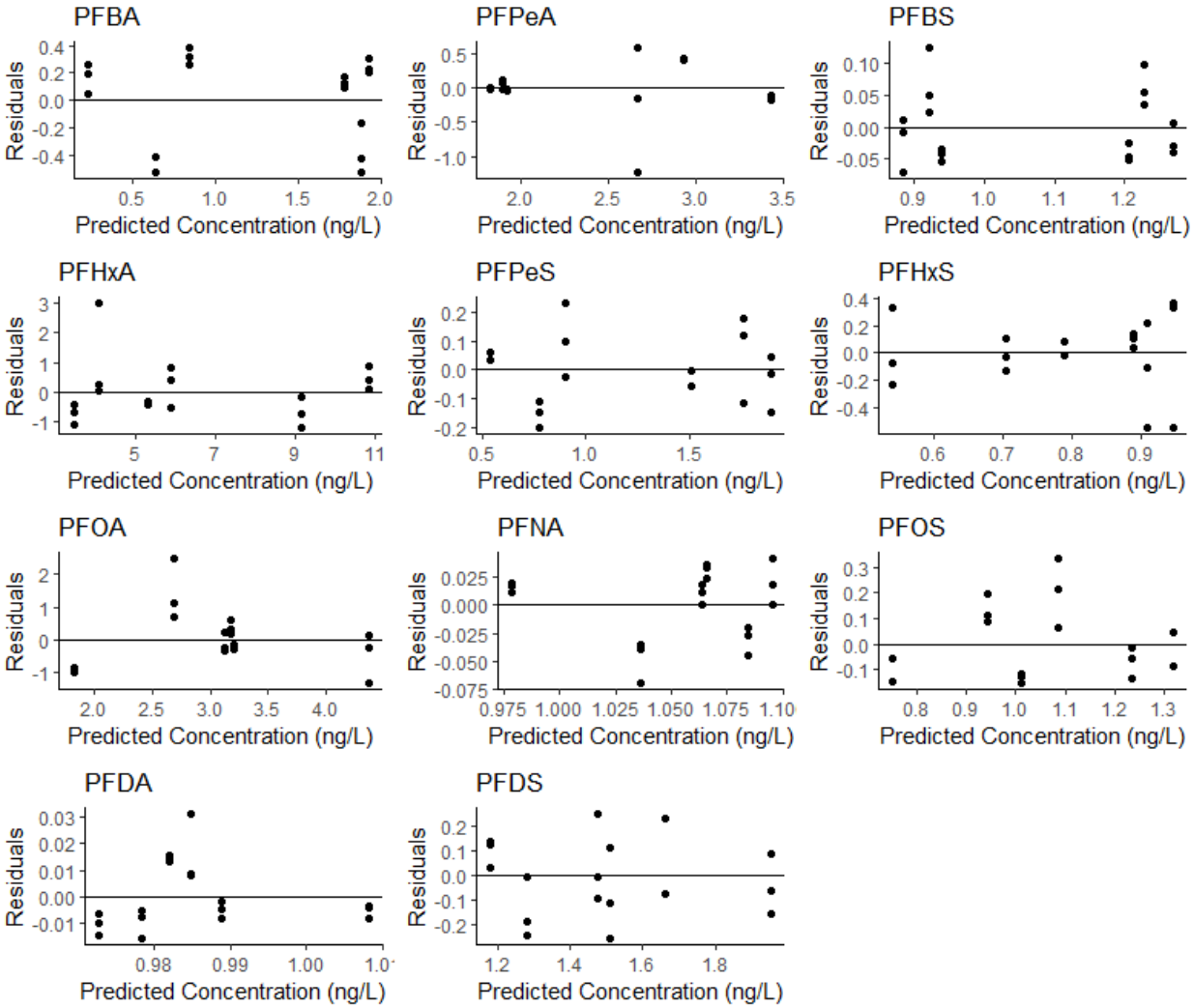


*Figure A1: Residual distributions for wastewater after box-cox transformations.*

*Table A1: box-cox lambdas for each compound for wastewater*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.46 |
| PFPeA | 0.22 |
| PFBS | 0.14 |
| PFHxA | 0.78 |
| PFPeS | -0.26 |
| PFHxS | 0.34 |
| PFOA | 0.02 |
| PFNA | -0.06 |
| PFOS | 0.26 |
| PFDA | 0.02 |
| PFDS | -0.18 |

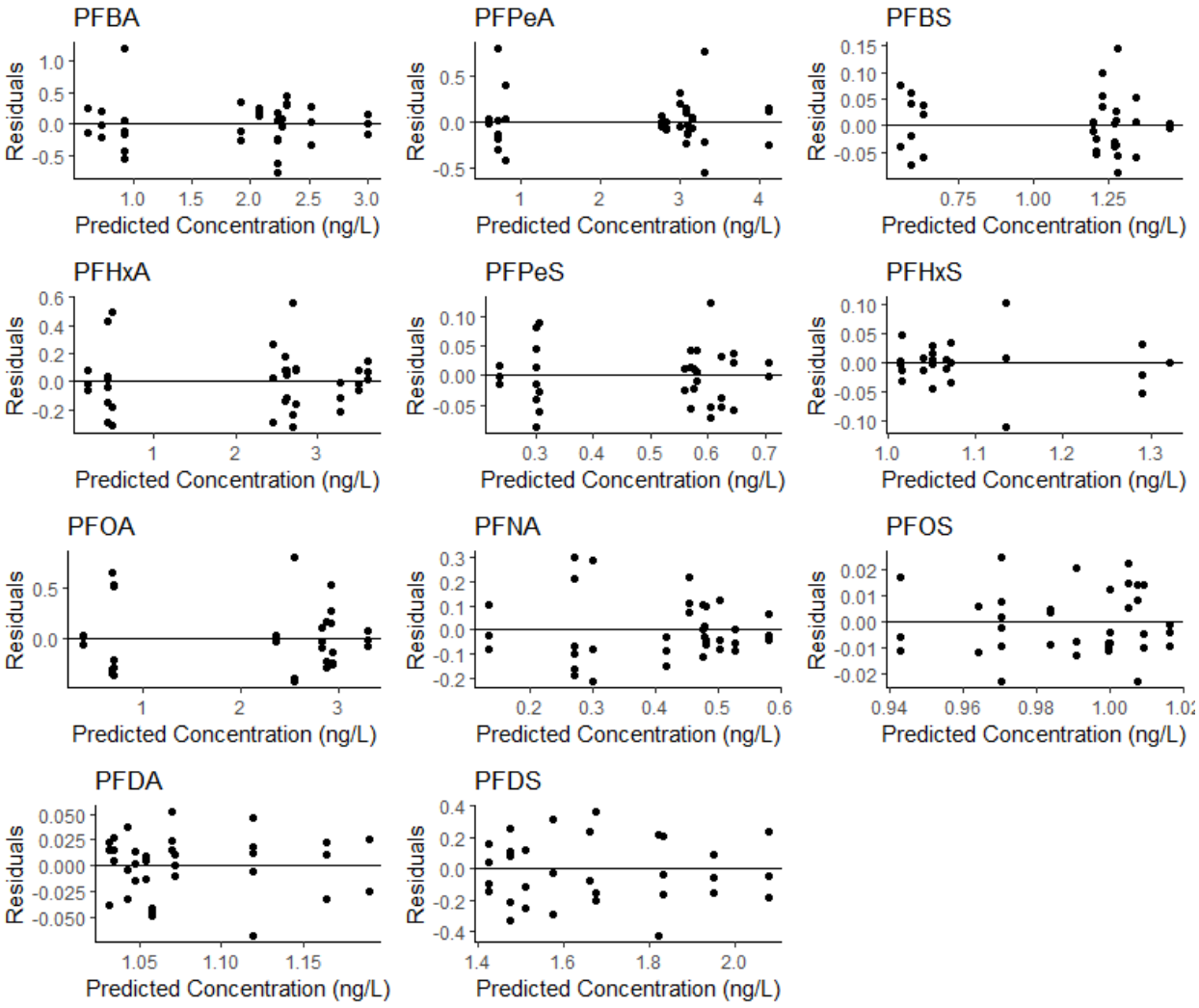


*Figure A2: Residual distributions for mainline spigots after box-cox transformations.*

*Table A2: box-cox lambdas for each compound for mainline spigots,*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.58 |
| PFPeA | 0.38 |
| PFBS | 0.14 |
| PFHxA | 0.42 |
| PFPeS | 0.22 |
| PFHxS | -0.06 |
| PFOA | 0.50 |

| | |
|---|---|
| PFNA | 0.62 |
| PFOS | 0.02 |
| PFDA | -0.06 |
| PFDS | -0.18 |

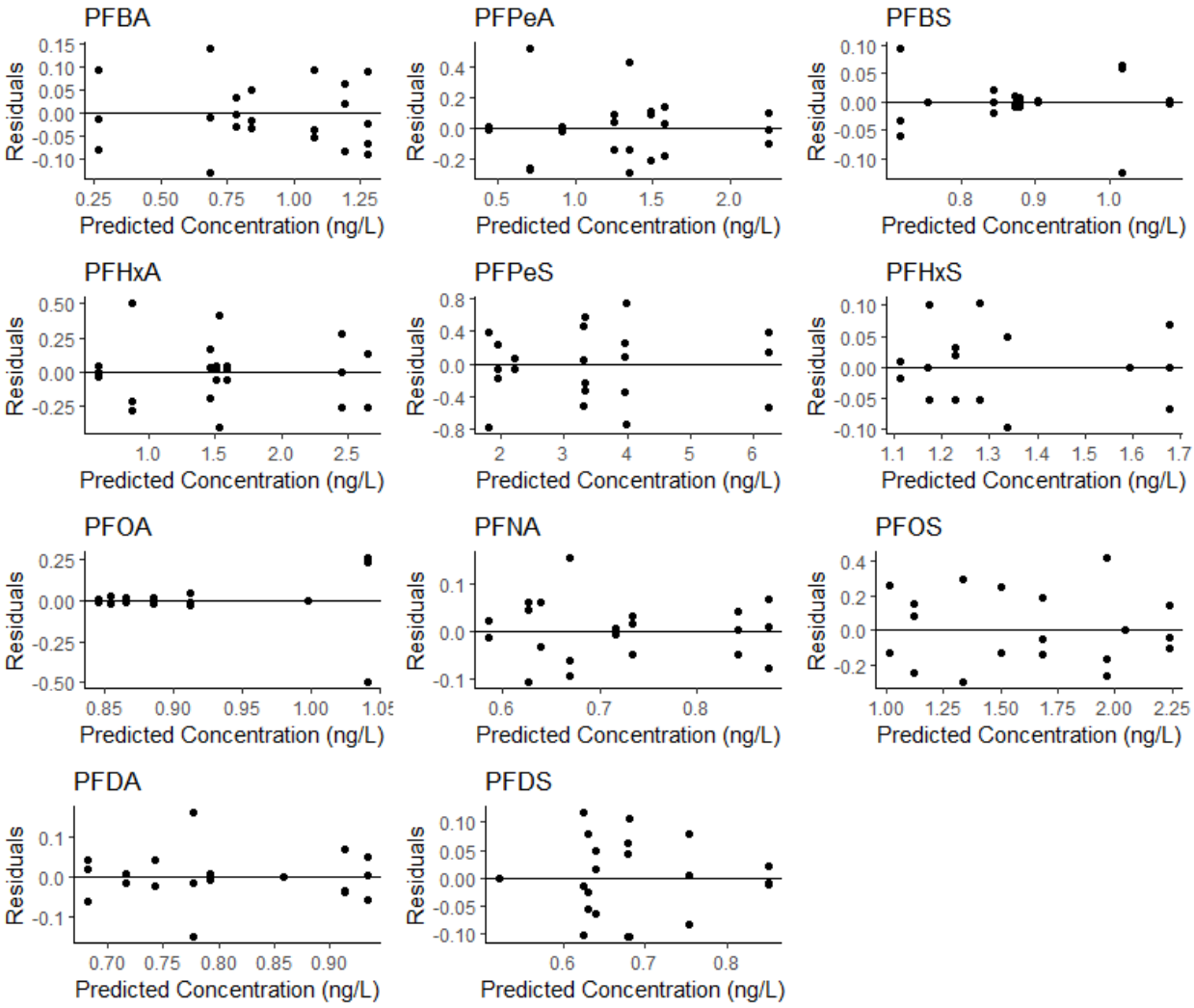


*Figure A3: Residual distributions for irrigation spigots after box-cox transformations.*

*Table A3: box-cox lambdas for each compound for irrigation spigots.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.22 |
| PFPeA | -0.30 |

| | |
|---|---|
| PFBS | 0.06 |
| PFHxA | -0.22 |
| PFPeS | -0.26 |
| PFHxS | -0.10 |
| PFOA | 0.10 |
| PFNA | 0.14 |
| PFOS | -0.22 |
| PFDA | 0.10 |
| PFDS | 0.14 |

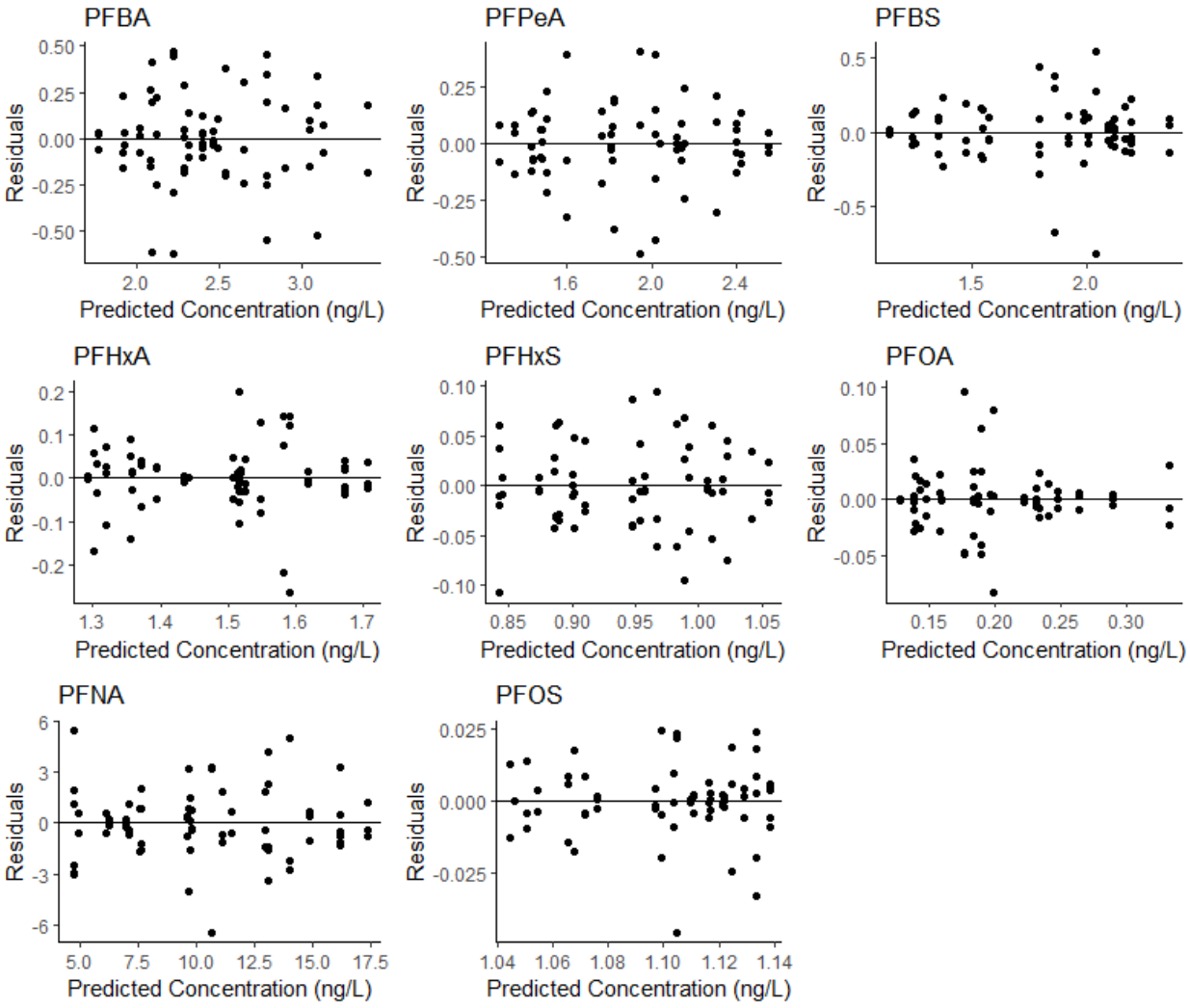


*Figure A4: Residual distributions for garden soils after box-cox transformations.*

*Table A4: box-cox lambdas for each compound for garden soils.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.18 |
| PFPeA | 0.18 |
| PFBS | 0.26 |
| PFHxA | 0.10 |
| PFHxS | -0.06 |
| PFOA | -0.26 |

| | |
|---|---|
| PFNA | 0.58 |
| PFOS | 0.02 |

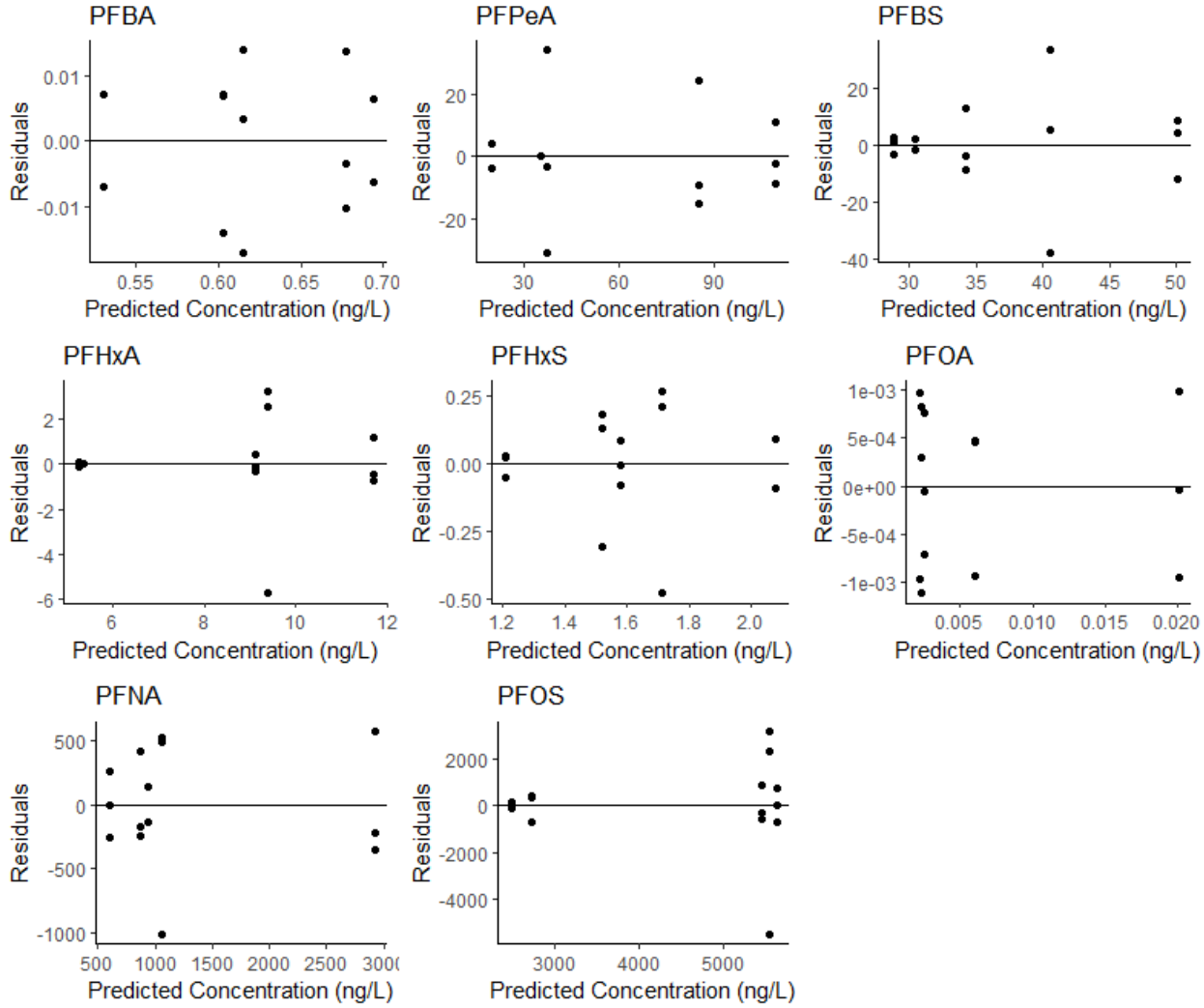


*Figure A5: Residual distributions for garden 1 soils after box-cox transformations.*

*Table A5: box-cox lambdas for each compound for garden 1 soils.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | -0.10 |
| PFPeA | 0.91 |
| PFBS | 1.19 |
| PFHxA | 0.46 |
| PFHxS | 0.26 |

| | |
|---|---|
| PFOA | -0.82 |
| PFNA | 1.64 |
| PFOS | 1.52 |

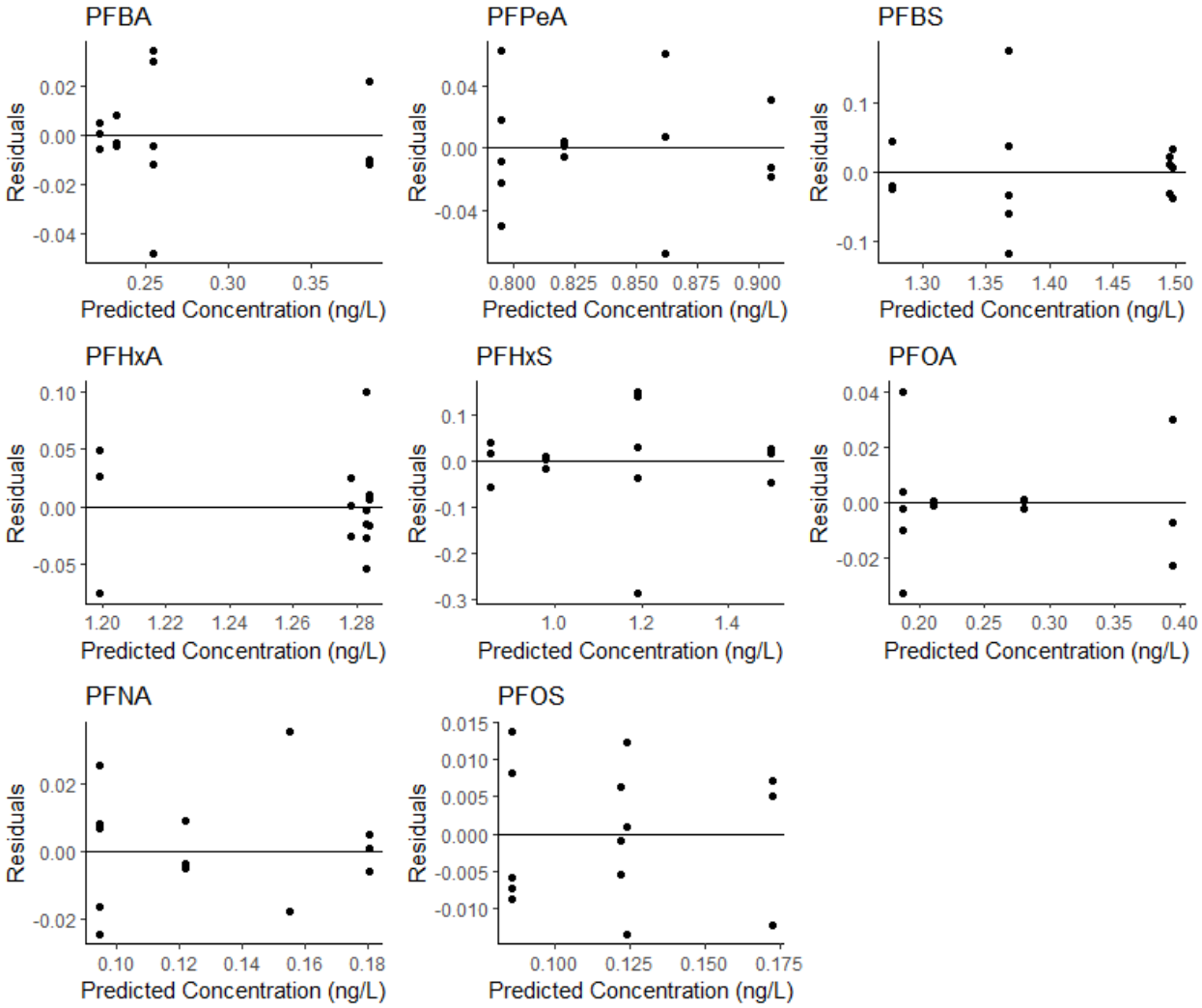


*Figure A6: Residual distributions for garden 2 soils after box-cox transformations.*

*Table A6: box-cox lambdas for each compound for garden 2 soils.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | -0.30 |
| PFPeA | -0.06 |
| PFBS | 0.14 |
| PFHxA | 0.06 |

| | |
|---|---|
| PFHxS | 0.18 |
| PFOA | -0.22 |
| PFNA | -0.54 |
| PFOS | -0.38 |

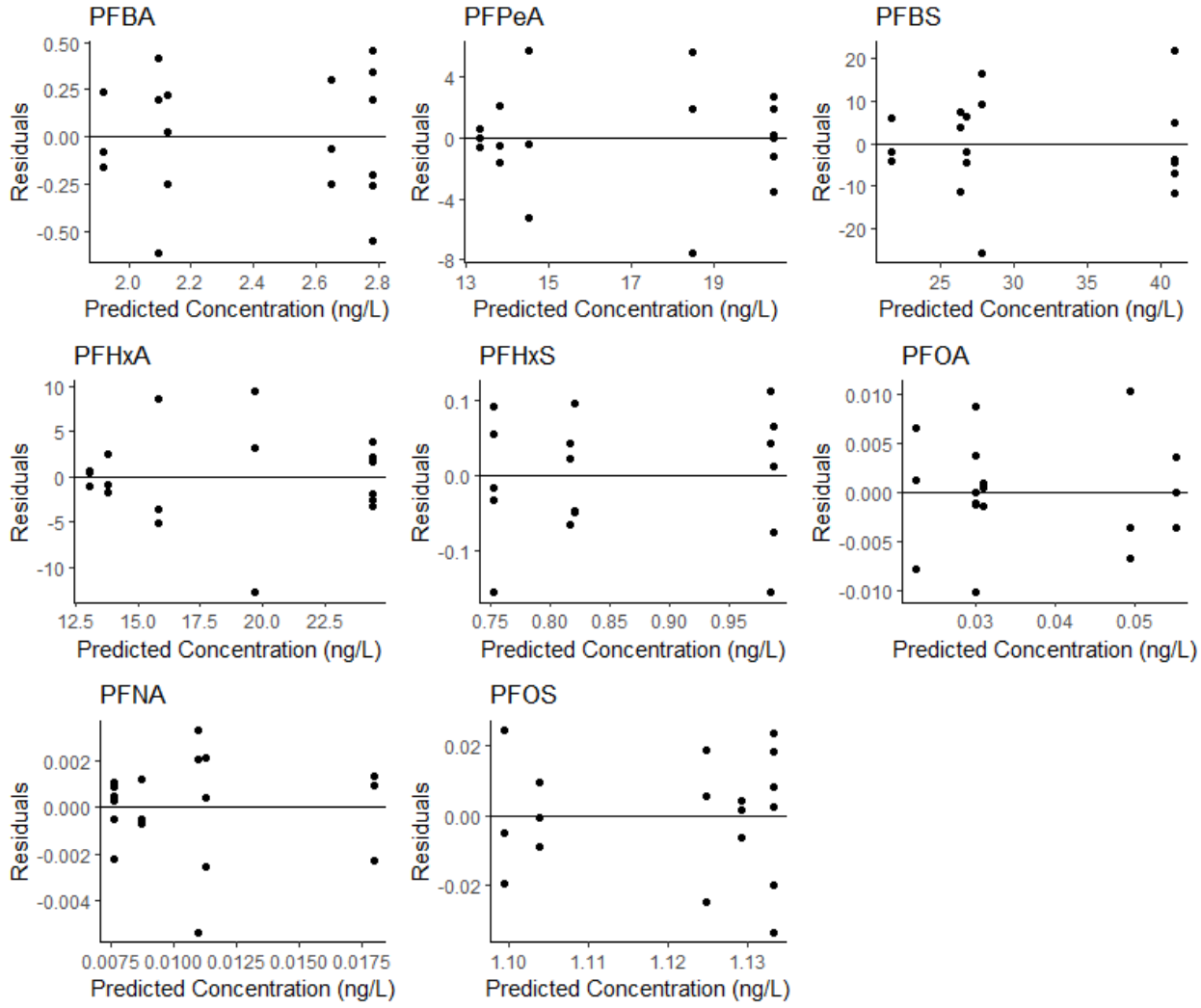


*Figure A7: Residual distributions for garden 3 soils after box-cox transformations.*

*Table A7: box-cox lambdas for each compound for garden 3 soils.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.18 |
| PFPeA | 0.62 |
| PFBS | 1.23 |

| | |
|---|---|
| PFHxA | 0.62 |
| PFHxS | -0.10 |
| PFOA | -0.54 |
| PFNA | -1.03 |
| PFOS | 0.02 |

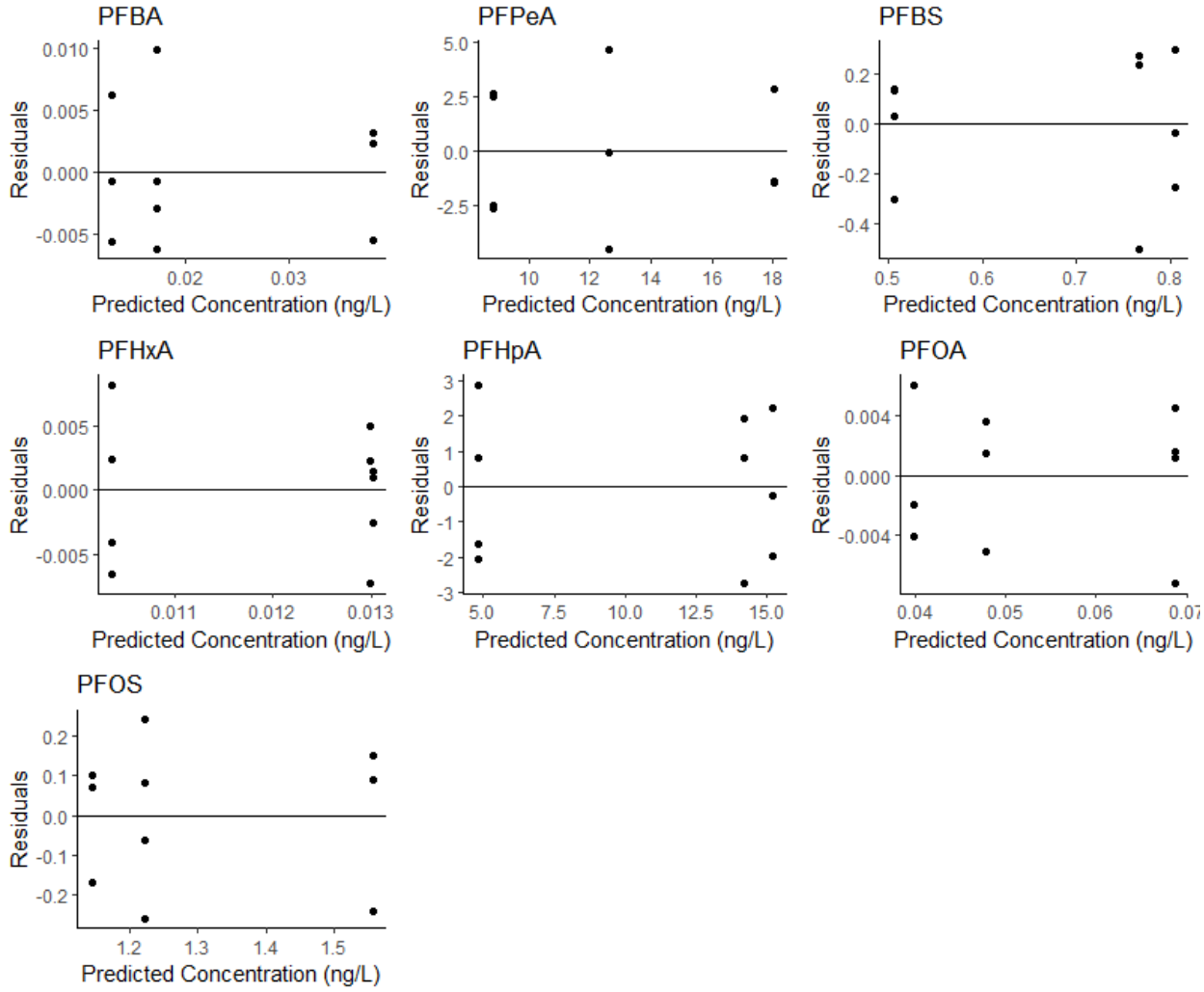


*Figure A8: Residual distributions for carrots after box-cox transformations.*

*Table A8: box-cox lambdas for each compound for carrots.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | -0.66 |
| PFPeA | 0.74 |
| PFBS | -0.66 |

| | |
|---|---|
| PFHxA | -1.27 |
| PFHpA | 0.91 |
| PFOA | 0.42 |
| PFOS | 0.18 |

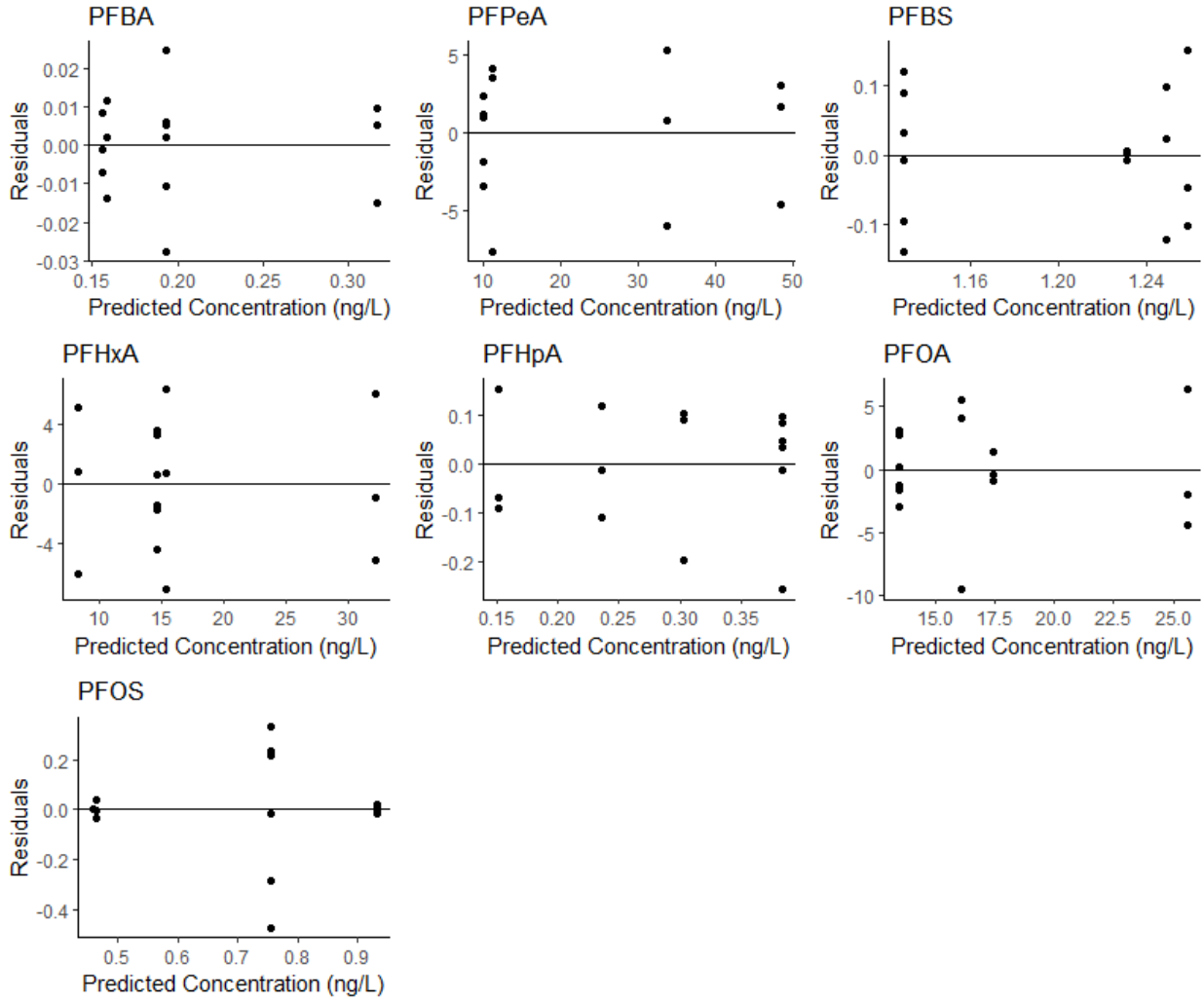


*Figure A9: Residual distributions for tomatoes after box-cox transformations.*

*Table A9: box-cox lambdas for each compound for tomatoes.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | -0.26 |
| PFPeA | 0.63 |
| PFBS | 0.10 |
| PFHxA | 0.58 |

| | |
|---|---|
| PFHpA | -0.74 |
| PFOA | 0.42 |
| PFOS | -0.30 |

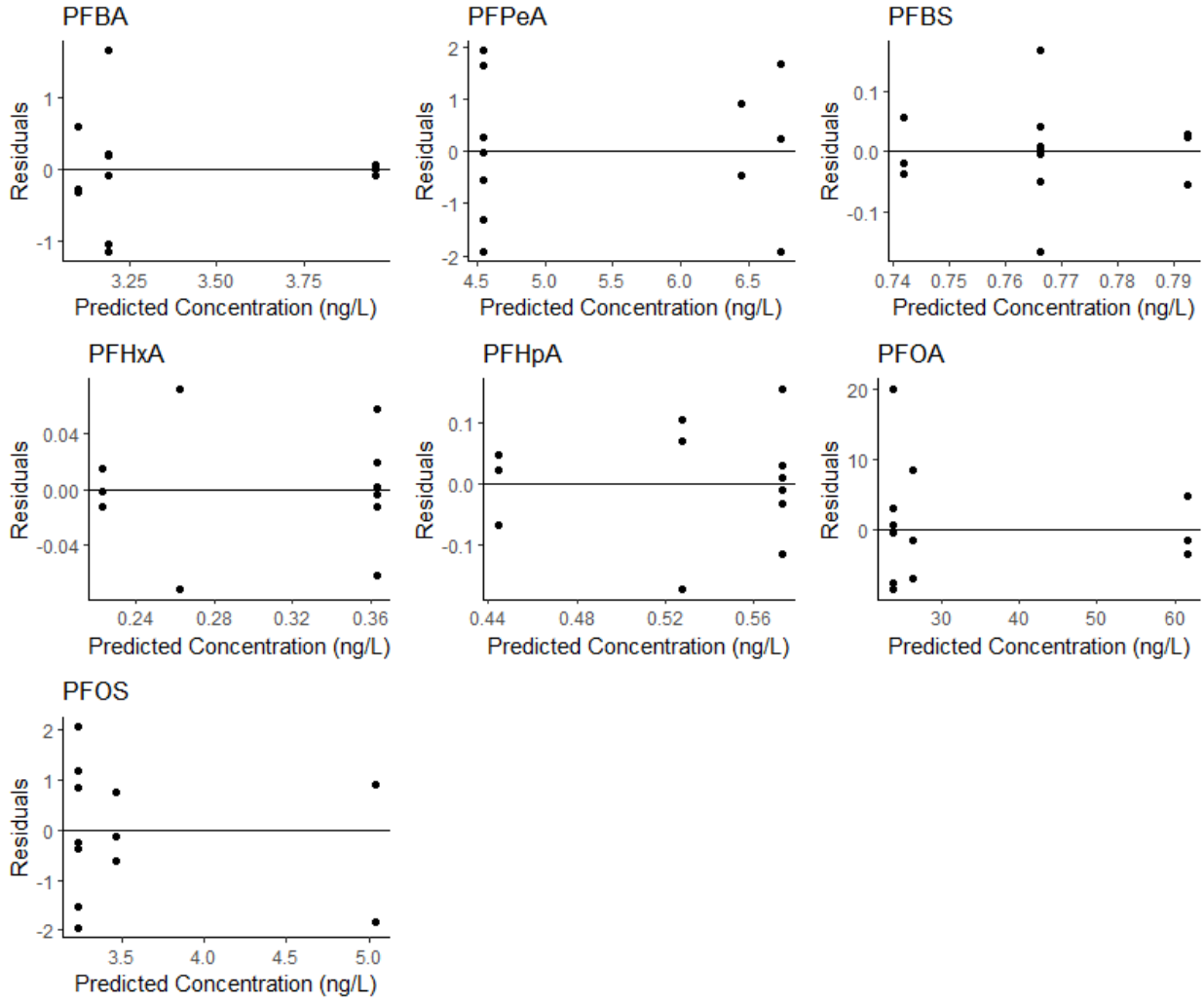


*Figure A10: Residual distributions for zucchinis after box-cox transformations.*

*Table A10: box-cox lambdas for each compound for zucchinis.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.18 |
| PFPeA | 0.42 |
| PFBS | -0.14 |
| PFHxA | -0.30 |
| PFHpA | -0.26 |

| | |
|---|---|
| PFOA | 0.51 |
| PFOS | 0.42 |

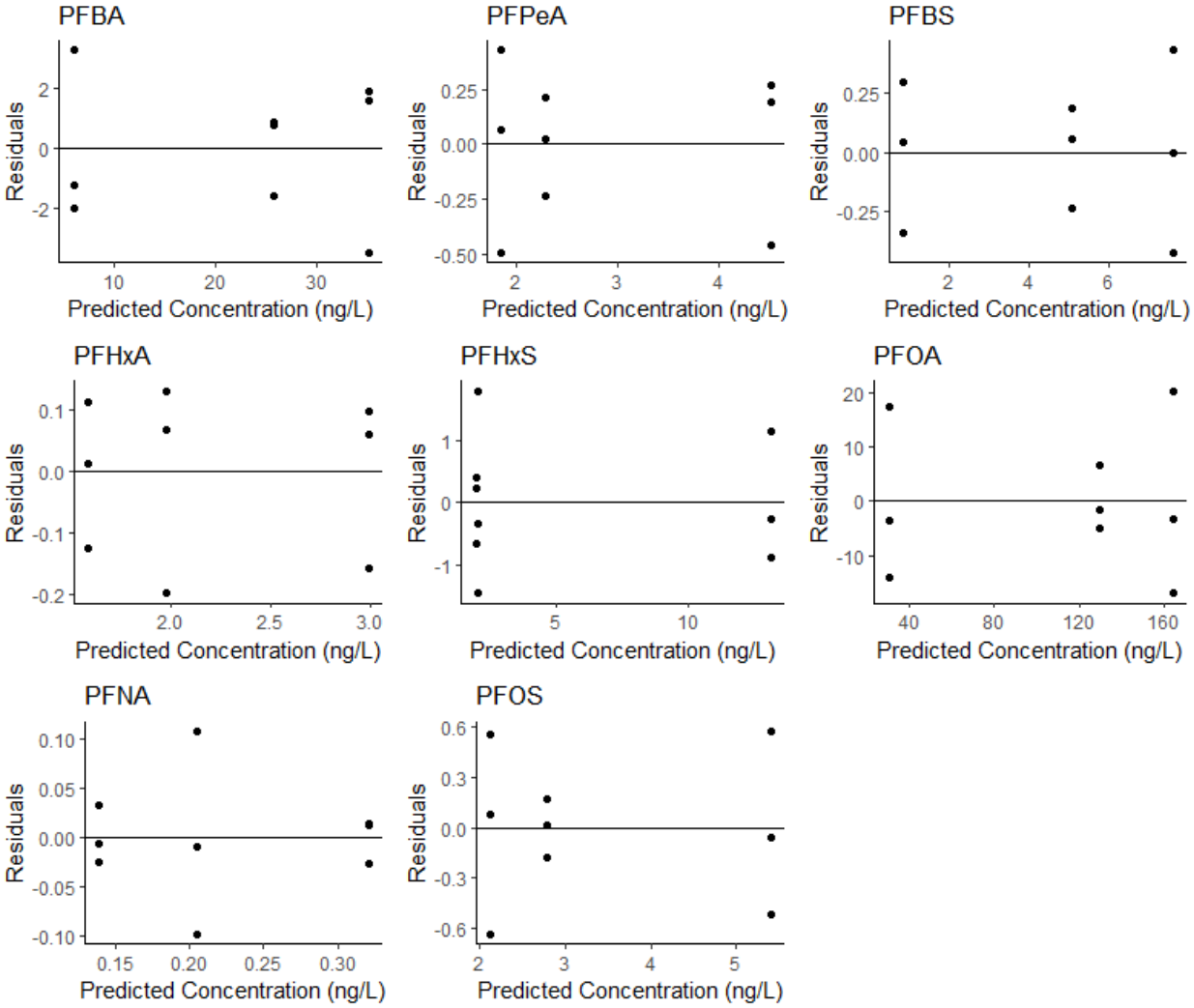


*Figure A11: Residual distributions for hay after box-cox transformations.*

*Table A11: box-cox lambdas for each compound for hay.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | 0.46 |
| PFPeA | 0.26 |
| PFBS | 0.26 |
| PFHxA | 0.18 |
| PFHxS | 0.46 |

| | |
|---|---|
| PFOA | 0.67 |
| PFNA | -0.67 |
| PFOS | 0.26 |
| PFDA | 0.42 |

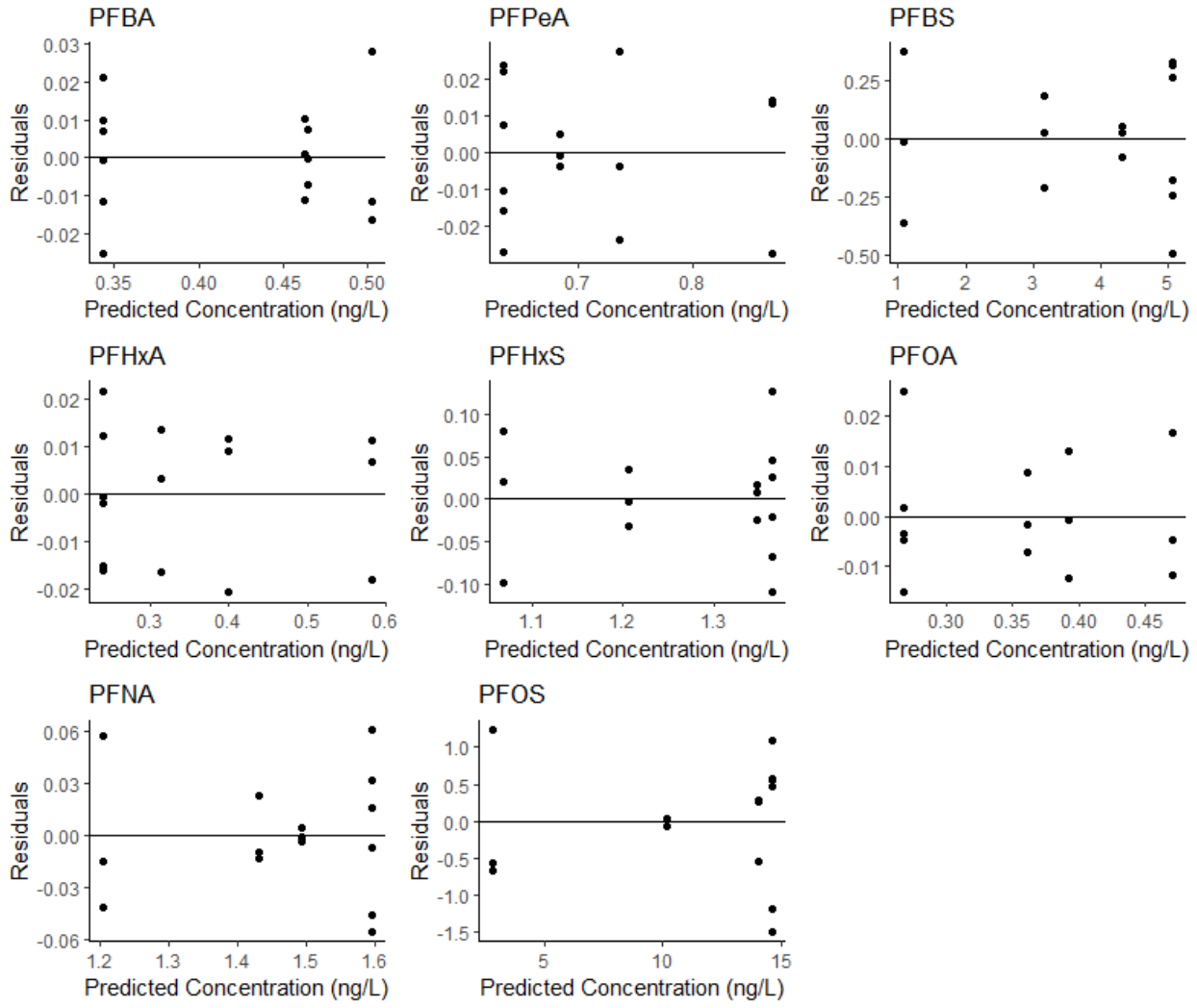


*Figure A12: Residual distributions for biosolids amended soils after box-cox transformations.*

*Table A12: box-cox lambdas for each compound for biosolids amended soils.*

| Compound | BoxCox Lambda |
|---|---|
| PFBA | -0.14 |
| PFPeA | -0.06 |

| | |
|---|---|
| PFBS | 0.22 |
| PFHxA | -0.18 |
| PFOA | -0.14 |
| PFNA | 0.26 |
| PFOS | 0.42 |
| PFDA | 0.10 |
| PFDS | -0.10 |

*Table A13: QQQ parameters used for MS analysis.*

| Compound Name | Precursos Ion | Product Ion | Fragmentor (V) | CE (V) | Cell Acc (V) | Ret Time (min) | Ret Window | Polarity |
|---|---|---|---|---|---|---|---|---|
| *M3PFBS* | 302 | 80 | 380 | 42 | 7 | 7.3 | 2 | Negative |
| *M3PFHxS* | 402 | 80 | 380 | 62 | 7 | 10.2 | 2 | Negative |
| *M4PFBA* | 217 | 172 | 380 | 2 | 5 | 3.5 | 2 | Negative |
| *M4PFHpA* | 367 | 322 | 380 | 4 | 7 | 10.1 | 2 | Negative |
| *M5PFHxA* | 318 | 273 | 380 | 4 | 7 | 8.7 | 2 | Negative |
| *M5PFPeA* | 268 | 223 | 380 | 4 | 7 | 6.74 | 2 | Negative |
| *M6PFDA* | 519 | 474 | 380 | 8 | 5 | 12.86 | 2 | Negative |
| *M8PFOA* | 421 | 376 | 380 | 4 | 7 | 11.1 | 2 | Negative |
| *M8PFOS* | 507 | 80 | 380 | 58 | 7 | 12.1 | 2 | Negative |
| *M9PFNA* | 472 | 427 | 380 | 8 | 3 | 12.1 | 2 | Negative |
| *PFBA* | 213 | 169 | 380 | 2 | 5 | 3.7 | 2 | Negative |
| *PFBS* | 299 | 99 | 380 | 34 | 7 | 7.3 | 2 | Negative |
| *PFBS* | 299 | 80 | 380 | 42 | 7 | 7.3 | 2 | Negative |
| *PFDA* | 513 | 469 | 380 | 8 | 5 | 12.86 | 2 | Negative |
| *PFDA* | 513 | 219 | 380 | 18 | 3 | 12.86 | 2 | Negative |
| *PFDS* | 599 | 99 | 380 | 66 | 7 | 13.5 | 2 | Negative |
| *PFDS* | 599 | 80 | 380 | 70 | 7 | 13.5 | 2 | Negative |
| *PFHpA* | 363 | 319 | 380 | 4 | 7 | 10.1 | 2 | Negative |
| *PFHpA* | 363 | 169 | 380 | 4 | 7 | 10.1 | 2 | Negative |
| *PFHpS* | 449 | 99 | 380 | 74 | 3 | 11.2 | 2 | Negative |
| *PFHpS* | 449 | 80 | 380 | 60 | 3 | 11.2 | 2 | Negative |
| *PFHxA* | 313 | 269 | 380 | 4 | 7 | 8.7 | 2 | Negative |
| *PFHxA* | 313 | 119 | 380 | 22 | 7 | 8.7 | 2 | Negative |
| *PFHxS* | 399 | 99 | 380 | 34 | 7 | 10.1 | 2 | Negative |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| *PFHxS* | 399 | 99 | 380 | 74 | 7 | 10.1 | 2 | Negative |
| *PFHxS* | 399 | 80 | 380 | 62 | 7 | 10.1 | 2 | Negative |
| *PFNA* | 463 | 419 | 380 | 8 | 3 | 12.1 | 2 | Negative |
| *PFNA* | 463 | 219 | 380 | 22 | 3 | 12.1 | 2 | Negative |
| *PFNS* | 549 | 99 | 380 | 54 | 5 | 12.86 | 2 | Negative |
| *PFNS* | 549 | 80 | 380 | 74 | 5 | 12.86 | 2 | Negative |
| *PFOA* | 413 | 369 | 380 | 4 | 7 | 11.1 | 2 | Negative |
| *PFOA* | 413 | 169 | 380 | 14 | 7 | 11.1 | 2 | Negative |
| *PFOS* | 499 | 80 | 380 | 58 | 7 | 12 | 2 | Negative |
| *PFPeA* | 263 | 219 | 380 | 4 | 7 | 6.74 | 2 | Negative |
| *PFPeA* | 263 | 169 | 380 | 14 | 7 | 6.74 | 2 | Negative |
| *PFPeS* | 349 | 99 | 380 | 30 | 7 | 8.95 | 2 | Negative |
| *PFPeS* | 349 | 80 | 380 | 34 | 7 | 8.95 | 2 | Negative |

*Table A14: QQQ parameters used for MS analysis.*

| ***Parameter*** | ***Value (+)*** | ***Value (-)*** |
|---|---|---|
| *Gas Temp (°C)* | 200 | 200 |
| *Gas Flow (l/min)* | 14 | 14 |
| *Nebulizer (psi)* | 20 | 20 |
| *SheathGasHeater* | 350 | 350 |
| *SheathGasFlow* | 7 | 7 |
| *Capillary (V)* | 3800 | 2400 |
| *VCharging* | 500 | 500 |

*Table A15 QQQ parameters used for MS analysis.*

*Ion Funnel Parameter*

| | |
|---|---|
| ***Neg High Pressure RF*** | 125 |
| ***Neg Low Pressure RF*** | 75 |

*Table A16: Relative percent differences. The highlighted red cells are over 50%. Empty cells did not have readable values due to matrix interferences.*

| PFBA | PFPeA | PFBS | PFHxA | PFPeS | PFHpA | PFOA | PFHpS | PFNA | PFOS | PFDA | PFNS | PFDS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 14% | NA | 18% | 10% | 27% | 3% | 5% | 0% | 4% | 10% | 15% | 7% | 0% |
| 17% | 4% | 14% | 10% | 6% | 24% | 19% | 11% | 24% | 3% | 16% | 71% | 98% |
| 3% | 0% | 2% | 10% | 10% | 4% | 2% | 29% | 6% | 34% | 10% | 32% | 66% |
| 2% | 53% | 50% | 30% | 35% | 31% | 19% | 12% | 25% | 13% | 41% | 45% | 41% |
| 26% | 30% | 20% | 11% | 10% | 1% | 19% | 30% | 7% | 17% | 16% | 36% | 1% |

| | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4% | NA | 5% | 1% | 29% | 1% | 4% | 6% | 11% | 13% | 32% | 12% | 73% |
| 0% | 14% | 21% | 12% | 4% | 9% | 1% | 36% | 11% | 44% | 18% | 38% | 23% |
| 6% | 9% | 10% | 9% | 20% | 4% | 1% | 7% | 17% | 3% | 12% | 9% | 123% |
| 15% | 0% | 6% | 5% | 18% | 3% | 6% | 7% | 5% | 12% | 16% | 23% | 150% |
| 2% | 5% | 11% | 9% | 1% | 6% | 4% | 3% | 5% | 1% | 15% | 9% | 17% |
| 4% | 28% | 8% | 9% | 7% | 6% | 10% | 3% | 2% | 8% | 2% | 1% | 6% |
| 3% | 22% | 7% | 3% | 1% | 2% | 3% | 7% | 19% | 22% | 0% | 4% | 9% |
| 0% | 2% | 4% | 2% | 4% | 1% | 1% | 2% | 1% | 21% | 6% | 4% | 6% |
| 8% | 15% | 6% | 13% | 6% | 5% | 2% | 19% | 7% | 11% | 10% | 4% | 21% |
| 1% | 6% | 4% | 5% | 18% | 4% | 0% | 1% | 3% | 22% | 0% | 6% | 14% |
| 9% | 9% | 5% | 5% | 9% | 1% | 24% | 2% | 20% | 2% | 9% | 5% | 31% |
| 4% | 8% | 8% | 1% | 21% | 1% | 7% | 9% | 5% | 6% | 5% | 3% | 29% |
| 3% | 25% | 14% | 13% | 5% | 6% | 10% | 24% | 12% | 23% | 12% | 1% | 7% |
| 17% | 19% | 19% | 10% | 6% | 25% | 11% | 22% | 28% | 13% | 3% | 24% | 33% |
| 32% | 20% | 2% | 5% | 8% | 10% | 3% | 8% | 5% | 11% | 15% | 9% | 8% |
| 25% | 24% | 3% | 3% | 17% | 4% | 0% | 14% | 0% | 3% | 10% | 29% | 3% |
| 10% | 16% | 0% | 19% | 11% | 7% | 34% | 3% | 20% | 21% | 26% | 15% | 14% |
| 27% | 1% | 18% | 1% | 13% | 2% | 12% | 26% | 6% | 12% | 3% | 28% | 8% |
| 5% | | 6% | 9% | 57% | 6% | 8% | 55% | 11% | 13% | 56% | 42% | 48% |
| 5% | 8% | 0% | 17% | 1% | 3% | 0% | 5% | 13% | 5% | 3% | 3% | 49% |
| 1% | 8% | 1% | 10% | 7% | 0% | 27% | 2% | 13% | 49% | 38% | 3% | 7% |
| 13% | 36% | 1% | 8% | 1% | 3% | 0% | 4% | 2% | 4% | 4% | 22% | 23% |
| 1% | 10% | 15% | 14% | 6% | 11% | 34% | 6% | 0% | 14% | 14% | 2% | 22% |
| 1% | 33% | 1% | 3% | 1% | 5% | 6% | 13% | 9% | 1% | 1% | 5% | 3% |
| 13% | 23% | 0% | 6% | 1% | 20% | 5% | 8% | 27% | 33% | 27% | 21% | 12% |
| 6% | 48% | 10% | 36% | 16% | 2% | 18% | 0% | 5% | 16% | 16% | 7% | 5% |
| 14% | 28% | 33% | 2% | 11% | 9% | 18% | 11% | 6% | 48% | 9% | 10% | 10% |
| 0% | 10% | 1% | 10% | 23% | 17% | 14% | 2% | 9% | 40% | 4% | 7% | 13% |
| 17% | 16% | 29% | 27% | 18% | 7% | 19% | 23% | 10% | 9% | 17% | 13% | 9% |
| 3% | 2% | 10% | 9% | 9% | 14% | 3% | 12% | 10% | 15% | 6% | 29% | 28% |
| 14% | 2% | 25% | 9% | 8% | 18% | 4% | 37% | 4% | 25% | 15% | 3% | 8% |
| 4% | 5% | 6% | 21% | 11% | 4% | 1% | 4% | 5% | 8% | 5% | 9% | 14% |
| 0% | 10% | 18% | 5% | 1% | 8% | 4% | 9% | 7% | 9% | 4% | 2% | 7% |

*Table A17: Garden soil data. Cells with red text are below detection and imputed. Highlighted cells are outliers.*

| Sample.ID | Run.Numb | Sample.na | Sample.Lo | Sample.Ty | Sample.Da | Veg | PFBA | PFPeA | PFBS | PFHxA | PFHxS | PFOA | PFNA | PFOS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 211908 | 20222 | N tom 1 | N Control | Veg Soil | 92521 | Tom | 883.07 | 45.63 | 1.71 | 32.55 | 3.10 | 224.21 | 2.70 | 22.54 |
| 211909 | 20222 | N tom 2 | N Control | Veg Soil | 92521 | Tom | 683.32 | 46.92 | 3.19 | 31.15 | 5.09 | 289.56 | 4.08 | 9.44 |
| 211910 | 20222 | n tom soil | N Control | Veg Soil | 92521 | Tom | 177.93 | 7.49 | 2.00 | 18.83 | 0.37 | 280.00 | 52.69 | 7.40 |
| 211129 | 82521 | G4 Zucchn | G1 | Veg Soil | 72121 | Zucchini | 38.93 | 109.72 | 18.08 | 36.83 | 7.53 | 118.49 | 62.23 | 299.49 |
| 211130 | 82521 | G4 Zucchn | G1 | Veg Soil | 72121 | Zucchini | 55.24 | 48.93 | 15.04 | 35.85 | 2.10 | 111.95 | 49.38 | 272.11 |
| 211131 | 82521 | G4 Zucchn | G1 | Veg Soil | 72121 | Zucchini | 49.96 | 7.97 | 17.13 | 33.46 | 6.71 | 105.46 | 35.74 | 322.47 |
| 211135 | 82521 | G4 Potato | G1 | Veg Soil | 72121 | Potato | 116.97 | 170.07 | 21.02 | 183.21 | 1.75 | 440.61 | 121.30 | 281.65 |
| 211136 | 82521 | G4 Potato | G1 | Veg Soil | 72121 | Potato | 163.05 | 193.97 | 30.35 | 243.48 | 2.26 | 593.33 | 146.09 | 322.73 |
| 211137 | 82521 | G4 Potato | G1 | Veg Soil | 72121 | Potato | 98.97 | 159.30 | 28.42 | 172.97 | 2.19 | 441.67 | 125.05 | 270.44 |
| 211147 | 82521 | G5 Lettuce | G2 | Veg Soil | 72121 | Lettuce | 19.32 | 10.16 | 5.03 | 28.80 | 0.46 | 85.45 | 38.09 | 308.32 |
| 211148 | 82521 | G5 Lettuce | G2 | Veg Soil | 72121 | Lettuce | 25.36 | 3.82 | 7.15 | 38.50 | 0.53 | 71.37 | 37.94 | 224.36 |
| 211149 | 82521 | G5 Lettuce | G2 | Veg Soil | 72121 | Lettuce | 25.69 | 44.68 | 4.87 | 6.86 | 0.28 | 47.31 | 20.81 | 179.10 |
| 211153 | 20622 | G6 tom so | G3 | Veg Soil | 72121 | Tom | 108.73 | 62.73 | 15.87 | 63.99 | 4.48 | 325.51 | 99.48 | 44.89 |
| 211154 | 20622 | G6 tom so | G3 | Veg Soil | 72121 | Tom | 67.55 | 66.81 | 9.00 | 52.66 | 5.72 | 286.07 | 65.69 | 324.52 |
| 211155 | 20622 | G6 tom so | G3 | Veg Soil | 72121 | Tom | 32.24 | 57.79 | 17.46 | 64.70 | 16.68 | 175.48 | 75.12 | 87.25 |
| 211159 | 83121 | Garlic Soil | G3 | Veg Soil | 72121 | Garlic | 95.88 | 45.52 | 1.91 | 21.67 | 0.40 | 558.63 | 88.09 | 112.80 |
| 211160 | 83121 | Garlic Soil | G3 | Veg Soil | 72121 | Garlic | 8.79 | 123.21 | 21.62 | 148.83 | 0.76 | 572.13 | 105.22 | 774.41 |
| 211161 | 83121 | Garlic Soil | G3 | Veg Soil | 72121 | Garlic | 156.61 | 161.58 | 18.76 | 218.61 | 6.31 | 640.60 | 107.80 | 435.52 |
| 211168 | 20622 | G6 kale so | G3 | Veg Soil | 72121 | Kale | 529.43 | 112.24 | 17.56 | 145.26 | 20.44 | 389.58 | 102.55 | 548.78 |
| 211169 | 20622 | G6 kale so | G3 | Veg Soil | 72121 | Kale | 637.99 | 124.29 | 18.55 | 182.16 | 161.60 | 501.37 | 158.58 | 112.23 |
| 211170 | 20622 | G6 kale so | G3 | Veg Soil | 72121 | Kale | 408.70 | 142.51 | 18.87 | 188.10 | 25.30 | 670.84 | 110.08 | 204.64 |
| 211183 | 82521 | G6 Onion S | G3 | Veg Soil | 72121 | Onion | 68.03 | 121.59 | 13.54 | 60.13 | 0.61 | 181.38 | 152.24 | 443.81 |
| 211184 | 82521 | G6 Onion S | G3 | Veg Soil | 72121 | Onion | 22.41 | 68.32 | 17.17 | 86.20 | 2.51 | 230.67 | 67.72 | 495.05 |
| 211185 | 82521 | G6 Onion S | G3 | Veg Soil | 72121 | Onion | 28.95 | 34.86 | 12.38 | 53.46 | 1.01 | 203.22 | 61.90 | 314.76 |
| 211355 | 83121 | Blue Curl S | G3 | Veg Soil | 72121 | Kale | 185.19 | 125.62 | 22.35 | 136.87 | 8.17 | 622.65 | 122.12 | 1091.71 |
| 211356 | 83121 | Blue Curl S | G3 | Veg Soil | 72121 | Kale | 84.20 | 150.84 | 15.48 | 131.14 | 5.32 | 1330.82 | 99.97 | 709.98 |
| 211357 | 83121 | Blue Curl S | G3 | Veg Soil | 72121 | Kale | 165.23 | 91.32 | 28.69 | 207.56 | 8.19 | 665.07 | 106.97 | 1380.03 |
| 211358 | 83121 | G5 Unassig | G2 | Veg Soil | 72121 | none | 62.71 | 52.50 | 6.67 | 50.13 | 4.81 | 1689.51 | 129.98 | 720.14 |
| 211359 | 83121 | G5 Unassig | G2 | Veg Soil | 72121 | none | 59.92 | 70.03 | 11.09 | 42.68 | 0.57 | 1954.62 | 48.31 | 755.92 |
| 211360 | 83121 | G5 Unassig | G2 | Veg Soil | 72121 | none | 179.25 | 127.22 | 21.25 | 209.31 | 2.18 | 783.69 | 106.12 | 793.71 |
| 211465 | 30722 | 1465 | G1 | Veg Soil | 72121 | Carrot | 466.08 | 369.46 | 36.84 | 233.72 | 12.06 | 1446.54 | 88.90 | 397.40 |
| 211466 | 30722 | 1466 | G1 | Veg Soil | 72121 | Carrot | 607.93 | 392.72 | 24.60 | 205.78 | 13.44 | 2130.90 | 90.11 | 372.33 |
| 211467 | 20222 | g4 car 3 | G1 | Veg Soil | 72121 | Carrot | 1079.51 | 50.41 | 2.16 | 16.47 | 2.23 | 1022.39 | 11.91 | 17.30 |
| 211481 | 30722 | 1481 | G2 | Veg Soil | 72121 | None | 105.78 | 12.72 | 7.66 | 29.99 | 3.01 | 2367.40 | 66.09 | 474.92 |
| 211482 | 30722 | 1482 | G2 | Veg Soil | 72121 | None | 95.51 | 30.44 | 4.84 | 58.61 | 5.02 | 4406.85 | 64.55 | 408.10 |
| 211525 | 30722 | N cucc soil | N Control | Veg Soil | 92521 | Cucumber | 38.11 | 12.65 | 5.64 | 31.19 | 0.49 | 432.02 | 25.63 | 25.71 |
| 211526 | 13122 | N cucc soil | N Control | Veg Soil | 92521 | Cucumber | 108.99 | 34.75 | 3.25 | 3.41 | 33.63 | 129.12 | 2.61 | 25.11 |
| 211527 | 13122 | N cucc soil | N Control | Veg Soil | 92521 | Cucumber | 41.92 | 24.87 | 7.42 | 20.79 | 3.92 | 3551.01 | 20.66 | 45.96 |
| 211539 | 32422 | 1539 | G3 | Veg Soil | 92521 | Carrot | 124.82 | 62.51 | 14.77 | 54.97 | 13.03 | 2332.46 | 56.36 | 203.20 |
| 211540 | 32422 | 1540 | G3 | Veg Soil | 92521 | Carrot | 386.96 | 82.81 | 11.22 | 165.63 | 2.42 | 661.40 | 46.91 | 89.79 |
| 211541 | 32422 | 1541 | G3 | Veg Soil | 92521 | Carrot | 186.90 | 54.57 | 10.21 | 44.22 | 12.63 | 963.57 | 46.09 | 130.04 |
| 211545 | 11522 | G4 soy soil | G1 | Veg Soil | 92521 | Soybean | 189.57 | 175.16 | 25.39 | 128.35 | 5.64 | 1331.01 | 79.58 | 150.87 |
| 211546 | 32422 | 1546 | G1 | Veg Soil | 92521 | Soybean | 134.23 | 107.11 | 17.46 | 108.32 | 4.68 | 3419.85 | 51.03 | 198.42 |
| 211547 | 32422 | 1547 | G1 | Veg Soil | 92521 | Soybean | 133.76 | 116.91 | 15.09 | 112.88 | 6.95 | 1061.69 | 54.88 | 203.09 |
| 211548 | 11522 | G4 tom 1 s | G1 | Veg Soil | 92521 | Tom | 40.45 | 32.66 | 18.56 | 37.01 | 19.18 | 1059.49 | 71.41 | 168.20 |
| 211550 | 11522 | G4 tom 3 s | G1 | Veg Soil | 92521 | Tom | 33.73 | 21.04 | 16.56 | 37.25 | 13.75 | 3359.20 | 59.30 | 179.68 |
| 211990 | 30722 | 1990 | PH Contro | Veg Soil | 92521 | Onion | 13.24 | 13.99 | 3.86 | 26.05 | 2.44 | 1683.71 | 48.17 | 3303.42 |
| 211992 | 30722 | 1992 | PH Contro | Veg Soil | 92521 | Onion | 36.77 | 4.08 | 4.15 | 18.96 | 4.12 | 1385.20 | 38.49 | 3155.87 |
| 211996 | 30722 | 1996 | N Control | Veg Soil | 92521 | Lettuce | 468.06 | 2.69 | 3.45 | 18.25 | 0.88 | 282.99 | 25.92 | 16.82 |
| 211998 | 30722 | 1998 | N Control | Veg Soil | 92521 | Lettuce | 252.82 | 5.42 | 7.51 | 10.81 | 0.30 | 179.85 | 18.78 | 11.60 |
| 212000 | 32422 | 2000 | N Control | Veg Soil | 92521 | Chard | 547.53 | 10.62 | 1.87 | 12.43 | 0.94 | 145.44 | 23.77 | 33.47 |
| 212002 | 32422 | 2000 | N Control | Veg Soil | 92521 | Chard | 501.82 | 6.68 | 1.87 | 13.19 | 2.06 | 151.18 | 25.89 | 41.05 |
| 212001 | 30722 | 2001 | N Control | Veg Soil | 92521 | Chard | 343.75 | 8.73 | 3.57 | 12.58 | 0.32 | 181.04 | 36.73 | 38.98 |
| 212003 | 12722 | 2003 | N Control | Veg Soil | 92521 | Onion | 220.93 | 20.80 | 2.92 | 16.86 | 1.09 | 185.13 | 23.77 | 11.37 |
| 212004 | 32422 | 2004 | N Control | Veg Soil | 92521 | Onion | 229.26 | 5.83 | 2.00 | 6.53 | 208.86 | 344.37 | 21.74 | 57.93 |
| 212005 | 30822 | 2005 | PH Contro | Veg Soil | 92521 | Zucchini | 112.41 | 11.68 | 7.02 | 29.78 | 12.12 | 2415.85 | 56.11 | 4101.09 |
| 212006 | 12722 | 2006 | PH Contro | Veg Soil | 92521 | Zucchini | 107.63 | 6.68 | 3.75 | 27.78 | 6.22 | 2449.64 | 78.79 | 6410.59 |
| 212007 | 32422 | 2007 | PH Contro | Veg Soil | 92521 | Zucchini | 362.80 | 4.37 | 3.01 | 13.75 | 2.34 | 140.47 | 19.44 | 9.24 |
| 212008 | 32422 | 2008 | N Control | Veg Soil | 92521 | Potato | 1117.44 | 10.52 | 1.66 | 441.98 | 0.81 | 7317.65 | 12.56 | 4.63 |
| 212009 | 32422 | 2009 | N Control | Veg Soil | 92521 | Potato | 615.43 | 6.61 | 6.00 | 100.30 | 0.66 | 259.77 | 18.47 | 15.82 |
| 212011 | 32422 | 2011 | N Control | Veg Soil | 92521 | Carrot | 79.77 | 12.15 | 1.74 | 23.04 | 0.43 | 2447.39 | 27.76 | 11.84 |
| 212012 | 12722 | 2012 | N Control | Veg Soil | 92521 | Carrot | 94.43 | 5.40 | 1.58 | 22.62 | 2.39 | 2460.96 | 25.72 | 30.12 |
| 212013 | 12722 | 2013 | N Control | Veg Soil | 92521 | Carrot | 139.71 | 5.65 | 1.64 | 16.89 | 0.34 | 2562.34 | 29.28 | 33.74 |
| 211476 | 61522 | G5 Tom so | G2 | Veg Soil | 92521 | Tom | 132.35 | 2.97 | 20.26 | 70.64 | 9.92 | 1081.78 | 41.65 | 268.91 |
| 211477 | 61522 | G5 Tom so | G2 | Veg Soil | 92521 | Tom | 140.45 | 7.26 | 17.84 | 66.92 | 10.17 | 1119.83 | 50.34 | 209.56 |
| 211478 | 61522 | G5 Tom so | G2 | Veg Soil | 92521 | Tom | 154.66 | 6.43 | 14.49 | 50.36 | 7.84 | 1078.45 | 51.31 | 244.33 |
| 211141 | 61522 | G5 Zucc so | G2 | Veg Soil | 72121 | Zucchini | 131.14 | 23.98 | 18.00 | 58.37 | 0.81 | 302.14 | 24.52 | 90.31 |
| 211142 | 61522 | G5 Zucc so | G2 | Veg Soil | 72121 | Zucchini | 128.60 | 29.20 | 18.93 | 78.48 | 0.91 | 306.28 | 21.90 | 117.89 |
| 211143 | 61522 | G5 Zucc so | G2 | Veg Soil | 72121 | Zucchini | 110.06 | 25.23 | 14.76 | 41.36 | 0.95 | 318.47 | 22.81 | 87.42 |

*Table A18: Biosolids and biosolids amended soil data. Cells with red text are below detection and imputed. Highlighted cells are outliers.*

| Sample.ID | Run.Numb | Sample.name | Sample.Lo | Sample.Ty | Sample.Da | Veg | PFBA | PFPeA | PFBS | PFHxA | PFPeS | PFHpA | PFHxS | PFOA | PFHpS | PFNA | PFOS | PFDA | PFNS | PFDS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 21823 | 72721 | Biosolids field new 3 | New Field | Hay Soil | 60921 | Hay | 277.95 | 585.57 | 741.61 | 553.82 | 4.94 | 432.99 | 150.01 | 1382.90 | 106.52 | 735.06 | 20291.68 | 5734.28 | 10.48 | 3258.91 |
| 21822 | 72721 | Biosolids field new 2 | New Field | Hay Soil | 60921 | Hay | 230.19 | 546.16 | 763.49 | 789.03 | 10.46 | 610.51 | 166.29 | 1543.15 | 104.62 | 785.16 | 25251.20 | 5965.81 | 16.81 | 3130.77 |
| 21821 | 72721 | Biosolids field new 1 | New Field | Hay Soil | 60921 | Hay | 200.54 | 476.00 | 665.32 | 465.42 | 2.33 | 374.10 | 99.12 | 1126.85 | 83.80 | 715.95 | 25336.05 | 4690.92 | 8.64 | 1635.01 |
| 21817 | 72721 | Biosolids field old 3 | Old Field | Hay Soil | 60921 | Hay | 163.47 | 171.76 | 186.24 | 136.75 | 0.52 | 210.21 | 21.47 | 592.52 | 18.98 | 313.46 | 6654.62 | 2939.78 | 7.07 | 241.81 |
| 21816 | 72721 | Biosolids field old 2 | Old Field | Hay Soil | 60921 | Hay | 151.99 | 270.40 | 230.91 | 207.32 | 1.10 | 262.11 | 35.49 | 940.03 | 21.46 | 474.55 | 6893.01 | 4073.96 | 4.92 | 230.08 |
| 21815 | 72721 | Biosolids field old 1 | Old Field | Hay Soil | 60921 | Hay | 88.16 | 87.14 | 131.75 | 132.06 | 0.25 | 149.56 | 14.32 | 756.95 | 32.43 | 327.52 | 6933.95 | 2356.65 | 6.92 | 209.80 |
| 211193A | 122421 | Hyrum Biosolids 1 | Biosolids | Biosolids | 72121 | Biosolids | 2450.48 | 2454.76 | 1987.58 | 3711.44 | 99.17 | 141.59 | 733.86 | 12418.84 | 50.70 | 4173.15 | 31384.90 | 9608.96 | 2031.28 | 14956.98 |
| 211193C | 122421 | Hyrum Biosolids 3 | Biosolids | Biosolids | 72121 | Biosolids | 1248.11 | 2833.98 | 1960.89 | 2699.96 | 29.69 | 71.03 | 291.61 | 12347.93 | 120.21 | 2649.26 | 30794.72 | 11253.86 | 756.55 | 10753.13 |
| 211193B | 122421 | Hyrum Biosolids 2 | Biosolids | Biosolids | 72121 | Biosolids | 1571.26 | 3812.31 | 1876.61 | 3787.46 | 37.70 | 47.89 | 129.98 | 17068.81 | 136.31 | 3127.39 | 35820.64 | 17866.97 | 601.42 | 8279.31 |
| 212029 | 32622 | Bio 3 1:20 | Biosolids | Biosolids | 91521 | Biosolids | 3292.78 | 1541.72 | 1271.49 | 2639.16 | 14.39 | 484.02 | 230.27 | 12788.44 | 265.80 | 2086.14 | 31594.73 | 13988.22 | 288.41 | 406.65 |
| 212027 | 32622 | Bio 1 1:20 | Biosolids | Biosolids | 91521 | Biosolids | 1664.73 | 1069.24 | 941.37 | 1609.76 | 4.31 | 384.95 | 42.31 | 10779.66 | 43.52 | 1259.36 | 18162.46 | 9061.92 | 85.39 | 313.40 |
| 212028 | 32622 | Bio 2 1:20 | Biosolids | Biosolids | 91521 | Biosolids | 1946.82 | 1017.54 | 1198.39 | 1972.49 | 7.87 | 303.30 | 72.81 | 5982.02 | 167.51 | 1394.71 | 19797.08 | 9308.02 | 156.93 | 215.46 |
| 211983 | 12722 | 1983 | Hay Contr | Hay Contr | 110921 | Hay | 225.89 | 7.50 | 0.23 | 23.07 | 0.53 | 19.71 | 4.08 | 218.87 | 1.35 | 46.06 | 207.74 | 5.27 | 14.94 | 32.25 |
| 211989 | 30722 | 1989 | Hay Contr | Hay Contr | 110921 | Hay | 251.87 | 7.59 | 5.46 | 18.17 | 0.09 | 9.13 | 9.73 | 159.11 | 0.92 | 17.34 | 19.21 | 12.34 | 0.88 | 5.73 |
| 211984 | 30722 | 1984 | Hay Contr | Hay Contr | 110921 | Hay | 201.82 | 16.47 | 1.36 | 17.43 | 0.04 | 10.41 | 0.60 | 243.60 | 1.98 | 12.03 | 22.20 | 4.74 | 0.05 | 3.83 |

*Table A19: hay data. Cells with red text are below detection and imputed. Highlighted cells are outliers.*

| Sample.ID | Run.Numb | Sample.na | Sample.Location | Sample.Type | Sample.Da | Veg | PFBA | PFPeA | PFBS | PFHxA | PFPeS | PFHpA | PFHxS | PFOA | PFNA | PFOS | PFDA |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 21825 | 32622 | 825 | New Field | Hay | 60921 | Hay | 2374.10 | 367.23 | 2792.23 | 464.36 | 60.27 | 186.07 | 304.39 | 2035.11 | 26.49 | 907.14 | 20.69 |
| 21824 | 32622 | 824 | New Field | Hay | 60921 | Hay | 1691.24 | 208.99 | 1826.40 | 309.04 | 56.93 | 112.25 | 243.78 | 1780.84 | 20.95 | 592.53 | 20.27 |
| 21826 | 32622 | 826 | New Field | Hay | 60921 | Hay | 2331.19 | 391.78 | 2263.19 | 496.94 | 69.23 | 180.34 | 219.15 | 2500.69 | 14.03 | 424.15 | 11.96 |
| 21818 | 32622 | 818 | Old Field | Hay | 60921 | Hay | 1159.37 | 15.39 | 500.45 | 60.89 | 3.19 | 20.29 | 5.44 | 1590.46 | 5.14 | 62.24 | 7.47 |
| 21819 | 32622 | 819 | Old Field | Hay | 60921 | Hay | 1169.13 | 24.24 | 551.08 | 51.47 | 1.29 | 17.98 | 1.78 | 1452.20 | 5.18 | 38.68 | 3.15 |
| 21820 | 32622 | 820 | Old Field | Hay | 60921 | Hay | 951.27 | 32.58 | 401.41 | 24.04 | 3.51 | 18.68 | 6.34 | 1393.02 | 6.24 | 50.85 | 3.37 |
| 211982 | 30822 | 1982 | Hay Control 32 | Hay Control | 110921 | Hay | 120.87 | 12.00 | 1.70 | 13.44 | 0.01 | 16.61 | 3.13 | 66.18 | 28.92 | 43.03 | 11.73 |
| 211987 | 22322 | 1987 | Hay Control 21 | Hay Control | 110921 | Hay | 28.81 | 3.25 | 0.08 | 8.26 | 0.01 | 8.69 | 0.33 | 331.62 | 5.73 | 4.70 | 4.16 |
| 211988 | 30722 | 1988 | Hay Control 12 | Hay Control | 110921 | Hay | 19.88 | 23.05 | 0.66 | 18.74 | 0.21 | 14.80 | 17.92 | 139.35 | 11.59 | 20.35 | 2.26 |

*Table A20: Rain data. Cells with red text are below detection and imputed. Highlighted cells are outliers.*

| Sample.ID | Run.Numb | Sample.na | Sample.Lo | Sample.Ty | Sample.Da | Veg | PFBA | PFPeA | PFBS | PFHxA | PFHpA | PFHxS | PFOA | PFNA | PFOS | PFDA | PFDS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 21528 | 62621 | UWRL 2 | UWRL | Rain | 51721 | none | 15.29 | 4.47 | 0.27 | 3.91 | 2.75 | 0.02 | 8.89 | 1.88 | 2.07 | 1.88 | 6.00 |
| 21527 | 61521 | UWRL 1 | UWRL | Rain | 51721 | none | 18.56 | 4.70 | 0.26 | 3.46 | 3.20 | 0.05 | 9.57 | 2.33 | 2.29 | 1.77 | 3.28 |
| 21522 | 61521 | Evans 2 | Evans | Rain | 51721 | none | 6.58 | 1.56 | 0.11 | 2.45 | 1.49 | 0.02 | 6.74 | 1.44 | 0.69 | 1.40 | 1.97 |
| 21523 | 61521 | Evans 3 | Evans | Rain | 51721 | none | 6.92 | 1.57 | 0.13 | 2.22 | 1.92 | 0.04 | 6.51 | 1.41 | 0.70 | 1.50 | 1.79 |
| 21490 | 51621 | UWRL 3 | UWRL | Rain | 42621 | none | 2.36 | 0.48 | 0.01 | 0.94 | 0.95 | 0.04 | 3.45 | 1.16 | 0.60 | 1.00 | 1.55 |
| 21488 | 51621 | UWRL 1 | UWRL | Rain | 42621 | none | 2.33 | 0.02 | 0.02 | 0.96 | 1.01 | 0.09 | 3.27 | 1.27 | 0.61 | 1.11 | 1.41 |
| 21489 | 51621 | UWRL 2 | UWRL | Rain | 42621 | none | 2.24 | 0.35 | 0.01 | 0.98 | 1.17 | 0.06 | 2.90 | 1.07 | 0.57 | 1.04 | 1.26 |
| 211371 | 90721 | Greenville | Greenville | Rain | 62621 | none | 1.03 | 0.32 | 0.04 | 0.42 | 0.23 | 0.004 | 22.34 | 0.17 | 0.31 | 0.28 | 0.54 |
| 21525 | 61521 | Drainage 2 | Drainage | Rain | 51721 | none | 8.84 | 0.65 | 0.07 | 1.48 | 2.02 | 0.006 | 3.81 | 0.96 | 1.34 | 0.96 | 0.39 |
| 21521 | 62621 | Evans 1 | Evans | Rain | 51721 | none | 11.98 | 2.12 | 0.18 | 1.96 | 1.70 | 0.02 | 7.98 | 1.32 | 4.10 | 1.25 | 0.36 |
| 21526 | 62621 | Drainage 3 | Drainage | Rain | 51721 | none | 14.91 | 0.96 | 0.06 | 1.94 | 1.34 | 0.005 | 4.65 | 1.16 | 0.81 | 0.96 | 0.34 |
| 21524 | 62621 | Drainage 1 | Drainage | Rain | 51721 | none | 10.72 | 0.93 | 0.07 | 0.79 | 0.48 | 0.06 | 1.28 | 0.36 | 3.54 | 0.15 | 0.34 |
| 211396 | 90721 | Greenville | Greenville | Rain | 80221 | none | 2.33 | 0.87 | 0.03 | 0.40 | 0.16 | 0.01 | 817.87 | 0.10 | 0.04 | 0.17 | 0.31 |
| 211399 | 90721 | Drainage 3 | Drainage | Rain | 80221 | none | 12.56 | 2.73 | 0.17 | 3.52 | 1.40 | 0.08 | 614.69 | 1.40 | 0.38 | 2.91 | 0.29 |
| 21516 | 62621 | House 2 | Hyrum | Rain | 51721 | none | 3.58 | 0.09 | 0.02 | 2.79 | 0.58 | 0.005 | 1.15 | 0.40 | 3.67 | 0.15 | 0.22 |
| 21484 | 51621 | Drainage 3 | Drainage | Rain | 42621 | none | 1.82 | 1.52 | 0.01 | 0.16 | 0.11 | 0.003 | 0.27 | 0.11 | 0.01 | 0.11 | 0.22 |
| 21942 | 62621 | E2 | Evans | Rain | 52621 | none | 5.35 | 1.12 | 0.06 | 0.74 | 1.38 | 0.01 | 0.99 | 0.71 | 0.29 | 0.21 | 0.21 |
| 21941 | 62621 | E1 | Evans | Rain | 52621 | none | 5.81 | 1.05 | 0.07 | 0.75 | 1.57 | 0.01 | 0.83 | 0.90 | 0.25 | 0.17 | 0.21 |
| 211400 | 90721 | 5SW 1 | 5SW | Rain | 80221 | none | 1.49 | 0.27 | 0.13 | 0.26 | 0.19 | 0.03 | 327.60 | 0.15 | 0.04 | 0.08 | 0.20 |
| 21944 | 70221 | Greenvile : | Greenville | Rain | 52621 | none | 4.82 | 1.11 | 0.10 | 1.48 | 2.12 | 0.006 | 0.91 | 1.23 | 0.04 | 0.23 | 0.19 |
| 211372 | 90721 | Greenville | Greenville | Rain | 62621 | none | 1.78 | 0.37 | 0.01 | 1.04 | 0.42 | 0.02 | 3.24 | 0.56 | 0.05 | 2.16 | 0.19 |
| 21517 | 61521 | House 3 | Hyrum | Rain | 51721 | none | 1.59 | 0.15 | 0.007 | 1.86 | 0.28 | 0.01 | 0.83 | 0.20 | 1.69 | 0.10 | 0.19 |
| 211405 | 90721 | TWDEF 3 | TWDEF | Rain | 80221 | none | 8.96 | 0.34 | 0.06 | 18.99 | 1.19 | 0.02 | 314.57 | 1.74 | 0.14 | 1.05 | 0.18 |
| 211402 | 90721 | 5SW 3 | 5SW | Rain | 80221 | none | 2.03 | 0.16 | 0.01 | 0.28 | 0.24 | 0.02 | 274.87 | 0.21 | 0.03 | 0.41 | 0.17 |
| 21480 | 51621 | 5SW 2 | 5SW | Rain | 42621 | none | 1.06 | 0.13 | 0.006 | 0.16 | 0.10 | 0.01 | 0.22 | 0.09 | 0.03 | 0.06 | 0.17 |
| 21515 | 62621 | House 1 | Hyrum | Rain | 51721 | none | 3.51 | 1.31 | 0.02 | 1.63 | 0.56 | 0.01 | 1.03 | 0.43 | 3.57 | 0.13 | 0.15 |
| 211389 | 90721 | UWRL 2 | UWRL | Rain | 73021 | none | 5.87 | 0.96 | 0.09 | 0.94 | 0.87 | 0.005 | 460.37 | 0.59 | 0.46 | 0.24 | 0.15 |
| 211401 | 90721 | 5SW 2 | 5SW | Rain | 80221 | none | 2.06 | 0.51 | 0.01 | 0.22 | 0.23 | 0.004 | 217.28 | 0.18 | 0.01 | 0.21 | 0.15 |
| 21514 | 62621 | Greenville | Greenville | Rain | 51721 | none | 10.89 | 0.80 | 0.06 | 0.89 | 0.47 | 0.02 | 1.21 | 0.27 | 3.34 | 0.18 | 0.15 |
| 211392 | 90721 | TWDEF 2 | TWDEF | Rain | 73021 | none | 10.20 | 2.10 | 0.13 | 7.22 | 1.16 | 0.02 | 453.96 | 0.94 | 0.17 | 0.73 | 0.14 |
| 21949 | 62621 | U3 | UWRL | Rain | 52621 | none | 6.22 | 1.01 | 0.06 | 0.64 | 1.51 | 0.006 | 1.11 | 1.04 | 0.25 | 0.22 | 0.13 |
| 21943 | 62621 | E3 | Evans | Rain | 52621 | none | 5.41 | 0.92 | 0.07 | 0.69 | 1.50 | 0.006 | 0.78 | 0.67 | 0.24 | 0.14 | 0.13 |
| 21477 | 51621 | House 2 | Hyrum | Rain | 42621 | none | 1.47 | 0.32 | 0.009 | 0.19 | 0.25 | 0.004 | 0.22 | 0.18 | 0.02 | 0.01 | 0.11 |
| 211397 | 90721 | Drainage 1 | Drainage | Rain | 80221 | none | 11.12 | 2.04 | 0.12 | 3.12 | 1.97 | 0.05 | 446.45 | 1.29 | 0.25 | 1.47 | 0.10 |
| 21518 | 61521 | 5SW 1 | 5SW | Rain | 51721 | none | 6.51 | 0.62 | 0.08 | 0.78 | 0.51 | 0.01 | 0.75 | 0.31 | 0.09 | 0.16 | 0.10 |
| 21940 | 62621 | 5SW3 | 5SW | Rain | 52621 | none | 2.40 | 0.16 | 0.03 | 0.24 | 0.40 | 0.05 | 0.44 | 0.20 | 0.32 | 0.07 | 0.10 |
| 21486 | 51621 | Evans 2 | Evans | Rain | 42621 | none | 1.36 | 0.20 | 0.008 | 0.14 | 0.14 | 0.02 | 0.27 | 0.17 | 0.10 | 0.06 | 0.10 |
| 21519 | 61521 | 5SW 2 | 5SW | Rain | 51721 | none | 6.36 | 0.64 | 0.09 | 0.78 | 0.42 | 0.005 | 0.66 | 0.27 | 0.04 | 0.19 | 0.09 |
| 21946 | 62621 | G3 | Greenville | Rain | 52621 | none | 5.88 | 0.93 | 0.09 | 0.71 | 1.74 | 0.01 | 0.82 | 1.10 | 0.23 | 0.16 | 0.09 |
| 21520 | 61521 | 5SW 3 | 5SW | Rain | 51721 | none | 5.33 | 0.27 | 0.08 | 0.69 | 0.55 | 0.01 | 0.72 | 0.29 | 0.02 | 0.16 | 0.09 |
| 211374 | 90721 | UWRL 1 | UWRL | Rain | 62621 | none | 3.43 | 0.35 | 0.05 | 0.33 | 1.00 | 0.01 | 3.19 | 0.47 | 0.07 | 0.27 | 0.08 |
| 21948 | 62621 | U2 | UWRL | Rain | 52621 | none | 6.33 | 0.71 | 0.09 | 0.73 | 1.62 | 0.03 | 1.37 | 1.09 | 0.19 | 0.20 | 0.08 |
| 21513 | 61521 | Greenville | Greenville | Rain | 51721 | none | 6.29 | 0.66 | 0.07 | 0.74 | 0.47 | 0.01 | 1.31 | 0.29 | 3.68 | 0.18 | 0.08 |
| 211393 | 90721 | TWDEF 3 | TWDEF | Rain | 80221 | none | 6.26 | 0.82 | 0.12 | 6.64 | 0.86 | 0.01 | 331.20 | 0.78 | 0.03 | 0.32 | 0.08 |
| 211394 | 90721 | Greenville | Greenville | Rain | 80221 | none | 2.03 | 0.25 | 0.03 | 0.31 | 0.14 | 0.004 | 213.08 | 0.08 | 0.010 | 0.12 | 0.07 |
| 211403 | 90721 | TWDEF 1 | TWDEF | Rain | 80221 | none | 13.52 | 0.23 | 0.11 | 21.24 | 0.86 | 0.03 | 324.46 | 0.89 | 0.13 | 0.86 | 0.07 |
| 21483 | 51621 | Drainage 2 | Drainage | Rain | 42621 | none | 1.57 | 0.14 | 0.006 | 0.20 | 0.11 | 0.02 | 0.41 | 0.15 | 0.02 | 0.09 | 0.07 |
| 21938 | 62621 | 5SW1 | 5SW | Rain | 52621 | none | 2.72 | 0.41 | 0.02 | 0.22 | 0.43 | 0.06 | 0.93 | 0.20 | 0.25 | 0.07 | 0.07 |
| 211404 | 90721 | TWDEF 2 | TWDEF | Rain | 80221 | none | 13.70 | 0.40 | 0.09 | 19.42 | 1.21 | 0.01 | 142.93 | 1.08 | 0.07 | 0.71 | 0.06 |
| 211390 | 90721 | UWRL 3 | UWRL | Rain | 73021 | none | 5.12 | 0.62 | 0.09 | 0.82 | 0.64 | 0.01 | 238.12 | 0.43 | 0.12 | 0.25 | 0.06 |
| 21474 | 51621 | Greenville | Greenville | Rain | 42621 | none | 3.77 | 0.67 | 0.01 | 0.43 | 0.34 | 0.01 | 0.71 | 0.29 | 0.04 | 0.11 | 0.06 |
| 21481 | 51621 | 5SW 3 | 5SW | Rain | 42621 | none | 1.46 | 1.30 | 0.01 | 0.16 | 0.11 | 0.003 | 0.19 | 0.11 | 0.07 | 0.05 | 0.06 |
| 211388 | 90721 | UWRL 1 | UWRL | Rain | 73021 | none | 5.81 | 1.78 | 0.05 | 0.92 | 0.76 | 0.02 | 297.96 | 0.51 | 0.29 | 0.28 | 0.05 |
| 211398 | 90721 | Drainage 2 | Drainage | Rain | 80221 | none | 9.97 | 1.75 | 0.10 | 3.66 | 1.56 | 0.02 | 840.24 | 1.14 | 0.48 | 1.28 | 0.05 |
| 211391 | 90721 | TWDEF 1 | TWDEF | Rain | 73021 | none | 4.57 | 0.74 | 0.12 | 10.58 | 0.70 | 0.03 | 524.26 | 0.50 | 0.21 | 1.28 | 0.05 |
| 211376 | 90721 | UWRL 3 | UWRL | Rain | 62621 | none | 3.00 | 1.61 | 0.02 | 0.77 | 1.11 | 0.03 | 2.83 | 0.63 | 0.53 | 0.50 | 0.05 |
| 21939 | 62621 | 5SW2 | 5SW | Rain | 52621 | none | 3.40 | 0.34 | 0.02 | 0.21 | 0.41 | 0.06 | 0.32 | 0.19 | 0.12 | 0.07 | 0.05 |
| 21479 | 51621 | 5SW 1 | 5SW | Rain | 42621 | none | 0.89 | 0.30 | 0.009 | 0.26 | 0.26 | 0.01 | 3.17 | 0.11 | 0.03 | 0.07 | 0.05 |
| 211377 | 90721 | House 1 | Hyrum | Rain | 62621 | none | 4.89 | 1.41 | 0.34 | 1.65 | 0.85 | 0.04 | 4.25 | 0.86 | 0.02 | 2.18 | 0.04 |
| 21947 | 62621 | U1 | UWRL | Rain | 52621 | none | 4.91 | 0.80 | 0.06 | 0.63 | 1.35 | 0.01 | 1.16 | 1.09 | 0.12 | 0.21 | 0.04 |
| 211375 | 90721 | UWRL 2 | UWRL | Rain | 62621 | none | 2.93 | 0.91 | 0.03 | 0.98 | 1.33 | 0.04 | 5.29 | 0.51 | 0.09 | 0.32 | 0.03 |
| 211373 | 90721 | Greenville | Greenville | Rain | 62621 | none | 1.19 | 0.14 | 0.03 | 0.30 | 0.17 | 0.004 | 3.44 | 0.07 | 0.02 | 0.12 | 0.03 |
| 21945 | 62621 | G2 | Greenville | Rain | 52621 | none | 5.95 | 1.04 | 0.07 | 0.81 | 1.81 | 0.01 | 0.94 | 1.13 | 0.19 | 0.21 | 0.02 |
| 21473 | 51621 | Greenville | Greenville | Rain | 42621 | none | 4.83 | 0.74 | 0.01 | 0.45 | 0.40 | 0.02 | 0.48 | 0.42 | 0.34 | 0.12 | 0.02 |
| 21475 | 51621 | Greenville | Greenville | Rain | 42621 | none | 2.44 | 0.35 | 0.010 | 0.28 | 0.25 | 0.01 | 0.45 | 0.26 | 0.06 | 0.09 | 0.02 |
| 21482 | 51621 | Drainage 1 | Drainage | Rain | 42621 | none | 1.22 | 0.15 | 0.006 | 0.18 | 0.11 | 0.01 | 0.18 | 0.12 | 0.01 | 0.08 | 0.02 |
| 21485 | 51621 | Evans 1 | Evans | Rain | 42621 | none | 1.45 | 0.31 | 0.01 | 0.17 | 0.13 | 0.003 | 0.24 | 0.16 | 0.03 | 0.07 | 0.02 |
| 21487 | 51621 | Evans 3 | Evans | Rain | 42621 | none | 1.12 | 0.25 | 0.008 | 0.15 | 0.13 | 0.003 | 0.14 | 0.19 | 0.15 | 0.04 | 0.02 |
| 21478 | 51621 | House 3 | Hyrum | Rain | 42621 | none | 1.43 | 0.19 | 0.007 | 0.21 | 0.22 | 0.004 | 0.19 | 0.14 | 0.02 | 0.03 | 0.02 |
| 21476 | 51621 | House 1 | Hyrum | Rain | 42621 | none | 1.03 | 0.18 | 0.007 | 0.16 | 0.14 | 0.01 | 0.16 | 0.11 | 0.01 | 0.03 | 0.02 |

*Table A21: Vegetable data. Cells with red text are below detection and imputed. Highlighted cells are outliers.*

| Sample.ID | Run.Numb | Sample.name | Sample.Lo | Sample.Ty | Sample.Da | Veg | PFBA | PFPeA | PFBS | PFHxA | PFHpA | PFOA | PFOS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 211900 | 20222 | N car 3 | N Control | Veg | 92521 | Carrot | 466.61 | 11.44 | 11.02 | 30.80 | 3.59 | 472.78 | 8.12 |
| 211901 | 20222 | N car 1 | N Control | Veg | 92521 | Carrot | 582.56 | 26.12 | 1.94 | 22.88 | 6.70 | 526.65 | 4.27 |
| 211902 | 20222 | N car 2 | N Control | Veg | 92521 | Carrot | 853.35 | 25.59 | 2.54 | 53.57 | 3.03 | 714.35 | 2.25 |
| 211903 | 20222 | N chard 1 | N Control | Veg | 92521 | Chard | 1853.12 | 490.40 | 31.80 | 43.30 | 26.71 | 323.85 | 63.93 |
| 211904 | 20222 | N chard 2 | N Control | Veg | 92521 | Chard | 2807.30 | 2088.26 | 27.29 | 36.79 | 25.82 | 193.42 | 2.11 |
| 211905 | 20222 | N chard 3 | N Control | Veg | 92521 | Chard | 1578.06 | 517.27 | 35.81 | 32.01 | 22.82 | 152.08 | 5.88 |
| 211912 | 20622 | PH green bean 2 | PH Contro | Veg | 92521 | Green bea | 2913.31 | 19.99 | 5.90 | 57.57 | 12.71 | 238.17 | 1.98 |
| 211913 | 20622 | PH green bean 3 | PH Contro | Veg | 92521 | Green bea | 2712.91 | 8.35 | 5.26 | 71.01 | 4.87 | 125.77 | 4.00 |
| 211917 | 20622 | PH zucc 1 0.767 yello | PH Contro | Veg | 92521 | Zucchini | 5788.64 | 81.55 | 6.32 | 52.29 | 19.69 | 1787.77 | 27.64 |
| 211918 | 20622 | PH zucc 1 green | PH Contro | Veg | 92521 | Zucchini | 70.18 | 9.75 | 10.53 | 27.85 | 6.86 | 246.59 | 1.85 |
| 211919 | 20622 | PH zucc 1 yellow | PH Contro | Veg | 92521 | Zucchini | 843.56 | 40.62 | 4.54 | 17.43 | 10.39 | 678.55 | 33.15 |
| 211920 | 20622 | PH zucc 2 yellow | PH Contro | Veg | 92521 | Zucchini | 805.98 | 73.58 | 36.29 | 23.88 | 9.04 | 519.55 | 11.98 |
| 211921 | 20622 | PH zucc 3 green | PH Contro | Veg | 92521 | Zucchini | 521.37 | 26.11 | 6.12 | 31.77 | 3.36 | 222.41 | 13.24 |
| 211922 | 20622 | PH zucc 3 yellow | PH Contro | Veg | 92521 | Zucchini | 812.33 | 35.02 | 6.87 | 29.27 | 7.89 | 560.74 | 51.06 |
| 211923 | 20622 | PH zucc green | PH Contro | Veg | 92521 | Zucchini | 52.61 | 15.81 | 1.64 | 872.58 | 10.54 | 250.67 | 3.52 |
| 211127 | 11522 | G4 zucc 1 | G1 | Veg | 72121 | Zucchini | 1959.90 | 40.83 | 11.92 | 146.08 | 5.71 | 3323.08 | 11.73 |
| 211126 | 30922 | G4 zucc .558 2 | G1 | Veg | 72121 | Zucchini | 1716.98 | 151.70 | 4.87 | 115.11 | 52.04 | 3113.43 | 29.71 |
| 211128 | 11522 | G4 zucc 3 | G1 | Veg | 72121 | Zucchini | 2118.11 | 97.07 | 10.04 | 170.37 | 7.15 | 4048.62 | 17.12 |
| 211133 | 11122 | G4 potato 2 | G1 | Veg | 72121 | Potato | 2878.36 | 24.76 | 2.02 | 445.30 | 5.43 | 7445.06 | 19.05 |
| 211137 | 20622 | G5 zucc 3 0.588 | G2 | Veg | 72121 | Zucchini | 1332.27 | 67.79 | 4.17 | 37.05 | 41.73 | 1123.92 | 67.25 |
| 211138 | 20622 | G5 zucc 1 | G2 | Veg | 72121 | Zucchini | 281.86 | 67.92 | 8.49 | 239.97 | 18.22 | 354.45 | 67.18 |
| 211139 | 20622 | G5 zucc 2 | G2 | Veg | 72121 | Zucchini | 303.66 | 110.24 | 4.02 | 1721.03 | 15.03 | 574.53 | 15.54 |
| 211144 | 13122 | G5 Lettuce 1 | G2 | Veg | 72121 | Lettuce | 800.34 | 140.84 | 8.86 | 108.78 | 18.08 | 10179.00 | 159.20 |
| 211145 | 13122 | G5 Lettuce 2 | G2 | Veg | 72121 | Lettuce | 494.62 | 154.14 | 4.32 | 102.59 | 31.37 | 11427.10 | 72.34 |
| 211146 | 13122 | G5 Lettuce 3 | G2 | Veg | 72121 | Lettuce | 777.96 | 84.90 | 1.16 | 115.66 | 40.40 | 11653.80 | 23.03 |
| 211150 | 11522 | G6 Tom 1 | G3 | Veg | 72121 | Tom | 1215.38 | 541.09 | 7.35 | 357.41 | 7.40 | 3531.09 | 16.48 |
| 211151 | 30822 | 1151 | G3 | Veg | 72121 | Tom | 964.05 | 518.00 | 7.98 | 502.73 | 15.54 | 1327.34 | 12.77 |
| 211152 | 11522 | G6 Tom 3 | G3 | Veg | 72121 | Tom | 1411.74 | 419.44 | 8.15 | 279.43 | 3.99 | 1724.83 | 9.78 |
| 211157 | 11522 | G6 Garlic 2 | G3 | Veg | 72121 | Garlic | 125.73 | 262.73 | 1.10 | 9.50 | 6.17 | 2216.20 | 2.40 |
| 211158 | 11522 | G6 Garlic 3 | G3 | Veg | 72121 | Garlic | 110.61 | 17.34 | 4.99 | 372.40 | 5.89 | 2342.03 | 10.91 |
| 211162 | 13122 | Kale blue 1 | G3 | Veg | 72121 | Kale | 1094.71 | 1569.63 | 51.38 | 68.66 | 12.16 | 9985.51 | 28.27 |
| 211163 | 11122 | Kale blue 2 | G3 | Veg | 72121 | Kale | 2063.97 | 471.67 | 59.65 | 186.29 | 35.52 | 5732.98 | 8.66 |
| 211164 | 11122 | Kale blue 3 | G3 | Veg | 72121 | Kale | 1165.02 | 291.87 | 53.85 | 459.75 | 129.01 | 14403.45 | 123.37 |
| 211171 | 30822 | 1171 | G3 | Veg | 72121 | Red Beet | 70.30 | 61.02 | 2.93 | 16.20 | 8.37 | 14419.44 | 60.62 |
| 211172 | 30822 | 1172 | G3 | Veg | 72121 | Red Beet | 104.70 | 15.57 | 4.28 | 22.01 | 11.69 | 8072.79 | 1.74 |
| 211173 | 11522 | Beet red 3 | G3 | Veg | 72121 | Red Beet | 194.63 | 28.74 | 2.85 | 22.42 | 6.50 | 7910.08 | 18.19 |
| 211174 | 11522 | Beet Gold 1 | G3 | Veg | 72121 | Gold Beet | 114.32 | 33.30 | 10.21 | 215.30 | 20.48 | 6474.36 | 5.51 |
| 211175 | 11522 | Beet Gold 2 | G3 | Veg | 72121 | Gold Beet | 371.30 | 138.81 | 9.67 | 107.21 | 129.81 | 6252.26 | 9.09 |
| 211176 | 11522 | Beet Gold 3 | G3 | Veg | 72121 | Gold Beet | 248.51 | 55.80 | 11.02 | 129.86 | 11.75 | 4941.17 | 1.63 |
| 211180 | 30822 | 1181 | G3 | Veg | 72121 | Onion | 165.45 | 171.56 | 3.03 | 20.34 | 5.81 | 5078.74 | 3.75 |
| 211181 | 30722 | 1182 | G3 | Veg | 72121 | Onion | 114.48 | 20.74 | 3.08 | 28.40 | 6.29 | 2448.24 | 1.53 |
| 211182 | 30822 | 1182 | G3 | Veg | 72121 | Onion | 73.57 | 11.23 | 2.62 | 24.92 | 4.67 | 2361.22 | 18.85 |
| 211454 | 11122 | G4 tom | G1 | Veg | 72121 | Tom | 70.82 | 78.92 | 19.20 | 83.69 | 40.79 | 1402.27 | 206.51 |
| 211456 | 30922 | G4 butt 1 | G1 | Veg | 72121 | Butternut | 464.45 | 21.44 | 1.83 | 18.34 | 13.72 | 369.19 | 1.43 |
| 211458 | 30922 | G4 butt 3 | G1 | Veg | 72121 | Butternut | 2604.28 | 27.27 | 1.05 | 86.03 | 19.86 | 537.26 | 3.10 |
| 211459 | 30922 | G4 soy bean 1 | G1 | Veg | 72121 | Soybean | 933.67 | 76.21 | 3.36 | 38.65 | 16.05 | 924.04 | 9.16 |
| 211460 | 20622 | G4 soybean 2 | G1 | Veg | 72121 | Soybean | 1265.91 | 17.36 | 8.53 | 2252.51 | 16.66 | 627.72 | 25.17 |
| 211462 | 30822 | G4 carrot 1 | G1 | Veg | 72121 | Carrot | 376.24 | 42.85 | 7.44 | 26.68 | 19.59 | 1695.79 | 19.09 |
| 211463 | 30822 | G4 carrot 2 | G1 | Veg | 72121 | Carrot | 732.51 | 58.20 | 1.00 | 23.53 | 17.13 | 1090.43 | 15.59 |
| 211464 | 30922 | G4 carrot 3 | G1 | Veg | 72121 | Carrot | 1564.99 | 42.98 | 0.95 | 58.22 | 23.21 | 1204.99 | 4.55 |
| 211471 | 30822 | G5 tom 2 | G2 | Veg | 72121 | Tom | 1570.12 | 202.43 | 4.20 | 36.99 | 3.46 | 796.68 | 1.35 |
| 211471 | 30922 | G5 tom 1 | G2 | Veg | 72121 | Tom | 844.43 | 285.45 | 6.61 | 113.99 | 3.33 | 743.39 | 1.26 |
| 211473 | 30922 | G5 tom 3 | G2 | Veg | 72121 | Tom | 1046.65 | 346.65 | 29.65 | 190.96 | 20.26 | 1008.35 | 1.18 |
| 211516 | 30822 | N tom 1 | N Control | Veg | 92521 | Tom | 475.71 | 46.09 | 0.91 | 78.88 | 3.20 | 467.70 | 2.72 |
| 211517 | 30822 | N tom 2 | N Control | Veg | 92521 | Tom | 466.00 | 20.78 | 1.41 | 104.99 | 3.08 | 251.32 | 1.11 |
| 211518 | 30922 | N tom 3 | N Control | Veg | 92521 | Tom | 948.89 | 46.10 | 3.14 | 52.74 | 15.70 | 335.94 | 1.04 |
| 211453 | 30722 | 1453 | G1 | Veg | 72121 | Tom | 95.68 | 8.09 | 3.24 | 4.08 | 4.88 | 82.14 | 13.22 |
| 211522 | 30822 | 1522 | N Control | Veg | 92521 | Cucumber | 1242.22 | 595.44 | 5.30 | 12.35 | 2.97 | 304.08 | 0.98 |
| 211523 | 30822 | 1523 | N Control | Veg | 92521 | Cucumber | 1427.49 | 698.47 | 3.34 | 1.00 | 2.86 | 354.89 | 0.92 |
| 211524 | 13122 | N cucc 3 | N Control | Veg | 92521 | Cucumber | 2378.19 | 12.41 | 1.76 | 20.76 | 17.59 | 410.29 | 31.88 |
| 211528 | 11522 | N green bean | N Control | Veg | 92521 | Green bea | 91.42 | 29.22 | 15.83 | 26.12 | 25.55 | 956.16 | 19.70 |
| 211536 | 30822 | G6 carrot 1 | G3 | Veg | 92521 | Carrot | 119.18 | 44.98 | 1.49 | 35.84 | 21.29 | 2591.96 | 2.90 |
| 211537 | 30822 | G6 carrot 2 | G3 | Veg | 92521 | Carrot | 169.95 | 29.37 | 0.87 | 27.88 | 14.59 | 1430.58 | 3.30 |
| 211538 | 30822 | G6 carrot 3 | G3 | Veg | 92521 | Carrot | 123.32 | 16.50 | 2.46 | 28.49 | 19.70 | 2249.28 | 0.86 |
| 212017 | 30822 | N carrot 1 | N Control | Veg | 92521 | Carrot | 224.23 | 11.75 | 1.95 | 79.61 | 9.45 | 521.09 | 0.81 |
| 212021 | 30822 | PH tom 2 | PH Contro | Veg | 92521 | Tom | 503.64 | 28.75 | 4.41 | 137.44 | 2.75 | 360.63 | 0.76 |
| 212021 | 30922 | Ph tom 2 | PH Contro | Veg | 92521 | Tom | 651.69 | 55.68 | 7.07 | 82.13 | 2.65 | 747.09 | 12.40 |
| 212022 | 30822 | PH tom 3 | PH Contro | Veg | 92521 | Tom | 332.70 | 47.32 | 9.17 | 140.90 | 3.76 | 705.41 | 65.38 |
| 212026 | 30922 | PH green bean soil1 | N Control | Veg | 92521 | Green bea | 129.76 | 4.92 | 1.22 | 25.61 | 27.92 | 1551.62 | 4.85 |
| 212027 | 30922 | PH green bean 2 | PH Contro | Veg | 92521 | Green bea | 6420.04 | 15.97 | 1.68 | 127.05 | 21.56 | 316.30 | 0.71 |
| 212028 | 30922 | PH green bean 2 | PH Contro | Veg | 92521 | Green bea | 3440.85 | 32.67 | 0.83 | 72.41 | 18.08 | 1340.33 | 0.66 |
| 211134 | 32422 | 1134 | G1 | Veg | 72121 | Potato | 3544.65 | 28.93 | 0.79 | 515.22 | 8.34 | 7371.46 | 7.6713 |
| 211162 | 32422 | 1162 | G3 | Veg | 72121 | Kale | 3051.90 | 1299.67 | 53.33 | 78.97 | 16.63 | 10961.55 | 22.18 |
| 211163 | 32422 | 1163 | G3 | Veg | 72121 | Kale | 1595.37 | 1311.19 | 31.80 | 159.69 | 22.91 | 11614.25 | 2.55 |
| 211164 | 32422 | 1164 | G3 | Veg | 72121 | Kale | 1431.94 | 183.30 | 28.22 | 338.72 | 89.78 | 14326.01 | 5.17 |
| 211454 | 32422 | 1454 | G1 | Veg | 72121 | Tom | 74.51 | 74.33 | 10.91 | 43.62 | 27.42 | 1180.41 | 245.90 |
| 211528 | 32422 | 1528 | N Control | Veg | 92521 | Green bea | 97.09 | 31.19 | 7.17 | 18.16 | 19.75 | 733.76 | 36.55 |
| 212014 | 32422 | 2016 | PH Contro | Veg | 92521 | Onion | 56.01 | 12.85 | 1.28 | 15.93 | 22.77 | 1864.68 | 21.88 |
| 212014 | 32422 | 2014 | PH Contro | Veg | 92521 | Onion | 192.71 | 3312.07 | 0.75 | 15.39 | 3.17 | 295.08 | 48.29 |
| 212015 | 32422 | 2015 | PH Contro | Veg | 92521 | Onion | 394.03 | 1811.02 | 0.71 | 3.09 | 2.55 | 340.38 | 16.63 |
| 211461 | 32422 | G4 soybean | G1 | Veg | 72121 | Soybean | 1252.59 | 7.20 | 3.66 | 5896.90 | 16.02 | 824.00 | 0.62 |

*Table A22: wastewater and spigot data. Cells with red text are below detection and imputed. Highlighted cells are outliers.*

| Sample.ID | Run.Numb | Sample.na | Sample.Lo | Sample.Ty | Sample.Da | Veg | PFBA | PFPeA | PFBS | PFHxA | PFPeS | PFHxS | PFOA | PFNA | PFOS | PFDA | PFDS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 21535 | 70221 | I1 | Hyrum WV | WW Influe | 61621 | none | 1.26 | 62.40 | 0.49 | 3.65 | 1.67 | 1.10 | 0.94 | 0.21 | 0.15 | 0.15 | 0.23 |
| 21536 | 70221 | I2 | Hyrum WV | WW Influe | 61621 | none | 1.57 | 200.52 | 0.46 | 2.96 | 0.62 | 0.99 | 0.76 | 0.24 | 0.25 | 0.18 | 0.35 |
| 21537 | 70221 | I3 | Hyrum WV | WW Influe | 61621 | none | 1.38 | 5.05 | 0.42 | 4.10 | 1.00 | 0.81 | 0.71 | 0.20 | 0.25 | 0.12 | 0.22 |
| 21538 | 70221 | Eff 1 | Hyrum WV | WW Efflue | 61621 | none | 5.11 | 17.06 | 3.22 | 22.69 | 0.12 | 0.47 | 7.20 | 0.17 | 0.65 | 0.23 | 0.29 |
| 21539 | 70221 | Eff 2 | Hyrum WV | WW Efflue | 61621 | none | 5.65 | 18.34 | 2.75 | 21.60 | 0.08 | 0.46 | 7.07 | 0.22 | 0.62 | 0.15 | 0.07 |
| 21540 | 70221 | Eff 3 | Hyrum WV | WW Efflue | 61621 | none | 5.24 | 16.78 | 2.84 | 20.89 | 0.09 | 0.66 | 7.70 | 0.12 | 0.56 | 0.26 | 0.16 |
| 21863 | 70221 | G4 1 | G1 | Spigot | 61621 | none | 0.40 | 0.21 | 0.12 | 0.11 | 0.004 | 0.13 | 0.17 | 0.03 | 0.11 | 0.10 | 0.27 |
| 21864 | 70221 | G4 2 | G1 | Spigot | 61621 | none | 0.33 | 0.22 | 0.10 | 0.15 | 0.007 | 0.04 | 0.29 | 0.03 | 0.14 | 0.09 | 0.14 |
| 21865 | 70221 | G4 3 | G1 | Spigot | 61621 | none | 0.28 | 0.44 | 0.14 | 0.12 | 0.005 | 0.13 | 0.19 | 0.08 | 0.06 | 0.11 | 0.06 |
| 21866 | 70221 | G5 1 | G2 | Spigot | 61621 | none | 0.59 | 0.21 | 0.10 | 0.19 | 0.010 | 0.10 | 0.17 | 0.06 | 0.07 | 0.04 | 0.29 |
| 21867 | 70221 | G5 2 | G2 | Spigot | 61621 | none | 0.38 | 0.33 | 0.09 | 0.15 | 0.006 | 0.11 | 0.20 | 0.01 | 0.09 | 0.09 | 0.30 |
| 21868 | 70221 | G5 3 | G2 | Spigot | 61621 | none | 0.42 | 0.17 | 0.13 | 0.14 | 0.02 | 0.20 | 0.20 | 0.07 | 0.02 | 0.04 | 0.38 |
| 99990 | 42122 | N1 | N Control | Spigot | 31622 | none | 0.17 | 0.07 | 0.009 | 0.009 | 0.0009 | 0.009 | 0.21 | 0.02 | 0.04 | 0.03 | 0.01 |
| 99992 | 42122 | N3 | N Control | Spigot | 31622 | none | 0.42 | 0.08 | 0.01 | 0.01 | 0.001 | 0.009 | 0.23 | 0.02 | 0.04 | 0.04 | 0.01 |
| 99993 | 42122 | N2 | N Control | Spigot | 31622 | none | 0.07 | 0.06 | 0.008 | 0.02 | 0.0007 | 0.008 | 0.28 | 0.03 | 0.04 | 0.04 | 0.01 |
| 211093 | 80321 | L1 Spigot 1 | L1 Spigot | Spigot | 72121 | none | 7.12 | 43.37 | 13.98 | 20.16 | 0.16 | 0.78 | 10.61 | 0.50 | 0.67 | 0.46 | 0.08 |
| 211094 | 80321 | L1 Spigot 2 | L1 Spigot | Spigot | 72121 | none | 6.53 | 42.50 | 14.44 | 18.88 | 0.09 | 0.83 | 10.20 | 0.37 | 0.82 | 0.38 | 0.08 |
| 211095 | 80321 | L1 Spigot 3 | L1 Spigot | Spigot | 72121 | none | 5.95 | 33.81 | 13.66 | 18.42 | 0.18 | 0.75 | 11.12 | 0.39 | 1.88 | 0.59 | 0.28 |
| 211096 | 80321 | L2 Spigot 1 | L2 Spigot | Spigot | 72121 | none | 4.30 | 20.24 | 8.22 | 10.06 | 0.07 | 0.41 | 5.45 | 0.32 | 0.48 | 0.32 | 0.02 |
| 211097 | 80321 | L2 Spigot 2 | L2 Spigot | Spigot | 72121 | none | 3.92 | 20.62 | 10.38 | 10.39 | 0.09 | 0.45 | 5.38 | 0.32 | 2.97 | 0.37 | 0.02 |
| 211098 | 80321 | L2 Spigot 3 | L2 Spigot | Spigot | 72121 | none | 3.97 | 18.67 | 5.80 | 8.75 | 0.09 | 0.46 | 5.60 | 0.28 | 2.18 | 0.27 | 0.16 |
| 211099 | 80321 | L3 Spigot 1 | L3 Spigot | Spigot | 72121 | none | 4.48 | 39.23 | 12.29 | 16.30 | 0.24 | 0.92 | 11.02 | 0.42 | 3.10 | 0.39 | 0.09 |
| 211100 | 80321 | L3 Spigot 2 | L3 Spigot | Spigot | 72121 | none | 3.30 | 18.99 | 4.17 | 8.43 | 0.07 | 0.28 | 4.62 | 0.25 | 0.97 | 0.37 | 0.05 |
| 211101 | 80321 | L3 Spigot 3 | L3 Spigot | Spigot | 72121 | none | 4.12 | 14.00 | 3.48 | 7.72 | 0.06 | 0.35 | 4.47 | 0.27 | 1.26 | 0.52 | 0.47 |
| 211102 | 80321 | L4 Spigot 1 | L4 Spigot | Spigot | 72121 | none | 3.79 | 20.05 | 5.85 | 11.47 | 0.08 | 1.30 | 7.40 | 0.25 | 0.29 | 0.42 | 0.09 |
| 211103 | 80321 | L4 Spigot 2 | L4 Spigot | Spigot | 72121 | none | 5.00 | 20.92 | 6.41 | 11.59 | 0.08 | 0.37 | 8.54 | 0.47 | 0.53 | 1.12 | 0.25 |
| 211104 | 80321 | L4 Spigot 3 | L4 Spigot | Spigot | 72121 | none | 5.83 | 14.86 | 4.57 | 9.36 | 0.06 | 0.97 | 7.75 | 0.29 | 0.57 | 0.47 | 0.03 |
| 211105 | | 1107 | | | 72121 | | 1.60 | 13.40 | 3.37 | 0.23 | 0.04 | 0.09 | 0.98 | 0.65 | 1.74 | 0.27 | 0.03 |
| 211106 | | 1105 | | | 72121 | | 2.78 | 14.16 | 3.70 | 6.15 | 0.09 | 0.32 | 62.60 | 0.20 | 0.33 | 0.28 | 0.01 |
| 211107 | | 1106 | | | 72121 | | 2.39 | 0.51 | 0.15 | 10.56 | 0.05 | 0.32 | 805.99 | 0.42 | 1.74 | 0.86 | 0.12 |
| 211108 | 80321 | L6 Spigot 1 | L6 Spigot | Spigot | 72121 | none | 0.57 | 0.27 | 0.06 | 0.99 | 0.02 | 0.01 | 1.79 | 0.43 | 0.03 | 3.87 | 0.03 |
| 211109 | 80321 | L6 Spigot 2 | L6 Spigot | Spigot | 72121 | none | 0.34 | 0.31 | 0.02 | 0.07 | 0.003 | 0.01 | 0.12 | 0.02 | 0.13 | 0.04 | 0.01 |
| 211110 | 80321 | L6 Spigot 3 | L6 Spigot | Spigot | 72121 | none | 0.91 | 0.24 | 0.05 | 0.02 | 0.002 | 0.01 | 0.16 | 0.09 | 0.04 | 0.08 | 0.02 |
| 211111 | 80321 | L7 Spigot 1 | L7 Spigot | Spigot | 72121 | none | 0.29 | 0.09 | 0.01 | 0.05 | 0.002 | 0.01 | 0.11 | 0.01 | 0.22 | 0.06 | 0.02 |
| 211112 | 80321 | L7 Spigot 2 | L7 Spigot | Spigot | 72121 | none | 0.81 | 1.64 | 0.04 | 0.02 | 0.002 | 0.01 | 0.16 | 0.03 | 0.09 | 0.07 | 0.04 |
| 211113 | 80321 | L7 Spigot 3 | L7 Spigot | Spigot | 72121 | none | 0.30 | 0.65 | 0.01 | 0.01 | 0.001 | 0.01 | 0.18 | 0.10 | 0.22 | 0.13 | 0.06 |
| 211114 | 80321 | Irrigation F | Surface W | Spigot | 72121 | none | 3.63 | 2.97 | 0.04 | 0.15 | 0.006 | 0.03 | 0.25 | 0.02 | 0.34 | 0.13 | 0.26 |
| 211115 | 80321 | Irrigation F | Surface W | Spigot | 72121 | none | 0.63 | 0.10 | 0.01 | 0.01 | 0.0010 | 0.02 | 0.12 | 0.08 | 0.20 | 0.17 | 3.18 |
| 211116 | 80321 | Irrigation F | Surface W | Spigot | 72121 | none | 0.19 | 0.43 | 0.05 | 0.11 | 0.004 | 0.02 | 1.51 | 0.31 | 0.14 | 2.06 | 0.12 |
| 211117 | 80321 | Irrigation ( | Surface W | Spigot | 72121 | none | 0.31 | 0.20 | 0.02 | 0.05 | 0.002 | 0.01 | 0.18 | 0.06 | 0.07 | 0.12 | 0.12 |
| 211118 | 80321 | Irrigation ( | Surface W | Spigot | 72121 | none | 0.97 | 0.19 | 0.05 | 0.17 | 0.009 | 0.01 | 0.24 | 0.03 | 0.25 | 0.08 | 0.08 |
| 211119 | 80321 | Irrigation ( | Surface W | Spigot | 72121 | none | 0.70 | 0.24 | 0.01 | 0.71 | 0.01 | 0.01 | 1.47 | 0.41 | 0.79 | 0.44 | 0.21 |
| 211120 | 80321 | G4 Spigot : | G1 | Spigot | 72121 | none | 2.03 | 0.15 | 0.09 | 0.16 | 0.90 | 0.04 | 0.65 | 0.15 | 0.24 | 0.54 | 0.05 |
| 211121 | 80321 | G4 Spigot : | G1 | Spigot | 72121 | none | 1.11 | 0.53 | 0.04 | 0.11 | 0.05 | 0.12 | 0.35 | 0.13 | 0.08 | 0.07 | 0.02 |
| 211122 | 80321 | G4 Spigot : | G1 | Spigot | 72121 | none | 1.20 | 0.81 | 0.06 | 0.34 | 0.05 | 0.04 | 0.28 | 0.07 | 0.24 | 0.01 | 0.07 |
| 211123 | 80321 | G5 Spigot : | G2 | Spigot | 72121 | none | 4.07 | 1.25 | 0.19 | 0.05 | 0.003 | 0.21 | 0.24 | 0.10 | 0.84 | 0.22 | 0.02 |
| 211124 | 80321 | G5 Spigot : | G2 | Spigot | 72121 | none | 2.13 | 1.39 | 0.18 | 0.59 | 0.01 | 0.21 | 0.37 | 0.09 | 0.11 | 0.22 | 0.18 |
| 211187 | 83121 | Influent 1 | Hyrum WV | WW Influe | 72121 | none | 0.01 | 229.92 | 1.37 | 6.41 | 8.10 | 1.43 | 11.59 | 1.06 | 1.12 | 0.72 | 0.26 |
| 211188 | 83121 | Influent 2 | Hyrum WV | WW Influe | 72121 | none | 0.01 | 230.89 | 0.66 | 6.06 | 5.72 | 0.05 | 9.23 | 0.98 | 1.64 | 0.69 | 0.81 |
| 211189 | 83121 | Influent 3 | Hyrum WV | WW Influe | 72121 | none | 0.04 | 222.95 | 0.81 | 12.02 | 4.67 | 0.53 | 20.44 | 1.76 | 1.24 | 2.21 | 0.60 |
| 211190 | 83121 | Effluent 1 | Hyrum WV | WW Efflue | 72121 | none | 2.25 | 22.58 | 7.41 | 16.19 | 0.15 | 0.32 | 10.01 | 0.53 | 3.81 | 0.78 | 0.08 |
| 211191 | 83121 | Effluent 2 | Hyrum WV | WW Efflue | 72121 | none | 3.16 | 20.58 | 5.19 | 13.94 | 0.09 | 0.19 | 11.57 | 0.36 | 1.71 | 0.84 | 0.03 |
| 211192 | 83121 | Effluent 3 | Hyrum WV | WW Efflue | 72121 | none | 1.91 | 16.69 | 5.82 | 15.00 | 0.08 | 0.54 | 9.21 | 0.40 | 2.70 | 0.88 | 0.03 |
| 211361 | 83121 | Influent 1 | Hyrum WV | WW Influe | 92521 | none | 0.22 | 201.11 | 0.39 | 7.66 | 8.25 | 2.04 | 15.80 | 1.03 | 2.20 | 1.25 | 0.12 |
| 211362 | 83121 | Influent 2 | Hyrum WV | WW Influe | 92521 | none | 0.16 | 222.71 | 0.23 | 7.56 | 6.90 | 0.07 | 7.61 | 1.08 | 2.19 | 1.22 | 0.17 |
| 211363 | 83121 | Influent 3 | Hyrum WV | WW Influe | 92521 | none | 0.07 | 217.16 | 0.46 | 7.54 | 6.82 | 2.21 | 13.34 | 1.17 | 3.24 | 1.01 | 0.05 |
| 211364 | 83121 | Effluent 1 | Hyrum WV | WW Efflue | 92521 | none | 3.85 | 14.15 | 4.60 | 11.18 | 0.24 | 0.67 | 6.63 | 0.30 | 1.86 | 0.38 | 0.02 |
| 211365 | 83121 | Effluent 2 | Hyrum WV | WW Efflue | 92521 | none | 4.00 | 13.94 | 4.34 | 10.28 | 0.21 | 0.03 | 6.91 | 0.27 | 1.43 | 0.45 | 0.03 |
| 211366 | 83121 | Effluent 3 | Hyrum WV | WW Efflue | 92521 | none | 4.24 | 15.14 | 5.61 | 8.37 | 0.21 | 0.11 | 9.11 | 0.36 | 2.11 | 0.53 | 0.04 |
| 211504 | 103621 | PH1 | PH Contro | Spigot | 102521 | none | 0.0005 | 0.4309 | 0.0333 | 0.0110 | 0.0138 | 0.009 | 0.002 | 0.0300 | 0.0291 | 0.0406 | 0.0287 |
| 211505 | 103621 | PH2 | PH Contro | Spigot | 102521 | none | 0.002 | 0.710 | 0.002 | 0.018 | 0.015 | 0.006 | 11.263 | 0.250 | 0.033 | 0.009 | 0.087 |
| 211506 | 103621 | PH3 | PH Contro | Spigot | 102521 | none | 0.01 | 0.38 | 0.001 | 0.03 | 0.006 | 0.004 | 13.81 | 0.02 | 0.02 | 0.03 | 0.02 |
| 211507 | 80321 | L5 Spigot 2 | L5 Spigot | Spigot | 92521 | none | 2.39 | 14.16 | 3.70 | 10.56 | 0.05 | 0.32 | 240.07 | 0.42 | 1.74 | 0.86 | 0.12 |
| 211508 | 80321 | L5 Spigot 3 | L5 Spigot | Spigot | 92521 | none | 4.10 | 14.96 | 3.62 | 8.45 | 0.11 | 0.41 | 143.30 | 0.31 | 0.43 | 0.54 | 0.10 |
| 211509 | 80321 | L5 Spigot 1 | L5 Spigot | Spigot | 92521 | none | 2.78 | 13.40 | 3.37 | 6.15 | 0.09 | 0.32 | 60.64 | 0.20 | 0.33 | 0.28 | 0.02 |

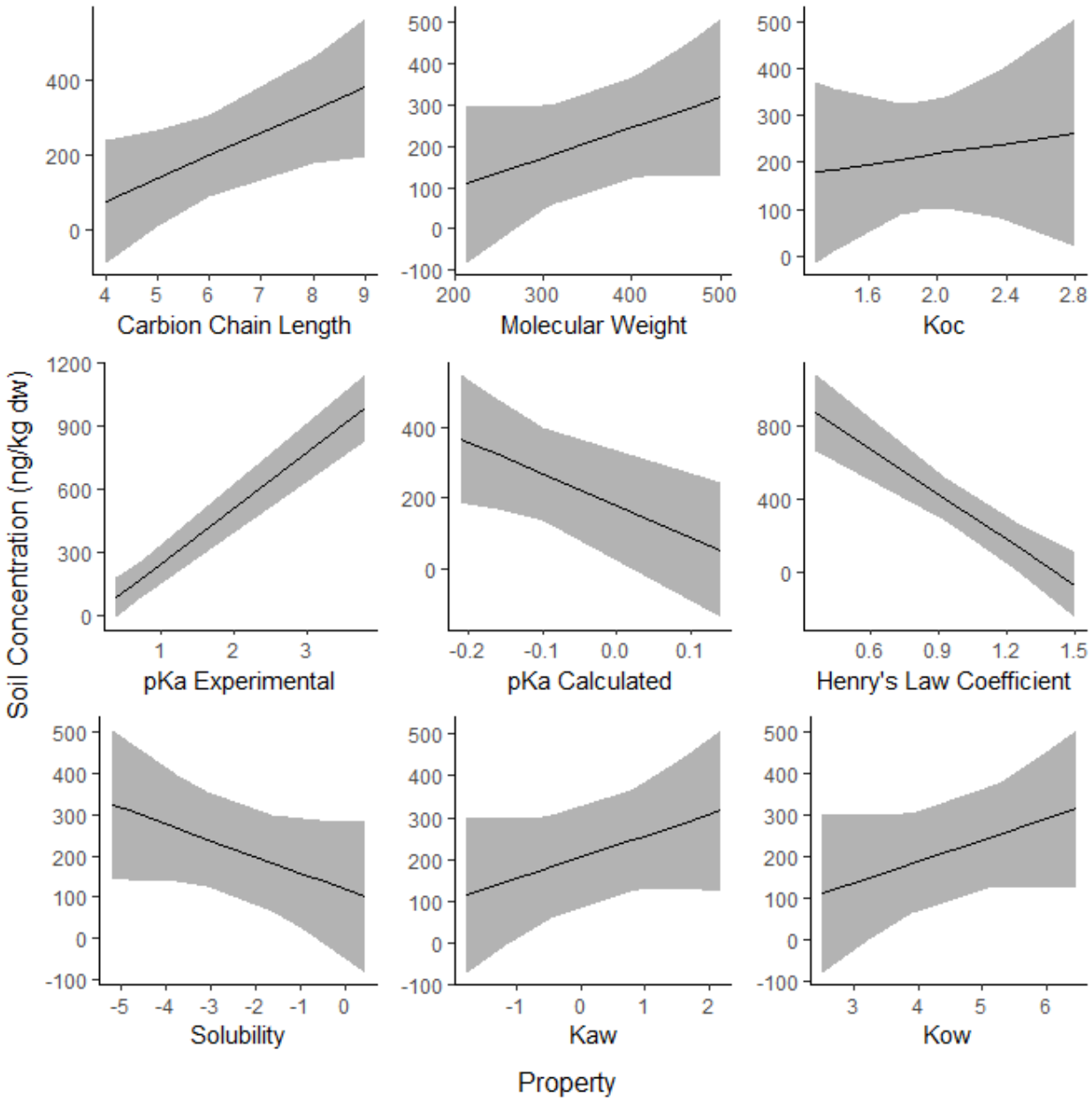


*Figure A13: a single linear model per property and its 95% confidence interval to find correlation between compounds and PFAS accumulation in soil.*

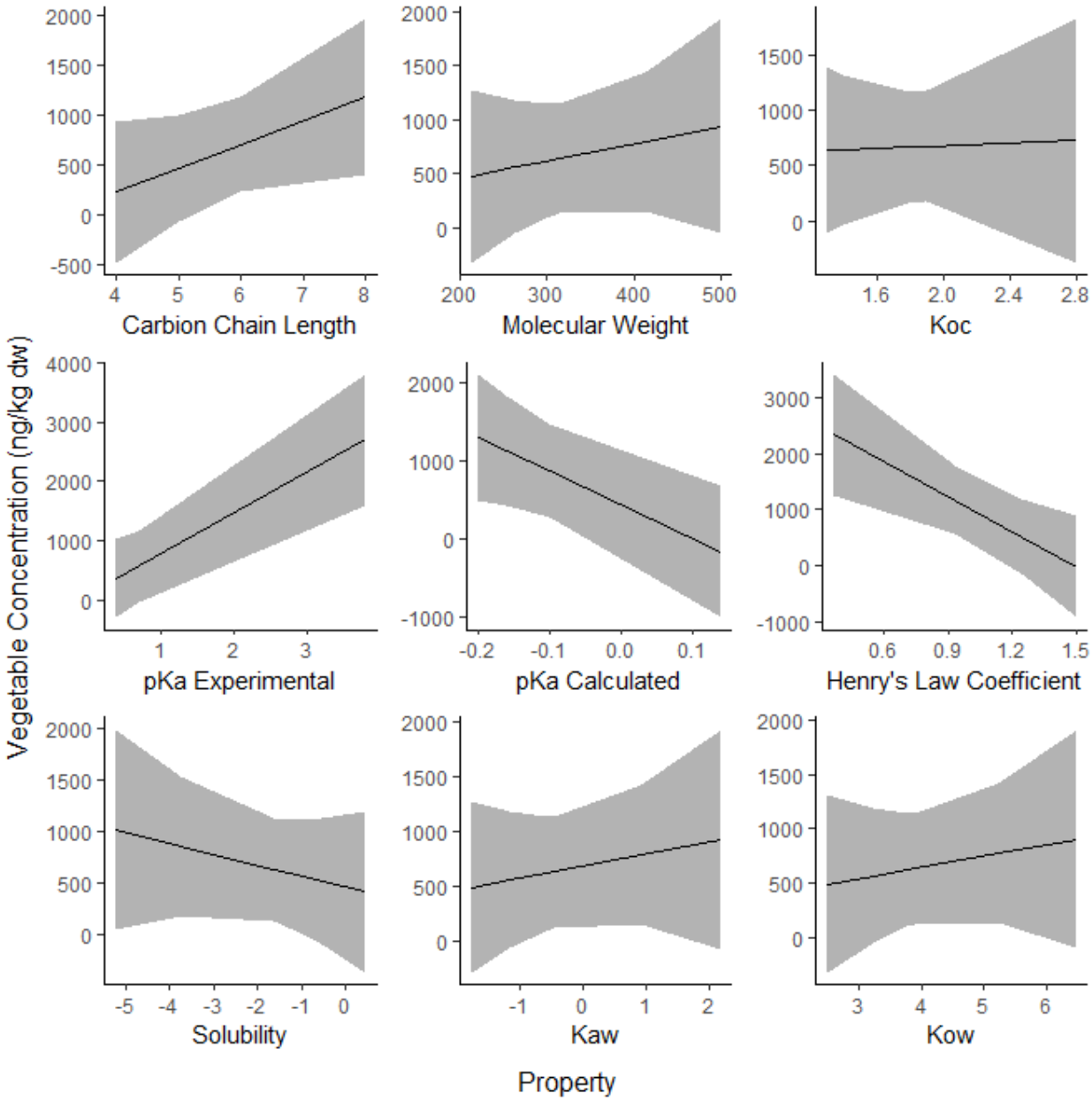


*Figure A14: A single linear model per property and its 95% confidence interval to find correlation between compounds and PFAS accumulation in vegetables.*

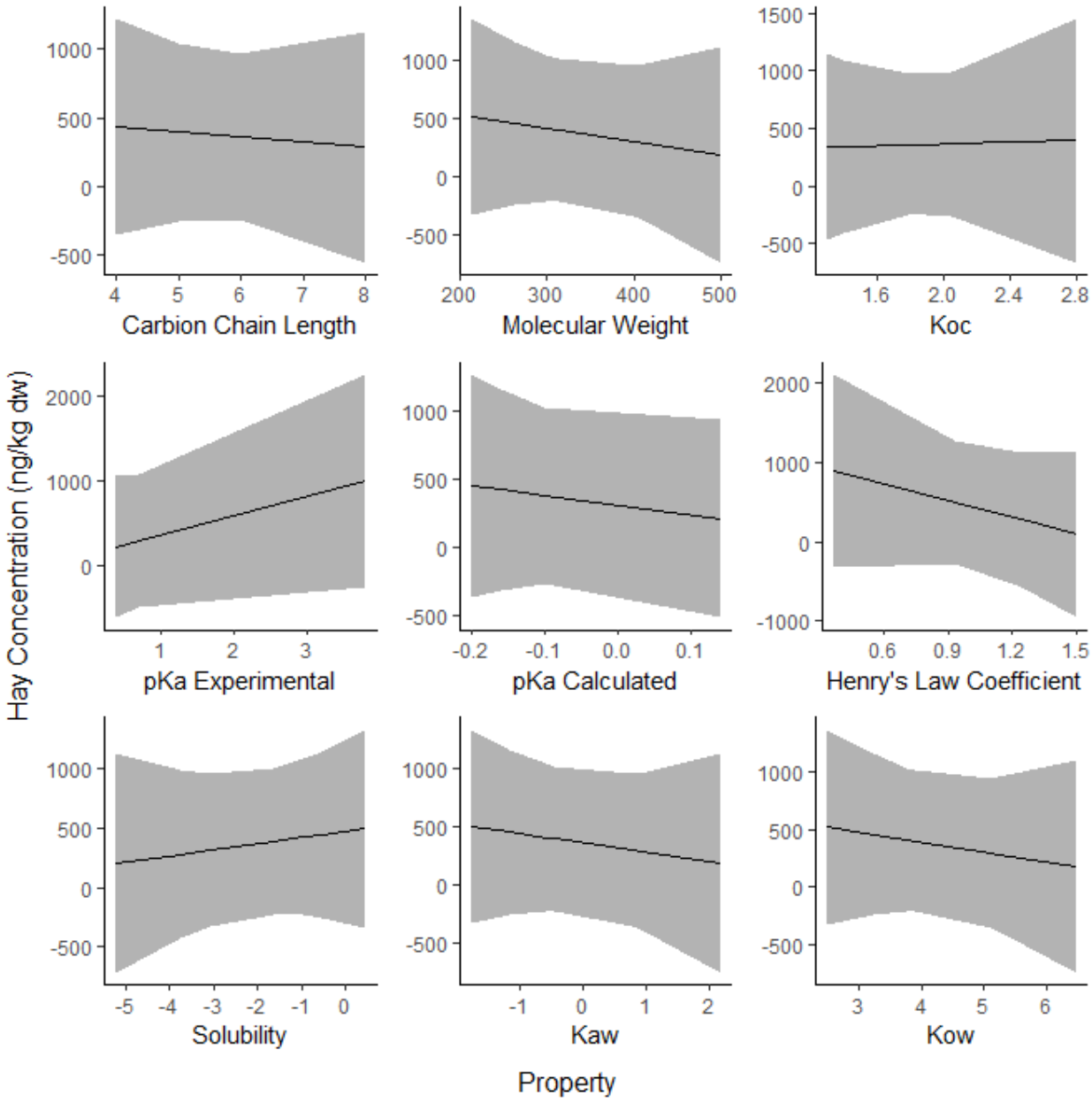


*Figure A15: A single linear model per property and its 95% confidence interval to find correlation between compounds and PFAS accumulation in Hay.*

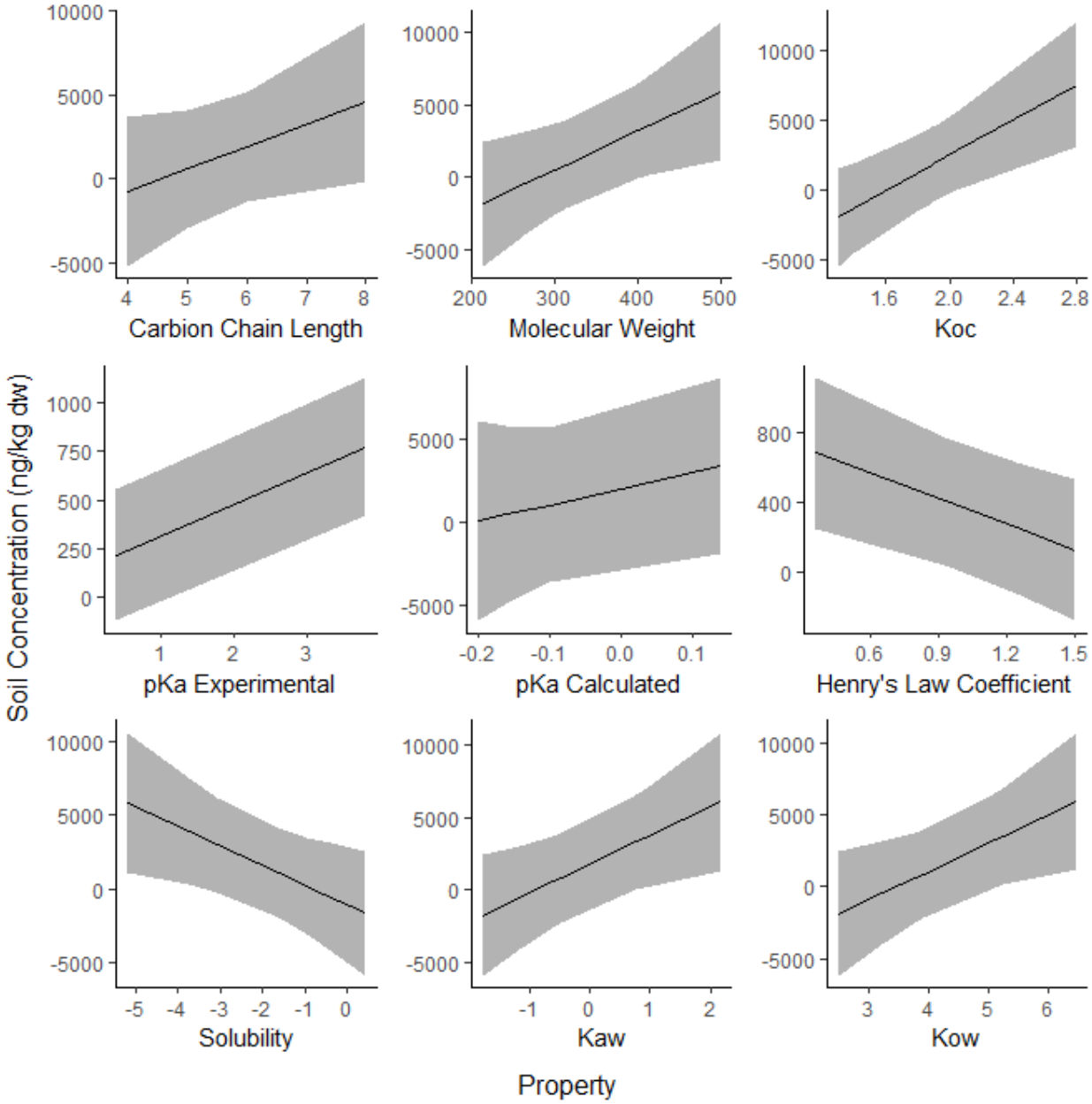


*Figure A16: A single linear model per property and its 95% confidence interval to find correlation between compounds and PFAS accumulation in Hay.*