

PFAS—emerging constituents of concern for mine closure studies

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Abstract

A screening level site investigation of per and/or polyfluoroalkyl substances (PFAS) was conducted at six legacy mining sites in North America; the sites have been inactive from 10 to 92 years before present. Soil and water samples were collected based on the location of historical infrastructure footprints, current monitoring networks (upstream and downstream of mine facilities), hydrogeological conditions, and flow gradients. Representative samples of groundwater, leach draindown solutions, and pit lake water were collected. Soil media samples included a vertical characterization of tailings storage facilities at locations near maintenance shops, processing plants, and pregnant leach solution ponds. The samples were sent to a third-party testing lab and analysed for 36 PFAS compounds included under Method 537 (modified).

The initial screening was successfully conducted, indicating the presence of nine PFAS compounds at five of the six legacy sites. The sample results were compared with five reference guidelines—U.S. EPA proposed MCLs, U.S. EPA Regional Screening Levels, Hawaii Department of Health, Health Canada, and Heads of EPAs Australia and New Zealand.

The results of this screening study will be used to advance the PFAS research at these mining sites related to mine closure planning and assessing the value of expanding the groundwater monitoring and/or establishing a continuous monitoring program at select locations. The voluntary monitoring provides the business with baseline data to inform mine closure issues and to prepare for potential future regulation of PFAS.

PFAS comprises more than 9000 chemicals (NIOSH 2022) and are characterised by a long carbon-fluorine backbone. They possess excellent thermal stability and chemical resistance, making them desirable for various industry applications. PFAS have been manufactured since the 1930s. However, the health risks related to PFAS only gained attention in the early 2000s. Due to their bio-accumulative nature, they persist in the food chain and assimilate into living organisms causing damage to the immune system, kidney, and liver and high risks of cancer, thyroid, and obesity.

PFAS have widespread applications in multiple industries, including electrical, printing, metal, laundry, textile, aerospace, and automotive. They are also widely used in consumer products like food packaging, non-stick cookware, waterproof carpets, cleaning reagents, and aqueous film-forming foam. In the mining industry, they are used as surfactants in cyanide and sulfuric acid leach solutions to enhance the recovery of gold and copper and as mist suppression agents in electroplating processes. PFAS assessment at both operational and legacy sites in North America was completed in a voluntary, exploration phase driven by corporate desire to identify and reduce potential future risks. There are currently no regulatory drivers for enforcement of PFAS standards at mining facilities. Assessments of PFAS at legacy sites is challenging given the typical age of information about specific chemical products and quantities historically used in the processing circuit.

Keywords: *PFAS in mining, mine closure, legacy sites, tailings, risk register, groundwater and soil impacts*

1 Introduction

The scope of this study was to complete a screening-level field investigation of per and/or polyfluoroalkyl substances (PFAS) by surveying six legacy (i.e., inactive between 10 to 92 years before present) mining and processing sites to verify and understand the presence of PFAS in former mining facilities/processes. The sites were chosen by SRK's mining client at their discretion to perform a voluntary, confidential study. PFAS assessment at legacy sites is challenging given the age of the sites, the lack of information about specific chemical products and quantities historically used, and in many cases, the layout of former processing facilities is not precisely known. The screening study was completed at legacy sites with a variety of mining facilities including heap leach pads/rock dump leach piles/in-situ leaching, tailings storage facilities (TSFs), pit lake waters, waste rock dumps, processing facilities and industrial infrastructure. A brief description of each of the sites is included in this study is presented in Section 1.1.

PFAS are ubiquitously present chemicals that are recalcitrant and are emerging constituents of concern. These compounds have a perfluorinated carbon backbone with a functional hydrophilic group. Due to the strong C-F bond, the molecule is highly hydrophobic, small, and electronegative—consequently, thermally, and chemically stable and resistant to any degradation (Rice et al. 2021). These properties make them water-, oil-, and stain-resistant, making them highly desirable for various products across several industries including electrical, textile, paper, metal, paint, cosmetics, fluoropolymer, and electrical. In addition, consumer goods like cleaning, food packaging material, non-stick cookware, firefighting foams etc., are manufactured using these forever chemicals (Bolan et al. 2021). Moreover, the functional groups at the end are hydrophilic, making them suitable surfactants, increasing wetting and dispersant effectiveness (Rice et al. 2021).

PFAS in the mining industry are applied to increase wetting properties of sulfuric acid and cyanide processing solutions used in copper, gold and uranium leaching and to enhance metal recovery. They are used as mist-suppression agents in electroplating processes (Wood 2021). The compounds are also used in proprietary chemicals and additives in sulphide ore concentrating, flotation processes, and maintenance shops.

Though these chemicals have been in application since the 1930s, their adverse health and environmental impacts were not widely known until recently. These artificial 'forever chemicals' are persistent and toxic. They tend to accumulate within the organism and cause serious health concerns. Due to the significant environmental and human health risks, these chemicals are now being studied more widely. The related health issues include increased cancer risks, obesity, thyroid, infertility, metabolism disruption (Lilienthal et al. 2017).

Given the significant health risks associated with them, PFAS regulations are evolving rapidly around the world. In early 2016, the U.S. Environmental Protection Agency (EPA) set the drinking water health advisory levels hexafluoropropylene oxide dimer acid (Gen X), for perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) at a combined level of 70 nanogram/litre (ng/L). PFOA and PFOS are the most commonly occurring contaminants in the PFAS family. In March 2023, the new proposed guidelines regulated PFOS and PFOA individually to an enforceable maximum contaminant level (MCL) of 4 parts per trillion (ppt). The guidelines further expanded to include a Hazard Index for perfluorobutanesulfonic acid (PFBS), perfluorononanoic acid (PFNA), perfluorohexanesulfonic acid (PFHxS), and Gen X (EPA, 2023). Currently regulatory agencies are focussing on promulgating standards for public drinking water. There are no regulations specific to mining, however, the regulations are expected to expand to this industry.

1.1 Site introduction

Site A consists of a former underground copper mine with an open pit and in-situ leach wellfield that operated in overlapping stages for more than 100 years until 2013. Copper sulphide ore was processed through milling, gravity and froth flotation, and concentration methods. Mixed sulphide-oxide tailings were also leached for a brief period. The leaching operation recovered copper through a precipitation plant method until a solvent extraction-electrowinning (SX-EW) plant was commissioned. After underground mining operations ceased in the late-1950s, the mill and concentrator buildings were demolished and

removed from the site. The open pit lake contains in-flowing groundwater, impacted stormwater runoff, and spent leaching process solutions. Reclamation was completed for select on-site TSFs by lime amendment, soil covering, and revegetation in the mid-2000s.

Site B consists of a former open pit copper mine (two pits) with a milling, flotation, concentration, and tailings disposal circuit that operated for almost 25 years. A rock dump leaching, and precipitation circuit operated concurrently for approximately 30 years until the early 1980s. The operators built the leach dumps as an evolving series of individual dumps and bermed paddocks coalesced into more extensive facilities with common downstream solution collection ponds to capture the pregnant leach solution (PLS) and process it in the precipitation plant. One open pit lake contains in-flowing groundwater, impacted stormwater runoff, and residual spent process solutions while the other contains in-flowing groundwater, stormwater runoff, and some residual water treatment solids. The inactive TSFs and rock dump leach facilities have not been reclaimed.

Site C consists of a former underground copper mine with numerous smaller underground mines, shaft, adits, and associated processing facilities that were operated nearly 60 years until the early 1930s. The operation had on-site milling and blast smelter facilities, TSFs, and waste rock dumps. Reclamation activities were largely completed in mid-2000s.

Site D contains a TSF that operated for more than 30 years and was covered with a dust cover and revegetated in the early 1960s. The TSF received tailings generated from a nearby copper sulphide milling and flotation operation. No beneficiation plant facilities were present at this site.

Site E consists of a former underground and open pit operation with a primary crusher for offsite handling of copper sulphide ore that operated for more than 45 years until the late 1990s. It also had a heap leach and in-situ leach operation with SX-EW plant, solid waste and wood landfills, and maintenance facilities. The site was reclaimed in the mid-2000s.

Site F consists of a former copper beneficiation facility with milling, froth flotation and concentration, flash smelting and refining facilities that operated more than 45 years until the late 1990s. Waste disposal included smelter slag, several TSFs, and a solid waste and wood landfill. The site was reclaimed in the mid-2000s.

2 Methodology

Based on site history, processing methods, current monitoring networks, reclamation cut-fill grading maps, and site-specific conditions, SRK selected representative groundwater and soil sampling locations that may have potentially been impacted by historical use of processing chemicals and materials containing PFAS. The following subsections summarize the field activities completed, as well as analytical methods used.

2.1 Pre-investigation activities

A significant effort was made on the pre-investigation activities which included reviewing information on the types and locations of historical processing operations conducted at each site, identifying the locations of former buried infrastructure areas with respect to reclamation cut and fill grading maps, identifying appropriate testing locations (in collaboration with the operator), developing internal workplans, and selecting analytical methods.

The test pit locations for soil sampling were selected based on knowledge about the historical infrastructure footprints. Samples were collected from soils and fill underlying the inert cover. In addition, tailings solids samples collected from previous sonic drillholes completed at four sites were analysed; the sample intervals were selected to represent the top, middle, and bottom layers of the TSF.

Groundwater sampling sites were defined based on the location of the monitoring wells and current hydrogeological conditions and flow gradients. SRK sampled locations that were upstream/upgradient and downstream/downgradient of the mine facilities to assess any potential impacts with respect to background levels. SRK also sampled pit lake water from three pits and from spent leach solutions.

2.2 Sampling procedure

Certain materials must be avoided during PFAS sample collection and handling to avoid contamination (Barfoot et al. 2022). Glass adsorbs PFAS, which might alter the results. Additionally, the sampling equipment should be free of any fluoropolymer compounds like Teflon, polytetrafluoroethylene, fluorinated ethylene propylene, ethylene tetrafluoroethylene, low density polyethylene, polyvinylidene fluoride. The standard equipment to be used for sampling should only be made of high-density polyethylene (HDPE), polypropylene, and silicone. The guidelines from the above paper were followed. Additionally, nitrile gloves were worn to avoid any contamination.

Test pit depths ranged from 1.2 to 4.2 m below ground surface (bgs). Materials collected using a decontaminated stainless-steel shovel were placed in several piles (based on depth). Tailings samples were collected from materials archived from sonic drilling characterization projects completed in 2017. For decontamination purposes, the sampling equipment was rinsed twice with deionized water and finally with PFAS-free water procured from the third-party testing lab. The samples were directly placed into the 500 mL HDPE sampling containers to avoid any external contamination. The HDPE containers were placed in a Ziploc bag and stored in a cooler to preserve temperature (4°C).

Water samples were collected in 500 ml HDPE bottles. The bottles were precleaned and needed no further preparation before taking the samples. The groundwater monitoring wells have dedicated low-flow pumps and tubing that were used to collect groundwater samples. Grab samples were collected at the drain down monitoring locations for drain down of spent processing solutions and pit lakes.

2.3 Quality assurance/quality control

The samples were collected following best practices. Duplicates were collected in groundwater after every 10 samples and in soils after every 20 samples. Equipment blanks (rinsates) were collected to verify the samples were not cross contaminated. To prepare an equipment blank, certified PFAS-free water was used to rinse the equipment. The rinsate was collected, labelled as a sample, and sent for analysis. Concentration of PFAS in field blanks were not detected; therefore, no external contamination was introduced during sampling activities.

It is important to highlight that the groundwater sampling equipment installed at the six legacy sites was not PFAS-free certified; this study did not assess for potential equipment bias by taking duplicate samples with PFAS-free pump equipment.

2.4 Water samples

At Site A, the water samples included groundwater, spent process solution, and pit lake water. The samples were collected in monitoring wells upstream and downstream monitoring of a reclaimed TSF. The upstream location is also the downgradient of another industrial plant operation, which was not investigated in this study. Pit lake A is believed to contain a mixture of stormwater runoff and spent processing solution and was expected to be the most concentrated location across all the sites included in this investigation.

For Site B, the samples were collected at monitoring wells located in known mining impacted areas including TSFs, leach dumps, and an impacted wash. Samples from pit lakes B1 and B2 were collected. Pit lake B1 contains a mixture of stormwater runoff and spent leach process solution. It was expected to be the most concentrated location on this site. Pit lake B2 is a blend of pit wall runoff stormwater, water treatment sludges and remedial water, and water in contact with inactive tailings and waste rock.

For Site C, samples were collected in wells upgradient and downgradient of a reclaimed TSF. A water sample from a historical shaft was also collected to assess the presence of PFAS in the underground workings.

At Site D, a well was sampled downstream of an inactive TSF. The sample was believed to be a blend of residual tailings seepage with the unimpacted groundwater of a nearby gulch.

At Site E, groundwater and spent process solution samples were collected for analysis. Upgradient and downgradient samples from the mine site were also collected.

For Site F, samples were collected upstream and downstream of the reclaimed TSFs.

2.5 Soil samples

Test pit soil samples ranged from 1.2 to 4.2 m bgs. The following information was logged in the field: Excavator type, Depth intervals of soil/material units, Grain size distribution, Unified Soil Classification System designation, colour, plasticity, moisture, maximum particle size, oxidation/reduction features, organic content, number/name of samples collected. From all the locations described below, samples were collected from the top, middle, and bottom sections of the test pits to get a vertical profile of the location. SRK had access to topographic and facility layout maps—before and after reclamation was completed at Site E and Site F— therefore, there was an understanding of cut and fill volumes and the estimated depth to the impacted soils below the overlying cover. This information guided SRK to define excavation depths targeted at each location, and to avoid sampling materials from the reclamation soil cover.

At Site A, locations with potentially impacted facilities were sampled including the soil within the footprint of a former power plant, tailings thickener, concentrator and shaft area, and downstream of a SX EW plant and process solution pond.

At Site B, samples were collected near previous mining infrastructure including a former milling and concentrator plant site and precipitation plant.

Mine tailings samples at Site C and D were collected from the top, middle, and bottom of the TSF to provide a vertical profile using sonic borehole samples. Sampling depths range from 0.3 m to 51 m below top of the TSFs surface. The soil samples were also taken from test pits near historical processing infrastructure.

At Site E, sampling locations included areas near the shafts, timber landfill, solid waste landfill, truck shop, and raffinate pond.

At Site F, sampling locations included areas near the former acid plant, truck maintenance shop, oxygen plant, refinery, tailings thickener, concentrator, electric shops, and tailings samples from one TSF (top, middle, and bottom).

2.6 Analytical methods

The samples in this study were analysed by a certified third-party lab for 36 PFAS compounds under the modified Method 537. The lab offered four packages with modified method 537 being the most comprehensive.

Currently, there are four methods to analyse PFAS in potable drinking water: Method 533, 537, modified 537, and 537.1. Methods 537, 537.1, and 533 can detect 14, 18, and 33 analytes, respectively. Method 533 targets the short-chain PFAS. Other methods (method 8327 and draft method 1633) can be used for non-potable water or other environmental media. Method 537 is strictly applicable for drinking water testing. Modified 537 is a modification of method 537, the laboratory, modifying, decides the changes to be made in the QC and other procedures. A few upcoming methods include total organic fluorine and total organic precursors. These methods do not target any specific PFAS compounds but identify the total organic fluorine and precursors. In method 537, the PFAS compounds are identified by solid phase extraction and liquid chromatography/tandem mass spectrometry (EPA, 2020).

3 Results and discussion

The following subsections discuss the results according to the sampling media and facility type. For comparison purposes, the results are categorized by facility type. The water and soil sample results are presented separately.

3.1 Water samples

Figure 1 shows the cumulative PFAS concentration detected at sampling locations according to facility types: tailings, spent leach process solutions, pit lakes, and other miscellaneous locations. The pit lakes measured the highest PFAS concentrations across all the facilities in this investigation. Water in Pit A is mainly composed stormwater runoff and spent processing solution, and it was expected to have the highest concentration and was targeted to determine and quantify the level of PFAS impact. Pit lake B1, believed to have a similar composition, is the second most concentrated location. The spent solution sample collected at Site E also presents PFAS concentrations above the laboratory Method Detection Limit. The well downgradient of the neighbouring industrial plant site and upgradient of the TSF (Site A) has higher concentration of PFAS compared to the downgradient TSF monitoring well so does not represent an unimpacted background sample. Groundwater samples collected downgradient of TSFs overall present significantly lower concentrations than those observed in samples collected from the pit lakes, spent leach solution, and monitoring wells downgradient of heap/dump leach facilities.

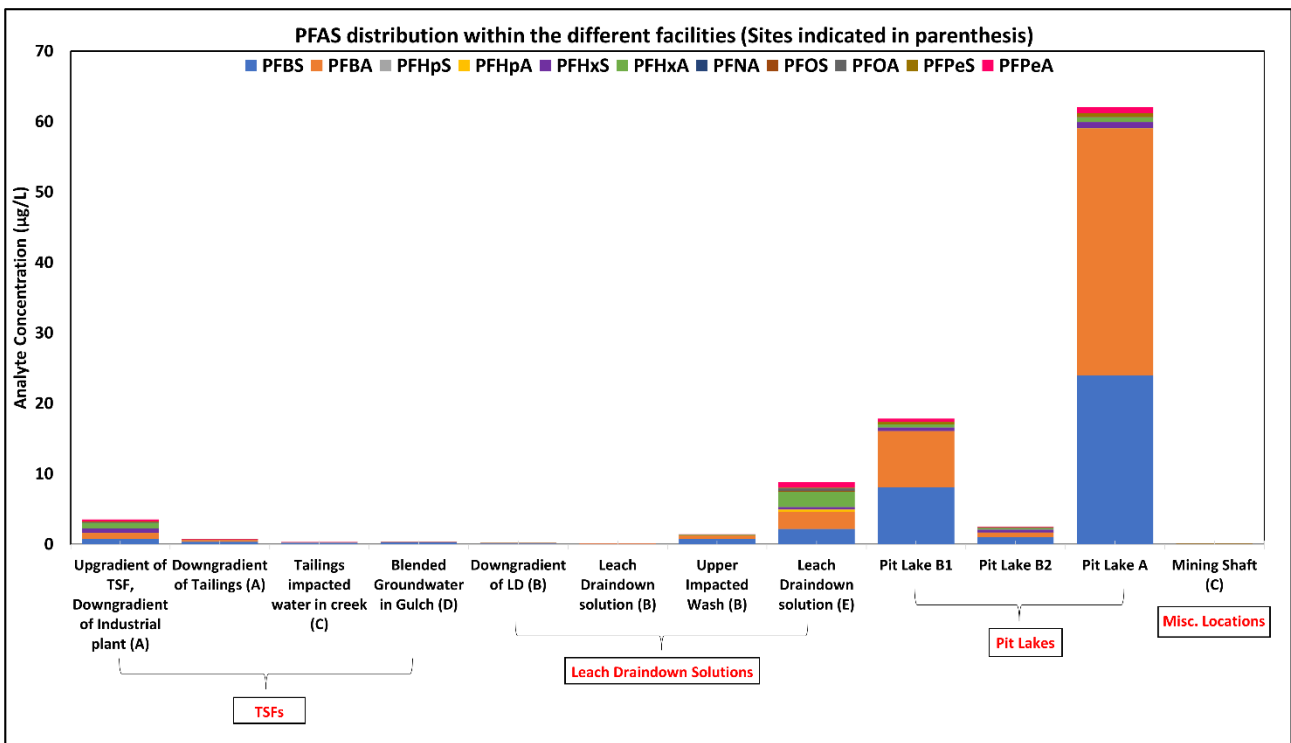


Figure 1 PFAS distribution according to mine facilities: water samples

Figure 2 and Figure 3 show the results of this study with respect to a comparative depiction of these analytes against four national and international guidelines. EPA has only proposed MCLs for drinking water (PFOS and PFOA). Among the 30 states in the USA that have published the guidelines restricting PFAS in water, Hawaii (regulating body: HDOH) has the most stringent guidelines. For this study SRK used the strictest guidelines including EPA, HDOH, Canadian (Health Canada), and Australian (HEPA). Nearly all the sample results exceed the HDOH and proposed EPA MCL for PFOS of 0.004 µg/L; only pit A exceeds the EPA screening level of 0.04 µg/L for PFOS. The Health Canada limit is listed but not graphed.

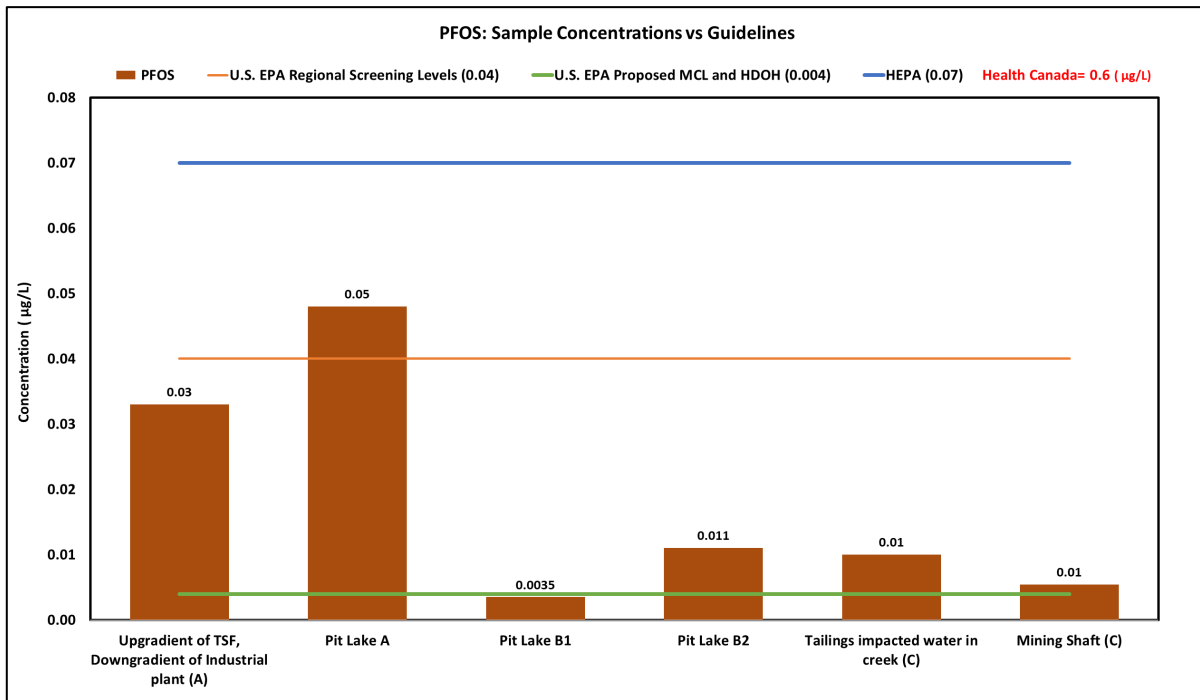


Figure 2 PFOS concentrations in water samples

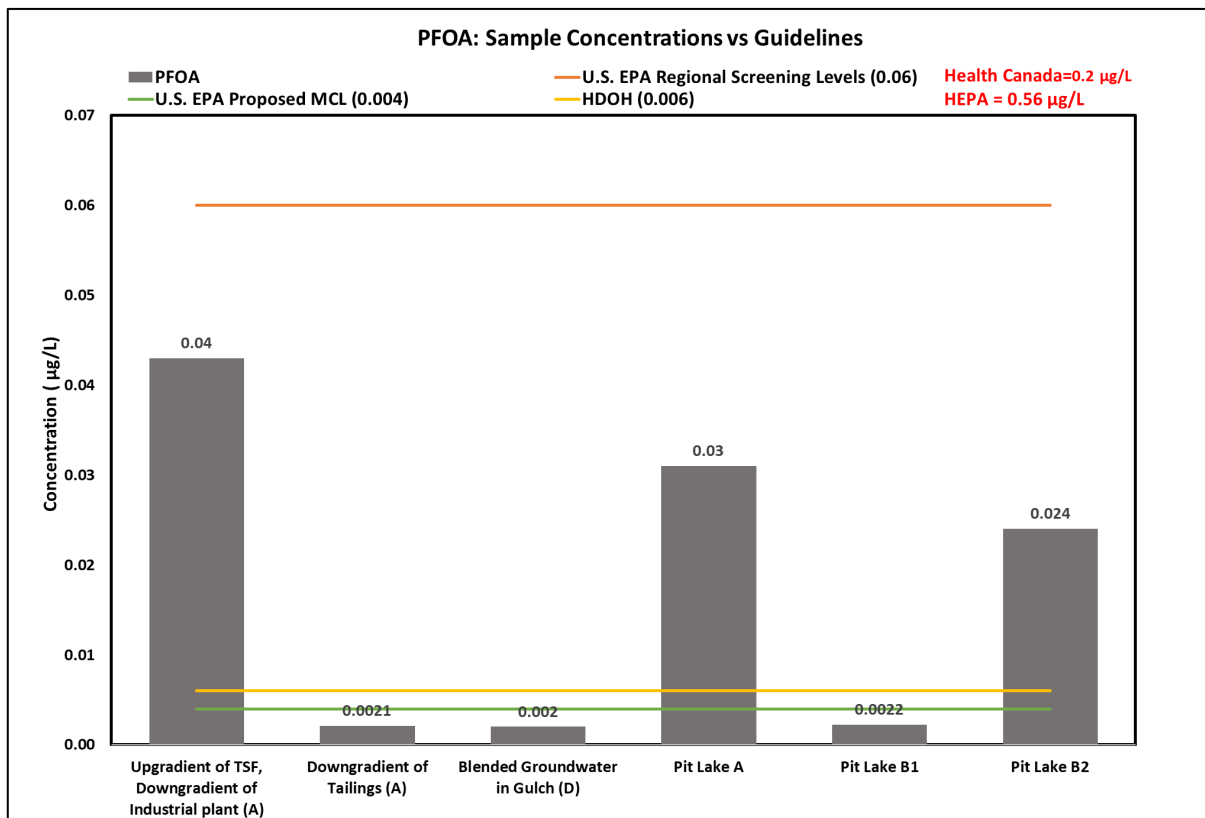


Figure 3 PFOA concentrations in water samples

Figure 3 shows all samples in which PFOA was detected. The HDOH and EPA proposed MCLs for PFOA are 0.006 and 0.004 µg/L, respectively. Samples from the pit lakes (A and B2) and the well that is upgradient of a TSF and downgradient of the neighbouring industrial plant site exceed those guidelines. The Health Canada and HEPA limits are listed but not graphed.

3.2 Solid samples

Figure 4 represents the PFAS concentrations ($\mu\text{g}/\text{kg}$) in solid samples. The soil taken near the former raffinate pond at site F has the highest solids concentrations. At a depth of range of 2.74 to 3.05 m bgs, the concentrations of PFOS and PFOA measured 21 and 23 $\mu\text{g}/\text{kg}$, respectively. Most of the locations at Site A, Site B, and Site C show presence of short-chain PFAS (i.e., PFBS and PFBA). PFAS were not detected at Site E. Site F shows presence of mainly long-chain PFAS (i.e., PFOA and PFOS).

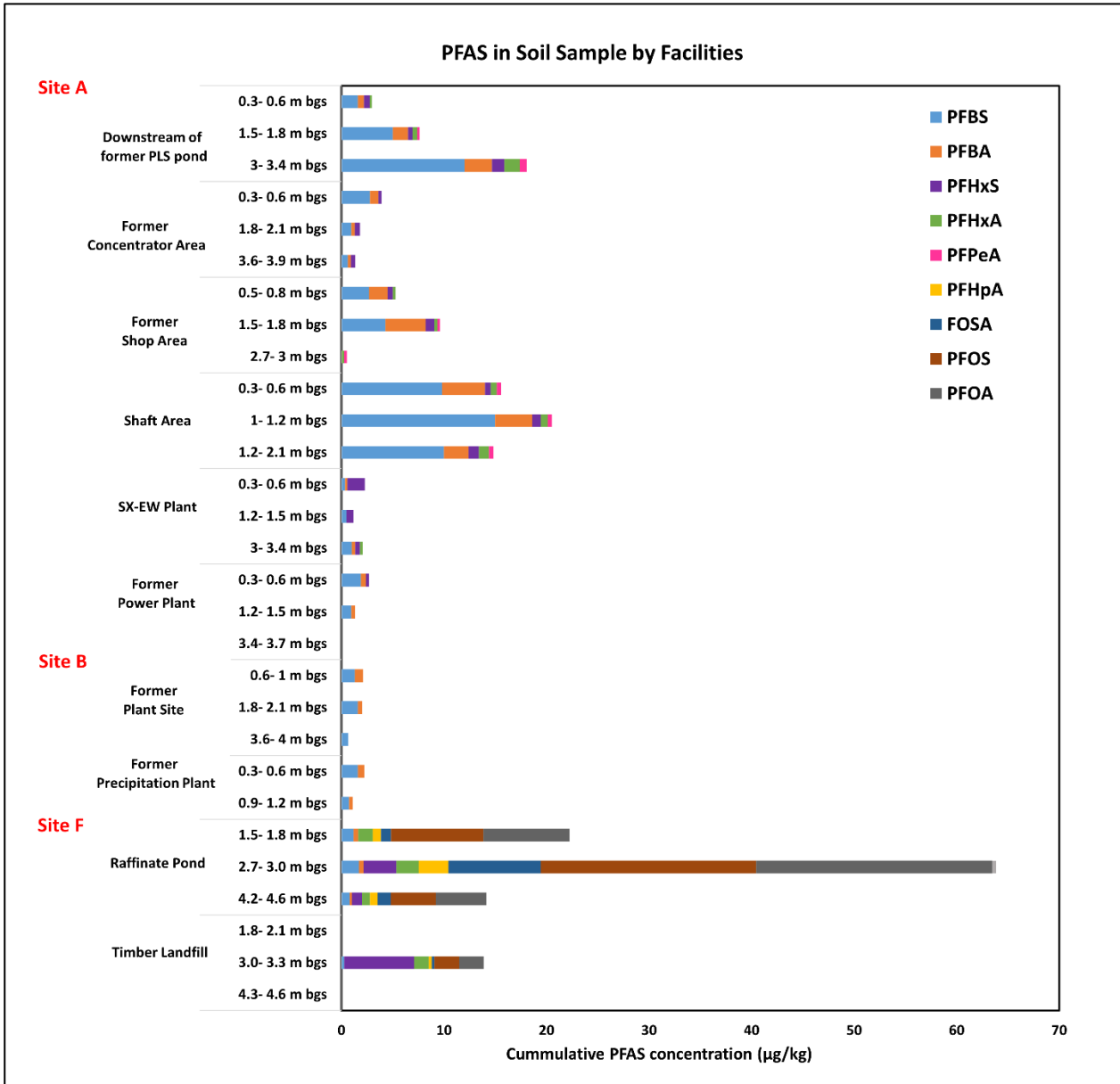


Figure 4 PFAS distribution according to mine facilities: solid samples

The tailings at Sites A, C and D measured short-chain PFAS compounds only (PFBS, PFBA and PFHxA) (Figure 5). No PFAS were detected at Site E for tailings samples. The concentration of PFAS in tailing samples is significantly lower than those observed in the facilities presented in the Figure 4.

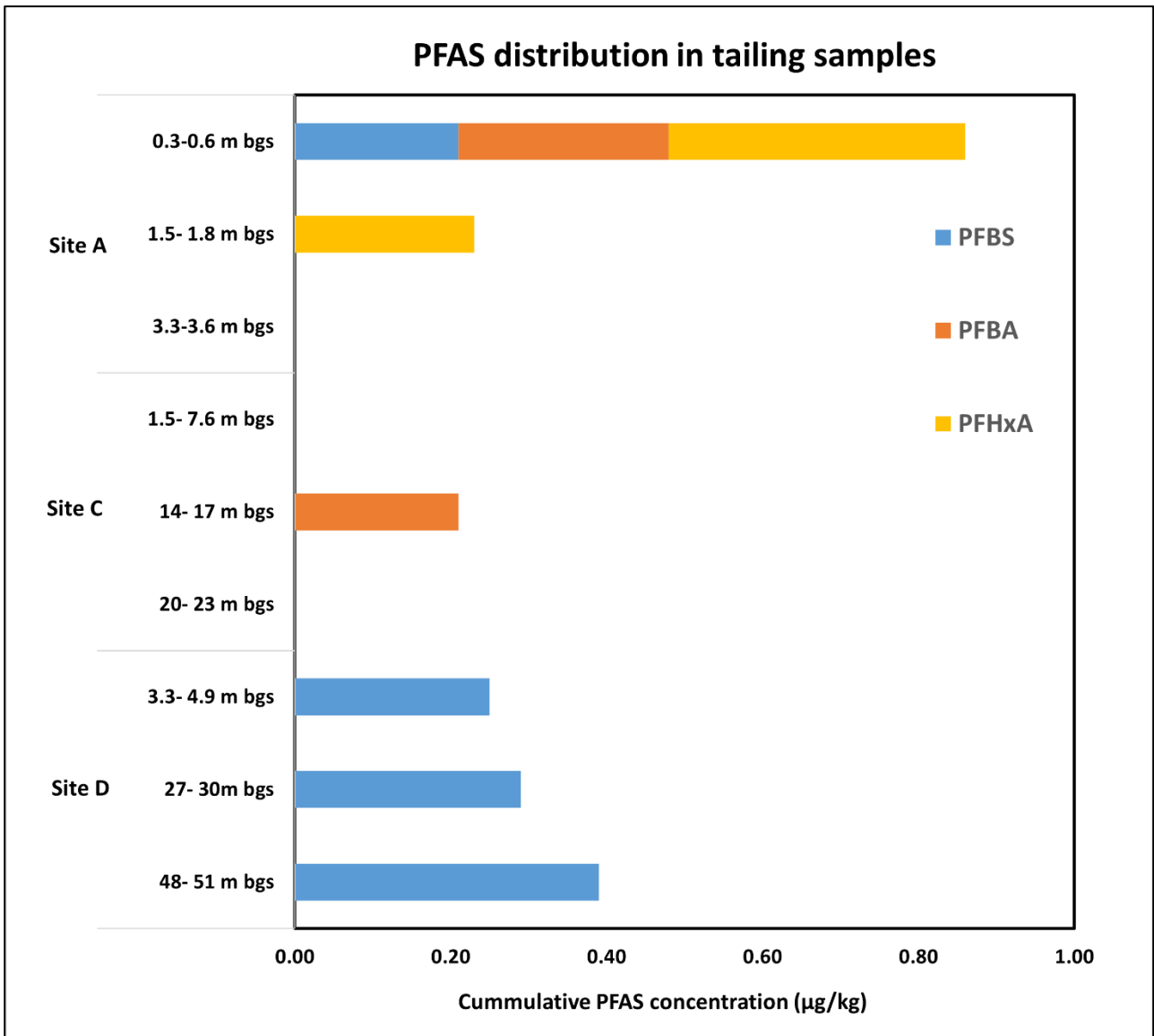


Figure 5 PFAS distribution in tailings samples

The strictest reference guidelines used in this study (HDOH, Canadian and Australian) are also applicable for soil (Figure 6, Figure 7). PFBS and PFHxS were selected to be compared with their respective guidelines because they are present in most of the solid samples.

PFBS (Figure 6) concentrations in most of the samples represented here exceed EPA USA regional screening levels (1.9 µg/kg) and HDOH (3.1 µg/kg). The Health Canada limit is listed for reference but not graphed. The locations downstream of the former PLS pond and shaft area were found to have the highest PFAS concentrations.

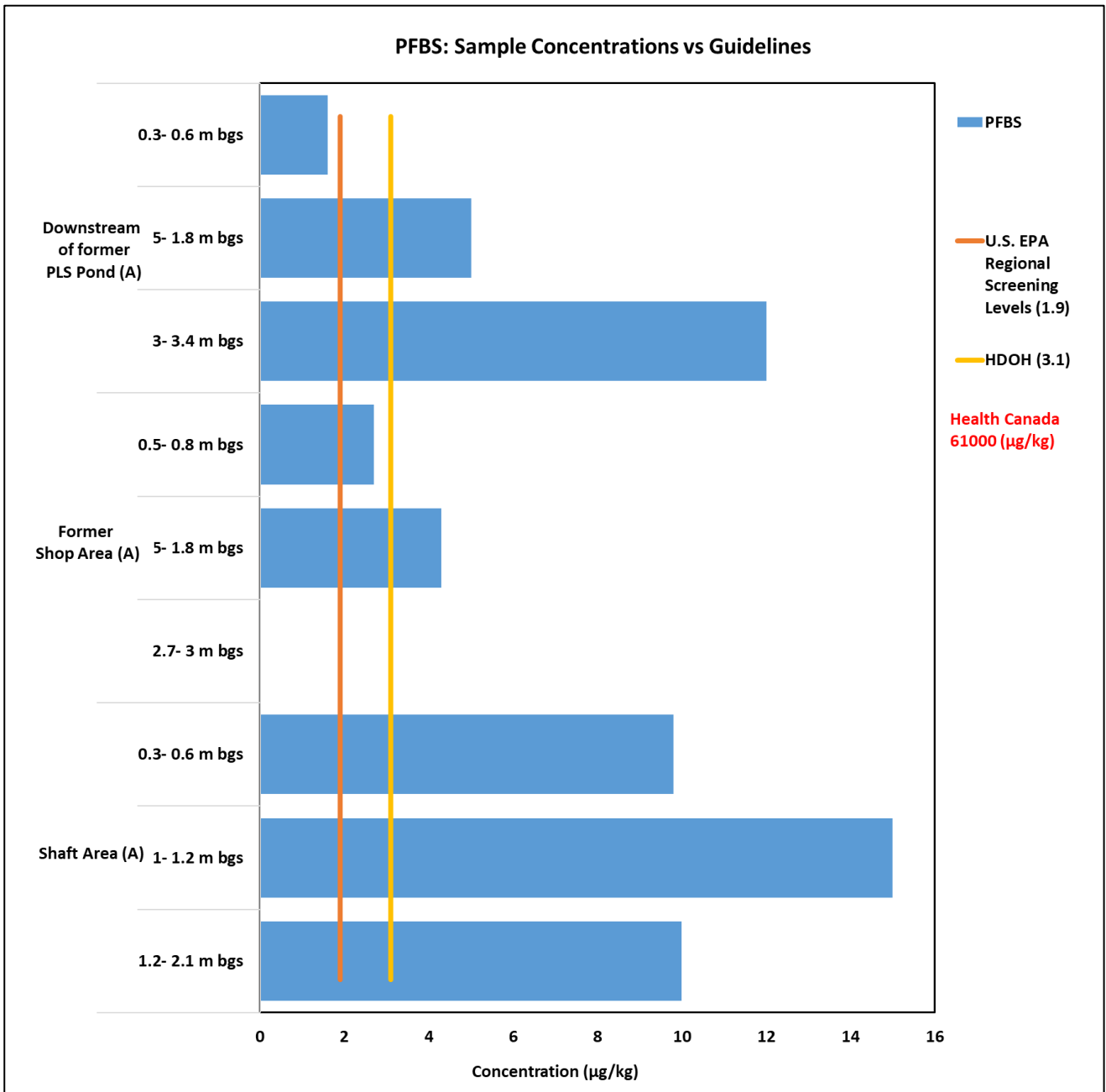


Figure 6 PFBS concentration in solid samples

Figure 7 compares PFHxS present in the samples against reference guidelines. The EPA regional screening level, HDOH limit, and HEPA limit in soil are 0.17 µg/kg, 3.7 µg/kg, and 0.01 µg/kg, respectively. The samples depicted in Figure 7 are from Site A and were chosen given the high concentration of PFHxS observed. Most samples from Site A exceed both EPA regional screening levels and HEPA. HDOH and Health Canada limits are listed but not graphed.

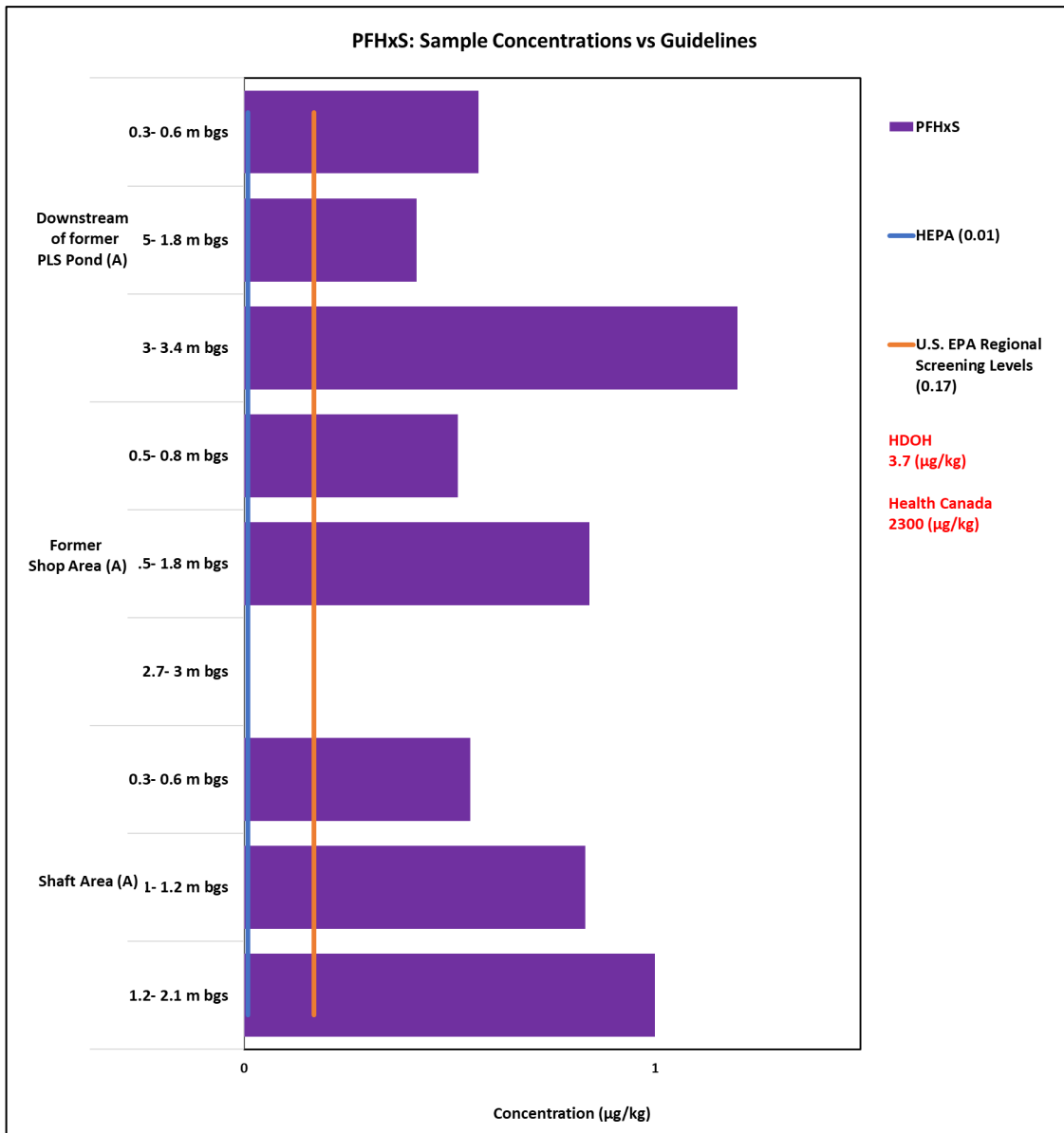


Figure 7 PFHxS concentration in samples vs guidelines

4 Conclusion

Mine closure studies have not typically evaluated the impact to groundwater and soils from PFAS compounds. Until now, these compounds have not been considered in baseline groundwater characterization programs prior to and during operations. Awareness is growing among mining operators about the risk these compounds have to the public and environment, and the need to characterize the potential presence and identify sources so they can be documented as a baseline condition, if present prior to operations, and fully addressed, as needed, during remedial closure designs. In advance of potential regulatory changes, it is important to update corporate risk registers to identify the potential change in regulatory standards. PFAS assessment at legacy sites is challenging given the age of information about specific chemical products and quantities and locations historically used.

This study was completed to assess the presence PFAS in six mining legacy sites based on the client’s desire to perform a voluntary, internal investigation. The study included groundwater, process water, and soils associated with inactive TSFs, pit lakes, leach dumps, former processing plants, and ancillary infrastructure (i.e., lube shops, truck maintenance shops, mine shaft areas). Analytical results broadly correlate with the

operational age of the facilities. The highest concentrations were noted in the most recently operated site (Site A). The site operated before 1930s (Site C) shows the lowest concentrations.

The results showed groundwater and soil samples related to TSFs measured low concentrations of PFAS, usually below the reference guidelines used for comparison purposes. Water samples collected downstream of former leaching facilities and spent leach process solution samples showed concentrations above the 2023 MCLs established for PFOS and PFOA by the EPA. Soil samples collected downstream of former PLS and raffinate ponds also exceeded reference EPA screening criteria.

The water samples collected from the three pit lakes presented concentrations generally exceeded all standards used for comparison purpose for PFOS and PFOA; the pit lake water in Pit A and Pit B1 consist of a mixture of stormwater runoff, residual leach processing solutions, precipitation, and inflowing groundwater.

Concentrations of short-chain PFAS in most of the soil samples near miscellaneous infrastructure including former power plants, processing plants, shaft areas, lube shops and truck shops were observed to be above at least one of the guidelines used for comparison purposes.

Presently, there are regulatory standards established for potable drinking water sources but none for water resources associated with mining and mineral processing facilities. There are limited soil standards set (i.e., EPA screening levels, Hawaii, Canada, Australia) for groundwater and surface water protection but no specific standards for industrial sites. PFAS regulations are evolving, currently focusing on public drinking water standards, but are expected to become stricter over time, expanding to other industries, including mining. The future mine operation focus will be to identify the existing impact to on-site and off-site receptors and to reduce and/or replace PFAS compounds going used in processing chemicals for PFAS-free chemicals.

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