

Sampling and Analysis Plan for LSL2 Underground Storage Tank Evaluation of PFAS in Soil: Protection of Groundwater

October 2024

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Richland, Washington 99354

Acronyms and Abbreviations

AFFF	Aqueous Film Forming Foam
CLARC	Cleanup Levels and Risk Calculation
COC	Contaminant of Interest
COV	Coefficient of Variation
DL	Detection Limit
DOE	U.S. Department of Energy
DQO	Data Quality Objectives
Ecology	Washington State Department of Ecology
EPA	Environmental Protection Agency
ft	Feet
in.	Inches
LBGR	Lower Bound of the Gray Region
LOD	Limit of Detection
LSL2	Life Sciences Laboratory II Building
MTCA	Model Toxics Control Act
NA	Not Applicable
Not Est.	Not Established
PFAS	Per- and polyfluoroalkyl substances (Note: Table 9 defines the abbreviations for specific PFAS substances)
PFOA	Perfluorooctanoic acid (CAS # 335-67-1)
PNNL	Pacific Northwest National Laboratory
PNSO	U.S. Department of Energy, Office of Science, Pacific Northwest Site Office
PQL	Practical Quantitation Limit
QA	Quality Assurance
QC	Quality Control
RL	Reporting Limit
SAP	Sampling and Analysis Plan
SOPs	Standard Operating Procedures
UBGR	Upper Bound of the Gray Region
UST	Underground Storage Tank
WAC	Washington Administrative Code

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1.0 Introduction

North of the Life Science Laboratory 2 (LSL2) Building on the campus of Pacific Northwest National Laboratory (PNNL), soil sampling associated with an underground storage tank removal indicated the presence of per- and polyfluoroalkyl substances (PFAS) that exceed the Washington State Model Toxics Control Act (MTCA) criteria for Soil Protective of Groundwater-Vadose (WAC 173-340-747). The 10,000-gallon underground storage tank (UST) was part of the Chemical Storage and Transfer Facility for LSL2. This Sampling and Analysis Plan (SAP) discusses the details of the proposed sampling to meet data quality objectives and support decisions for the future actions associated with the pit where the tank had been located and the fate of the staged soil from removal of the tank.

1.1 Content and Organization

This SAP gathers all pertinent information relevant to the sampling and analysis of soil at the removal site for a former 10,000-gallon UST and reference sites in the vicinity of the excavation site. To support these objectives, the document is organized as follows (in accordance with the guidance in EPA 1988):

- Section 1.0, *Introduction*: Provides introductory material on the former tank location, background on the tank, initial soil sampling, and proposed project schedule.
- Section 2.0, *Data Quality Objectives*: Establishes the process and steps for the acquisition of data.
- Section 3.0, *Quality Assurance Project Plan*: Establishes the quality requirements for data collection, including planning, implementation, and assessment of sampling, field measurements, and laboratory analysis.
- Section 4.0, *Field Sampling Plan*: An overview of the field work to be conducted by a subcontract. Their plan will include information related to field activities, sampling design, instrumentation/equipment, field sampling methods, and sample handling.
- Section 5.0, *Health and Safety Plan*: Provides information on health and safety-related procedures during sampling efforts.

1.2 Background on the Underground Storage Tank, R-C-1662

The underground storage tank (UST; R-C-1662) previously located to the north of the LSL2 Building (Figure 1 and Figure 2) was installed when the building was constructed in 1975. The storage tank was originally designed and permitted (WAC 173-360) to contain diesel fuel that fed the LSL2 boilers in conjunction with a second (20,000 gallon) tank that fed the LSL2 generator. Post construction, facility management determined that the generator and boilers could both operate using the 20,000-gallon tank. The 10,000-gallon tank was removed from service in 1986 and the underground storage tank permit with Ecology was cancelled. In 1988, the 10,000-gallon tank and associated piping was repurposed for fire suppression collection (e.g., emergency spill and overflow containment, which is exempted from UST regulation) and was plumbed to receive building fire sprinkler containment water from the Life Science Laboratory 2A (LSL2A), also referred to as the Chemical Storage and Transfer Facility (CSTF). The fire suppression system in LSL2A at that time contained aqueous film forming foam (AFFF).

a known contributor to PFAS contamination. There are no indications that the fire suppression system in LSL2A was ever activated.

In 2015, the piping connecting the 10,000-gallon tank was isolated from LSL2A when the building was refurbished and the AFFF system was removed. Piping from the building to the tank was capped with epoxy, isolating the tank from potential sources. Subsequently, the 10,000-gallon tank and associated infrastructure was converted into a hazardous waste storage area.

In 2022 a sinkhole developed in the LSL2 north parking lot adjacent to the tank, and the area was blocked off. Consideration was given that the tank could have been failing and been a factor in the developing sinkhole. The plan was to remove the tank and repair the parking lot to make it functional again. On October 23, 2023, the tank was removed from below the parking lot.

In preparation for removal of the 10,000-gallon tank, the inside of the tank was observed, sampled and tested. The water in the underground tank was analyzed and was positive for PFAS compounds.

Fulcrum Environmental Consulting, Inc., was contracted to complete a Site Assessment and plan for further soil sampling (Fulcrum 2023a). The assessment included determining if the tank and surrounding soils showed evidence of diesel fuel and chemicals used in fire suppression systems. There was no evidence of a release of diesel fuel from the underground tank system. Field screening did not find evidence of diesel fuel in the surrounding soils. However, soil samples had detectable levels of PFAS compounds above levels for MTCA criteria for Soil Protective of Groundwater–Vadose (WAC 173-340-747), but below levels for MTCA Soil Method B Direct Contact Noncancer.

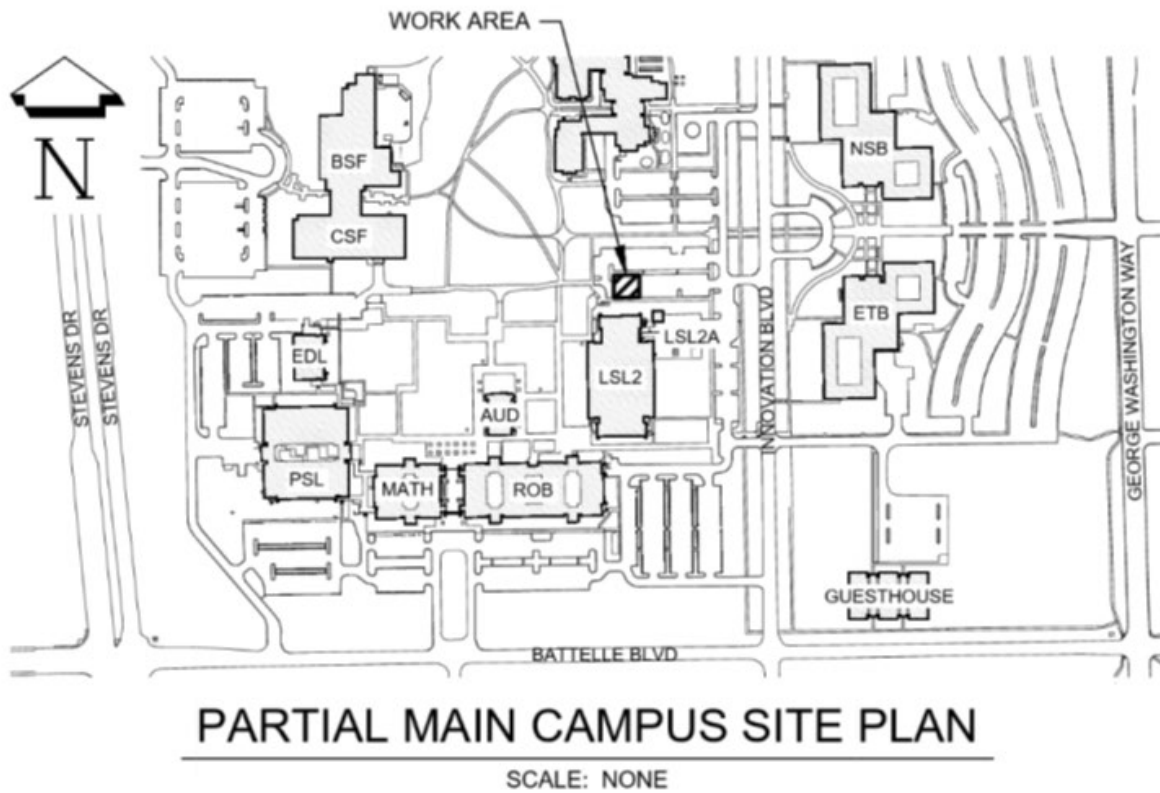


Figure 1. PNNL campus and the area surrounding the location of the 10,000-gallon underground storage tank (Work Area).

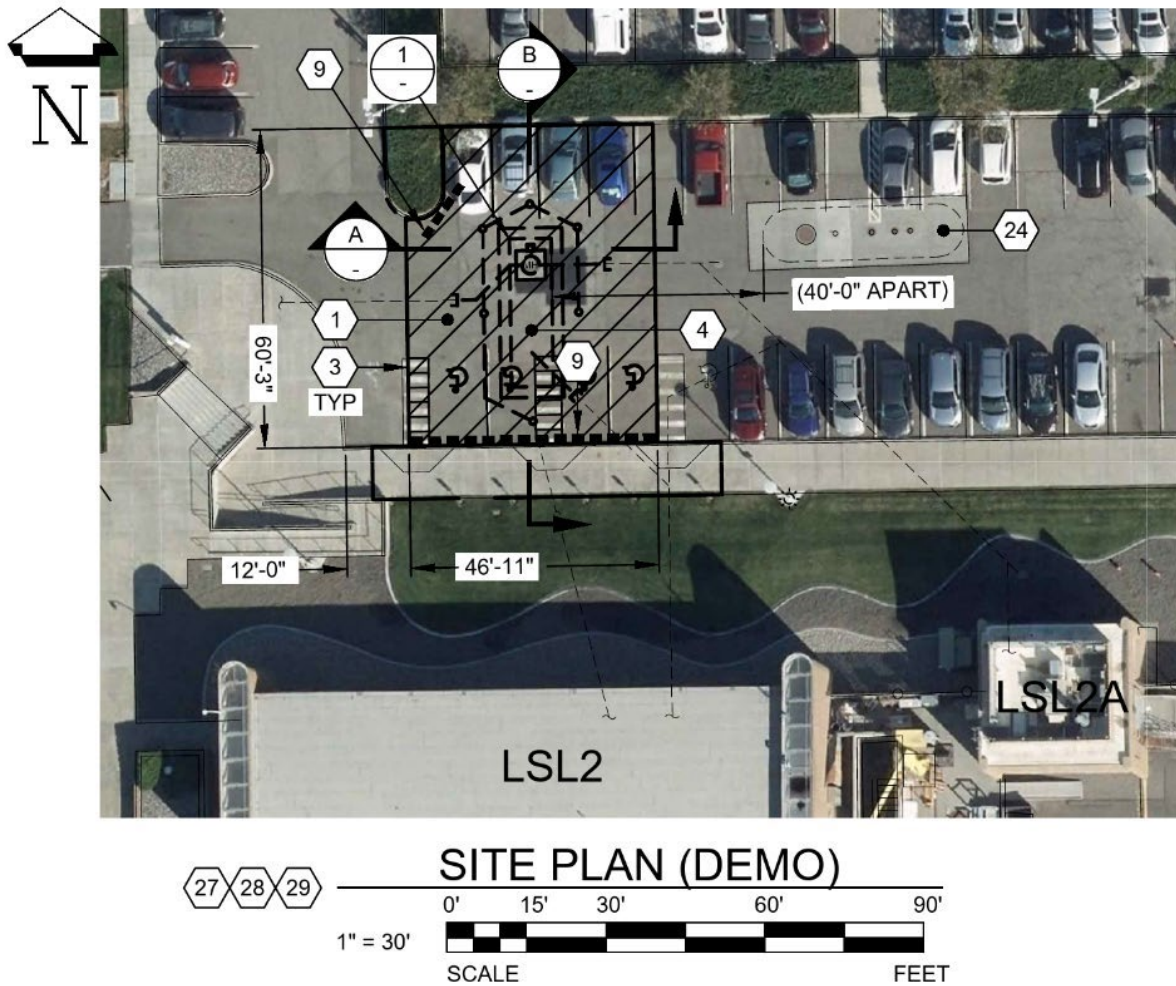


Figure 2. Site plan for the removal of 10,000-gallon underground storage tank.

1.3 Initial Sampling Summary

In early October 2023, the 10,000-gallon tank was excavated, and soil samples were collected during the excavation. Fulcrum collected twelve samples of site soils from excavation extents, below fuel piping, and from the excavated stockpile. Soil samples were collected at the interface between the backfill and native soils. In addition, soil samples were collected from the stockpile of soil removed from the excavation of the tank. Soil sampling was completed consistent with WAC 173-360A.

Laboratory analyses were completed on soil samples using draft EPA Method 1633. The soil samples from the Tank Pit and the stockpile of soil were completed in two different batches, resulting in some differences in the reporting limits for the PFAS substances. Sampling results are discussed further in Section 2.1.3.

2.0 Data Quality Objectives

This section defines the Data Quality Objectives (DQOs) for the collection of soil samples associated with an underground storage tank removal that indicated the presence of PFAS compounds that exceed the MTCA criteria for Soil Protective of Groundwater–Vadose (WAC 173-340-747) using the seven steps of the DQO process that are described in U.S. Environmental Protection Agency (EPA) QA/G-4, *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA 2006). It is formatted as a DQO worksheet rather than a typical DQO report for brevity.

The use of the DQO process assures the collection of the type, quantity, and quality of data needed to reach defensible decisions or make credible estimates for the presence of PFAS in the soil around the excavation site (hereto forward called Tank Pit), in the stockpiled excavated soil (hereto forward called Stockpile), and in reference areas in the vicinity of the former tank (hereto forward called Reference Areas).

2.1 Step 1: State the problem

2.1.1 Are the members of the planning team identified?

Name	Affiliation	Role
Mike Stephenson	PNNL	Decision/Risk
Dan Edwards	PNNL	Environmental Compliance
Deb Fagan	PNNL	DQO support/statistics
Amoret Bunn	PNNL	Environmental Engineer
John Wilson	PNNL	Visual Sample Plan Subject Matter Expert
Clara Stickney	PNNL	Summer intern
Minsuh Chun	PNNL	Summer intern
Abby Friedman	DOE PNSO	Project Manager
Jennifer Lind	WA State Dept of Ecology	Stakeholder

2.1.2 Who are the primary decision makers?

U.S. Department of Energy, Office of Science, Pacific Northwest Site Office (PNSO) and Washington State Department of Ecology

2.1.3 What is the conceptual site model and what is a concise description of the problem? Site history and summary of the current problem

- A 10,000-gallon tank below the parking lot north of building LSL2 was installed when LSL2 was constructed in 1975 and was originally designed and permitted (WAC 173-360) to contain diesel fuel that fed the LSL2 boilers in conjunction with a second (20,000 gallon) tank that fed the LSL2 generator. Figure 1 shows the location of the tank within the PNNL campus.
 - From past schematic drawings of the 10,000-gallon tank, the estimated dimensions of the tank are 95 in. (7.9 ft) diameter and 28 ft length (R-M-1217 1989).
 - Depth below surface was 6 ft. and the depth below the tank was 14 ft (R-M-1217 1989).
- In 1986, the tank was removed from service and the underground storage tank permit with the Washington State Department of Ecology was cancelled.

- In 1988, the tank and associated piping were repurposed for fire suppression collection (e.g., emergency spill and overflow containment, which is exempted from UST regulation) and was plumbed to receive building fire sprinkler containment water from the Life Science Laboratory 2A (LSL2A), also referred to as the Chemical Storage and Transfer Facility.
- In 2015, the piping connecting the tank was isolated from LSL2A when the building was refurbished and the AFFF system was removed. The structure was converted into a hazardous waste storage area.
- In July 2022 a sinkhole began forming over the tank (see Figure 2) and in June 2023 the decision was made to remove the tank. It is clear from the picture in Figure 3 that runoff from the parking lot was entering the soil beneath the parking lot as a result of the sinkhole (and probably other cracks in the parking lot).
- Tank removal activities began October 2023 (Fulcrum 2023a).
- Fulcrum completed a site assessment in November of 2023 (Fulcrum 2023b). The site assessment cited the Washington State Department of Ecology guidance, *Site Assessment Guidance for Underground Storage Tank Systems* (Ecology 2022), as an appropriate best practice.



Figure 3. The location in the parking lot where the tank and tank access covers re located, facing north, taken July 2022.

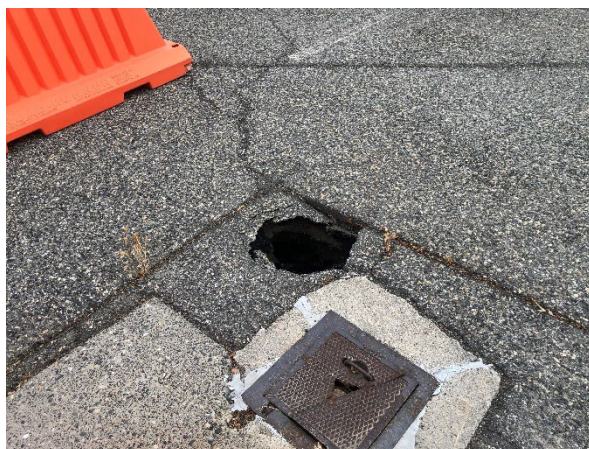


Figure 4. The sinkhole is forming near the northwest corner of the tank access cover, within the area demarcated by safety cones. This photo was taken in June 2023. Subsidence since 2022 is evident.

- Historical document research did not reveal any information that AFFF was ever stored in the tank (Battelle 1988).
- In 2022, a dip test indicated that 14 inches of liquid were present in the tank.
- A dip test in October 2023 showed that 15.1 inches of liquid were present in the tank (see Figure 5). The liquid was removed and tested prior to tank excavation. Tank liquid was sampled and analyzed for VOCs, petroleum products, metals and PFAS. PFAS above the limit of detection were present (Fulcrum 2023a).



Figure 5. Water is being removed from the tank prior to excavation.

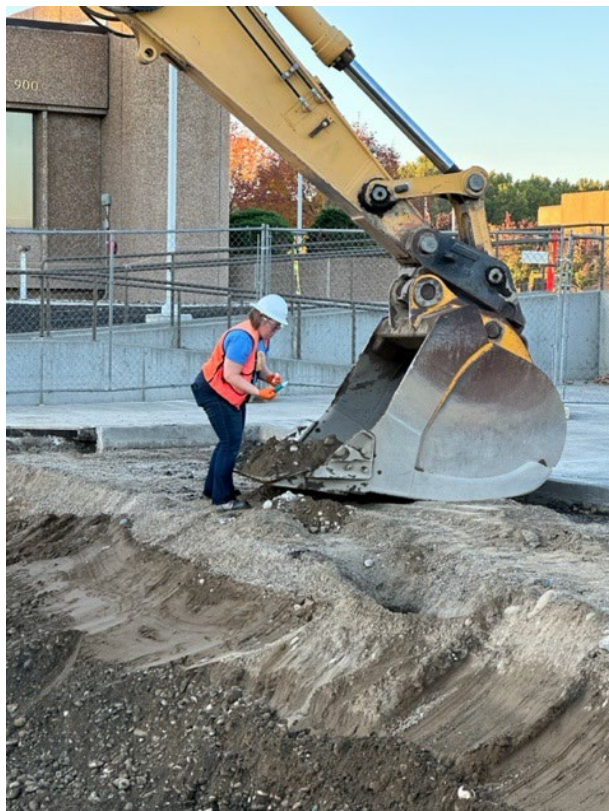


Figure 6. Soil is being sampled from a bucket of excavated material.

- While the tank was being removed, twelve soil samples were collected from soils surrounding the tank (see Figure 6). Samples were analyzed for petroleum hydrocarbons and PFAS. Figure 7 shows the location of soil samples in context of the excavation area. Note, the sample numbers indicate the sequence and depth of sampling. For example, 07.14 was the seventh sample collected at 14 ft below grade. The sample collected at 14 ft was the deepest sample, located under the tank and was collected after the tank was removed.

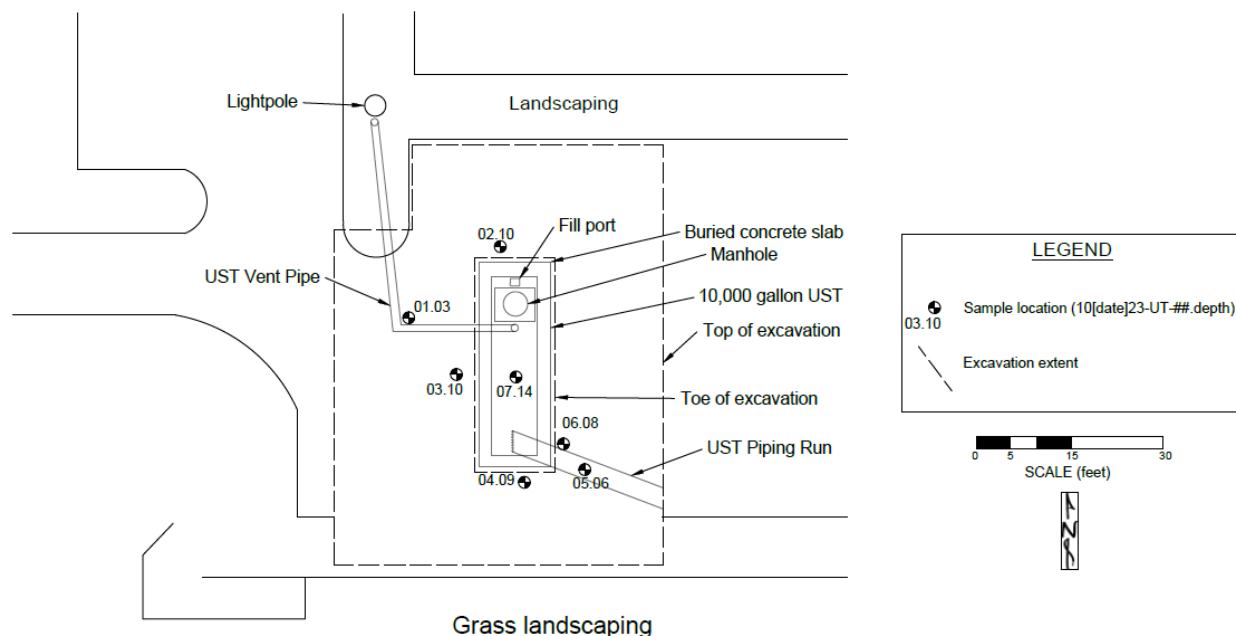


Figure 7. Schematic of the excavation area (from Fulcrum 2023b).

- Per Fulcrum, 2023b:
 - Heavy oil range hydrocarbons were detected in 10 of the 12 samples, including 5 samples from the staged pile (this does not include the duplicate sample collected). Detected concentrations in the soils collected at the site range between 22.6 mg/kg to 25.3 mg/kg. The MTCA A clean up level for heavy oil range hydrocarbons is 2000 mg/kg. Detected concentrations from the staged pile range between 30.7 mg/kg to 247 mg/kg.
 - One sample collected from the staged pile showed a naphthalene concentration of 0.0397 mg/kg. The MTCA A cleanup value is 5 mg/kg.
 - No samples had values that exceeded detection limits for diesel range organics, benzene, toluene, ethylbenzene, or xylene.
 - Samples were analyzed for 64 PFAS using Draft EPA Method 1633:
 - PFAS compounds were detected in 12 out of 13 samples. Detected compounds include perfluorobutanoic acid (PFBA), perfluorononanoic acid (PNFA), perfluorooctane sulfonic acid (PFOS), perfluorooctanoic acid (PFOA), perfluoroheptanoic acid (PFHpA), perfluoro-3-methoxypropanoic acid (PFMPA), and perfluorodecanoic acid (PFDA). Table 1 summarizes the results, with "<" indicating a non-detect and the associated detection limit reported. [Note, the information on cleanup levels has been superseded by more recent guidance from Ecology (2024) and EPA (89 FR 32532).]

Figure 8 shows the data in boxplots for both untransformed and log transformed values. The log transformed values are negative because the log of a number less than one is negative. The log transformed boxplots are helpful because they tend to show the structure of the data when untransformed data are highly skewed. Tank Pit and Stockpile samples are differentiated by color, while the detect status is differentiated by symbol.

Table 1. Historical data sorted by depth. Units are in µg/kg.

Sample ID	Location	Depth (ft)	Matrix	PFBA ¹	PFNA ¹	PFOS ¹	PFOA ¹	PFHpA ²	PFMPA ²	PFDA ²	PFBS ¹	PFHxS ^{1,3}	PFHxA ¹
101823-UT-01.03	UST vent pipe, beneath elbow	3	Near tank	<0.14	<0.044	0.14	0.15	0.042	<0.042	<0.038	<0.027	ND	<0.077
101923-UT-05.06	UST piping run, center	6	Near tank	0.91	<0.044	<0.052	0.067	<0.030	0.075	<0.039	<0.027	ND	<0.078
102323-UT-06-08	East sidewall, north of piping run	8	Near tank	<0.14	<0.044	<0.051	0.04	<0.030	<0.042	<0.038	<0.027	<0.033	<0.078
101923-UT-04.09	South sidewall, center	9	Near tank	<0.14	<0.044	<0.051	0.083	<0.030	<0.042	<0.038	<0.027	ND	<0.077
101923-UT-13.09	South sidewall, center	9	Dup. of 04.09	<0.14	<0.044	<0.051	0.083	<0.030	<0.042	<0.038	<0.027	ND	<0.077
101923-UT-02-10	North sidewall	10	Near tank	<0.14	<0.044	<0.051	<0.036	0.061	<0.041	<0.038	<0.027	ND	<0.077
101923-UT-03.10	West sidewall, center	10	Near tank	<0.14	0.12	0.33	0.12	<0.030	<0.042	<0.039	<0.027	ND	<0.078
102323-UT-07-14	Pit bottom, center	14	Near tank	<0.14	<0.044	<0.052	<0.036	<0.029	<0.041	<0.038	<0.027	<0.033	<0.077
102323-UT-08	NA	NA	Stockpile	<0.14	<0.044	0.1	0.059	0.041	<0.041	<0.038	<0.027	<0.032	<0.077
102323-UT-09	NA	NA	Stockpile	<0.14	<0.044	0.12	0.073	0.034	<0.042	<0.038	<0.027	<0.033	<0.078
102323-UT-10	NA	NA	Stockpile	<0.14	<0.044	0.11	0.064	0.038	<0.042	<0.039	<0.027	<0.033	<0.077
102323-UT-11	NA	NA	Stockpile	<0.14	<0.044	0.072	0.059	0.038	<0.041	0.045	<0.027	<0.033	<0.077
102323-UT-12	NA	NA	Stockpile	0.3	<0.044	0.067	<0.036	<0.029	<0.041	<0.038	<0.027	<0.033	<0.077

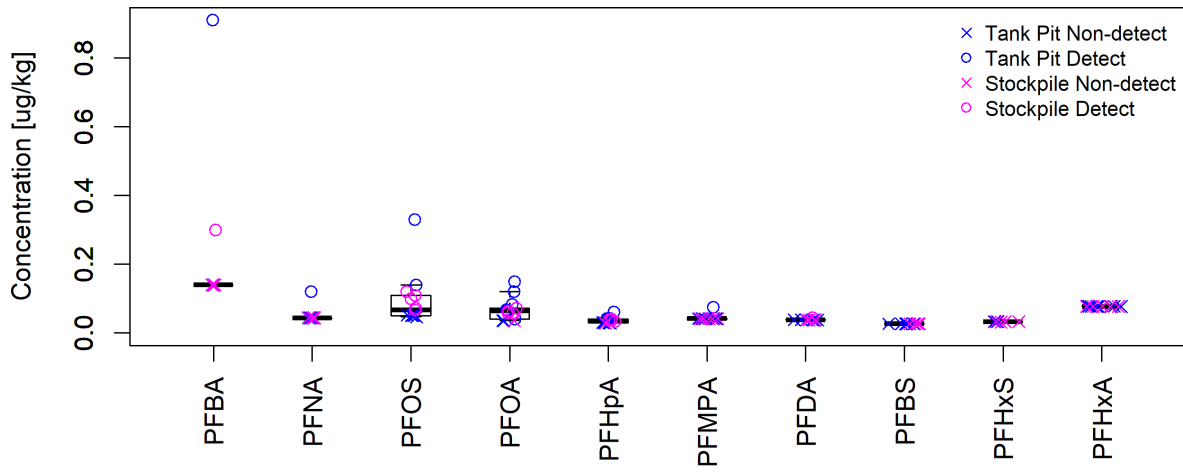
¹ MTCA CLARC value established for soil concentrations protective of groundwater (June 2024).

² No established MTCA CLARC value for soil concentrations protective of groundwater (June 2024).

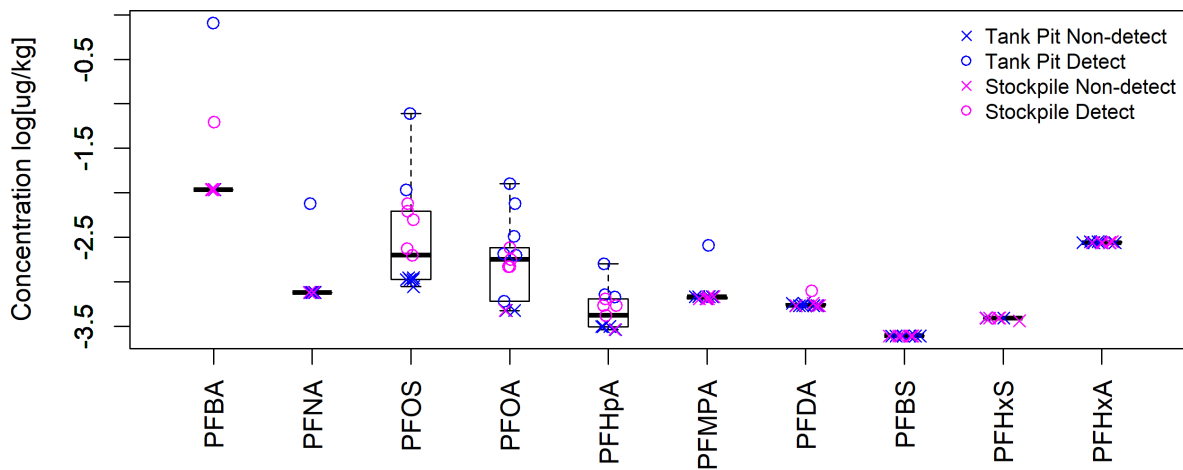
³ Historical samples were analyzed in different batches, resulting in some values having a report limit (RL), and some without.

ND = Not Detected, no detection limit provided in analytical report for these batch of samples (Fulcrum 2023b).

Blue values are the analyses from the Tank Pit that are greater than the detection limit (DL) of the analytical method (Fulcrum 2023b).



(a) Units are ug/kg



(b) Units are log(ug/kg)

Figure 8. Boxplots of the PFAS compounds from Table 1. Panel (a) shows untransformed values; panel (b) shows log transformed values. Non-detects are shown at the detection limits.

- Updates on regulations related to analysis and compliance with PFAS substances:
 - EPA Method 1633 (EPA 2024) was finalized January 2024. Fulcrum 2023a used the draft version of Method 1633 for the samples collected in October 2023.
 - EPA published the final rule for *PFAS National Primary Drinking Water Regulation* on April 26, 2023 (89 FR 32532).
 - MTCA updated CLARC in July 2024 to incorporate EPA’s final rule on for PFAS drinking water regulations, and as a result, the individual PFAS substances cleanup criteria for soil protective of groundwater changed compared to the levels considered in the Fulcrum site assessment (Fulcrum 2023b).
 - Table 2 provides a review of existing information in 2023 (Ecology 2023) and current CLARC cleanup values (Ecology 2024), in comparison to the analytical methods of the

historical samples (Fulcrum 2023b). This shows how updates in regulations require the laboratory analyzing the samples to have a lower reporting limit. Approaches for using the historical samples require the reporting limit to be considered where the laboratory reported “ND” or not detected.

- Because the location of the tank is beneath a parking area where vehicles and machinery operate daily, and where runoff from storm and irrigation water may traverse, and because there is no documented evidence that the tank leaked, there is some question as to whether the levels of PFAS compounds found in the historical samples are related to the tank.
- Because PFAS contamination is ubiquitous in the environment (Ecology 2023), particularly in areas where humans have had a large presence, the possibility exists that anthropogenic levels might exceed cleanup values.

Table 2. Analytical information from historical sampling for PFAS compounds and regulatory guidance on cleanup levels.

Abbreviation	CAS #	Fulcrum Laboratory Analyte LOD ¹ (µg/kg)	Fulcrum Laboratory Analyte DL ¹ (µg/kg)	Fulcrum Laboratory Analyte RL ² (µg/kg)	MTCA Soil Concentrations Protective of Potable Groundwater ³ (µg/kg)	MTCA Soil Concentrations Protective of Groundwater Vadose @ 13°C (Eq. 747-1) ⁴ (µg/kg)
PFBA	375-22-4	0.4	0.1436	0.14	Not Est.	44
PFNA	375-95-1	0.1	0.0441	0.044	0.080	0.089
PFOS	1763-23-1	0.15	0.0516	0.047-0.052	0.170	0.046
PFOA	335-67-1	0.1	0.0362	0.036	0.063	0.025
PFHpA	375-85-9	0.1	0.0296	0.029-0.03	NA	NA
PFMPA	377-73-1	0.2	0.0417	0.041-0.042	NA	NA
PFDA	335-76-2	0.1	0.0384	0.038-0.039	NA	NA
PFBS	375-73-5	0.1	0.0269	0.027	1.800	25
PFHxS	355-46-4	0.1	0.0328	ND-0.033	0.410	0.062
PFHxA	307-24-4	0.16	0.0775	0.077-0.078	Not Est.	35

LOD = Limit of Detection, identified in Site Assessment report (Fulcrum 2023a).

DL = Detection Limit, identified in Site Assessment report (Fulcrum 2023a).

RL = Reporting Level, as reported by Pace Analytical Services, LLC (Fulcrum 2023b).

Not Est. = Not established (Ecology 2023)

NA = Not applicable (Ecology 2023, 2024)

¹ Reference is Fulcrum 2023a

² Reference is Fulcrum 2023b

³ Reference is Table 5, Ecology 2023

⁴ Reference is Ecology 2024

Red values are levels where historical samples (Fulcrum 2023; Table 1) exceeded MTCA 2023 levels (Ecology 2023).

Blue values indicate decreases in MTCA CLARC values published in June 2024 that are now below DL reported by Fulcrum 2023b.

2.2 Step 2: Identify the decision

2.2.1 What are the principal study questions?

Do the observed levels of PFAS in soil around the tank indicate that groundwater may be impacted?

2.2.2 What are the alternative actions that could result from resolution of the principal study questions?

Figure 9 identifies decisions and alternatives related to the principal study question and a detailed discussion follows here.

The appropriate statistical comparisons between the observed values and the soil concentrations that are protective of groundwater (WAC 173-340-474) depends upon the statistical distribution of the population of PFAS values (see Box 1 in Figure 9), which will be estimated using the ProUCL software (EPA 2022) with observed values from field sampling. The statistical comparisons will be based on WAC 173-340-740(7), Compliance Monitoring, which has several criteria against which the measurements shall be evaluated. As noted with Box 2 of Figure 9, field observations will be compared against are Cleanup Levels and Risk Calculation (CLARC) values, specifically the MTCA soil concentrations protective of groundwater Vadose @ 13°C (Table 2)(Ecology 2024).

If there is an indication that groundwater may be impacted, a series of follow-up questions result (leading to Box 3 of Figure 9. If there is no indication that groundwater may be impacted, then PNNL will proceed with the MTCA Soil Method B Direct Contact Noncancer cleanup values (Ecology 2024).

If the soil concentrations exceed the CLARC values for protection of groundwater, then PNNL will prepare site-specific cleanup values using the equations in WAC 173-340-747 (Box 3 in Figure 9). This will include using available information on PFAS distribution coefficients (K_d) and Henry's law constants from available literature. Data for the Hanford formation will be used for the other geological media properties in Equation 474-1, e.g., water-filled soil porosity (θ_w), air-filled soil porosity (θ_a), and dry soil bulk density (ρ_b). Past evaluations of the PNNL campus indicate that the subsurface in the region of the LSL2 building is within the Hanford formation (Figure 10; Freedman et al. 2010).

Site-specific cleanup values for soil protective of groundwater will then be compared (Box 4 in Figure 9) to the upper one sided 95% confidence limit for the true mean of PFAS concentrations in Tank Pit soil (WAC 173-340-740(7)(d)(i)(A), or as appropriate for the distribution of the field soil sample results). If the upper 95% confidence interval for the means based on the observed values are less than the site-specific cleanup values, then groundwater is protected and PNNL will proceed to use the MTCA Soil Method B Direct Contact Noncancer cleanup values (Ecology 2024). If the results are greater than the site-specific values, then PNNL will develop a 1-D advection/dispersion transport model.

The 1-D advection/dispersion transport model will provide further evidence as to whether PFAS compounds found in the Tank Pit soil are likely to transport to groundwater (Box 5 in Figure 9). The outcome of the model is to determine the breakthrough for the concentrations of PFAS substances at the water table (i.e., time-varying concentrations of PFAS at the water table). The groundwater/vadose zone interface is approximately 45 ft below ground surface (Figure 10; Freedman et al. 2010). Only PFAS substances that were evaluated in the historical samples will be evaluated with the new samples collected as a result of this DQO and SAP. Only the results of samples taken in the Tank Pit will be considered for evaluation with the 1-D

advection/dispersion transport model because we cannot associate stockpile samples (Table 1) with any particular location or depth within the Tank Pit. Model parameterization will involve many of the parameters associated with equations in WAC 173-340-747 and discussed in the setting of PFAS soil concentrations in Ecology 2023. As stated in Ecology 2023, WAC-173-340-747 does not account for some of the unique transport characteristics of PFAS, and the modeling will address some of these aspects based on the latest available information. By using a model, e.g., STOMP (Subsurface Transport Over Multiple Phases; <https://www.pnnl.gov/projects/stomp>), stochastic simulations will provide the likelihood of breakthrough based on the range of information still being developed for PFAS substances, e.g., K_d . Initial conditions will include the presence of an asphalt parking lot and estimates of reduction of water infiltration by that barrier. Over time, the transport model can consider increased infiltration based on parking lot deterioration or removal.

The outcome of the 1-D advection/dispersion transport model will provide the times at which the concentrations of individual PFAS substances are likely to exceed the concentrations for protection of groundwater (Box 6 in Figure 9). Because the model is stochastic, the results will provide a range of breakthrough times within the timeframe of institutional controls. The outcome of the model, along with additional samples and site-specific evaluations using Equation 747-1, will provide information to determine if the PFAS substances will impact groundwater in the future. If the conclusion is that groundwater is not likely to be impacted, PNNL will proceed with the MTCA Soil Method B Direct Contact Noncancer cleanup values (Ecology 2024). If the results indicate that groundwater may be impacted, actions are yet to be determined.

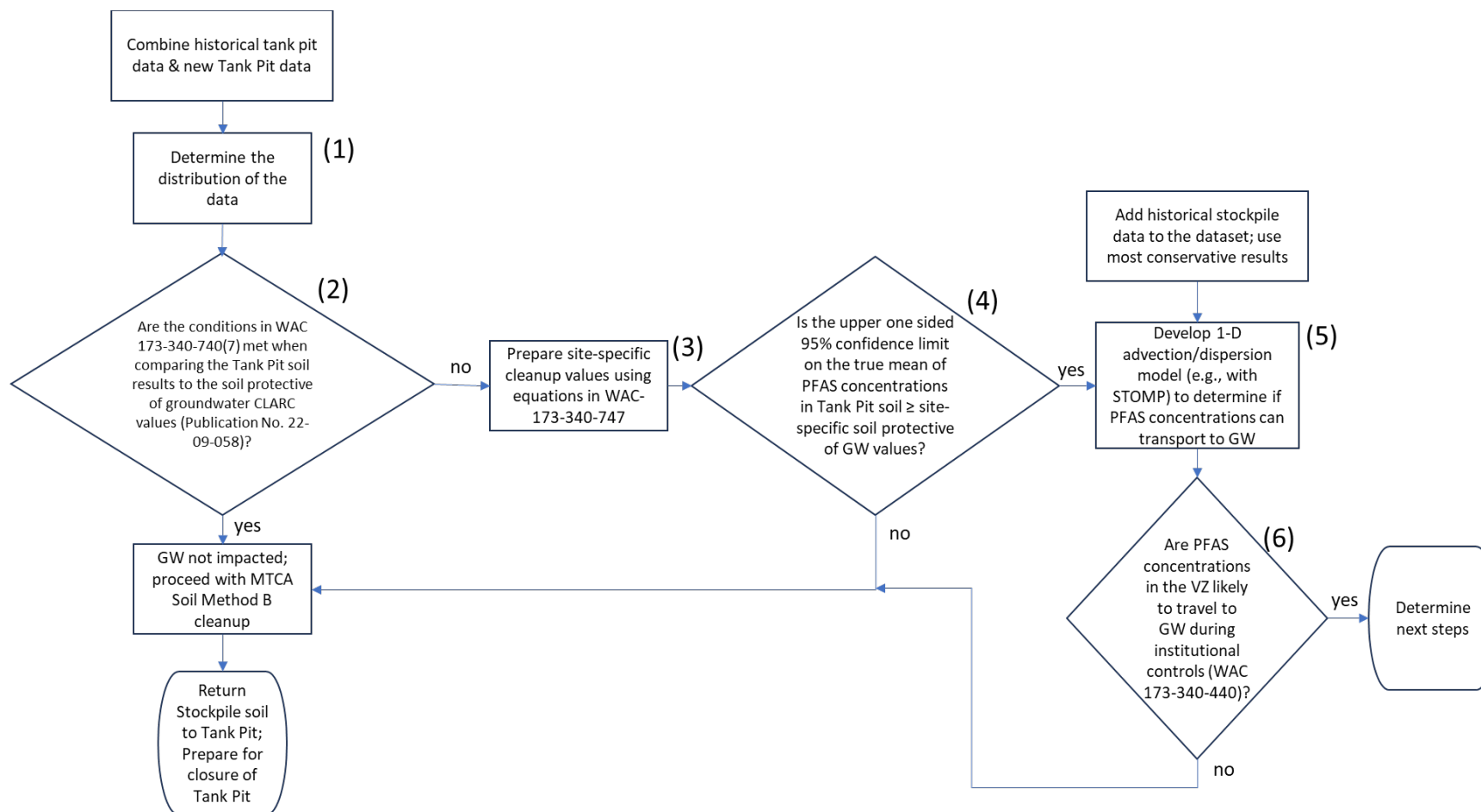


Figure 9. Decision flow diagram with major questions and alternative actions. Numbered shapes are discussed in Section 2.2.2.

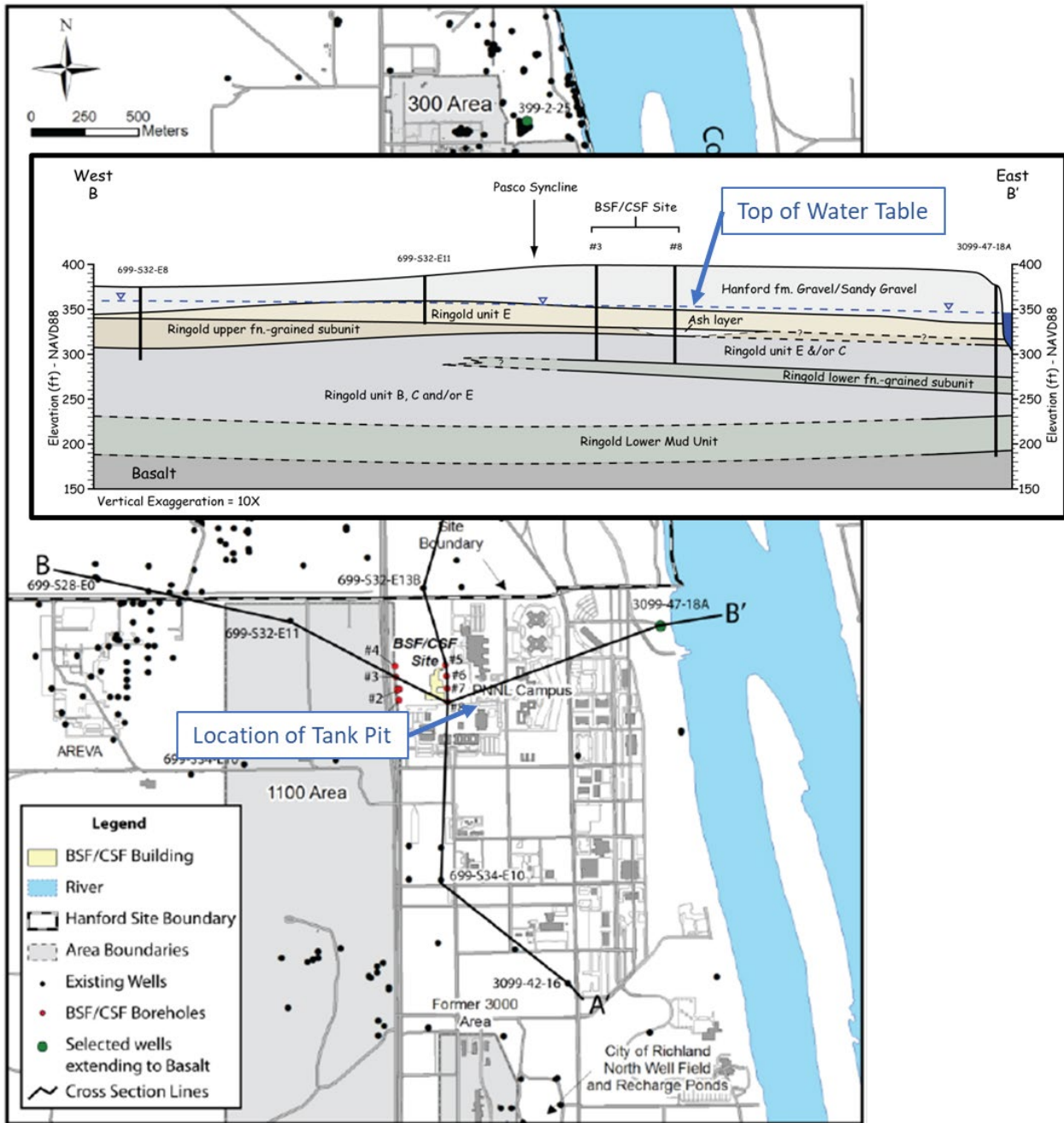


Figure 10. Geologic cross section in the vicinity of LSL2, from West to East (B-B'), showing the vadose zone (dominated by the Hanford formation) and the depth to groundwater (~45 ft below ground surface)(Freedman et al. 2020).

Step 3: Identify the Inputs to the Decision

2.2.3 What is the information required to resolve the study question?

Historical data with detection limits, historical documentation, and area maps with locations of underground infrastructure document the history of the site, provide a geographical frame of

reference, and show the locations of historical samples relative to the tank and piping locations. Relevant WAC codes and publications that contain cleanup values and equations for deriving site-specific values are also required.

A schematic of the excavation area (from Fulcrum 2023b) is shown in Figure 11, where the red circles indicate at least one PFAS compound detected above CLARC values (Ecology 2024), and green circles indicate that all PFAS compounds are below the detection limit. The historical sample collected at 14 ft was collected after the tank was removed from the excavation pit (from below the tank). Samples at 10 ft are approximately at the midpoint of the tank's diameter. Note that the detected values that exceed CLARC values are located to the west and south of the tank location and that the non-detects are located directly below as well as north of the tank location. Table 1 shows the data associated with Figure 10, with non-detects reported with a "<" and the detection limit (Fulcrum, 2023b).

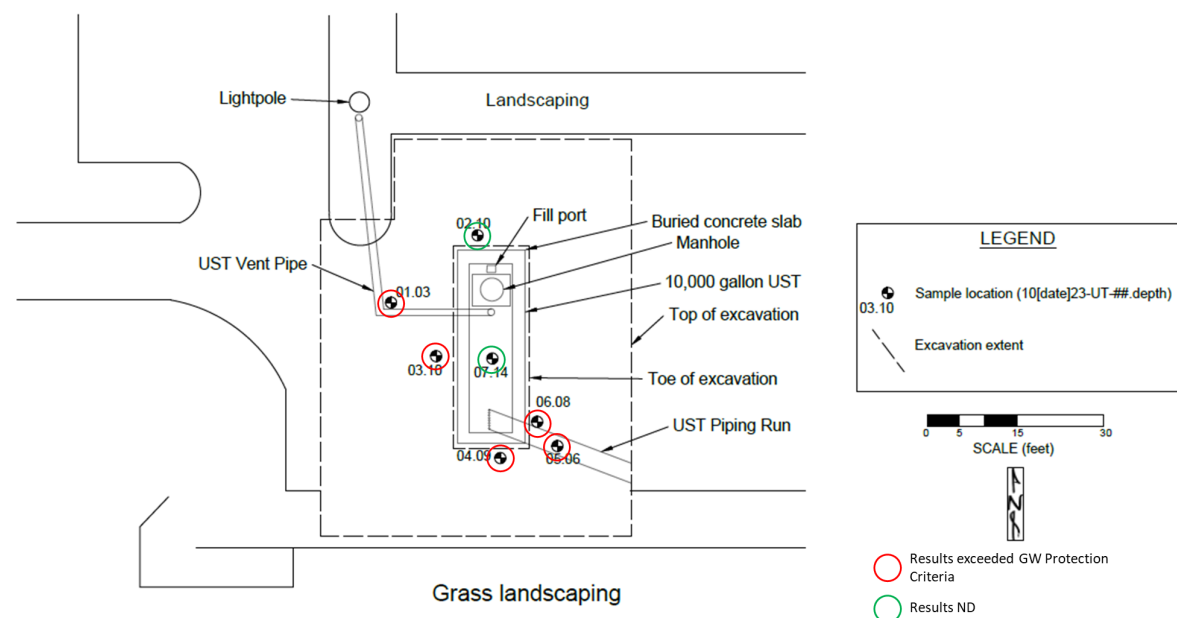


Figure 11. Schematic of the tank site from Fulcrum 2023b, with sample locations color coded by whether historical data exceed CLARC values or whether all values are non-detects.

2.2.4 What are the sources for each item of information?

Site history, documentation of the tank removal, and historical site data are from Battelle 1988, Fulcrum 2023a, and Fulcrum 2023b. Relevant regulations are downloaded from the Washington State Department of Ecology websites as described in Section 2.3.3.

2.2.5 What information is needed to establish the action level?

Washington State Department of Ecology has determined that PFAS fall under the Model Toxics Control Act (MTCA). The regulations are listed below, and cleanup levels are shown in Table 3.

- WAC 173-340-440 sets the measures undertaken to limit or prohibit activities that may interfere with the integrity of an interim action or cleanup action or that may result in exposure to hazardous substances at a site.

- WAC 173-340-740 sets unrestricted land use cleanup levels for soil, including MTCA Method B. Also, WAC 173-340-740(7) identifies how to conduct compliance monitoring is with soil cleanup levels.
- WAC 173-340-745 sets soil cleanup levels for industrial properties (MTCA C).
- WAC 173-340-747 sets cleanup levels for groundwater protection.

Additional requirements for establishing MTCA C cleanup levels are whether the site meets the definition of an industrial property under MTCA Cleanup Regulations (WAC 173-340-745(1)(a)(i)) and whether institutional controls are in place.

Table 3. MTCA Maximum Values from CLARC (Ecology 2024).

Source	PFAS Substances (units are in µg/kg)										
	PFHpA	PFOS	PFOA	PFNA	PFBA	PFMPA	PFBS	PFDA	PFHxS	PFHxA	PFPeA
MTCA Soil Method C Noncancer – Industrial	Not Est.	11000	11000	8800	3500000	NA	1100000	NA	34000	1800000	NA
MTCA Soil Method B Direct Contact Noncancer	Not Est.	240	240	200	80000	NA	NA	NA	NA	40000	NA
MTCA Soil Protective of Groundwater - Vadose	Not Est.	0.046	0.025	0.089	44	NA	25	NA	0.062	35	NA
WA Board of Health Drinking Water State Action Levels	NA	0.015	0.01	0.009	NA	NA	0.345	NA	0.065	NA	NA

Not Est. = Not established (Ecology 2023)
 NA = Not applicable (Ecology 2023, 2024)

2.2.6 Are the appropriate measurement methods available to provide the necessary data?

Soils will be analyzed for PFAS substances using EPA Method 1633 (EPA 2024), with only compounds listed in Table 2 reported.

2.2.7 Have the important inputs and practical constraints been considered?

Constraints for collecting additional samples from the site include:

- OSHA constraints limit the ability of personnel to enter the Tank Pit to collect samples (confined space issues).
- Additional samples from the Tank Pit will have to be collected via soil in excavator bucket, similar to the method used to collect historical Tank Pit samples (Figure 6).
- The depths at which samples can be collected are constrained by the soil excavation equipment capabilities. The contractor believes that samples down to 20 ft can be collected via excavator bucket.
- The Tank Pit has been open for several months, during which time it has been exposed to runoff from the parking lot as well as irrigation runoff from the landscaping to the south (Figure 12). This runoff may have introduced anthropogenic contamination, thus, the sampling plan will include removal of several inches of soil from the bottom of the Tank Pit before additional samples can be collected. For comparison purposes, the current stockpile of soil removed from the excavation pit remains under tarps located elsewhere on the PNNL campus (Figure 13).
- New samples will be analyzed by EPA Method 1633 (EPA 2024), in accordance with requirements for WAC 173-340-740(7). While EPA Method 1633 does not describe the soil fraction for analysis, WAC 173-340-740(7)(a) specifies that the soil aliquot should have a size fraction of 0.2 mm or less. The historic samples included percent moisture determination and this should be a requirement for new sample analyses as well (WAC 173-340-740(7)(a)).



Figure 12. Recent photos (July 2024) of the site show evidence of runoff entering the Tank Pit and fine sediments deposited in the bottom.



Figure 13. Recent photo of the stored stockpiled soil removed from the Tank Pit during excavation.

2.2.8 What are the statistical issues related to the design strategy?

A statistical design was not used to determine sampling locations or the number of samples that were collected from the Tank Pit during excavation, nor for samples collected from the stockpile of removed soil (Fulcrum 2023a and b). However, sample locations were selected to represent the soils surrounding the tank, and other infrastructure, and were not biasedly based on soil discoloration or other observed characteristics. Therefore, the historical samples are deemed representative of the volume of soil that surrounded the tank. The samples from the stockpile were collected after the soil was relocated twice, which resulted in extensive mixing prior to sampling. Therefore, for the purpose of assessing the statistical distribution of PFAS concentrations at the site and calculating statistics for comparison with cleanup values, only the historical and new tank pit samples will be used.

VSP will be used to determine the number of samples needed from the tank pit to perform valid statistical comparisons with cleanup values. Because historical samples are deemed representative, the number of new samples collected will be the difference between the number of historical samples and the number needed to make an appropriate statistical comparison.

To convey information, VSP will place notional sample locations for additional soils to be collected from the site. Actual sample locations will be based on the ability of the excavator to reach an area to obtain a bucket of soil, which will then be brought to the surface for sampling. The approximate location from which the soil was collected will be recorded as the sample location, similar to the procedure used to document the locations of the historical Tank Pit samples (Fulcrum 2023b).

2.3 Step 4: Define the boundaries of the study

2.3.1 Are the characteristics of the population of interest defined?

The site population of interest is the volume of soil that may have been impacted through leaks in the 10,000-gallon tank, as well as the soil beneath the tank down to a depth of 20 ft. Collecting samples down to this depth will allow both site-specific cleanup value calculations and an assessment of the likelihood of PFAS to infiltrate to groundwater at concentrations comparable to cleanup values.

2.3.2 Is the geographical area defined for the study?

We have defined the site boundary to be the outer dotted line demarcating the excavation area under investigation as shown in Figure 7. The vertical sampling boundary is bounded by the inner dotted line in Figure 7, down to a depth of approximately 20 ft, which is the limiting reach of an excavator that will be used for sampling. Transport modeling will extend the vertical extent from 20 ft depth to groundwater depth.

2.3.3 Is the population divided into strata each having relatively homogeneous characteristics?

This volume identified in Section 2.4.2 is not expected to have soil strata; conditions at the site are a homogeneous mix of sand, fine soil/silt, and cobble down to groundwater (Freedman et al. 2010).

2.3.4 What are data to be collected?

Additional data will be collected from soil at the site at depths down to 20 ft. Samples will be analyzed for the PFAS compounds shown in Table 2. Practical quantitation limits (PQL) down to the level of CLARC 2024 levels are required, as Ecology 2023 states that cleanup levels may often be below the PQL.

2.4 Step 5: Develop a decision rule

2.4.1 What is the statistical parameter that characterizes the population being studied?

For compliance monitoring, the analysis of the historical and new samples is described for both acute and chronic exposure (WAC 173-340-740(7)(c)(iv)(A) and (B), respectively). MTCA cleanup levels for PFAS substances accounts for noncancer and carcinogenic risk (Ecology 2023, Section 3.2). In both cases, the levels are based on lifetime exposure to the source, which equates to chronic exposure, and thus, WAC 173-340-740(7)(c)(iv)(B) would apply. Therefore, the parameter of interest is the true mean concentration in soils surrounding the tank to a depth of approximately 20 ft.

Furthermore, WAC 173-340-740(7)(c)(iv)(B)(d)(i)(A) specifies that the statistic that shall be used is an upper one-sided 95% confidence limit (UCL) for the true mean soil concentration. Additional instructions are in place within WAC 173-340-740(7)(c) that specify how the data shall be analyzed and how the upper confidence limit shall be calculated.

In addition to evaluate the UCL for the mean, under WAC 173-340-740(7)(e), no single sample shall be greater than twice the cleanup value, and less than 10% of the samples shall not be greater than the cleanup value. WAC 173-340-740(7)(e) also includes specific instructions for performing the required calculations when some sample results are less than the PQL and/or RL.

2.4.2 What are the action levels of the study?

For box 2 of Figure 9, the cleanup values in Table 2 are the ones against which the data will be evaluated. Box 4 of Figure 9 also uses the true mean concentration as the parameter of interest but compares the upper 95% UCL for the mean to site-specific values, which are yet to be calculated.

2.4.3 What is the decision rule for the study?

The decision rules are outlined in Figure 9. If the upper one-sided 95% UCL for the mean does not exceed the cleanup value, and the maximum observed value does not exceed twice the cleanup value, and less than 10% of the observed values are less than the cleanup value for all PFAS compounds of interest, then the conclusion is that groundwater is not impacted.

If one or more of the criteria above are not met, then PNNL will calculate site-specific cleanup values using equations in WAC 173-340-747. If the upper one-sided 95% UCL for the mean does not exceed the site-specific value for all PFAS compounds, the conclusion is that groundwater is not impacted.

If the upper one-sided 95% UCL for the mean exceeds the site-specific cleanup value for one or more PFAS compounds of interest, PNNL will develop a 1-D dispersion model as discussed in Section 2.2.2.

2.5 Step 6: Specify Tolerable Limits on Decision Errors

2.5.1 What are the decision errors?

There are two types of decision errors associated with statistical hypothesis testing, as shown in Figure 14. The goal of statistical sampling is to acknowledge and control for these errors to the extent possible by collecting a sufficient number of samples in a statistically rigorous way.

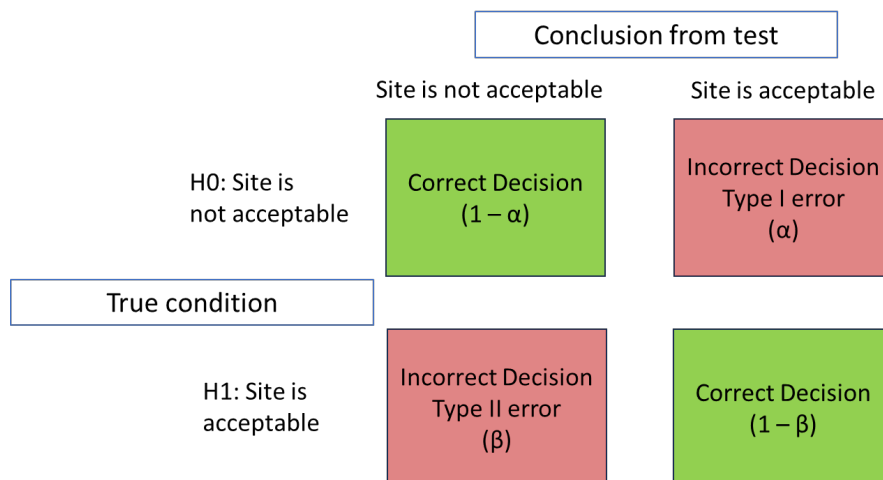


Figure 14. Decision error matrix, with the true condition as rows and the decision based on data as columns.

The decision errors for this DQO are:

- Type I: incorrectly that the true mean concentration is less than or equal to the cleanup value, and
- Type II: incorrectly concluding that the mean concentration is greater than the cleanup level.

For this DQO, we have determined that the acceptable Type I and Type II error rates are 5 and 10%, respectively. The 5% acceptable Type I error rate is based on the discussions in Sections 2.5.1 and 2.5.2. The 10% acceptable Type II error rate is a typical selection for environmental investigations and, for this site, results in a number of samples that is reasonable for statistical comparisons as well as practical for site limitations discussed in Section 2.4.

2.5.2 What are the null and alternative hypotheses upon which the decision will be evaluated?

In statistical designs, the number of samples is usually based on the most egregious error. In this case, that is the Type I (alpha) error that corresponds to the mistaken determination that the mean concentration is less than or equal to the cleanup value. To guard against this error, the null and alternative hypotheses are stated as follows:

H0: site mean \geq cleanup value, and

H1: site mean $<$ cleanup value.

2.5.3 What are some potential consequences for the decision errors?

- If we mistakenly determine that the site mean is less than or equal to the cleanup value, that is, that H1 is true, then we will mistakenly conclude that groundwater is protected. The risk associated with this false determination is that we leave potentially contaminated soils in the ground that may eventually impact groundwater.
- If we mistakenly determine that the site mean exceeds the cleanup value, that is, that H0 could be true, then we will mistakenly conclude that groundwater is not protected. In this case, we move through subsequent actions shown in Figure 9 and Section 2.5.

2.5.4 Is the gray region specified?

For the statistical hypotheses shown in Section 2.5.2, the one sample Student's t-test (t-test) assuming a lognormal distribution is used as the basis for determining the number of samples. The t-test assumes population values are normally distributed, the observations are independent, and the data are a random sample from the population. The last three assumptions are required for nearly all statistical tests. The normality assumption is discussed in more detail here.

As seen from the boxplots in Figure 8, the historical data are not normally distributed; however, the PFOA data could be lognormally distributed. The lognormal QQ-plot of the PFOA data is shown in Figure 15, with non-detects and detects specified by different symbols. The diagonal line in the QQ plot is the trend the data are expected to follow if the concentrations on the y-axis are lognormal. Note that the data do follow this line, even in the tails, which is a common place for deviations in environmental datasets. WAC 173-340-740(7)(d)(i)(B) specifies that D'Agostino's test or the W test can be used to get a p-value for the hypothesis test that data are from a normal distribution. Because the generally accepted minimum number of samples for D'Agostino's test is 20 (our number of samples is 13), we use the W test. The W test is the Shapiro-Wilk test for normality, which, when performed on the lognormal-transformed historical data for PFOA, gives a p-value of 0.2258, indicating the lognormality assumption is reasonable.

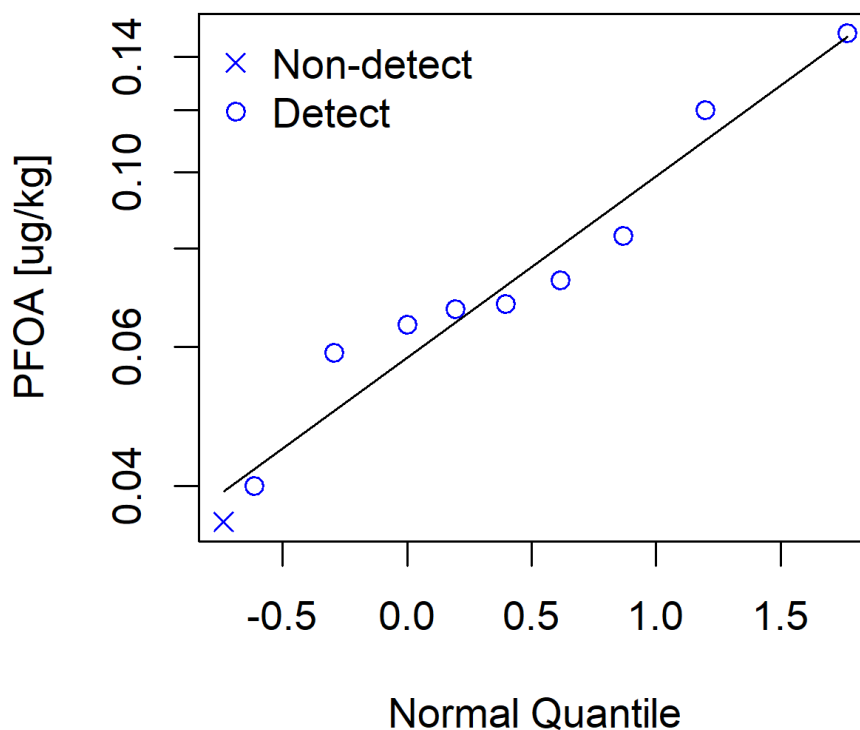


Figure 15. Lognormal QQ-plot of the historical PFOA concentrations.

The concepts related to number of sample calculations using the one sample t-test are best illustrated by a performance goal diagram, as shown in Figure 16. The performance goal diagram is a graphical representation of the equation used to calculate the number of samples. This equation considers the bounds of the gray region, the hypothesis test of interest, the acceptable error rates, and observed uncertainty in the historical data.

The boundaries of the gray region are the PFAS concentrations at which we wish to control the error probabilities. For the null and alternative hypotheses in Section 2.5.2, the Type I error is controlled at the null hypothesis value; therefore, the upper bound of the gray region (UBGR) is the cleanup value (and the concentration at which the acceptable Type I error is achieved). The lower bound of the gray region (LBGR) is the concentration at which the acceptable Type II error is achieved. The main driver for the number of samples calculation is width of the gray region relative to the estimated standard deviation, or the ratio (Δ/σ) , where $\Delta = UBGR - LBGR$. The smaller the relative shift, the more samples required to achieve acceptable error tolerances.

The green line in Figure 16 represents the historical standard deviation. We use PFOA as the basis for the number of samples because it has the highest number of detected values in the samples from the Tank Pit and so provide the best estimate of a standard deviation. The curved red line in Figure 16 is the line for the upper one-sided t-test that intersects the UBGR at the desired confidence level (or 1, the desired Type I error) and the LBGR at the desired Type II error. The equation of this line determines the number of samples.

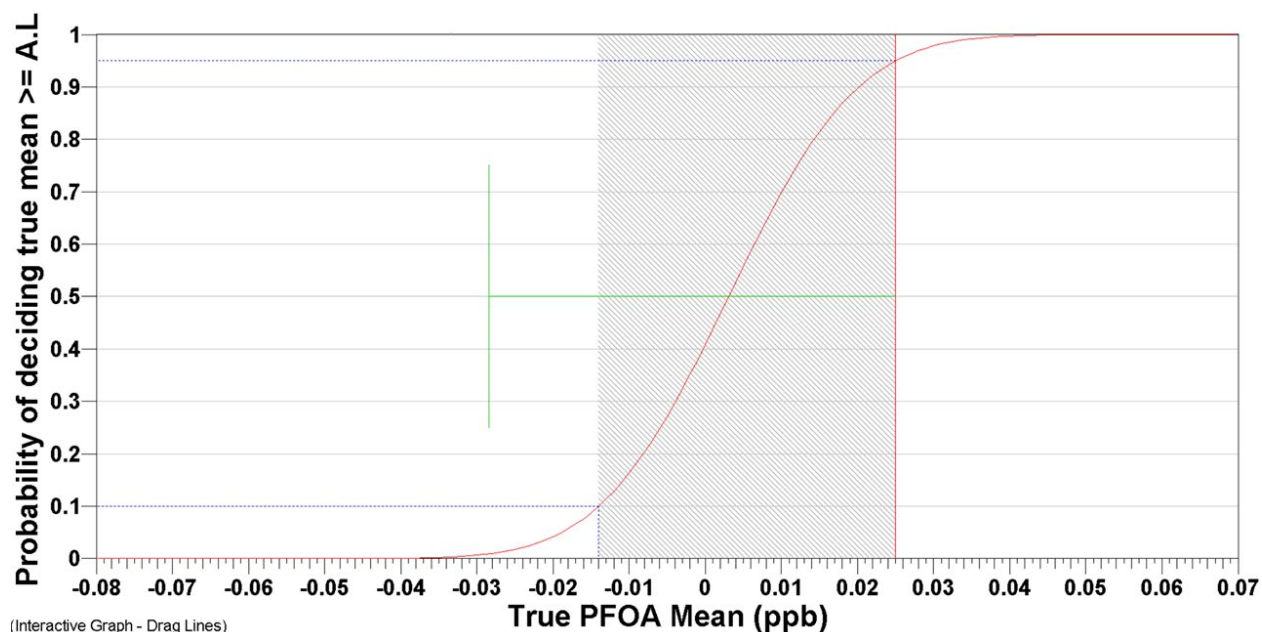


Figure 16. Performance goal diagram for the upper one-sided Student's t-test for PFOA, which has the highest number of detects and lowest number of non-detects (6 and 2, respectively).

Table 4 shows the inputs to the number of samples equation for the upper one-sided t-test. The historical PFOA results from the Tank Pit were used to estimate the standard deviation because it has the highest number of detected values (and hence the lowest number of non-detects) and so can provide the best estimate of a standard deviation. We chose the LBGR that would result in a relative shift of 0.74, which means that the width of the gray region is 74% of the standard deviation estimate. This results in a number of samples that is reasonable to collect given the configuration and size of the Tank Pit (see Section 2.6 for discussion). Note that the LBGR is negative because the cleanup value (UBGR) is small relative to the estimated standard deviation. This can happen when the UBGR is very small and near (or below) the DL of the analytical method. The important quantity is the relative shift, which is always positive for an upper one-sided test.

Table 4. Data Quality Objectives inputs to determine the number of samples. These values result in the curved red line in Figure 15.

Parameter	Value
UBGR	0.025 $\mu\text{g}/\text{kg}$
LBGR	-0.039 $\mu\text{g}/\text{kg}$
Type I (α) error	0.05
Type II (β) error	0.10
Standard Deviation	0.053 $\mu\text{g}/\text{kg}$
Relative shift	0.74

2.6 Step 7: Optimize the Design

The performance goal diagram in Figure 16 indicates that the minimum number of samples for the site is 18. Since 8 samples were collected from the Tank Pit during excavation (Table 1), we will collect an additional 10 samples from the Tank Pit at a deeper depth. These combined data will support the decisions and actions outlined in Section 2.2.

Because the Tank Pit has been open to the environment and has received runoff for several months (see Figure 11), 2.5 feet of soil will be removed from the bottom of the Tank Pit before the new samples are collected. Figure 17 shows a graphic that identifies the soil to be removed as well as the volume from which new samples will be collected.

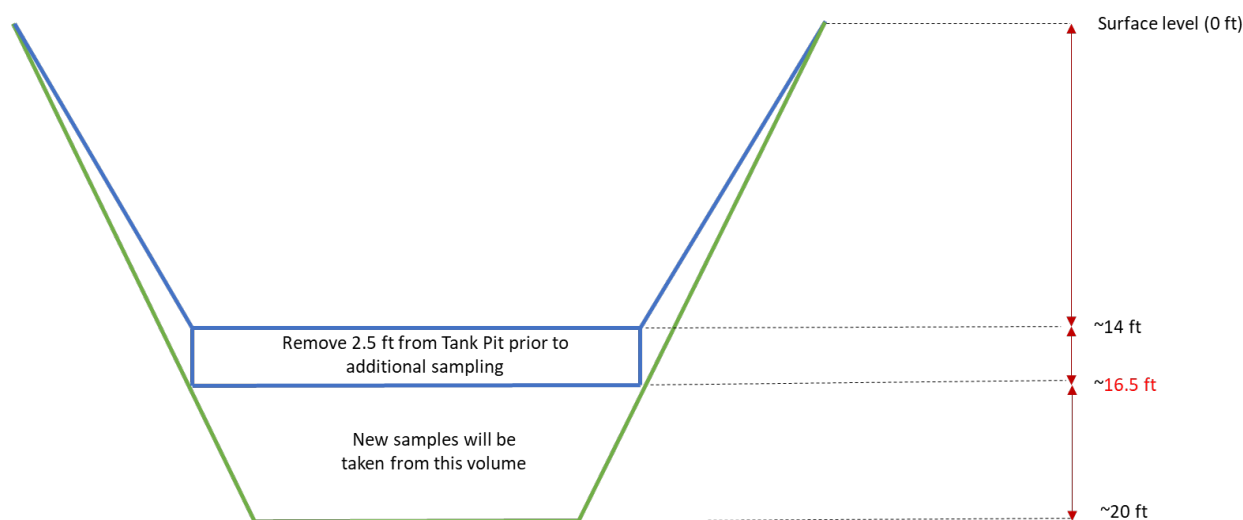


Figure 17. Drawing illustrating the current depth of Tank Pit (14 ft), depth for removal of exposed soils (2.5 ft), and new sampling depths (16.5 – 17 ft, and 19.5 – 20.0 ft).

Because PFOA has the highest number of detected values (Table 1), we use it as the basis for calculating the number of samples. The standard deviation of the historical PFOA samples from the tank pit is 0.053 $\mu\text{g}/\text{kg}$ (the green vertical line in Figure 16). The relative shift was set at 0.74, so that the LBGR = -0.039 $\mu\text{g}/\text{kg}$. The LBGR is less than zero because the null hypothesis value is less than the standard deviation. Negative concentrations, while not possible for organic compounds, do not pose an issue when determining the number of samples (this happens when the cleanup value is very low in comparison to the capability of the analytical method and the observed variability). The relative shift is the important parameter for planning sampling.

Samples from the Tank Pit will be collected in a manner similar to the historical sampling (Figure 6), where the sample is collected from soil in the excavator bucket. Table 1 summarizes the historical sampling at depth as these are key considerations in the approach for sampling further in the Tank Pit.

Sampling in the excavator bucket will minimize risk to worker by eliminating the need for the sampler to enter the Tank Pit. Notional sample locations were randomly selected from an approximate area that represents the current bottom of the Tank (see the blue area in Figure 18). Two samples will be collected from each area, at approximate depths of 17 and 20 ft below grade. Seventeen feet represents the soil at a depth that is unlikely to be impacted from recent runoff, and 20 feet is the maximum depth that the excavator can reach given the current

configuration of the site (Figure 17). An additional two samples were added as duplicates for the Tank Pit for quality control purposes. The two additional samples will be randomly selected from the excavator bucket during sampling (at different lifts, extending from North to South).

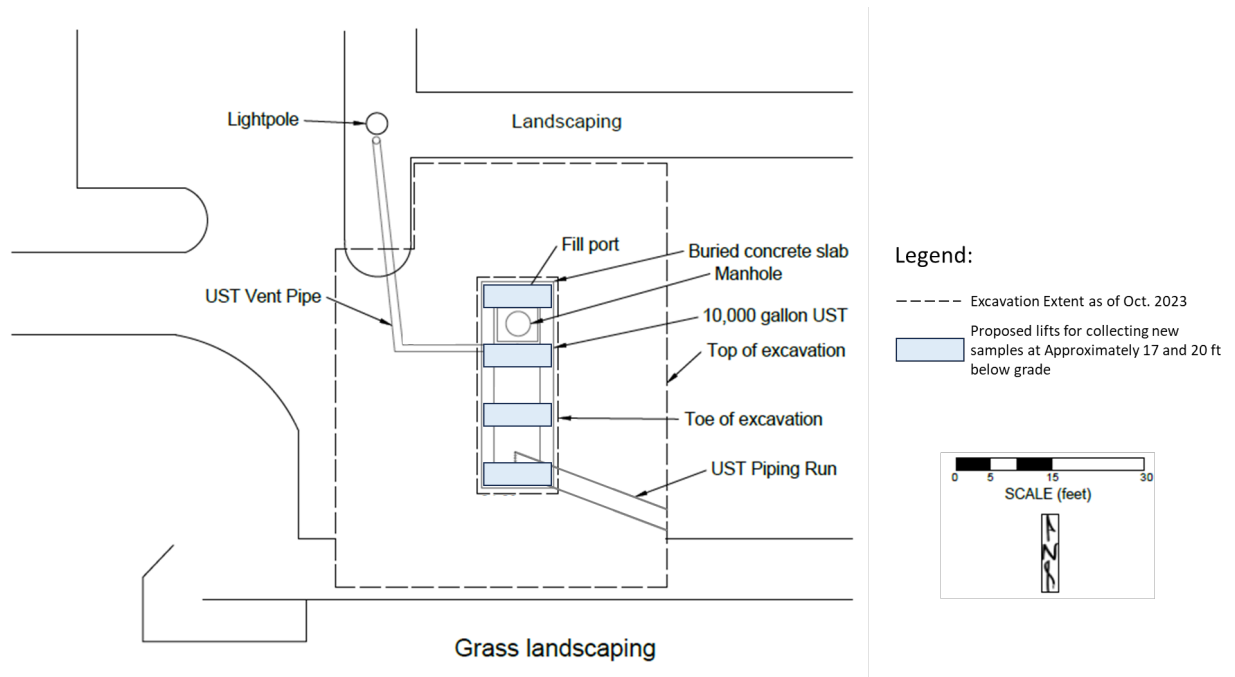


Figure 18. Proposed lifts for collecting new samples within Tank Pit.

3.0 Quality Assurance Project Plan

This quality assurance project plan establishes the quality requirements for data collection, including planning, implementation, and assessment of sampling, field measurements, and laboratory analysis. This section builds off the DQOs identified in the previous section.

3.1 Project Management and Task Organization

This section addresses the basic aspects of project management, which demonstrate that the project has defined goals, the project team understands the goals and the approaches used, and the planned outputs are appropriately documented. Project management roles and responsibilities discussed in this section apply to the major activities for the evaluation of PFAS in soil at the LSL2 10,000-gallon tank site.

A sub-contractor, acquired by PNNL, is responsible for planning, coordinating, collecting, preparing, packaging, and shipping samples to the analytical laboratory, as defined in the contractor's respective contracts and procedures.

3.1.1 Problem Definition

PNNL must collect sufficient quantity and quality of PFAS analyses to demonstrate that the stockpiled soil can be returned to the Tank Pit, the area closed for re-application of asphalt and the parking lot reopened.

3.1.2 Special Training

PNNL will be responsible for determining that personnel supporting the project have completed the required training.

The subcontractor's programs shall provide for the indoctrination, training, qualification, and certification as necessary of personnel performing or managing activities to assure that their proficiency or certifications are achieved and maintained. The subcontractor is responsible for demonstrating that its personnel have obtained suitable proficiency or certification before assigning personnel to support the PFAS sampling effort are met.

3.1.3 Documents and Records

The subcontractor's programs are responsible for maintaining documents and records in accordance with their quality program requirements and the Disposition Program requirements identified in their procurement document(s).

3.1.4 Sampling Process Design (Experimental Design)

The subcontractor will define a process prior to initiating sampling. The sample process will be similar to that used in 2023 (Fulcrum 2023a) for collecting the historical samples. Sampling sites may be moved from the notional sites identified in Figure 18 based on several reasons:

- Surveys of underground utilities are in the vicinity of the sampling location.
- Concerns that sampling could be disruptive to 20,000-gallon diesel UST.

- Safety concerns for the workers.

Other considerations for the sampling of the Tank Pit:

- In the Tank Pit, the top 30 in. (2.5 ft) will be removed before sampling begins. The Tank Pit has been exposed since excavation in October 2023, and there is evidence of sediments at the deepest level of the pit and plants growing (Figure 12), indicators that the soil may have become contaminated.
- Soil removed during sampling effort will be placed in the vicinity of the Tank Pit, then pushed back into the excavation pit at the end of sampling.
- Excavation will be completed from the North to the South of the excavation pit. Historical samples indicate that the PFAS substances are in lower concentration from the North to the South (Figure 11).
- Soil samples will be collected in lifts from 16.5 -17.0 ft and then from 19.5 -20.0 ft (Figure 7). Sampling of the soil will be from the excavator bucket, with effort to minimize collection of soil that is in contact with the bucket (Figure 6).
- Number of samples to collect are discussed in Section 2.6.

3.1.5 Sample Handling and Custody

The subcontractor will define sample handling and custody process for the samples to be collected. Direction on sample handling for the analytical methods will be based on directions from the contract analytical laboratory. A similar process as used previously is anticipated (Fulcrum 2023a and b), and will be consistent with Ecology 2023 and EPA Method 1633 (EPA 2024).

3.1.6 Analytical Methods

The approved analytical method for soil is: *Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS* (EPA 2024). Fulcrum 2023b used the draft version of Method 1633. Of key importance is that the LOD, DL and RL of the analytical laboratory for the PFAS substances to be reported are less than the MTCA Soil Concentrations Protective of Groundwater Vadose @ 13°C (Table 2; Ecology 2024).

The subcontractor will select an approved laboratory to perform the analytical testing methods using laboratory-specific standard operating procedures (SOPs). The laboratory-specific SOPs shall address specific quality control (QC) procedures, QC frequencies, QC limits, and corrective action requirements. The approved laboratory providing analytical services shall have a corrective action program addressing analytical system failures and documenting the effectiveness of corrective actions.

To follow WAC 173-340-740(7), Compliance Monitoring, the analytical laboratory will report the practical quantitation limits (PQL) as well as the LOD, DL and RL for the analyses. These various limits are required as these limits describe the sensitivity of the analytical procedures of the laboratory. Each limit is specific to the analyte, sample matrix, test method, instrumentation and the analyst/laboratory performance. They are key to the evaluation and interpretation of the analytical results.

Analytical laboratory will also report the percent moisture in each soil sample. The percent moisture will be used to report all results as dry weight.

Table 5 presents the reporting limits for PFAS in soil based on the laboratory used for the historical samples (Fulcrum 2023b). Actual detection limits will be applied and controlled in accordance with the approved laboratory's quality assurance program.

Table 5. Reporting Limits for Target PFAS

Target Analyte Name	Abbreviation	CAS #	Reporting Limit ¹ (µg/kg)
Perfluoroalkyl carboxylic acids			
Perfluorobutanoic acid	PFBA	375-22-4	0.14
Perfluoropentanoic acid	PFPeA	2706-90-3	0.061
Perfluorohexanoic acid	PFHxA	307-24-4	0.077
Perfluoroheptanoic acid	PFHpA	375-85-9	0.029
Perfluorooctanoic acid	PFOA	335-67-1	0.036
Perfluorononanoic acid	PFNA	375-95-1	0.044
Perfluorodecanoic acid	PFDA	335-76-2	0.038
Perfluoroundecanoic acid	PFUnA	2058-94-8	0.030
Perfluorododecanoic acid	PFDoA	307-55-1	0.038
Perfluorotridecanoic acid	PFTTrDA	72629-94-8	0.031
Perfluorotetradecanoic acid	PFTeDA	376-06-7	0.030
Perfluoroalkyl sulfonic acids			
Acid Form			
Perfluorobutanesulfonic acid	PFBS	375-73-5	0.027
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	0.032
Perfluorohexanesulfonic acid	PFHxS	355-46-4	0.033
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	0.025
Perfluorooctanesulfonic acid	PFOS	1763-23-1	0.051
Perfluorononanesulfonic acid	PFNS	68259-12-1	0.039
Perfluorodecanesulfonic acid ²	PFDS	335-77-3	0.033
Perfluorododecanesulfonic acid ²	PFDoS	79780-39-5	0.028
Fluorotelomer sulfonic acids			
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2FTS	757124-72-4	0.15
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2FTS	27619-97-2	0.14
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2FTS	39108-34-4	0.13
Perfluorooctane sulfonamides			
Perfluorooctanesulfonamide	PFOSA	754-91-6	0.047
N-methyl perfluorooctanesulfonamide	NMeFOSA	31506-32-8	0.034
N-ethyl perfluorooctanesulfonamide	NEtFOSA	4151-50-2	0.059
Perfluorooctane sulfonamidoacetic acids			
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9	0.048

Target Analyte Name	Abbreviation	CAS #	Reporting Limit ¹ (µg/kg)
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6	0.026
Perfluorooctane sulfonamide ethanols			
N-methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7	0.39
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2	0.44
Per- and Polyfluoroether carboxylic acids			
Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6	0.10
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4	0.099
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1	0.042
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5	0.038
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6	0.059
Ether sulfonic acids			
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1	0.081
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid ²	11Cl-PF3OUdS	763051-92-9	0.11
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7	0.048
Fluorotelomer carboxylic acids			
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5	0.21
2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA	914637-49-3	1.1
3-Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4	0.99

¹ Reporting Limits are from Fulcrum 2023b, using the draft EPA Method 1633.

² These analytes may not perform as well as others in some matrices (see EPA 2024, Section 1.6): PFDS, PFDoS, and 11CLPF3OUdS in aqueous samples; PFDoS and 11CLPF3OUdS in biosolid samples; and PFDoS in tissue samples.

If a failure in the analytical method occurs or is suspected (e.g., receipt of erroneous or incomplete laboratory results), the PNNL Environmental Compliance representative shall be notified and decide if action is required (e.g., resampling may be necessary).

3.1.7 Quality Control

Procedures must be followed in the field and laboratory to obtain reliable data. Field personnel will collect QC samples to evaluate the potential for cross-contamination and to provide information pertinent to field variability. Field QC (Table 6) for sampling will require the collection of field duplicate (as described in Section 2.7). Laboratory QC (Table 6) samples estimate the precision and bias of the analytical data. Approved laboratory procedures shall identify specific laboratory QC procedures, including frequency, limits, and corrective action requirements.

Table 6. Quality Control Checks

QC Sample Type	Purpose	Frequency
<i>Field QC</i>		
Field duplicates	Estimate precision, including sampling and analytical variability.	Over the sampling event, a field duplicate will be collected each day of sampling. Field duplicates will be collected as defined in Section 2.7
Equipment rinsate blank	Verify adequacy of sampling equipment decontamination.	If only disposable equipment is used, then an equipment rinsate blank is not required. Otherwise, one per decontamination activity.
<i>Laboratory QC</i>		
Method blank	Assess response of an entire laboratory analytical system.	One per batch, 20 samples maximum, or as identified by the method guidance.
Matrix spike	Identify analytical (preparation and analysis) bias; possible matrix effect on the analytical method used.	As identified by the method guidance, or one per batch, 20 samples maximum.
Matrix duplicate or matrix spike duplicate	Estimate analytical bias and precision.	As identified by the method guidance, or one per batch, 20 samples maximum.
Laboratory control samples	Assess method accuracy.	As identified by the method guidance, or one per batch, 20 samples maximum.
Surrogates (organic analyses only)	Estimate recovery/yield.	As identified by the method guidance.

3.1.8 Data Management

Data management includes the processes used to generate, validate, interpret, track, store, and retrieve information. PNNL will meet all policies and procedures in accordance with data management.

The subcontractor's programs are responsible for data management in accordance with their quality program requirements and the Disposition Program requirements identified in the procurement document(s) (e.g., Interagency Work Order).

3.2 Assessment and Oversight

The purpose of assessments is to verify that the project requirements are effectively implemented as prescribed. PNNL's assessment and oversight program is discussed in *How Do I*, and meet all policies and procedures for conducting work with PNSO.

The subcontractor's programs are responsible for assessment and oversight in accordance with their quality program requirements and the Disposition Program requirements identified in the procurement document(s) (e.g., Interagency Work Order).

3.3 Data Validation and Usability

The subcontractor's programs are responsible for data review, verification and validation in accordance with their quality program requirements and the Disposition Program requirements identified in the procurement document(s) (e.g., Interagency Work Order).

All coordination and execution of data review, verification and validation activities, and handling/storage of deliverables, and submittals will be performed in accordance with PNNL's quality program requirements for review, verification, and validation of analytical data. These requirements will meet those identified in MCTA 173-340-747 and based on other relevant guidance (e.g., Ecology 2023).

3.3.1 Data Review, Verification and Validation

The data review and verification will be performed by the subcontractor in accordance with their quality program requirements for review and verification of analytical data.

The data review and verification activities include, but are not limited to, verification of required deliverables, review for completeness (samples were analyzed as requested), review of the laboratory case narrative, use of the correct analytical method or procedure, reporting of the correct PFAS compounds, verification of achieved practical quantitation limits (PQL), transcription errors (e.g., sample ID), correct application of dilution factors and conversion factors, appropriate reporting of units (e.g., dry weight versus wet weight), evaluation of holding times and preservation (when applicable), and verification or addition of applicable qualifiers for 100% of the sample results.

Data validation will be determined by PNNL. The criteria for data validation are based on a graded approach. Five levels of validation have been defined, Level A through Level E. Level A is the lowest level and is the same as verification. Level E is a 100% review of data (e.g., calibration data; calculations of representative samples from the data set). To meet the objectives of the this SAP, Level E data validation is required.

Level E is defined as:

- Verification of required deliverables, review of laboratory case narrative, verification of requested versus reported analyses
- Verification of transcription errors
- Evaluation of field blanks, field duplicates, and field splits (if information is provided), evaluation and qualification of results based on method blank result criteria
- Evaluation and qualification of sample results based on matrix spikes, laboratory control samples, laboratory duplicate, and chemical and tracer recovery criteria, as appropriate to the method
- Evaluation of requested versus achieved analyte PQLs, and evaluation and qualification of results based on analytical holding time criteria
- Qualification of results based on initial and continuing instrument calibration and other QC check criteria performed as required by the particular analytical method
- Calculation checks of both sample and QC results will be performed at a frequency of 20%, or at least one sample and one complete QC sample series will be recalculated, whichever is greater

3.3.2 Data Quality Assessment

The quality assurance objective of this plan is to develop implementation guidance for providing data of known and appropriate quality. Data quality indicators describe data quality by evaluation for identified data needs and the work activities identified in this SAP. The applicable QC guidelines, quantitative target limits, and levels of effort for assessing data quality are dictated by the intended use of the data and the nature of the analytical method. The principal data quality indicators are precision, bias or accuracy, representativeness, comparability, completeness, and sensitivity. These data quality indicators are defined for the purposes of this document in Table 7. The data quality indicators will be evaluated during the data quality assessment process.

As PFAS analyses are evolving, laboratory operations and analytical services shall be in compliance with a recognized guidance as identified in Ecology 2023 and DOE 2023.

The criteria for data quality indicators are listed in Table 7.

Table 7. Data Quality Indicators

Data Quality Indicator	Definition	Example Determination Methodologies	Project-Specific Information	Examples of Corrective Actions
Precision	<p>The measure of agreement among repeated measurements of the same property under identical or substantially similar conditions; calculated either as the range or as the standard deviation.</p> <p>May also be expressed as a percentage of the mean of the measurements, e.g., relative range, relative percent difference, or relative standard deviation (coefficient of variation [COV]).</p>	<p>Use same analytical instrument to make repeated analyses on same sample.</p> <p>Use same method to make repeated measurements of same sample by laboratory.</p> <p>Split a sample in field and submit both for sample handling, preservation and storage, and analytical measurements, if applicable.</p> <p>Collect, process, and analyze co-located samples for information about sample acquisition, handling, shipping, storage, preparation, and analytical processes and measurements.</p>	<p>Field precision: A field duplicate sample will be collected randomly each day of sampling. Sampling field duplicate is described in Section 2.7.</p> <p>Laboratory precision: Analysis of laboratory duplicate or matrix spike duplicate samples.</p> <p>One duplicate sample will be analyzed per sample set (i.e., group of samples collected from a media and analyzed using the same laboratory method), exclusive of batch leach testing and physical properties analyses, and as applicable to the method.</p> <p>Split samples will be evaluated like duplicate samples.</p> <p>The required laboratory analytical precision is $\leq 30\%$ for radiological and $\leq 35\%$ for chemical batch laboratory replicate sample relative percent differences.</p>	<p>If duplicate data do not meet objective:</p> <ul style="list-style-type: none"> Evaluate apparent cause (e.g., sample heterogeneity). Request reanalysis or re-measurement. Qualify the data before use.
Accuracy	<p>A measure of the overall agreement of a measurement to a known value; includes a combination of random error (precision) and systematic error (bias) components of sampling and analytical operations.</p>	<p>Analyze a reference material or reanalyze a sample to which a material of known concentration or amount of pollutant has been added (a spiked sample); usually expressed as percent recovery or percent bias.</p>	<p>Laboratory accuracy determination based on matrix spike and matrix spike duplicate results.</p> <p>The required laboratory analytical accuracy is 70–130% for batch laboratory control sample percent recoveries. Additional accuracy criteria include analysis-specific evaluations preformed for matrix spike, tracer, and/or carrier recoveries, as appropriate to the method.</p>	<p>If recovery does not meet objective:</p> <ul style="list-style-type: none"> Qualify the data before use. Request reanalysis or re-measure.

Data Quality Indicator	Definition	Example Determination Methodologies	Project-Specific Information	Examples of Corrective Actions
Representativeness	A qualitative term expressing “the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition” (ANSI/ASQ 1995).	Evaluate whether measurements are made, and physical samples are collected in such a manner that the resulting data appropriately reflect the environment or condition being measured or studied.	<p>Samples will be collected as described in the sampling design.</p> <p>Random sampling satisfies requirements for Class 3 land area sampling (NRC 2000).</p> <p>Judgment sampling investigates areas most likely to be contaminated, based on current information.</p>	<p>If results are not representative of the system sampled:</p> <ul style="list-style-type: none"> • Identify the reason the result is not representative. • Reject the data, or, if the data are otherwise usable, qualify the data for limited use and define the portion of the system the data represent. • Redefine sampling and measurement requirements and protocols. • Resample and reanalyze.
Comparability	A qualitative term expressing the measure of confidence that one data set can be compared to another and can be combined for the decision(s) to be made.	Compare sample collection and handling methods, sample preparation and analytical procedures, holding times, stability issues, and quality assurance protocols.	<p>Sampling personnel will use the same sampling protocols.</p> <p>Samples will be submitted to the same laboratories when possible (based on laboratory contracts) for analysis by the same methods; thus, data results will be comparable.</p>	<p>If data are not comparable to other data sets:</p> <ul style="list-style-type: none"> • Identify appropriate changes in data collection and/or analysis methods. • Identify quantifiable bias, if applicable. • Qualify the data as appropriate. • Resample and/or reanalyze if needed. • Revise sampling/analysis protocols for future comparability.

Data Quality Indicator	Definition	Example Determination Methodologies	Project-Specific Information	Examples of Corrective Actions
Completeness	A measure of the amount of valid data needed to be obtained from a measurement system.	Compare the number of valid measurements completed (samples collected or samples analyzed) with those established by the project's data needs.	The percent complete will be determined during data validation. Minimum required for COIs will be 80% complete (does not apply to non-COIs).	<p>If the data set does not meet completeness objective:</p> <ul style="list-style-type: none"> • Identify appropriate changes in data collection and/or analysis methods. • Identify quantifiable bias, if applicable. • Qualify the data as appropriate. • Resample and/or reanalyze, if needed. • Revise sampling/analysis protocols for future comparability.
Sensitivity	The capability of a method or instrument to discriminate among measurement responses representing different levels of the variable of interest.	Determine the minimum concentration or attribute to be measured by a method (method detection limit), by an instrument (instrument detection limit), or by a laboratory (quantitation limit). PQL is the lowest level that can be routinely quantified and reported by a laboratory.	Verify that sensitivity, as measured by detection limits, is appropriate for the action levels.	<p>If sensitivity does not meet the objective:</p> <ul style="list-style-type: none"> • Request reanalysis or re-measure. • Qualify/reject the data before use.

4.0 Field Sampling Plan

The field sampling plan includes the sampling objectives, locations and number of samples, documentation, equipment, methods, sample handling and sample waste management. The subcontractor will be responsible for meeting the information provided in this report and develop their own field sampling plan. This is expected to meet the historical sampling plan as in Fulcrum 2023 and b.

The subcontractor is responsible for collecting information during excavation that verifies the pre-selected locations discussed in Section 2.7 (Figure 17). These include notes on location for soil collected out of the Tank Pit, as well as the depths of soil collected.

The objectives of the field sampling plan may be completed with one round of soil sampling. The results from laboratory analyses will be evaluated in accordance with the decision rule in Section 2.5.

4.1 Field Sample Locations

Notional field sample locations have been identified in Figure 17 for the Tank Pit. Sampling process is discussed further in Section 3.1.4

4.2 Sampling Methods

Soil samples will be collected in three different ways by the subcontractor: In the Test Pit, an excavator will remove approximately 30 inches in the bottom of the hole, and from the sides of excavation area, before sampling. The excavator bucket will then be used to retrieve soil from 16.5-17 ft below grade. The excavator will then remove an additional layer of soil to allow sampling from 19.5-20 ft below grade. Subcontractor's sampling plan will have more details.

Soil samples will be stored in the appropriate container and preserved for shipment to the laboratory in accordance with the subcontractor's sampling plan (Ecology 2023 and EPA 2024).

To summarize, considering the minimum number of samples, design optimization, and quality control samples, the total number of samples to be collected with the new sampling effort is 10 samples at the Tank Pit and one field duplicate per day of sampling.

4.3 Sample Handling

The subcontractor's sample plan will have details on sample handling. Their plan will meet guidance (Ecology 2023; DOE 2023) and specifics called out for the analytical method (EPA 2024).

5.0 Health and Safety Plan

PNNL will meet all health and safety requirements for staff to be associated with this project. The subcontractor will have their own health and safety plan that will meet requirements that are set forth by PNNL in the contract.

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