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MEMORANDUM

To: James Fish— Environmental Program Specialist, Alaska Department of Environmental Conservation

From: Integral Consulting Inc.

Date: September 30, 2024

Subject: Work Plan Technical Memorandum— Fish Tissue Sampling and Analysis for PFAS REV 1, Williams Alaska Petroleum, Inc., Former North Pole Refinery, North Pole, Alaska

Project No.: CF2052

Integral Consulting Inc. (Integral) has prepared this Work Plan Technical Memorandum on behalf of Williams Alaska Petroleum, Inc. (Williams) to collect up to ten composite fish tissue samples for analysis of PFAS from the North Gravel Pit at the Former North Pole Refinery, North Pole, Alaska.

The need for this action was identified following detection of PFAS in sediment and surface water samples collected from North Gravel Pit in 2022, and the field confirmation that this water body also supports fish. This information was presented in a revised ecological conceptual site model submitted to ADEC in June of 2023 (Integral 2023).

FISH TISSUE SAMPLING

Williams' subcontractor, Shannon & Wilson, performed a qualitative fish survey under the direction of Integral using baited minnow traps at the Site in the fall of 2022. Angling was also completed within the gravel pits, but no fish were captured in either pit as part of this effort. The investigation activities determined that North Gravel Pit contained longnose suckers (*Catostomus catostomus*) and lake chubs (*Couesius plumbeus*). The traps did not collect any fish from the much smaller South Gravel Pit. Hence, the sampling effort described below will focus exclusively on North Gravel Pit.

Under the direction of Integral, Shannon & Wilson field personnel will deploy 10 baited minnow traps in shallow (< 5 ft. deep) water along the entire shoreline of North Gravel Pit.

Each trap will be securely closed with a clip to which is attached a length of rope and a small float. The traps will be evenly spaced around the pond using waders or a small boat. Approximate locations are provided on Figure 1, but they may change due to field conditions. If possible, the traps will be placed close to submerged structure (e.g., boulders, aquatic vegetation, sunken wood, overhanging branches, etc.) that are more likely to attract fish. Each trap will be baited with dog chow to lure in fish from the surrounding area. The traps will be submerged in the water for 24 hours. If the traps do not contain sufficient fish for a sample, the traps will be re-deployed for a second 24-hour interval to increase capture and success of the sample event. Each trap will be deployed for no more than two 24-hour intervals.

The field team will retrieve the traps one by one at the end of each 24-hour period. Based on the 2022 sampling effort, it is expected that the traps will catch longnose suckers and lake chubs. However, it is also possible that fish from additional species, which were missed in 2022, may also be collected. Regardless, no fish larger than about 100 mm are expected to be caught due to the relatively small entrance openings into the minnow traps.

All fish present in a minnow trap will be counted, measured (to the nearest mm), weighed (to the nearest 0.1 g), and keyed out to the lowest-possible taxonomic level (preferably down to species). Specimens will be composited per species and by trap location when possible. If insufficient mass is available for a species in a given trap, specimens will be composited among multiple traps by species with trap compositing identified and documented. Up to five composite samples will be targeted for two species (assume this will be longnose suckers and lake chubs) for a total of up to 10 composite fish tissue samples.

As directed by the laboratory, fish will be double bagged with Ziplock bags (confirmed PFAS-free) rather than wrapped in aluminum foil, as is typical. The double bagged samples will be placed on dry ice for shipping to the laboratory for tissue processing and PFAS analysis. The need for placing the fish on dry ice - instead of water ice - is predicated on the fact that it may take longer than 24 hours for the samples to reach the laboratory (see USEPA, 2024 for details).

Each sample bag will be labeled using a permanent marker to provide required information, such as sampling location, sampling date, and sample ID. All pertinent field information will be recorded in a dedicated field notebook and/or sample forms, as applicable. Photographs will be taken of trap deployment, representative fish, minnow traps, and sampling locations from around the North Gravel Pit. Field procedures will be conducted in accordance with Section 10 of ADEC's field sampling guidance (ADEC 2022a). Prior to sample collection, an Aquatic Resources Permit Application will be

submitted to the Alaska Department of Fish and Game (ADF&G) for approval to take fish for scientific purposes. If ADF&G requires any modification to the proposed sample collection methods, Integral will notify ADEC of the proposed change in protocols prior to sampling. A collection report will be submitted to ADF&G following sampling activities.

Sample Handling and Quality Control Sampling

The up to ten composite fish samples from North Gravel Pit will be collected and shipped with completed chain-of-custody documentation to an ADEC-certified analytical testing laboratory for tissue processing and PFAS analysis using USEPA Method 1633 as described in the Sample Processing and Analysis section below. One equipment blank will be collected from the tissue processing equipment in the laboratory for quality control purposes. One field duplicate will be collected if enough tissue mass is available from one of the composited fish samples. Every attempt will be made to prepare a representative duplicate in terms of species composition, size range, and number of individual fish compared to the original sample.

Off-site background or reference fish samples will not be obtained for this project.

Sample Processing and Analysis

Pace Analytical Services in Green Bay, WI will process the fish tissue samples prior to analysis. Each composite will be thoroughly blended (whole fish with skin) into a smooth and consistent paste. Between each composite sample, all the processing equipment will be thoroughly cleaned following the protocol outlined in USEPA (2024). The tissue paste from each composite sample will be placed in a 100-mL, wide-mouth, HDPE sample jar with liner-less HDPE or polypropylene caps (USEPA, 2024). The up to ten tissue samples will be labelled and then frozen until shipped out for analysis.

Pace Analytical Services in Minneapolis, MN will analyze the up to ten samples for 40 PFAS compounds using U.S. Environmental Protection Agency (EPA) Method 1633. Sampling and reporting will follow the ADEC guidance *Guidelines for Data Reporting Technical Memorandum 22-01* (ADEC 2022b).

The results of these analyses will be validated by EcoChem, who performed a compliance validation (USEPA Stage 2A). That validation will be based on the EPA National Functional Guidelines for Organic Data Review (USEPA 2017, 2020).

SCHEDULE/REPORTING

The fish sampling effort at North Gravel Pit will be completed in late September 2024. The tissue processing and analysis is expected to be completed by late November 2024. Following completion of field mobilization, fish sampling, tissue processing, and analytical activities, the findings of this work will be reported to ADEC in the first quarter of 2025 consistent with AAC 75.335 and project-specific requirements.

The tissue residue analytical chemistry data will be used to support a dietary dose model for estimating site-specific ingestion of PFAS by piscivorous wildlife (e.g., belted kingfisher, mink) foraging for fish at North Gravel Pit.

REFERENCES

ADEC. 2022a. Field sampling guidance. Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Program. January.

ADEC. 2022b. Guidelines for Data Reporting. Alaska Department of Environmental Conservation Division of Spill Prevention and Response Contaminated Sites Program. August.

Integral. 2023. Updated Ecological Conceptual Site Model Version 2 for Per-and Polyfluoroalkyl Substances at the Former North Pole Refinery, North Pole, AK. Integral Consulting Inc. June 2023.

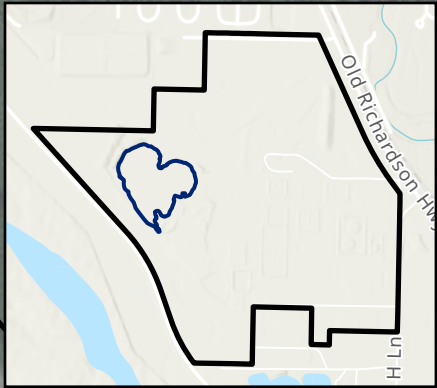
Integral. 2024. Updated Site Characterization Report for PFAS – REV1, Williams Alaska Petroleum, Inc., Former North Pole Refinery, North Pole, AK. Integral Consulting Inc. January 5.

USEPA. 2017. EPA Contract Laboratory Program national functional guidelines for organic Superfund methods data review. EPA-540-R-2017-002. U.S. Environmental Protection Agency, Washington, DC. January.

USEPA. 2020. EPA Contract Laboratory Program national functional guidelines for organic Superfund methods data review. EPA-540-R-20-005. U.S. Environmental Protection Agency, Washington, DC. November.

USEPA. 2024. Method 1633: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. EPA 821-R-24-001. January.

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Legend:

- Proposed Fish Sampling Location
- North Gravel Pit
- Site Boundary

Aerial Source: Esri, Maxar (2021)

0 100 200
Feet

N

Figure 1.
Proposed Fish Sampling Locations

DEC comments to Work Plan Technical Memorandum --- Fish Tissue Sampling and Analysis for PFAS, Williams Alaska Petroleum, Inc., Former North Pole Refinery,
 From Integral Consulting, Inc. to James Fish, ADEC; August 16, 2024, and

DEC comments to Work Plan Technical Memorandum --- Onsite Soil and Groundwater Characterization, Williams Alaska Petroleum, Inc., Former North Pole Refinery,
 From Integral Consulting, Inc. to James Fish, ADEC; August 16, 2024

Num.	Page	Paragraph or Section	Comment	
Fish Tissue Sampling Work Plan Comments				Response
1	2	2 nd	The text states that the minnow traps will be retrieved after 24 hours. If the traps do not contain sufficient fish for a sample, DEC requests re-deploying the traps to increase capture and success of the sample event.	The text has been updated to state that the exercise will be repeated (i.e. the trap will be deployed for a second 24-hour interval) if sufficient fish are not captured for a sample.
2	2	3 rd	The text states, “All fish present in a minnow trap will be counted, measured (to the nearest mm), weighed (to the nearest 0.1 g), and keyed out to the lowest-possible taxonomic level (preferably down to species). All fish captured by a given trap will be combined across species with all the fish captured by an adjacent trap to create up to five composite samples representing five equal sections of the North Gravel Pit.” Please clarify the following: <ul style="list-style-type: none"> ○ Please provide more information (preferably including a figure) regarding the planned distribution of the 10 minnow traps in the North Gravel Pit and determination of the “five equal sections of the North Gravel Pit.” 	Approximate trap locations are provided on Figure 1. As noted in the text, trap locations will be placed close to submerged structures (e.g., boulders, aquatic vegetation, sunken wood, overhanging branches) that are more likely to attract fish, where possible. Regarding compositing, the text will be revised to: “Specimens will be composited per species and by trap location when possible. If insufficient mass is available for a given trap after 48 hours, specimens will be composited among multiple traps by species. Up to five composite samples will

			<ul style="list-style-type: none"> ○ Will the description of “adjacent” traps for fish compositing be pre-determined based on trap location or determined in the field based on capture or other factors? ○ Will the composite samples be species-specific or a combination of species? <ul style="list-style-type: none"> ○ If not species-specific, how will variability between adjacent traps be addressed when compositing the fish? Will fish species or size or other variables be considered when determining which fish to composite together? 	<p>be targeted for each species (longnose suckers and lake chubs) for a total of up to 10 composite fish tissue samples.”</p>
3	2		<p>In addition to the minnow traps, please consider using a fyke net or gill net set from shore to collect fish – these can still be set with a small unmotorized boat or canoe. This will potentially allow capture of fish > 100mm in length. It is possible that larger fish may have a different body burden of PFAS. If larger fish are captured, they could be composited separately from the smaller fish and/or organ-specific PFAS analysis may also be possible. Please comment.</p>	<p>If sufficient tissue mass is not collected with minnow traps, additional methods will be assessed for future sampling events.</p>

Soil and Groundwater Characterization Work Plan Comments				Response
1	2	3 rd paragraph	<p>Please confirm how the middle soil sample interval for the soil borings will be established to clarify the text description, “one from the 6-in. interval corresponding to the midpoint of the soil boring.”</p> <ul style="list-style-type: none"> ○ How will “the 6-in. interval corresponding to the midpoint of the soil boring” be determined? Since the soil boring depth will vary based on the water table depth at each location, how will this depth be known before the soil boring is advanced? 	<p>Generally speaking, groundwater is relatively shallow at this time of year, between 5.5 to 7.5 feet bgs, so the approximate midpoint can be estimated prior to advancing the boring. That estimate was used to make protocols based on the type of tools used (hand clearing tools or macrocore). Text has been added to clarify these protocols.</p>
2	3	Sample Handling and Quality Control Sampling	<p>The text states that, “Field quality control samples will be collected at a minimum of 1 field duplicate per every 20 samples and 1 field equipment blank per sampling team/equipment per day per the ADEC guidance document referenced above.”</p> <p>However, the ADEC guidance requires a minimum of 1 field duplicate per every 10 samples. Please revise.</p>	<p>The correct number of duplicates was completed during field sampling (1 per 10 samples). The text has been revised.</p>
3	3	Schedule/Reporting	<p>While DEC understand some sampling has occurred already, the text states that “The sampling will be completed in late July 2024.” Please confirm whether the sampling has already occurred or provide a revised sample schedule if additional sampling will occur.</p>	<p>The sampling was completed successfully between July 23-28, 2024.</p>