

APPENDIX A: INNOTECH ALBERTA 2020 CONTRACT RESEARCH REPORT



Petroleum Hydrocarbon Fractions 2 and 3: Evaluation of Management Limits

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EXECUTIVE SUMMARY

In the Alberta Tier 1 guidelines, generic management limits for PHC F2 and F3 can often become limiting and potential drivers for remediation in the case that the eco-contact exposure pathway can be eliminated (*i.e.*, 1.5 m below ground surface). Based on a previous phase of this project, it was recommended that empirical testing be conducted to validate generic inputs for two factors that are used in calculating PHC F2 and F3 management limits: 1) potential exposure of workers in trenches to PHC F2 vapours, and 2) potential effects of PHC F2 and F3 on buried infrastructure.

To generate data for modelling vapours that could potentially accumulate in a trench, experimental testing was conducted to assess volatilization of PHC F2 alone and when spiked into fine- and coarsegrained soils with moisture levels representative of potential *in situ* conditions. Method development was conducted to measure both total PHC F2 vapours in headspace above distillate and spiked soil, as well as specific sub-fractions in the PHC F2 range (>nC₁₀-nC₁₆), including aliphatic and aromatic compounds in both the nC₁₀₋₁₂ and nC₁₂₋₁₆ ranges. Headspace concentrations were provided to MEMS as input parameters for trench models. Vapours from PHC F3 were not tested as concentrations were assumed to be very low given low volatility of compounds in the >nC₁₆-nC₃₄ range.

Potential effects of PHC F2 and F3 on buried infrastructure, including water pipe and fibre optic cables, was assessed through a literature review followed by piping material incubation in PHC F2 and F3 distillate, and testing of water within one type of piping wrapped in PHC F2-soaked batting.

The literature review found that fibre optic cables are generally buried at depths shallower than the zone of interest for this project. Water piping, however, is buried a minimum of 2.59 m below curb top to avoid freezing, and is therefore at a depth below the eco-contact exposure pathway. It was found that polyvinyl chloride (PVC), high density polyethylene (HDPE) and fibre-reinforced plastic (FRP) are the most common polymer types used for water pipe, with PVC being the most common.

Representative pipe material (PVC, HDPE and FRP) was obtained with an inner diameter of 150 mm (6 in) and thickness required to withstand 150 kPa pressure (>1 cm). Coupons (4 cm²) were cut and incubated for 80 days in pure PHC F2 and F3 distillate with water and no incubation as a control. Post-incubation, the coupons were weighed, tested for hardness and thickness, and examined under 10X magnification. Based on the average and standard deviation of three replicates, the majority of pre- and post-incubation metrics were not significantly different, with exception of the weight of HDPE incubated in PHC F2 (increase) and hardness decrease in PVC following incubation in both PHC F2 and F3. Notable differences could not be detected *via* observation of the coupons, with or without magnification.

Samples of water were taken from inside PVC pipe wrapped in PHC F2-soaked batting after 80 days. It was found that concentrations of PHC F2 in the water exceeded the Alberta Tier 1 groundwater remediation guideline of 1.1 mg/L, with an average concentration of 1.97 mg/L and with an elevated concentration (2.2 mg/L) in the control that was never exposed to PHC F2 distillate. Examination of chromatograms led to the conclusion that the source of PHC F2 was not distillate, but likely a chemical resulting from adhesive or, less likely, the PVC itself.



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Petroleum Hydrocarbon Fractions 2 and 3: Evaluation of Management Limits

SIMONE LEVY

1.0 INTRODUCTION

1.1 Background

In 2019, Millennium EMS Solutions Ltd. (MEMS) was contracted by PTAC-AUPRF to complete the second phase of a 2018 PTAC project (18-RRRC-08) re-evaluating the management limits for F2 and F3 petroleum hydrocarbons (PHCs; MEMS, 2019). Two sets of management limits for PHC fractions F2 and F3 are currently applicable in Alberta; a generic set in the Alberta Tier 1 Soil and Groundwater Remediation guidelines (Tier 1; AEP, 2019) applicable to all land uses, and another set specific to remote parts of the province in the Green Area (AESRD, 2014). The generic management limits for PHC F2 and F3 can often become limiting and potential drivers for remediation in cases where the eco-contact exposure pathway can be eliminated.

Management limits for PHC fractions indicate concentrations where factors other than toxicity, such as aesthetics, mobile free-phase formation and explosion hazards may be of concern. The report from Phase 1 of this project recommended further investigation of the data supporting two of the retained factors: 1) potential exposure of workers in trenches to PHC F2 vapours and 2) potential effects of PHC F2 and F3 on buried infrastructure. The activities described herein were designed to provide additional data for those two factors for re-calculation of the management limits.

2.0 PART 1: EXPOSURE OF WORKERS IN TRENCHES TO PHC F2 VAPOURS

The CCME (2008) limits for exposure of workers in trenches were established based on a trench vapour model published by VDEQ (2005); however, the model and key default inputs do not appear to have been validated against real-world measurements (MEMS, 2019). Bench-scale experiments described here were designed to define input concentrations for the air mixing models within the air space of a utility trench. Input parameters for the models require both concentrations of PHC F2 and four PHC F2 sub-fractions: C_{10} - C_{12} aromatic compounds; C_{10} - C_{12} aliphatic compounds; $C_{>12}$ - C_{16} aromatic compounds, and $C_{>12}$ - C_{16} aliphatic compounds. Testing was not conducted with PHC F3 distillate due to low volatility of its subcomponents.

Activities conducted in Part 1 of the project included:

- Characterization of pure PHC F2 distillate by gas chromatograph mass spectrometer (GC-MS) and flame ionization detector (GC-FID);
- Method development and range finding for headspace equilibrium setup;
- Testing headspace at equilibrium with pure PHC F2 distillate; and





• Testing evolution of PHC F2 vapours from coarse- and fine-grained soils spiked with PHC F2 distillate, with various moisture levels.

2.1 PHC F2 Distillate Characterization

2.1.1 Methods

A sample of previously-distilled PHC F2 was tested by injecting a liquid sample into a gas chromatograph – mass spectrometer (GC-MS; 25 m 0.25 mm column 0.25 μ mMS5 film with single quad mass spec analyzer (Agilent brand)). Using the apex of nC₁₀, nC₁₂ and nC₁₆, the aliphatic and aromatic concentrations of nC₁₀-nC₁₂ and nC₁₂-nC₁₆ were calculated using the Robinson whole oil method¹ (Robinson, 1971). This method is a baseline technique to resolve the mass spectrum into saturates and aromatic spectra in a sample within boiling point range of 200 to 1,100 F°. The method allows determination of up to 4 saturated and 21 aromatic compound types. It uses the low-resolution mass spectrum and the number average molecular weight, obtained from distillation data, of the unseparated sample.

The apex at each of nC_{10} , nC_{12} and nC_{16} was identified on the chromatograph to establish the concentration of PHC F2 parameters in distillate, as some compounds were present outside the C_{10} - C_{16} range.

The cut point and boiling point of the PHC F2 distillate were assessed following ASTM D2887 methodology (ASTM, 2019).

2.1.2 Results

The chromatogram provided in Figure 1 shows the distribution of components within the PHC F2 distillate. Most components (93.31%) fall between the apices of nC_{10} and nC_{16} , with approximately 3.63% below the apex of nC_{10} and 3.05% beyond the apex of nC_{16} (Table 1). Aliphatic and aromatic sub-fractions within total PHC F2 in the two desired ranges (C_{10-12} and C_{12-16}) were also determined and are provided in Table 1. Appendix A includes full analysis in the form of cut points and boiling points for PHC F2, analyzed at inception of this project, and historical (2012) analyses of PHC F3.



¹. The method is known as the Robinson Whole Oil Method because in contrast to many other methods (such as D2786 or D3239) it does not require a prior separation of a sample into saturates and aromatic fractions. The method reports four saturated hydrocarbon types, twelve aromatic HC types, three thiophenic and six unidentified aromatic groups.



Figure 1. Analysis of PHC F2 distillate and indication of peak apex categories.

Table 1. Total PHC F2 hydrocarbon and sub-component weight % fraction in pure PHC F2 distillate, by apex to apexgrouping (see Figure 1).

Component or sub-fraction	<c10 apex<="" th=""><th>пС₁₀ арех - пС₁₂ арех</th><th>nC₁₂ apex - nC₁₆ apex</th><th>>nC₁₆ apex</th></c10>	пС ₁₀ арех - пС ₁₂ арех	nC ₁₂ apex - nC ₁₆ apex	>nC ₁₆ apex
	Wt%	Wt%	Wt%	Wt%
Total hydrocarbons	3.63	32.25	61.06	3.05
Aliphatic sub-fraction	2.2	21.8	38.9	1.6
Aromatic sub-fraction	1.4	10.4	22.2	1.5

2.2 Headspace method development and range finding

2.2.1 Methods

Three activities were conducted as part of method development and range finding, in support of the soil incubation experiments. All experiments were conducted at room temperature (~21°C), and the petri dish in Tedlar[®] bag setup was developed to maintain atmospheric pressure.

2.2.1.1 Headspace vapour distribution

To establish a maximum potential headspace concentration of PHC F2, 3 mL of PHC F2 distillate was placed in a petri dish inside a re-sealable 3L Tedlar[®] bag. The bag was evacuated by a vacuum and then filled with 1,500 mL of laboratory-grade air. The petri dish lid was removed, and the bag was incubated at room temperature for one week after which it was deemed that a maximum headspace concentration had been reached in the bag. A headspace sample was analyzed by removing 100 mL of headspace gas into a set of two thermal desorption tubes. The samples were analyzed *via* GC-FID. The results were used to establish 1) the ideal sample extraction volume for testing PHC F2 vapours while meeting required detection limits, and 2) optimizing capacity and number of thermal desorption tubes.



2.2.1.2 Headspace saturation above pure PHC F2

The second task in method development was designed to establish an ideal ratio of air to PHC F2 distillate for subsequent testing. Pure PHC F2 distillate (1.5 mL, approximate volume of PHC F2 in soil at 10,000 ppm in subsequent tests) was placed into a petri dish inside each of three sealed 3L Tedlar[®] bags. Each bag was evacuated by a vacuum and then filled with either 300 mL, 500 mL or 800 mL of laboratory-grade air. Petri dish lids were removed, and the bags were placed in a fume hood at room temperature (~21°C) for one week. Three thermal desorption tube sets (*i.e.*, one desorption tube, in line with a second one in case of breakthrough) were used to collect a 50 mL headspace gas sample. Samples were run on the GC-FID to determine the concentration of total hydrocarbon in headspace. Results were compared to identify an ideal headspace volume to: 1) ensure saturation at steady state, 2) leave space in the bag to allow expansion, and 3) to facilitate removal of the petri dish lid, which can be hindered with too small a bag or insufficient headspace.

2.2.1.3 Method reproducibility

Three replicate tests were set up using 1.5 mL of pure PHC F2 distillate in a petri dish inside a 3L Tedlar[®] bag with 500 mL of air injected following evacuation. After equilibrium was reached, 3 sets of thermal desorption tubes were collected from each bag. Average equilibrium concentrations (*e.g.*, total PHC and sub-fractions) were calculated from 3 replicated tests and will be used as input to the designated trench model for comparison against the exposure limits set out in Table C7 of the Tier 1 guidelines document (AEP, 2019): 1.0 mg/m³ for aliphatic compounds and 0.2 mg/m³ for aromatic compounds. Replicates of identical setup were also used to determine sample and method repeatability *via* standard deviation.

2.2.2 Results

2.2.2.1 Headspace vapour distribution

Based on the equilibrium testing setup described in section 2.1.2.1, the chromatogram output was overlain on the chromatogram of distillate analysis (Figure 2). The vapour above the F2 fraction does not have the same distribution as the original distillate due to higher volatility of parameters within the lower carbon range (C_{10} - C_{12}). As would be expected, the components below n C_{10} appear in the vapour phase and will affect the equilibrium of the F2 fraction.







2.2.2.2 Headspace saturation above pure PHC F2

The bags with differing headspace volumes (300, 500 and 800 mL) all had the same concentration and distribution of components (Figure 3). Based on this finding, it can be interpreted that the headspace was saturated with PHC F2 vapour, irrespective of the headspace volume. A headspace volume of 500 mL was recommended for subsequent tests based on slightly higher concentrations than 300 or 800 mL.





Figure 3. Overlain chromatograms of PHC F2 distillate in petri dishes within Tedlar[®] bags with differing volumes of injected headspace after complete evacuation.

2.2.2.3 Method reproducibility

The results in Table 2 show expected method deviations under ideal bench-scale setup for saturation with pure PHC F2 distillate. These results can be compared with results obtained from volatilization of PHC F2 in soil to determine whether variability between replicates is within an acceptable range.

 Table 2.
 Total PHC F2 concentrations in headspace at steady state, with breakdown of aliphatic and aromatic components within C10-12 and C12-16 ranges.

	PHC F2		C ₁₀₋₁₂		C ₁₂₋₁₆			
Rep	(C ₁₀₋₁₆)	Total GC- FID	Aliphatic	Aromatic	Total GC-FID	Aliphatic	Aromatic	
1	1.328	1.230	0.757	0.473	0.098	0.047	0.051	
2	1.287	1.193	0.735	0.458	0.094	0.046	0.048	
3	1.432	1.323	0.810	0.513	0.109	0.052	0.057	
Average	1.349	1.249	0.767	0.481	0.100	0.049	0.052	
St. Dev	0.067	0.067	0.039	0.028	0.008	0.003	0.005	



2.3 Testing evolution of PHC F2 vapours from spiked soils

2.3.1 Methods

2.3.1.1 Setup and soil characterization

Setup photographs of spiked soil within petri dishes in Tedlar[®] bags are provided in Figure 4. Previously characterized coarse- and fine-grained soils were spiked with the PHC F2 distillate to a target of 10,000 mg/kg. The exact concentration was difficult to achieve, especially in coarse-grained soil, as some of distillate intended for spiking became adhered to the mixing container. Several attempts were made to accurately spike the soils. All work was conducted in a -14°C walk-in freezer to minimize losses through volatilization. Three representative samples of each batch of soil were taken and submitted for laboratory analysis to confirm PHC F2 concentrations and assess variability in each batch.



Figure 4. Experimental setup for spiked soils including mixing distillate into soil, weighing once in petri dish for density calculation, placement into Tedlar[®] bag, and evacuation.

A) spiking soil in a refrigerated vessel, within a -14°C walk-in freezer; B) weighing tailings in 100 mL glass petri dish; and C) Evacuating air from bags prior to adding known amount of laboratory-grade air.

Once spiking concentrations were in the desired range, the soils were moistened to representative field conditions. Coarse-grained soil with limited moisture holding capacity was moistened to approximately 1.5% by weight. Two moisture levels were created with fine-grained soil to represent soil in the unsaturated (14%) and saturated (24%) zones. Four replicates of each soil was placed into a 100 mL glass petri dish and compacted to a target density: Alberta Tier 1 guidelines' default soil bulk density estimates for undisturbed samples for coarse and fine soil are 1.7 kg/L and 1.4 kg/L (dry soil basis), respectively. Lids were placed on the petri dishes, which were then inserted into re-sealable Tedlar® bags. Bags were clamped prior to removing air using a vacuum hose. Soil moistening was conducted in a 4°C walk-in refrigerator.

2.3.1.2 Headspace analysis

'Time 0' was defined as the time when the petri dish lids were removed in the bag after 500 mL of labgrade air was injected into each bag. One of the bags was monitored with a MiniRAE 3000 photoionization detector (PID) calibrated to isobutylene (Figure 5). The PID was used to monitor concentrations of volatile organic compounds (VOCs) in headspace. At steady state, or when the headspace VOC concentration



plateaued in the bag monitored by PID (*i.e.*, average concentrations within approximately 10% variability over a predetermined period), headspace samples were taken from the 3 replicate bags for analysis by flame ionization detector (total VOCs) and gas chromatograph (PHC F2 sub-fractions).



Figure 5. Photoionization detector attached to Tedlar[®] bag containing spiked soil. 1 of 4 replicates containing PHC F2-spiked soil at approximately 10,000 mg/kg.

Where replicate samples were taken, the average and standard deviation of the replicates were calculated and reported. In some cases the standard deviation was reported as a percent of the average to compare the variability of results between sample types.

Aliphatic and aromatic components were analyzed on GC-MS data using the Robinson method (Robinson, 1971).

2.3.2 Results

2.3.2.1 Setup and soil characterization

The final soil concentrations of PHC F2 and the moisture levels as reported by the laboratory are provided in Table 3. Given difficulties in spiking the soils, it was not possible to achieve a uniform concentration in all soils. However, based on headspace saturation tests conducted in the earlier part of the experiment, the starting concentration in soil does not need to be calibrated as headspace would be saturated regardless.

The time to peak PID reading and the reading itself are also reported, in the case that trench vapour modeling could incorporate information on time to 'steady state' in a closed system.

2.3.2.2 Headspace analysis

Results of headspace analyses, including total PHC F2, nC_{10-12} , nC_{12-16} , and their aliphatic and aromatic subcomponents, are provided in Table 3. Results will be used as model input parameters at which point interpretation can be made as to their impact.



Parameter or sub-component						Headspace concentration (µg/mL air)							
		Average							nC ₁₀₋₁₂			nC ₁₂₋₁₆	
Soil Type	Average soil moisture (%)	F2 (mg/kg) (Standard deviation; % standard deviation)	Average Density in Petri Dish Reps	Time to peak PID reading (mins)	Peak PID reading (ppm equivalent)	Replicate	PHC F2 (nC ₁₀₋ 16)	Total GC- FID	Aliphatic	Aromatic	Total GC- FID	Aliphatic	Aromatic
						Control	0.003	0.003	-	-	<0.001	-	-
		0 202		58	560	1	1.136	0.999	0.657	0.342	0.137	0.068	0.069
Coarse	1.43	9,393 (1,111; 11.8%)	1.52			2	1.048	0.937	0.614	0.323	0.111	0.055	0.056
course						3	0.887	0.804	0.527	0.277	0.083	0.043	0.040
						Average	1.023	0.913	0.599	0.314	0.110	0.055	0.055
						St. Dev	-	0.081	0.054	0.027	0.022	0.010	0.012
				122	479	Control	0.006	0.006	-	-	<0.001	-	-
		12 167				1	1.006	0.883	0.561	0.323	0.123	0.062	0.061
Fine	14 23	(499.	1.55			2	0.868	0.759	0.480	0.279	0.109	0.056	0.053
	1 1120	3.8%)		120		3	0.912	0.790	0.518	0.272	0.122	0.064	0.059
		,				Average	0.929	0.811	0.520	0.291	0.118	0.061	0.058
						St. Dev	-	0.053	0.033	0.023	0.006	0.003	0.003
						Control	0.002	0.002	-	-	<0.001	-	-
		C 772				1	0.280	0.231	0.078	0.153	0.049	0.021	0.028
Fino	24.40	6,//3 (153·	1 5 2	77	300	2	0.270	0.218	0.049	0.169	0.052	0.018	0.034
Tille	24.40	2.3%)	1.52	,,	333	3	0.314	0.248	0.067	0.181	0.066	0.026	0.040
		2.3707				Average	0.288	0.232	0.065	0.168	0.056	0.022	0.034
						St. Dev	-	0.012	0.012	0.011	0.007	0.003	0.005

 Table 3.
 Summary of soil and headspace results for petri dish within Tedlar[®] bag setup.



3.0 PART 2: EFFECTS OF PHC F2 AND F3 ON BURIED INFRASTRUCTURE

The previous review by MEMS also indicated a need for better understanding of the potential impact of PHC F2 and F3 hydrocarbons on buried infrastructure. The review and experiments described in this report aimed to identify the most common types of polymer used in buried infrastructure applications, and specifically those which might be buried at least 1.5 m bgs, such as water piping that is buried deeper to avoid impact in freeze-thaw cycles. Based on the review, the Part 2 testing program included obtaining representative material samples for testing potential material degradation when incubated in PHC F2 and F3 distillates, and potential impact to drinking water inside a typical pipe.

Activities conducted in Part 2 included:

- Literature review on buried infrastructure materials and depths;
- Incubation of piping material coupons in PHC F2 and F3 distillates; and
- Incubation of water in piping with a coating of PHC F2 distillate. •

3.1 Literature Review on Buried Infrastructure Materials and Depths

3.1.1 Methods

Innotech Alberta's Corrosion Engineering group, with direction from the Reclamation group as needed, conducted a literature review and consultation with third parties to inform potential experiments on the impact of hydrocarbons to water piping and fibre optic cable materials. The review was completed as follows:

- Engineering and literature sources were reviewed, and relevant third parties (e.g., TELUS, EPCOR) • were consulted for information on the characteristics of water piping and fibre optic materials – polymer types, range of wall thickness, installation practices (*i.e.*, depth, outer covering of fibre optic cables).
- For each polymer type, physical and chemical specifications (i.e., chemical compatibility, PHC diffusion rate, typical mechanical properties) were tabulated.
- Recommendations were provided as to potential testing of piping materials.

3.1.2 Results

March 2020

The full review is included in Appendix B; a short outline is provided here.

A representative of TELUS provided information on material and installation of fibre optic cables. They indicated that fibre optic cables are typically made of poly-steel-poly (PSP) cables, installed within conduits made of high-density polyethylene (HDPE) and occasionally polyvinyl chloride (PVC). Cables are generally installed between 1.1 and 1.5 metres below ground surface. Based on this burial depth above the zone of interest for this project (>1.5 m bgs), a decision was made not to pursue direct testing of the potential



impact of PHC F2 and F3 to fibre optic cables themselves; however, both HDPE and PVC were included for testing based on their prevalence in water piping.

Information on water piping was obtained through the Alberta Government's Standards and Guidelines for Municipal Waterworks, Wastewater and Storm Drainage Systems (Government of Alberta, 2012). The most common material types are PVC, HDPE and fibre-reinforced plastic (FRP), with PVC being the most common. Piping is generally buried a minimum of 2.59 m below curb top. The minimum inner diameter is 150 mm (6 in) based on a pressure rating of 150 kPa.

Chemical resistivity of PVC, HDPE and FRP were identified *via* literature sources. Select hydrocarbons were found to soften or swell the rigid structure of PVC, potentially weakening the structure to the point of allowing permeation of certain hydrocarbon types or components. Based on specifications provided by polymer manufacturers and the National Association of Corrosion Engineers (NACE International), resistivity ratings were established for PVC, HDPE and FRP. It should be noted that the testing temperatures for all three materials were generally at room temperature and above, with a minimum of 15°C. In many cases a lower chemical resistivity was reported at higher temperatures; however, in a water piping scenario, temperatures in soil below 2.5 m bgs would likely be lower than even 15°C.

Testing of the three most common material types (PVC, HDPE and FRP) *via* coupon incubation was recommended based on installation depths within the zone of interest (*i.e.,* below 1.5 m bgs) and potential susceptibility to hydrocarbon impact based on literature findings. Only PVC was used in the water incubation test.

3.2 Incubation of piping material coupons in PHC F2 and F3 distillates

3.2.1 Methods

Three polymer types were obtained from local suppliers, including polyvinyl chloride (PVC), high density polyethylene (HDPE) and fibre-reinforced plastic (FRP). Three replicates of each polymer type were cut into in 4 x 4 cm coupons, and were placed in sealed jars with pure PHC F2 and F3 distillates (Figure 6). Two controls were set up for each polymer type: no incubation and incubation in distilled water. After 80 days of incubation, coupons were removed from distillates and cleaned with a 1% Liquinox detergent solution. Surface changes were noted and photographs taken through a microscope at approximately 10X magnification. Weights (0.0001 g scale; \pm 0.0003 g) of each coupon were taken pre- and post-incubation coupon thickness was measured using calipers and results were compared from pre- and post-incubation. Hardness of all coupons was measured with a Shore D durometer, and incubated coupons were compared with equivalent controls (non-incubated and those incubated in water).

Where replicate samples were taken, the average and standard deviation of the replicates were calculated and reported.





Figure 6. Polyvinyl chloride (PVC), fibre-reinforced plastic (FRP) and high density polyethylene (HDPE) coupons (5 x 5 cm) incubated in (A) PHC F2 distillate; (B) PHC F3 distillate; (C) water.

3.2.2 Results

Results are presented including visual observation and empirical testing of coupons.

3.2.2.1 Visual observation and magnification of post-incubation coupons

Based on visual observations, including those under 10X magnification, it appears that incubation in PHC F2 and F3 noticeable impact on PVC, HDPE and FRP coupons (Figure 7).





Figure 7. Photographs of one representative coupon of each polymer type post-incubation in water, PHC F2 or PHC F3 distillate. Photos of coupons (top right) overlain on magnified image of same coupon.

3.2.2.2 Empirical testing of coupons to compare weight, thickness and hardness pre- and postincubation

Figure 8 shows the results of 16 cm² coupons incubated in PHC F2 and F3 distillates, water, and nonincubated. The difference in average weight and standard error (SEM) were compared. If the difference between the averages of the initial weight and final weight was greater than 1.96 SEM (p < 0.05) then the difference is considered statistically significant. HDPE exposed to PHC F2 showed a statistically significant difference in weight (3% and 2.6% increase in weight for after exposure and washed, respectively). This indicates that PVC may have F2 adsorbed onto the surface of the material. The other materials showed no reportable difference in weight. Standard errors were generally low (<10% of mean), and no significant trends are observed that would indicate the material was being altered through exposure to distillates.

The thickness of each coupon was measured with calipers after washing. The average, standard deviation (SD), and SEM were calculated for washed measurements. The difference in average thickness and standard error were compared. If the difference between the averages of the exposed coupon thickness and control was greater than 1.96 SEM (p < 0.05) then the difference is considered statistically significant. No coupons showed a reportable difference in thickness.



For Shore D hardness, each coupon was tested at 5 points after exposure (or control) and after washing. The average, SD, and SEM were compared to controls (and water exposed). If differences between F2 and F3 exposed were greater that 1.96 SEM (p < 0.05), the difference was considered statistically significant. PVC for both F2 and F3 showed a decrease in hardness (2.9% for F2 exposed and 5.4% for F3 exposed). The other materials showed no reportable difference in hardness.

Data corresponding to the graphs in Figure 8 is provided in Appendix C.





Figure 8. Graphs of average ± standard deviation (n = 3) for PVC (A-C), HDPE (D-F) and FRP (G-I) coupons incubated in PHC F2 and F3 distillates, water, and non-incubated (control).



3.3 Incubation of water in water piping with a coating of PHC F2 distillate

A second experiment aimed to determine whether water within piping could potentially be impacted by PHC F2; PHC F3 components were not tested as they are not soluble in water.

3.3.1 Methods

In the review described in 2.2.1, PVC was determined to be the most common polymer type for water piping. The minimum inner diameter of piping was 150 mm, with corresponding thickness of approximately 11 mm. The pipe was obtained and cut into 15 cm sections, which were then capped on both ends and sealed with Permatex Plastic Welder 2-part epoxy and silicone caulking, identified as a suitable sealant that would not leach hydrocarbons to the water and would not interact with PHC F2. The pipes were then wrapped in PHC F2-soaked cotton batting and covered in neoprene rubber. The incubation was carried out over 80 days. One water test from each of the 3 replicate pipes was submitted to Element Environmental Laboratories in Edmonton, Alberta, for analysis of PHC F2 concentration in water.



Figure 9. Cross section of PVC piping with ruler, showing piping diameter and thickness.



Figure 10. (A) Assembled piping incubation setup prior to installation of distillate-soaked batting and enclosure; (B) final setup including batting and closure.

3.3.2 Results

The results of water testing after incubation within PVC piping exposed to PHC F2 show that all parameters are below appropriate drinking water guidelines with exception of PHC F2 in one control and two out of



three replicates exposed to PHC F2 (Table 4). Based on internal quality control protocol, it is not possible that samples were mixed up during sampling (*i.e.,* control swapped with 'PHC F2 exposed water column 2'), and the laboratory re-ran samples with the same results. Investigation was undertaken to identify whether the apparent detection of PHC F2 could related to chemicals leaching from the PVC, as another inconsistency was identified in the absence of PHC F1 in water, despite it having been identified in the distillate. It was deemed unlikely that PHC F2 could be leaching from PVC based on a brief review of literature (US EPA, 2002; Stantec, 2003).

PHC F2 analysis is not specific to petroleum hydrocarbons and identifies any compound eluting in the F2 range as PHC F2. Chromatograms of PHC F2 typically show a complex spectrum similar to Figure 1, reflecting the large number of hydrocarbon compounds present in PHC F2. The chromatograms from the apparent PHC F2 detections in the water within the incubated pipes are provided in Appendix D and are quite different, showing just a couplet of peaks near C13. This suggests that the cause of these apparent PHC F2 detections is two individual chemicals (or two similar isomers of one chemical) and not PHC F2. The identity of these two chemicals is not known, but possible sources include the adhesive used to attach the end cap of the pipe or, less likely, the PVC itself.



Table 4. Summary of analytical results for water incubated inside PVC piping wrapped in PHC F2-soaked batting for 80 days.

Properties and Parameters	Analyte	Units	PHC F2 Exposed Water Column Rep 1	PHC F2 Exposed Water Column Rep 2	PHC F2 Exposed Water Column Rep 3	Control Water Column	Alberta Tier 1 Groundwater Remediation Guideline*
Hydrocarbons							
F1 Hydrocarbons $(C_6 \text{ to } C_{10})$	F1 Hydrocarbons (C_6 - C_{10}) (PHCF1_ C_6 - C_{10})	mg/L	<0.1	<0.1	<0.1	<0.1	2.2 (1)
F2 Hydrocarbons $(C_{10} \text{ to } C_{16})$	F2c C ₁₀ -C ₁₆	mg/L	2.2	<0.1	1.5	2.2**	1.1 (1)
F3 Hydrocarbons $(C_{16} \text{ to } C_{34})$	F3 C ₁₆ -C ₃₄	mg/L	0.2	<0.1	<0.1	0.6	-
F3+ Hydrocarbons (C ₃₄₊)	F3+ C ₃₄ +	mg/L	<0.1	<0.1	<0.1	<0.1	-
BTEX							
Benzene	F1 Benzene (71- 43-2)	mg/L	<0.001	<0.001	<0.001	<0.001	0.005 (1)
Toluene	F1 Toluene (108- 88-3)	mg/L	<0.0004	<0.0004	<0.0004	<0.0004	0.021
Ethylbenzene	F1 Ethylbenzene (100-41-4)	mg/L	<0.0010	< 0.0010	<0.0010	<0.0010	0.0016 (1)
<i>m,p,o</i> -xylene	F1 m,p-Xylene (1330-20-7)	mg/L	<0.001	<0.001	<0.001	<0.001	0.02 (1)

Bold values exceed relevant parameter guidelines.

Notes:

* Values for Natural Areas - All Water Uses (coarse-grained soil)

**Elevated concentration of PHC F2 in 'Control' considered a discrepancy that could not be reconciled in this study.

Greyed out values are below detection limit

Bold values exceed Alberta Surface Water guidelines

Applicable Guidelines

Environmental Quality Guidelines for Alberta Surface Waters (Surface Water Guidelines; AEP, 2018) for the protection of freshwater aquatic life (PAL) and agricultural uses

Notes

All guidelines are structured as the most stringent unless otherwise specified Nitrite guidelines based upon actual sample values associated with chloride concentrations

Sulfate guidelines based upon actual sample values associated with hardness concentrations

(1) Potable water

4.0 CONCLUSIONS

Method development was conducted to measure both total PHC F2 vapours in headspace above distillate and spiked soil, as well as specific sub-fractions in the PHC F2 range (>nC10-nC16), including aliphatic and aromatic compounds in both the nC10-12 and nC12-16 ranges. Headspace concentrations were provided

to MEMS as input parameters for trench models. Vapours from PHC F3 were not tested as concentrations were assumed to be very low given low volatility of compounds in the >nC16-nC34 range.

A literature review of potential impact of PHC F2 and F3 on underground utilities found that fibre optic cables are generally buried at depths shallower than the zone of interest for this project (>1.5 m bgs), while water piping is buried a minimum of 2.59 m below curb top. The most common material types for water piping were found to be PVC, HDPE and FRP.

Representative pipe material of each type indicated (PVC, HDPE and FRP) was obtained with an inner diameter of 150 mm (6 in) and thickness required to withstand 150 kPa pressure (>1 cm). Coupons (4 cm2) were cut and incubated for 80 days in pure PHC F2 and F3 distillate with water and no incubation as a control. Post-incubation, the coupons were weighed, tested for hardness and thickness, and examined under 10X magnification. Based on the average and standard deviation of three replicates, preand post-incubation metrics were not significantly different, with exception of the weight of HDPE incubated in PHC F2 (increase) and hardness decrease in PVC following incubation in both PHC F2 and F3. Notable differences could not be detected *via* observation of the coupons, with or without magnification.

Samples of water were taken from inside PVC pipe wrapped in PHC F2-soaked batting after 80 days. It was found that concentrations of PHC F2 in the water exceeded the Alberta Tier 1 groundwater remediation guideline of 1.1 mg/L, with an average concentration of 1.97 mg/L and with an elevated concentration (2.2 mg/L) in the control that was never exposed to PHC F2 distillate. Examination of chromatograms led to the conclusion that the source of PHC F2 was not distillate, but likely a chemical resulting from adhesive or, less likely, the PVC itself.

5.0 **RECOMMENDATIONS**

Once existing trench vapour models have been run using input data from this experiment, it may be of use to further evaluate the models to ensure all assumptions are valid in a true trench scenario. There are several factors that may require further evaluation, such as whether the source of vapours is continuous from the soil, and true air exchange rates. Results could also be compared with field data, if available.

6.0 **REFERENCES**

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APPENDIX A: PHC F2 AND F3 DISTILLATE CHARACTERIZATION

SimDis Expert 8.9

Thu Jul 25 09:08:07 2019

File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\FL19_1237-001.D\FL19_1237-001_FID1_A.CDF Sample: FL19_1237-001 Parameter: EZCHROMD2887-ALDRICH

23-Jul-19, 02:36:52 Operator: SYSTEM

Page 1

Boiling Point Table (%Off) ASTM D2887

FID(0) Channel

%Off	BP(C)	%Off	BP(C)	%Off	BP(C)
IBP	162.5	38.00	217.2	76.00	259.8
1.00	166.3	39.00	218.9	77.00	261.3
2.00	169.6	40.00	219.7	78.00	262.6
3.00	173.7	41.00	220.8	79.00	263.9
4.00	174.3	42.00	222.6	80.00	264.7
5.00	175.4	43.00	224.0	81.00	265.5
6.00	178.9	44.00	225.6	82.00	266.8
7.00	180.0	45.00	227.0	83.00	268.4
8.00	181.6	46.00	228.2	84.00	269.7
9.00	182.9	47.00	228.9	85.00	270.3
10.00	184.3	48.00	230.1	86.00	270.7
11.00	186.8	49.00	230.9	87.00	271.1
12.00	188.1	50.00	231.6	88.00	271.7
13.00	189.2	51.00	232.8	89.00	273.2
14.00	190.8	52.00	234.4	90.00	275.0
15.00	192.9	53.00	235.1	91.00	276.8
16.00	194.6	54.00	235.5	92.00	278.4
17.00	195.7	55.00	235.8	93.00	279.8
18.00	196.0	56.00	236.3	94.00	281.4
19.00	196.3	57.00	237.8	95.00	283.5
20.00	196.5	58.00	239.2	96.00	286.0
21.00	196.9	59.00	241.0	97.00	287.0
22.00	198.5	60.00	242.6	98.00	289.0
23.00	200.2	61.00	244.3	99.00	294.5
24.00	201.8	62.00	245.9	FBP	298.9
25.00	202.5	63.00	247.2		
26.00	203.2	64.00	248.5		
27.00	204.6	65.00	249.4		
28.00	206.6	66.00	250.1		
29.00	207.9	67.00	251.1		
30.00	209.4	68.00	251.9		
31.00	210.7	69.00	252.8		
32.00	212.4	70.00	253.4		
33.00	213.9	71.00	253.8		
34.00	215.3	72.00	254.1		
35.00	216.0	73.00	254.8		
36.00	216.4	74.00	256.3		
37.00	216.7	75.00	258.0		

Recovery: 100.00 @499.7C Analysis Area: 0.36960e+06 Detector RF: 1.00000e+00 R.Time Date: 7/25/2019

Start Time: 0.555 min. Start Signal: 0.099 pA Sample Amt: 0.0000 R.Factor Date: 7/25/2019

End Time: 28.068 min. End Signal: 0.734 pA Solvent Amt: 0.0000

R.Time File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\RT-INIT_D\RT-INIT_FID1_A.CDF R.Factor File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\RF-INIT_D\RF-INIT_FID1_A.CDF Blank File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\BLK4_D\BLK4_FID1_A.CDF



Thu Jul 25 10:40:29 2019

Page 1

File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\FL19_1237-001.D\FL19_1237-001_FID1_A.CDF Sample: FL19_1237-001 Parameter: EZCHROMD2887-ALDRICH

23-Jul-19, 02:36:52 Operator: SYSTEM

Cut Point Table-1 (%Off)

ASTM D2887

FID(0) Channel

<u>Cut(C)</u>	<u>%Off</u>	Name
(-0.5, 36.1)	0.00	C4 TO C5
(36.1,68.7)	0.00	C5 TO C6
(68.7,98.4)	0.11	C6 TO C7
(98.4, 125.7)	0.16	C7 TO C8
(125.7, 150.8)	0.15	C8 TO C9
(150.8, 174.1)	3.21	C9 TO C19
(174.1, 195.9)	13.97	C10 TO C11
(195.9, 216.3)	18.28	C11 TO C12
(216.3, 235.4)	17.98	C12 TO C13
(235.4, 253.5)	16.58	C13 TO C14
(253.5,270.6)	15.46	C14 TO C15
(270.6, 286.8)	11.04	C15 TO C16
(286.8, 301.9)	2.61	C16 TO C17
(301.9, 316.3)	0.14	C17 TO C18
(316.3, 330.1)	0.13	C18 TO C19
(330.1, 343.2)	0.13	C19 TO C20
(343.2, 356.5)	0.04	C20 TO C21
(356.5, 368.6)	0.00	C21 TO C22
(368.6, 380.1)	0.00	C22 TO C23
(380.1, 391.2)	0.00	C23 TO C24
(391.2, 401.8)	0.00	C24 TO C25
(401.8,412.0)	0.00	C25 TO C26
(412.0, 421.9)	0.00	C26 TO C27
(421.9, 431.3)	0.00	C27 TO C28
(431.3, 440.4)	0.00	C28 TO C29
(440.4, 449.2)	0.00	C29 TO C30
(449.2, 457.6)	0.00	C30 TO C31
(457.6,465.7)	0.00	C31 TO C32
(465.7,474.0)	0.00	C32 TO C33
(474.0, 481.0)	0.00	C33 TO C34
(481.0, 489.0)	0.00	C34 TO C35
(489.0, 496.0)	0.00	C35 TO C36
(496.0, 503.0)	0.00	C36 TO C37
(503.0, 509.0)	0.00	C37 TO C38
(509.0, 516.0)	0.00	038 10 039
(516.0, 522.0)	0.00	C39 TO C40
(522.0, 528.0)	0.00	C40 TO C41
(528.0, 534.0)	0.00	C41 TO C42

Recovery: 100.00 @347.1C Analysis Area: 6.35746e+06 Detector RF: 1.00000e+00 R.Time Date: 7/25/2019 Start Time: 0.910 min. Start Signal: 0.039 pA Sample Amt: 0.0000 R.Factor Date: 7/25/2019

End Time: 17.013 min. End Signal: 0.702 pA Solvent Amt: 0.0000

R.Time File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\RT-INIT.D\RT-INIT_FID1_A.CDF R.Factor File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\RF-INIT.D\RF-INIT_FID1_A.CDF Blank File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\BLK4.D\BLK4_FID1_A.CDF



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Page 2

File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\FL19_1237-001.D\FL19_1237-001_FID1_A.CDF Sample: FL19_1237-001 Parameter: EZCHROMD2887-ALDRICH

23-Jul-19, 02:36:52 Operator: SYSTEM

Cut Point Table-1 (%Off)

ASTM D2887 FID(0) Channel

Cut(C)	%Off	Name
(534.0, 540.0)	0.00	C42 TO C43
(540.0, 545.0)	0.00	C43 TO C44
(545.0, 550.0)	0.00	C44 TO C45
(550.0, 556.0)	0.00	C45 TO C46
(556.0, 561.0)	0.00	C46 TO C47
(561.0, 566.1)	0.00	C47 TO C48
(566.1, 570.0)	0.00	C48 TO C49
(570.0, 575.0)	0.00	C49 TO C50

Recovery: 100.00 @347.1C Analysis Area: 6.35746e+06 Detector RF: 1.00000e+00 R.Time Date: 7/25/2019 Start Time: 0.910 min. Start Signal: 0.039 pA Sample Amt: 0.0000 R.Factor Date: 7/25/2019 End Time: 17.013 min. End Signal: 0.702 pA Solvent Amt: 0.0000

R.Time File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\RT-INIT.D\RT-INIT_FID1_A.CDF R.Factor File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\RF-INIT.D\RF-INIT_FID1_A.CDF Blank File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\BLK4.D\BLK4_FID1_A.CDF



Thu Jul 25 09:08:07 2019

Page 1

File: X:\3\DATA\FL19_1237 D2887 2019-07-22 12-10-25\FL19_1237-001.D\FL19_1237-001_FID1_A.CDF Sample: FL19_1237-001 Parameter: EZCHROMD2887-ALDRICH 23-Jul-19, 02:36:52 Operator: SYSTEM

Distillation Chart ASTM D2887

ASTIVI D2887

FID(0) Channel





PHC F3 Distillate Characterization – 2012

250 Karl Clark Road Edmonton, Alberta Canada T6N 1E4		Fuels & Lubricants			Page 1 of 2			
Order Id:	FL12_0936						Reported: 1	9-Mar-2020
PO#:	3910360						Revision: 0	1
Report To:					Invoice To:			
Alberta Innov	ates - Technology	Futures			Alberta Innovates	 Technology Futures 		
250 Karl Clar	k Road				250 Karl Clark Roa	ad		
Edmonton, A	B, T6N 1E4				Edmonton, AB T	6N 1E4		
Bonnie Drozo	lowski				Bonnie Drozdowsk	ki		
Sample ID:	FL12_0936-00	10	Description:	F3: 287-481°C			Date Sampled:	2012-08-02
Product:	Crude Oil						Date Recieved:	2012-08-02
				-			 	

Test	Method	Parameter	Results	Units	Date Tested	Notes
ASTM D2887		Distillation IBP	278.2	°C	2012-08-28	
ASTM D2887		1%	283	°C	2012-08-28	
ASTM D2887		2%	287.1	°C	2012-08-28	
ASTM D2887		3%	290.3	°C	2012-08-28	
ASTM D2887		4%	293.7	°C	2012-08-28	
ASTM D2887		5%	295.2	°C	2012-08-28	
ASTM D2887		10%	302.8	°C	2012-08-28	
ASTM D2887		20%	318.6	°C	2012-08-28	
ASTM D2887		30%	337.6	°C	2012-08-28	
ASTM D2887		40%	356	°C	2012-08-28	
ASTM D2887		50%	374.6	°C	2012-08-28	
ASTM D2887		60%	394.1	°C	2012-08-28	
ASTM D2887		70%	414.1	°C	2012-08-28	
ASTM D2887		80%	434.2	°C	2012-08-28	
ASTM D2887		90%	456.8	°C	2012-08-28	
ASTM D2887		95%	473.1	°C	2012-08-28	
ASTM D2887		96%	477.5	°C	2012-08-28	
ASTM D2887		97%	483	°C	2012-08-28	
ASTM D2887		98%	490.6	°C	2012-08-28	
ASTM D2887		99%	502.6	°C	2012-08-28	
ASTM D2887		Final Boiling Point	513.3	°C	2012-08-28	
ASTM D4052		Density @ 15°C	879.7	kg/m3	2012-08-29	
ASTM D4052		Relative Density @ 15/15°C	0.8805		2012-08-29	

<u>DRAFT</u>

Thu Aug 23 13:41:18 2012

Page 1

Sample: FL12-0936-001C Operator: eg Parameter: EZCHROMD2887

Boiling Point Table (%Off)

ASTM D2887

Carbon (0) Channel

%Off	BP(C)	%Off	BP(C)	%Off	BP(C)
IBP	278.2	38.00	352.5	76.00	426.3
1.00	283.0	39.00	354.5	77.00	428.4
2.00	287.1	40.00	356.0	78.00	430.5
3.00	290.3	41.00	357.0	79.00	432.0
4.00	293.7	42.00	359.0	80.00	434.2
5.00	295.2	43.00	361.2	81.00	436.3
6.00	296.9	44.00	363.3	82.00	438.5
7.00	299.3	45.00	365.4	83.00	440.3
8.00	301.3	46.00	367.5	84.00	442.4
9.00	302.0	47.00	368.6	85.00	444.7
10.00	302.8	48.00	370.2	86.00	447.1
11.00	303.6	49.00	372.6	87.00	449.1
12.00	305.5	50.00	374.6	88.00	451.5
13.00	307.8	51.00	376.7	89.00	454.1
14.00	309.6	52.00	378.9	90.00	456.8
15.00	311.5	53.00	380.1	91.00	459.2
16.00	313.7	54.00	381.9	92.00	462.4
17.00	315.5	55.00	384.1	93.00	465.6
18.00	316.3	56.00	386.4	94.00	469.0
19.00	317.4	57.00	388.7	95.00	473.1
20.00	318.6	58.00	390.6	96.00	477.5
21.00	320.9	59.00	391.9	97.00	483.0
22.00	323.0	60.00	394.1	98.00	490.6
23.00	324.9	61.00	396.4	99.00	502.6
24.00	327.0	62.00	398.6	FBP	513.3
25.00	329.2	63.00	400.8		
26.00	330.1	64.00	402.0		
27.00	331.0	65.00	404.0		
28.00	333.0	66.00	406.3		
29.00	335.4	67.00	408.5		
30.00	337.6	68.00	410.7		
31.00	339.6	69.00	412.1		
32.00	341.8	70.00	414.1		
33.00	343.3	71.00	416.3		
34.00	344.2	72.00	418.4		
35.00	345.9	73.00	420.6		
36.00	348.2	74.00	422.0		
37.00	350.4	75.00	424.1		

Recovery: 100.00 @535.0C Analysis Area: 9.09376e+05 Detector RF: 1.00000e+00 R.Time Date: 8/23/2012 Response Factor: 4.45685e-06

Start Time: 0.000 min. Start Signal: 0.051 pA Sample Amt: 0.0000 R.Factor Date: 8/20/2012 End Time: 31.139 min. End Signal: 1.743 pA Solvent Amt: 0.0000

R.Time File: O:\SimDData\HPChem\2\DATA\FL120936-D2887 2012-08-22 16-12-55\RTINIT.D\RTINIT_FID1_A.CDF R.Factor File: O:\SimDData\EPChem\2\DATA\FL120936-D2887 2012-08-22 16-12-55\BK5.D\BK5_FID1_A.CDF Blank File: O:\SimDData\HPChem\2\DATA\FL120936-D2887 2012-08-22 16-12-55\BK5.D\BK5_FID1_A.CDF



Thu Aug 23 13:41:18 2012

Page 1

File: O:\SimDData\HPChem\2\DATA\FL120936-D2887 2012-08-22 16-12-55\FL12-0936-001C.D\FL12-0936-001C_**______A12D_Z3**:14:28 Sample: FL12-0936-001C Operator: eg Parameter: EZCHROMD2887

Distillation Chart

ASTM D2887

Carbon (0) Channel





APPENDIX B: UTILITY DESIGN, SPECIFICATIONS AND POTENTIAL IMPACT DUE TO PETROLEUM HYDROCARBONS

1.0 Fibre Optic Cables and Other Buried Utilities

TELUS was contacted for information on materials and installation practices for fibre optic cables based on their prevalence throughout the province of Alberta. The TELUS representative, Manager of Environment, stated that in most cases fibre cable is placed in conduits, rather than direct buried.

Ducts are made of high-density polyethylene (HDPE) and occasionally PVC. Most cable applications are Prysmian LT2.0 Poly-Steel-Poly (PSP) cables. When asked about potential points of weakness, they stated that at joints, the conduits could be susceptible to hydrocarbon ingress and the cables could then be impacted. Main fibre ducts are typically buried between 1.1 and 1.5 metres below ground surface (m bgs); however, from the main line to individual residences they could be as shallow as 0.45 m bgs (Brian Daniel, *pers. comm*).

Based on the information provided, it was determined that fibre optic cables should be omitted from the testing program based on their installation above the zone of interest (*i.e.*, below 1.5 m bgs).

2.0 Municipal Waterworks

The Alberta Government's Standards and Guidelines for Municipal Waterworks, Wastewater and Storm Drainage Systems (GoA, 2012 a and b) provide a general design basis for municipal water distribution systems. The minimum depth of a water distribution pipe is specified as 2.5 m and the minimum inner diameter is 150 mm (6 in). Otherwise, pipelines are to be sized according to a minimum distribution pressure of 150 kPa [1, 2].

The cities of Edmonton and Calgary provide more specific design standards [3, 4]. There are four types of polymer pipes that can potentially be used: polyvinyl chloride (PVC), flexible polyvinyl chloride (FPVC), polyethylene (PE), and high-density polyethylene (HDPE). FPVC, PE, and HDPE pipes require approval on a project specific basis, whereas PVC does not. Approved pipe types are given in the design standards, including manufacturer, model, and nominal sizes; both cities use the same models for PVC and HDPE.

The minimum nominal pipe size for a water main in both cities is 100 mm (4 in), which is restricted to residential cul-de-sacs. Otherwise, nominal pipe sizes start at 150 mm (6 in). The depth of water mains below curb top is also given in the City of Edmonton's design standards based on the specific diameters given in Table B1.

Diameter, mm (in)	Minimum depth of invert below curb top (m)
150 (6)	2.59
200 (8)	2.62
250 (10)	2.64
300 (12)	2.67
350 (14)	2.70
400 (16)	2.72
450 (18)	2.75

Table B1. Depth of water mains



For the specified diameters above, pipe dimensions were tabulated according to inner diameter and thickness for each polymer type. For PVC and HDPE pipes, there are a variety of manufacturers and thus a dimension range is given (Tables B2 and B3) [5-13].

NPS Size	Min ID, mm (in)	Max ID, mm (in)	Min Thickness, mm (in)	Max Thickness, mm (in)
6	149.10 (5.87)	165.61 (6.52)	9.73 (0.383)	16.43 (0.647)
8	202.95 (7.99)	217.17 (8.55)	12.75 (0.502)	15.98 (0.629)
10	248.77 (9.794)	266.19 (10.48)	15.65 (0.616)	19.76 (0.778)
12	295.81 (11.646)	316.74 (12.47)	16.59 (0.653)	23.09 (0.909)
14	345.44 (13.6)	373.13 (14.69)	15.49 (0.61)	21.59 (0.85)
16	392.684 (15.46)	424.18 (16.7)	17.68 (0.696)	24.61 (0.969)
18	454.66 (17.9)	475.49 (18.72)	19.81 (0.78)	19.81 (0.78)

Table B2. PVC pipe dimension ranges.

 Table B3.
 HDPE pipe dimension ranges.

NPS Size	Min ID, mm (in)	Max ID, mm (in)	Min Thickness, mm (in)	Max Thickness, mm (in)
6	135.86 (5.349)	141.50 (5.571)	15.29 (0.602)	15.93 (0.627)
8	176.78 (6.96)	185.55 (7.305)	19.91 (0.784)	20.90 (0.823)
10	220.45 (8.679)	227.61 (8.961)	24.82 (0.977)	25.63 (1.009)
12	261.37 (10.29)	270.76 (10.66)	30.48 (1.2)	29.44 (1.159)
14	287.02 (11.3)	313.69 (12.35)	32.33 (1.273)	35.33 (1.391)
16	328.17 (12.92)	356.87 (14.05)	36.96 (1.455)	40.18 (1.582)
18	369.06 (14.53)	399.80 (15.74)	41.55 (1.636)	45.03 (1.773)

EPCOR's distribution and transmission piping system comprises of 52.5% PVC, 0.3% HDPE, 0.2% fibrereinforced plastic (FRP), with the remainder being non-polymeric materials. For the specified polymeric materials, PVC spans the largest size range, from 100 mm (4 in) to 900 mm (35.5 in), with 150 mm (6 in) being the smallest PVC distribution size and 100 mm (4 in) PVC being used for services.

3.0 Material Properties

The chemical properties of PVC and PE were investigated previously to determine the effect of structure on diffusion rate [14]. It was found that more polar penetrants showed higher permeation rates due to their ability to soften or swell the rigid structure of PVC. Alkanes and aliphatic hydrocarbons had less risk of permeation because their non-polarity decreased ability to soften PVC. Conversely, PE permeation rates were higher for aromatic and aliphatic hydrocarbons, alkanes with increased molar weight, and nonpolar compounds in general. HDPE shows greater resistance than PE or LDPE although it is still susceptible to the compounds mentioned prior.

To quantify chemical resistance, a numerical rating was assigned to each compound as given in Table B4.


Rating	Resistivity
5	Resistant
3	Conditional/questionable
0	Not resistant

 Table B4.
 Resistivity Ratings.

Tables B5-B7 show chemical resistance of polymers for select hydrocarbons based on manufacturer and NACE data [15-18]. Unless specified otherwise, the concentration of each compound is 100%. The total resistivity ratings summed up for PVC, HDPE, and FRP were 148, 91, and 104, respectively.

PHC Fraction Chemical		Chemical Formula	Temperature Range (°C)	PVC Resistivity Rating
Unspecified (C<6) Methane		CH ₄	23 – 60	5
	Acetylene	C ₂ H ₂	21 – 65	5
	Propane	C ₃ H ₈	21 – 65	5
	Butadiene	C₄H ₆	21 – 26	3
			26 – 65	0
	Butane	C ₄ H ₁₀	21 – 65	5
	Pentane	C ₅ H ₁₂	23 – 60	3
	Natural gas	N/A	21 – 65	5
F1 (C6 - C10)	Benzene	C ₆ H ₆	15 – 21	0
			21 – 57	0
			57 – 65	0
	Cyclohexane	C ₆ H ₁₂	21 – 65	5
	Hexane	C ₆ H ₁₄	21 – 65	0
	Trimethylpropane	C_6H_{14}	21 – 26	3
			26 – 65	0
	Toluene	C ₇ H ₈	21 – 51	0
			51 – 65	0
	Heptane	C ₇ H ₁₆	21 – 26	5
			26 – 37	0
-			37 – 65	0
	Xylene	C ₈ H ₁₀	23 – 60	0
	Gasoline	N/A	21 – 26	3
			26 – 57	0
			57 – 65	5

 Table B5.
 Chemical Resistivity of PVC.



PHC Fraction Chemical		Chemical Formula	Temperature Range (°C)	PVC Resistivity Rating
	Naphtha	N/A	15 – 21	5
			21 – 65	5
F2 (C10 - C16)	Naphthalene	C ₁₀ H ₈	21 – 26	0
			26 – 65	0
	Jet fuels, general	N/A	15 – 21	5
			21 – 65	5
	Jet fuels, JP-4, JP-5	N/A	23 – 60	3
	Kerosene	N/A	15 – 21	5
			21 – 65	5
F3 (C16 - C34)	Diesel fuels	N/A	15 – 21	5
			21 – 65	5
	Lubricating oils	N/A	21 – 57	5
			57 – 65	3
	Motor oil	N/A	23 - 60	5
	Hydraulic oil	N/A	23	5
			60	0
	Transformer oil	N/A	15 – 21	5
			21 – 60	5
	Mineral oil	N/A	15 – 21	5
			21 – 150	5
	Paraffin	N/A	15 – 21	5
			21 – 65	5
	Silicone oil	N/A	23	5
			60	0

Table B6. Chemical Resistivity of HDPE.

PHC Fraction	Chemical	Chemical Formula	Temperature Range (°C)	HDPE Resistivity Rating
Unspecified (C<6)	Methane	CH ₄	23 – 60	5
	Propane	C_3H_8	21 – 65	5
	Butadiene	C_4H_6	21 – 26	0
			26 – 65	0
	Butane	C_4H_{10}	21 – 65	3
	Pentane	C_5H_{12}	23 – 60	3
	Natural gas	N/A	21 – 65	5
F1 (C6 - C10)	Benzene	C_6H_6	15 – 21	3
			21 – 57	0
			57 – 65	0
	Cyclohexane	C ₆ H ₁₂	21 – 65	0



PHC Fraction	Chemical	Chemical Formula	Temperature Range (°C)	HDPE Resistivity Rating
	Hexane	C ₆ H ₁₄	21 – 65	0
	Trimethylpropane	C ₆ H ₁₄	21 – 26	5
			26 – 65	3
	Toluene	C ₇ H ₈	21 – 51	0
			51 – 65	0
	Heptane	C ₇ H ₁₆	21 – 26	3
			26 – 37	0
			37 – 65	0
	Styrene	C ₈ H ₈	21 – 26	0
			26 – 65	0
	Ethylbenzene	C ₈ H ₁₀	15 – 21	0
	Xylene	C ₈ H ₁₀	23 – 60	0
	Octane	C ₈ H ₁₈	21 - 60	5
	Gasoline	N/A	21 – 26	3
			26 – 57	0
			57 – 65	0
	Naphtha	N/A	15 – 21	3
			21 – 65	0
F2 (C10 - C16)	Naphthalene	C ₁₀ H ₈	21 – 26	0
			26 – 65	0
	Jet fuels, general	N/A	15 – 21	5
			21 – 65	3
	Kerosene	N/A	15 – 21	3
			21 – 65	0
	Stoddard solvent	N/A	21 – 26	5
			26 – 51	3
F3 (C16 - C34)	Diesel fuels	N/A	15 – 21	5
			21 – 65	0
	Fuel oils		15 – 21	5
			21 – 65	0
	Lubricating oils	N/A	21 – 57	0
			57 – 65	0
	Transformer oil	N/A	15 – 21	3
			21 - 60	0
	Mineral oil	N/A	15 – 21	5
			21 – 150	0
	Paraffin	N/A	15 – 21	5
			21 - 65	3



PHC Eraction	Chomical	Chemical	Temperature	FRP Resistivity
	Chemical	Formula	Range (°C)	Rating
F1 (C6 - C10)	Benzene	C_6H_6	15 - 21	3
			21 - 57	3
			57 - 65	0
	Hexane	C_6H_{14}	21 – 65	0
	Toluene	C ₇ H ₈	15 - 21	5
			21 – 51	0
			51 – 65	5
	Heptane	C ₇ H ₁₆	21 – 26	5
			26 – 37	5
			37 – 65	5
	Styrene	C ₈ H ₈	21 - 26	0
	Xylene	C ₈ H ₁₀	23 - 60	5
	Isooctane	C ₈ H ₁₈	15 - 21	5
			21 - 65	0
	Gasoline	N/A	21 – 26	5
			26 – 57	5
			57 – 65	5
	Naphtha	N/A	21 - 65	5
F2 (C10 - C16)	Naphthalene	C ₁₀ H ₈	21 - 26	5
	Diphenyl	C ₁₂ H ₁₀	21 - 51	5
	Dodecene	C ₁₂ H ₂₄	21 - 51	5
	Anthracene (10% concentration)	C ₁₄ H ₁₀	15 - 21	3
	Kerosene	N/A	21 - 65	5
F3 (C16 - C34)	Diesel fuels	N/A	21 - 65	5
Fuel oils			15 - 21	5
			21 - 65	5
	Transformer oil	N/A	21 - 60	5

Table B7.	Chemical Resistivity of FRP
	chemical resistivity of the

Other various physical and mechanical properties for PVC and HDPE are given in Table B8 [19-20].



Property	PVC Value	HDPE Value
Shore D hardness	80	59 - 64
Impact strength (20°)	20 kJ/m ²	26 – 35 kJ/m ²
Ultimate tensile strength	52 MPa	33 MPa
Elastic tensile modulus	3.0 – 3.3 GPa	9.7 – 13.8 GPa
Elongation at break	50 – 80%	600 – 1,350%
Softening point	80 – 84°C	125°C
Maximum continuous service temperature	60°C	82°C
Coefficient of thermal expansion	7 x 10 ⁻⁵ K	2.4 x 10 ⁻⁴ °C

Table B8. Physical and mechanical properties of specified polymers.

Based on the above data and analysis, it was recommended that the potential impact of PHC F2 and F3 on PVC piping should be evaluated, as it is likely the most prevalent polymer used in Alberta's water distribution piping network. Although HDPE is not as widely used as PVC, it could also be evaluated as it is more susceptible to permeation by hydrocarbons and therefore posits the worst-case scenario. Likewise, it was recommended that the smallest pipe sizes (NPS 4 and 6) be tested.



4.0 APPENDIX B REFERENCES

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APPENDIX C: PVC, HDPE AND FRP COUPON WEIGHTS, SHORE D HARDNESS, AND THICKNESS

1.0 PVC, FRP AND HDPE Coupon Weights

		PVC Coupon Weight (g)			
				After	
	Coupon #	Initial Weight	After PHC Exposure	Wash	
sed	#1	24.1678	24.1686	24.1657	
Expo	#2	22.4652	22.4677	22.4632	
F2	#3	22.5206	22.5206	22.5193	
sed	#4	22.4330	22.4509	22.4345	
Expo	#5	24.4830	24.5052	24.4845	
F3	#6	24.3325	24.3437	24.3331	
Water	#7	22.7694	22.7881		
Control	#8	23.8547	23.8507		

		FRP Coupon Weight (g)			
				After	
	Coupon #	Initial Weight	After PHC Exposure	Wash	
sed	#1	13.8202	13.8175	13.8141	
;odx	#2	16.1732	16.1699	16.1656	
F2 I	#3	15.3120	15.3089	15.3043	
sed	#4	15.0620	14.9121	15.0420	
Expo	#5	15.0468	15.0729	15.0572	
F3	#6	14.9024	15.0560	14.8978	
Water	#7	14.3347	14.3878		
Control	#8	15.2205	15.2029		

		HDPE Coupon Weight (g)			
				After	
	Coupon #	Initial Weight	After PHC Exposure	Wash	
sed	#1	11.4042	11.7713	11.7193	
Expo	#2	11.2983	11.6287	11.5821	
F2	#3	11.5079	11.8639	11.8134	
sed	#4	11.4232	11.5320	11.5153	
Expo	#5	10.9088	11.0196	11.0028	
F3	#6	10.7188	10.8190	10.8053	
Water	#7	12.0097	12.0118		
Control	#8	13.3776	13.3763		



2.0 PVC, FRP AND HDPE Coupon Thickness

	PVC		
	Coupon		
	#	Thickness (mm)	
sed	#1	11.22	
Expo	#2	10.21	
F2 E	#3	10.72	
ed	#4	10.44	
Expo	#5	11.25	
F3 I	#6	11.34	
Water	#7	10.87	
Control	#8	10.90	

FRP

	Coupon	
	#	Thickness (mm)
sed	#1	6.91
sodx	#2	7.09
F2	#3	6.87
sed	#4	6.93
Expo	#5	6.97
F3	#6	6.90
Water	#7	6.97
Control	#8	6.87

HDPE

		TIDLE
	Coupon	
	#	Thickness (mm)
sed	#1	7.40
Expo	#2	7.53
F2	#3	7.19
sed	#4	7.62
sodx	#5	7.09
F3 I	#6	7.29
Water	#7	7.22
Control	#8	7.16



		PVC S	hore D Har	dness Unw	ashed	PVC	Shore D Ha	irdness Wa	shed
	Coupon								
	#	1	2	3	4	1	2	3	4
sed	#1	82	82	82	80	81	81	82	82
Expo	#2	83	81	81	82	84	82	82	82
F2 I	#3	81	81	81	83	82	81	82	83
sed	#4	78	78	78	79	85	83	81	82
sodx	#5	79	80	80	80	82	82	82	83
F3 I	#6	80	80	80	82	82	83	82	81
Water	#7	84	84	87	85				
Control	#8	84	84	83	85				

3.0 VC, FRP AND HDPE Coupon Shore D Hardness

		FRP SI	hore D Har	dness Unw	ashed	FRP	Shore D Ha	rdness Wa	shed
	Coupon								
	#	1	2	3	4	1	2	3	4
sed	#1	88	91	92	89	89	92	94	93
Expo	#2	90	89	92	91	93	91	91	94
F2	#3	91	96	92	92	94	94	92	91
sed	#4	89	88	87	92	91	91	91	89
i xbo	#5	91	90	90	91	91	91	94	92
F3 I	#6	89	91	92	91	90	90	92	94
Water	#7	86	89	88	89				
Control	#8	91	90	95	89				

		HDPE S	Shore D Ha	rdness Unv	vashed	HDPE	Shore D H	ardness Wa	ashed
	Coupon								
	#	1	2	3	4	1	2	3	4
sed	#1	68	62	69	63	66	69	63	62
Expo	#2	72	70	66	68	64	63	68	64
F2 I	#3	64	70	74	72	68	61	68	63
sed	#4	68	67	64	64	68	65	66	65
sodx	#5	67	68	66	68	64	68	66	66
F3 I	#6	67	66	66	68	66	72	69	64
Water	#7	71	69	74	70				
Control	#8	66	70	68	66				



APPENDIX D: ORIGINAL LABORATORY REPORTS

e	ele	ment		Element Bay #5, : Caigary, T1Y SL3,	2712-37 Avenue N.E. Alberta Canada	T: +1 (403) 291-2022 F: +1 (403) 291-2021 E: Info.Calgary@elemer W: element.com	nt.com	
Report Trans	smission	n Cover Page						
Bill To:	InnoTech 1 Oil Path	Alberta Inc.	Project ID: Project Name:	33910453.300 PTAC F2	1	Lot ID: Control Number:	141	2099
Attn:	Devon, A T9G 1A8 Allan Mai	B, Canada	Project Eddation: LSD: P.O.:			Date Received: Date Reported: Report Number	Mar 6 Apr 2 2509	3, 2020 7, 2020 552
Sampled By: Company:	Victor Ba Innotech	chmann Alberta	Proj. Acct. code:	33910453.300)1	report number.	2008	
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- Mar 20, 2020 Sample 1412099-2; 7014553: The repeated result for TEH analysis did not differ significantly from the original; it is within expected Mar 20, 2020 - Sample 1412099-3; 7014554: The repeated result for TEH analysis did not differ significantly from the original; it is within expected
- precision of the test.
- Mar 20, 2020 Sample 14120994; 7014555: The repeated result for TEH analysis did not differ significantly from the original; it is within expected precision of the test. • Mar 20, 2020 - Samples 1-4: the repeated results for F2-F4 analysis did not differ significantly from the original; it is within expected precision of the
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· Apr 27, 2020 - Report was issued to include additional services requested by Simone Levy of InnoTech on April 27: CTGM service requested on sample(s) 1, 2, 3, 4. Previous report 2501200.

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Analytical R	eport					
Bill To: Attn: Sampled By: Company:	InnoTech Alberta Inc. 1 Oil Path Dr. Devon, AB, Canada T9G 1A8 Allan Mah Victor Bachmann Innotech Alberta	Project ID: 3 Project Name: P Project Location: LSD: P.O.: Proj. Acct. code: 3	3910453.3001 'TAC F2 3910453.3001	Lo Control Nur Date Rece Date Repo Report Nur	ot ID: 1412099 mber: ived: Mar 6, 2020 orted: Apr 27, 2020 mber: 2509552	
		Reference Number Sample Date Sample Time Sample Location	1412099-1	1412099-2	1412099-3	
		Sample Description	PTAC F2 Water Column Rep 1 / 3.7°C	PTAC F2 Water Column Rep 2 / 3.7°C	PTAC F2 Water Column Rep 3 / 3.7°C	
		Matrix	Water	Water	Water	
Analyte		Units	Results	Results	Results	Nominal Detection
Mono-Aromatio	Hydrocarbons - Water					
Benzene		mg/L	< 0.001	< 0.001	<0.001	0.001
Toluene		mg/L	< 0.0004	< 0.0004	< 0.0004	0.0004
Ethylbenzene		mg/L	< 0.0010	< 0.0010	<0.0010	0.0010
Total Xylenes (m,p,o)	mg/L	< 0.001	< 0.001	<0.001	0.001
Volatile Petrole	um Hydrocarbons - Water					
F1 -BTEX		mg/L	<0.1	<0.1	<0.1	0.1
F1 C6-C10		mg/L	<0.1	<0.1	<0.1	0.1
Extractable Pet	troleum Hydrocarbons - Wate	er 🛛				
F2 C10-C16		mg/L	1.8	<0.1	1.3	0.1
F3 C16-C34		mg/L	0.3	<0.1	<0.1	0.1
F3+ C34+		ma/L	<0.1	<0.1	<0.1	0.1

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Analytical Report

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---------------------------------------------------------------------------------------------------------------	----------------------------------------------------------------------------------------------------------------------------------------------------	--------------------------------------------------	----------------------------------------------------------------------------------	---------------------------------------------------	--

Reference Number 1412099-4

Sample Date

Sample Time

Sample Location

Sample Description PTAC Control Water Column / 3.7°C

Matrix Water

mauix	water			
Units	Results	Results	Results	Nominal Detection Limit
mg/L	<0.001			0.001
mg/L	< 0.0004			0.0004
mg/L	<0.0010			0.0010
mg/L	<0.001			0.001
mg/L	<0.1			0.1
mg/L	<0.1			0.1
mg/L	1.7			0.1
mg/L	0.6			0.1
mg/L	<0.1			0.1
	maurix Units mg/L mg/L mg/L mg/L mg/L mg/L mg/L mg/L	matrix water Units Results mg/L <0.001	Water Water Units Results Results mg/L <0.001	Water Water Units Results Results Results mg/L <0.001

Approved by:

Jimmy Tran **Operations Manager**

Lh

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Evaluation of PHC F2 and F3 Management Limits March 2020









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Quality Control							
Bill To: Inno 1 Oi Dev T9G Attn: Alla Sampled By: Vict Company: Inno	oTech Alberta Inc. il Path Dr. on, AB, Canada 5 1A8 n Mah or Bachmann otech Alberta	Project ID: Project Name: Project Location: LSD: P.O.: Proj. Acct. code:	33910 PTAC 33910	0453.3001 C F2 0453.3001	Lot ID: Control Number: Date Received: Date Reported: Report Number:	1412099 Mar 6, 2020 Apr 27, 2020 2509552	
Extractable Petro	oleum Hydrocarbons	-					
Water							
Blanks	Units	Measu	red	Lower Limit	Upper Limit		Passed QC
F2 C10-C16	µg/mL		0	-0.2	0.2		yes
F3 C16-C34	µg/mL		0	-0.2	0.2		yes
F3+ G34+	µg/mL		U	-0.2	0.2		yes
Date Acquired:	March 06, 2020						_
Calibration Check	Units	% Recov	ery	Lower Limit	Upper Limit		Passed QC
F2 C10-C16	µg/mL	94	.92	80	120		yes
F3 C16-C34	µg/mL	98	08.80	80	120		yes
Date Acquired:	µg/mL March 06, 2020	90	.82	80	120		yes
bute noquired.	11010100,2020						
Mono-Aromatic	Hydrocarbons - Wate	r					
Blanks	Units	Measu	red	Lower Limit	Upper Limit		Passed QC
Benzene	ng		0	-0.002	0.002		yes
Toluene	ng		0	-0.0015	0.0015		yes
Ethylbenzene	ng		0	-0.0015	0.0015		yes
Total Xylenes (m,	p,o) ng		0	-0.002	0.002		yes
Styrene	ng		0	-0.002	0.002		yes
Date Acquired:	March 07, 2020						
Calibration Check	Units	% Recov	ery	Lower Limit	Upper Limit		Passed QC
Benzene	ng	109	.00	80	120		yes
Toluene	ng	96	.60	80	120		yes
Ethylbenzene	ng	95	.40	80	120		yes
Total Xylenes (m,	p,o) ng	88	.67	80	120		yes
Styrene	ng	85	.60	80	120		yes
Date Acquired:	March 07, 2020						
Volatile Detroleu	m Hydrocarbons W	ator					
Planks	In riyul Ocarbons - w	Maacu	rod	Lower Limit	Upper Limit		Paccod OC
E1_BTEX	onics	measu	0	_0.3	03		rasseu ujo
F1 C6-C10	na		ŏ	-0.300	0.300		ves
F2 C10-C16	na		0	-0.3	0.3		Ves
Date Acquired:	March 07, 2020		-				,
Calibration Check	Unite	% Recov	erv	Lower Limit	Upper Limit		Passed OC
F2 C10-C18	000	110	00	80	120		vec
Date Acquired:	March 07, 2020	110			120		100
wate Augureu.							







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Methodology	y and Notes						
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Method of A	nalysis						
Method Name		Reference		Meth	hod	Date Analysis Started	Location
BTEX-CCME - W	Vater	US EPA		Vola Sam Hea Chro 5021	tile Organic Compounds in Various ple Matrices Using Equilibrium dspace Analysis/Gas omatography Mass Spectrometry, 1/8260	Mar 7, 2020	Element Calgary
TEH-CCME - Wa	ater	EPA/CCM	E '	Sep: Extra	aratory Funnel Liquid-liquid action/CCME, EPA 3510/CCME	Mar 19, 2020	Element Calgary
Deferre				"Re	ference Method Modified		
EPA/CCME US EPA	Environment US Environn	tal Protectio nental Prote	n Agency Tes ction Agency	t Met Test	hods - US/CCME Methods		
Comments:							

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Project Name:	PTAC F2	Phone:	780-450-5170		Phone:	(78	0) 450-5210				Sam	ple Cu	stody		
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1	PTAC	F2 Water C	Column Rep 1			Water	Grab	S	×						
2	PTAC	: F2 Water O	Column Rep 2			Water	Grab	ß	×						
3	PTAC	F2 Water C	Column Rep 3			Water	Grab	ß	×						
4	PTA	C Control W	ater Column			Water	Grab	ы	×						
5															
6															
7									_						
8									-					-	
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APPENDIX B: INNOTECH ALBERTA 2022 CONTRACT RESEARCH REPORT



Petroleum Hydrocarbon Fraction 2: Evaluation of Management Limit – Flux Rate Experiment Summary (Phase 3)

Prepared by:

Simone Levy and Victor Bachmann, InnoTech Alberta

InnoTech Alberta

250 Karl Clark Road Edmonton, Alberta T6N 1E4 Canada

May 30, 2022

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1.0 INTRODUCTION

Management limits for Petroleum Hydrocarbon (PHC) fractions indicate concentrations where factors other than toxicity, such as aesthetics, mobile free-phase formation and explosion hazards may be of concern. Two sets of management limits for PHC fractions F2 (C_{10} - C_{16}) and F3 ($C_{>16}$ - C_{34}) are currently applicable in Alberta; a generic set in the Alberta Tier 1 Soil and Groundwater Remediation guidelines (Tier 1; AEP, 2019) applicable to all land uses, and another set specific to remote parts of the Green Area (AESRD, 2014). The generic management limits for PHC F2 and F3 can often become limiting and potential drivers for remediation in the case that the eco-contact exposure pathway can be eliminated.

In 2018, Millennium EMS Solutions Ltd. (MEMS) was contracted by PTAC-AUPRF to complete a project entitled 'Re-evaluating the management limits for F2 and F3 petroleum hydrocarbons – Phase 1 and 2' (PTAC AUPRF#18-RRRC-08; Millennium EMS Solutions Ltd., 2019). To meet the recommendations from Phase 1 of the project, further investigation of data supporting potential exposure of workers in trenches to PHC F2 vapours was undertaken. In 2020, MEMS completed project 19-RRRC-08 with a report entitled *Re-Evaluation of F2 and F3 Petroleum Hydrocarbon Management Limits – Phase 2* (Millennium EMS Solutions Ltd., 2020). As part of the project, MEMS sub-contracted InnoTech to conduct experiments to generate equilibrium vapour concentrations for PHC F2 aliphatic and aromatic sub-fractions. The concentrations generated through the experiments were used by MEMS as input parameters for air mixing models simulating the air space of a utility trench. Revised threshold values for the Exposure of Workers in Trenches to PHC F2 vapours were then calculated and used to inform management limits.

Phase 2 resulted in a recommendation to establish flux rates of F2 sub-fractions from the surface of soil samples to further support recommended management limits. As opposed to maximum headspace concentrations at equilibrium (which were derived in Phase 2), empirical flux rates can be combined with trench air exchange rates in a simple model to calculate the 'trench worker scenario' management limit component with a higher degree of confidence. Phase 3, reported herein, builds on the previous experimental work and Phase 2 recommendations. Phase 3 measured F2 flux rates from the surface of fine- and coarse-grained soils containing approximately 10,000 mg/kg PHC F2. Phase 3 was completed in two parts: Part 1 included assessing the flux rate from samples within Tedlar® bags (i.e., a closed system), while Part 2 assessed the flux rate in an open system configuration with a controlled air exchange.

In general, headspace concentrations reported by the photoionization detector were not consistent with those collected for analysis by flame ionization detector. Flux rate experiments were conducted in two different ways: within a closed system using Tedlar[®] bags, and an open air exchange system more closely emulating a trench scenario. The data will be used as empirical input parameters for guideline derivation and the results from the two experimental designs will be compared to establish optimal experimental setup for the needs of this project.

2.0 METHODS

Method development and range finding for appropriate setup were reused from the previous Phase 2 project conducted by InnoTech for MEMS (InnoTech Alberta, 2020), including:

- Characterization of pure PHC F2 distillate by gas chromatograph mass spectrometer (GC-MS) and flame ionization detector (GC-FID)
- Method development and range finding for headspace equilibrium setup
- Testing headspace at equilibrium with pure PHC F2 distillate

Coarse- and fine-grained soil samples were spiked to approximately 10,000 mg/kg with PHC F2 distillate. In Part 1 of these experiments, samples were incubated in Tedlar[®] bags and headspace vapour samples were taken periodically from the headspace for analysis by FID of total VOCs, PHC F2 and the following sub-fractions:

- C₁₀-C₁₂ aromatic compounds
- C₁₀-C₁₂ aliphatic compounds
- C_{>12}-C₁₆ aromatic compounds
- C_{>12}-C₁₆ aliphatic compounds

In Part 2, a flow-through system was designed using a mass flow controller to introduce air into a 500 mL jar containing spiked soil. Periodic samples were also taken for analysis of the same components.

The experiments followed the tasks described below, including calibration of a photoionization detector (PID) for monitoring headspace combustible vapour concentrations (CVCs), soil spiking and analysis, flux rate experiments, and data analysis.

2.1 Soil spiking

Previously characterized coarse- and fine-grained soils (Appendix A) were spiked with PHC F2 distillate (characterization reported in InnoTech Alberta, 2020) to a target of 10,000 mg/kg as requested by MEMS. Spiking was conducted in a -14°C walk-in freezer to minimize losses through volatilization. Soils were moistened to approximately 14% (w/w) in fine-grained soil and 1.5% (w/w) in coarse-grained soil, based on their respective water holding capacities. Following spiking, one random grab sample of each batch of soil was taken and submitted for laboratory analysis of BTEX and PHC F1-F4 components to confirm the concentration.

2.2 Experimental Setup

2.2.1 Part 1 - Closed System

Once spiking results were received and deemed satisfactory by MEMS, soil was placed into 100 mL glass petri dishes (Diameter 89mm, Surface Area 62.2cm²; Part 1; Figure 1), compacted and weighed for calculation of bulk density. Petri dish samples were prepared as follows for 3 replicated 'runs' of the flux rate experiment:

- Bag 1: 1 sample for headspace monitoring via PID (petri dish + 200 mL lab grade air in Tedlar bag);
- Bag 2: 1 sample for headspace sampling (petri dish + 200 mL lab grade air in Tedlar bag); and,
- Sufficient soil to submit samples for soil analysis of BTEX and PHC F1-F4 parameters (reported as Run 1/2/3 – C/F).



Figure 1. Experimental setup: soil compacted in glass petri dishes within a Tedlar[®] bag.

For each soil type, 3 repetitions in duplicate (2x) of a petri dish was place inside a tedlar bag (total of 12 bags and petri dishes) and immediately evacuated of headspace using a manual 1 L syringe, taking care not to over-pressurize the bags. Samples were maintained in a -15°C freezer until they were transferred to a 4°C fridge for 24-hours. Samples were removed from the fridge to laboratory air temperature (~21°C) for 4 hours prior to starting experiments.

2.2.2 Part 2 - Controlled Air Exchange

2.2.2.1 Controlled Air Exchange Evaluation

The second part of the experiment included the use of a mass flow controller (MFC; Alicat Scientific MC-Series¹). Preliminary trials were conducted to refine the experimental setup as follows:

- 1) Ensure the mass flow controller (MFC; Alicat Scientific MC-Series²) can accurately and consistently deliver air to a vessel at the desired flow rate (46 air exchanges per hour³).
- 2) Conduct range finding to confirm that volatile organic carbon (VOC) concentrations in headspace are within a suitable range for analysis by GC-FID (Gas chromatography with flame

¹ Alicat Scientific MC-Series Gas Mass Flow Controllers rapidly reach setpoints and maintain stable control of mass flow, volumetric flow, or pressure across a flow range of 0.01% to 100% of full scale <u>https://www.alicat.com/models/mc-gas-mass-flow-controllers/</u>

² Alicat Scientific MC-Series Gas Mass Flow Controllers rapidly reach setpoints and maintain stable control of mass flow, volumetric flow, or pressure across a flow range of 0.01% to 100% of full scale <u>https://www.alicat.com/models/mc-gas-mass-flow-controllers/</u>

³ Flow rate to be determined based on container headspace volume.

ionization detection) over the 60-minute experiment. Use data to select an optimally sized vessel based on soil to headspace ratio (500 mL or 1 L).

3) Confirm that air introduced into the vessel will circulate adequately to effectively exchange the headspace by conducting a smoke test.

To address points 1) and 2), fine-grained PHC F2-spiked soils remaining from Part 1 were placed into 500 mL and 1 L vessels to 16 mm thickness. The Alicat was connected to a supplied airline and set to a flow rate of 46L/hour (23 air exchanges an hour), introducing air into the vessels. No fluctuation was observed on the Alicat read-out, which was indicative of its ability to consistently deliver air to the vessels at the target rate. Range finding samples were taken from headspace of each vessel at 60 minutes and analyzed for total PHC. Concentrations were within acceptable range for analysis well above detectable limits. Based on results, the 500 ml vessel was selected for the full experiment.

To address point 3), a smoke test was performed to evaluate air flow within the vessel containing PHC F2-spiked soil. The Alicat was daisy chained to two 500 mL vessels. The first jar (connected to the Alicat) was filled with smoke and connected to the second jar for observation. The Alicat was connected to a supplied airline and set to a flow rate of 46L/hour (23 air exchanges an hour). A light was projected through the observation jar to observe the smoke flow pattern. At the selected flow rate, the smoked air flowed into the observation and covered the bottom of the jar (Figure 2a). The whole volume of the observation vessel filled with smoked air before exiting through the exhaust port (Figure 2b).



Figure 2. Testing the flow of air through the 500ml vessels using smoke. A) The initial path of air into the test vessel from the top port flows to the bottom and covers the bottom of the vessel. B) The smoke fills the vessel as it travels out of the vessel through the exhaust port.

2.2.2.2 Experimental Setup

Coarse-grained soils were used from Part 1, while fine-grained soils were freshly spiked for Part 2 experiments due to insufficient quantity. Experiments were conducted in triplicate for each of the soil types.

Soil was placed into the bottom of a pre-weighed 500 mL jar and was compacted to a total height of 16 mm, equivalent to soil in petri dishes in Part 1. Each jar was then re-weighed for soil bulk density calculation. The jar lids were fitted with inflow and outflow ports. The inflow port was used to connect an air input tube through an Alicat mass flow controller (MFC)⁴, which drew laboratory grade air from a cylinder at an equivalent flow rate of ~46 air exchanges per hour. A MiniRAE 3000 photoionization detector (PID), previously calibrated to hexane at concentrations of approximately 200 and 1,000 ppm, was used to monitor the headspace by inserting a tube into the outflow. Two soil types were tested (fine and coarse) in 3 repetitions for each soil type. The bulk spiked soils were stored in a -14°C freezer. One day before each experiment, the spiked soil was transferred to a 4°C fridge to thaw. Samples were prepared in the 500 mL vessels the day of each run(s) and stored in the 4°C fridge. The samples were removed from the fridge approximately an hour before the start of each run to allow them to reach room temperature (21°C).

2.3 Flux Rate Experiments

2.3.1 Part 1 - Closed System

A MiniRAE 3000 photoionization detector (PID) was calibrated to hexane at concentrations of approximately 200 and 1,000 ppm. The instrument was used to monitor Bag 1 headspace by inserting a tube into the port in the Tedlar[®] bag (Figure 3).

At 'time 0', 2 L of laboratory grade air was injected into Bag 1 for headspace monitoring via PID and Bag 2 for sampling. The PID cycles 500 mL per minute, but analysis is nondestructive and the sample volume is returned to the bag.

Four samples were taken from bag 2 over the flux duration based on the volatile organic compounds (VOCs) on PID output curves (Figure 3). For each sample, one thermal desorption tube set (*i.e.* one desorption tube in line with a second one in case of breakthrough) were used to collect a 25 mL headspace gas sample (total removal of 100 mL from 2 L headspace). The tube set was used to determine the concentration of total hydrocarbons by GC-FID and was used to determine the aromatic/aliphatic composition by ASTM D2786 (ASTM, 2016; modified).



Figure 3. PID connected to Tedlar bag containing a petri dish filled with spiked soil.

Experiments were run three times, referred to as Run 1, 2 and 3 with a designation for coarse- (- C) or fine-grained (- F). From the three runs for each respective soil type, average concentrations of total VOCs

⁴ Alicat Scientific MC-Series Gas Mass Flow Controllers rapidly reach setpoints and maintain stable control of mass flow, volumetric flow, or pressure across a flow range of 0.01% to 100% of full scale <u>https://www.alicat.com/models/mc-gas-mass-flow-controllers/</u>

in headspace at select sample time points were averaged and a standard deviation calculated. These were plotted on a graph with the PID curve presented as mol/mol (units).

Data from headspace samples was submitted for calculation of aliphatic and aromatic concentrations of nC_{10} - nC_{12} and nC_{12} - nC_{16} following the Robinson whole oil method⁵ (Robinson, 1971). This was conducted using the apex of nC_{10} , nC_{12} and nC_{16} , the aliphatic and aromatic concentrations of nC_{10} - nC_{12} and nC_{12} - nC_{16} . This method is a baseline technique to resolve the mass spectrum into saturates and aromatic spectra in a sample within boiling point range of 200 to 1,100 F°. The method allows determination of up to 4 saturated and 21 aromatic compound types. It uses the low-resolution mass spectrum and the number average molecular weight, obtained from distillation data, of the unseparated sample.

Compound classes are as follows:

Saturates:

- Paraffins
- Monocycloparaffins
- Dicycloparaffins
- Tricycloparaffins

Aromatics:

- Alkylbenzenes
- Aromatic Sulfur
- Benzocycloalkanes
- Benzodicycloalkanes
- Benzonaphthiophenes
- Benzothiophenes
- Benzpyrenes/Perylenes
- Chrysenes
- •CnH2n-32/CnH2n-46
- •CnH2n-36/CnH2n-26S

2.3.2 Part 2 - Controlled Air Exchange

- •CnH2n-38/CnH2n-28S
- •CnH2n-40/CnH2n-30S
- •CnH2n-42/CnH2n-32S
- •CnH2n-44/CnH2n-34S
- Diaromatics
- Dibenzanthracenes
- Dibenzothiophenes
- Fluorenes
- Monoaromatics
- Naphthalenes

- Naphthocycloalkanes
- Pentaaromatics
- Phenanthrenes
- Phenanthrocycloalkanes
- Pyrenes/Benzofluorenes
- Tetraaromatics
- Triaromatics
- Unidentified

At 'time 0', coverings (used to minimize volatilization of VOCs) were removed, lids were secured onto jars, and the air supply was connected via MFC (Figure 4). Headspace samples were taken from each replicate at 15, 30 and 60 minutes after exposing soil to atmosphere. For each headspace sample, one thermal desorption tube set (*i.e.* one desorption tube, in line with a second one in case of breakthrough) was used to collect a 25 mL headspace gas sample. The tube set was used to determine the concentration of total hydrocarbons by GC-FID. Data was analyzed for aromatic/aliphatic composition by ASTM D2786 (ASTM, 2016; modified) from each of the samples at 60 minutes only based on direction from MEMS.

⁵ The method is known as the Robinson Whole Oil Method because in contrast to many other methods (such as D2786 or D3239), it does not require a prior separation of a sample into saturates and aromatic fractions. The method reports four saturated hydrocarbon types, twelve aromatic HC types, three thiophenic and six unidentified aromatic groups.



Figure 4. Experimental setup, controlled air exchange with coarse-grained soil.

3.0 RESULTS AND DISCUSSION

Results of the trial are summarized in the following sections. Original laboratory reports are included in Appendix B. A summary of average headspace concentrations from all experiments are included in Appendix C.

3.1 Part 1 – Flux Rate in Closed System

3.1.1 Soil spiking and preparation

Results of spiking coarse- and fine-grained soils with PHC F2 distillate are provided in Table 1, including composite samples taken directly following spiking (post-spike composite) and samples representative of soil at the start time of each run (Run 1/2/3 - C or - F). The post-spike spike composite was collected after the soil samples were spiked and moistened. Samples associated with a run were collected at the same time as filling the petri dishes for the associated run.

Sample description	Moisture	Soil Bulk Density	Benzene	Toluene	Ethylbenzene	Xylenes	PHC F1	PHC F2	PHC F3	PHC F4		
Sample description			(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)	(mg/kg)		
Coarse-grained soil												
Post-spike composite	3.29	n/a	n/a	n/a	n/a	n/a	n/a	10,100	225	<100		
Run 1 - C	2.14	1.15	<0.005	0.53	0.029	0.32	230	6,820	146	<100		
Run 2 - C	2.34	1.15	<0.005	0.46	0.025	0.3	278	7,300	157	<100		
Run 3 - C	2.32	1.15	<0.005	0.47	0.026	0.32	301	7,320	153	<100		
Run 1-3 - C Average (PRSD*)	2.27 (4)	1.15	<0.005	0.49 (6)	0.027 (6.30)	0.31 (3.0)	270 (11)	7,146 (3)	152 (3)	<100		
Fine-grained soil												
Post-spike composite	12.2	n/a	n/a	n/a	n/a	n/a	n/a	13,200	253	<100		
Run 1 - F	12.4	1.94	<0.005	0.27	<0.005	0.17	230	8,100	364	<100		
Run 2 - F	12.3	1.82	<0.005	0.33	0.015	0.25	341	8,020	358	<100		
Run 3 - F	12.3	1.65	<0.005	0.16	0.008	0.14	126	12,200	537	<100		
Run 1-3 - F Average ((PRSD*))	12.33 (0.4)	1.81 (6.6)	<0.005	0.25 (28)	0.012 (29.17)	0.19 (24)	232 (38)	9,440 (21)	420 (20)	<100		

Table 1. Laboratory analytical results for PHC F2-spiked soil samples; average and composite results for PHC F2 highlighted in blue as the parameter of interest.

* Percent Relative Standard Deviation

A decrease in total PHC F2 was seen between the initial sample at the time of spiking and the samples tested after storage and transfer into petri dishes in both soil types. Total PHC F2 decreased by 29.2% and 28.4% in coarse- and fine-grained soils, respectively. Based on this data, to ensure accurate starting concentrations, samples were submitted for analysis following the same preparation steps as those in the experiments (*i.e.*, transfer to petri dishes and evacuation of headspace within Tedlar[®] bag).

In coarse-grained soils, concentrations of PHC F2 were relatively consistent with a percent relative standard deviation (%RSD) of 3.23%; however, one elevated concentration was reported for fine-grained soil (Run 3-F), resulting in a %RSD of 20.68% for PHC F2 and high variability in other measured parameters. The sample with elevated PHC F2 and other hydrocarbon components may have been the result of inconsistent spiking of PHC F2 distillate into soil, which can be particularly challenging in fine-grained soil. However, it is notable that headspace concentrations measured with sample Run 3-F were in the same concentration range as the previous two runs.

3.1.2 Flux Rate Experiments

Results for time to equilibrium and flux rates experiments are provided in the following sections. These include PID versus GC-FID results and general correlation through experiments; hydrocarbon compounds in headspace over time in three replicated experiments for each soil type; and an evaluation of the ratio of saturate and aromatic sub-fractions to evaluate consistency.

3.1.2.1 Coarse-grained soil

Total PHC concentrations as measured by GC-FID were plotted with a representative curve of headspace vapour concentrations as measured by PID in ppm (mol/mol; Figure 5 and Appendix C). Orange circles represent the average total PHC concentration in headspace of the 3 replicate runs at or around the sample time indicated. Error bars represent standard deviation of replicates. At point 4 (1:30:00), only 2 replicates were averaged as the third was compromised due to equipment malfunction. In Figure 5, the standard deviation was relatively small for two sample points (point 2 and 3) and is not visible on the graph.



Figure 5. Representative coarse-grained soil monitored via photoionization detector (PID) plotted with average total PHC vapour in headspace +/- standard deviation (n=3 where SD shown); note, error bars on points 2 and 3 not visible at 0.005 and 0.02, respectively; n=2 for point 4, no SD calculated.

Analytical results from the GC-FID generally follow the trend of those measured by PID. Units of PID (ppmv or mol/mol) and GC-FID (μ g/mL) concentrations differ, and it was determined that conversion of PID data to match FID units may introduce error based on assumptions around molecular weights of the compounds in headspace. The PID curve in Figure 5 was chosen as that most representative of the GC-FID results curve; however, there was some variation in the curves generated by the PID despite consistent calibration. It is therefore not recommended that concentrations from the PID be used as the basis for reporting. However, the PID curve was used to determine approximate time to equilibrium for timing samples for GC-FID analysis, targeting the relatively flat portion of the curve.

A full dataset of headspace analyses for coarse-grained soil samples is provided in Table 2; select parameters and sub-fractions will be used by MEMS as model input parameters.

Replicated	Sample Time on PID Curve	Sample Lab ID	Total PHC	PHC F2	nC	10 apex-nC 12a	pex	nC12apex-nC16apex			
experiment and sample point on PID Curve					Total	Saturate sub- fraction	Aromatic sub- fraction	Total	Saturate sub- fraction	Aromatic sub- fraction	
Run 1 - C			P6/	P6/ 111	P6/	P6/	P6/ 111	P6/ 111	P6/	P6/ 111	
time 1	00:51	FL21-2006-006	0.631	0.484	0.417	-	-	0.067	-	-	
time 2	03:00	FL21-2006-007	0.872	0.665	0.581	0.436	0.145	0.084	0.065	0.019	
time 3	06:00	FL21-2006-008	1.23	0.942	0.797	0.596	0.201	0.145	0.111	0.033	
time 4	08:30	FL21-2006-009	1.57	1.202	1.053	0.785	0.268	0.149	0.113	0.036	
time 5	45:00	FL21-2006-010	2.02	1.502	1.370	1.034	0.336	0.132	0.100	0.032	
Run 2 - C											
time 1	0:10:00	FL21-2006-011	1.328	0.970	0.864	0.634	0.231	0.106	0.081	0.025	
time 2	0:40:00	FL21-2006-012	2.027	1.472	1.332	0.962	0.370	0.139	0.099	0.040	
time 3	1:00:00	FL21-2006-013	2.055	1.498	1.362	0.987	0.375	0.136	0.097	0.039	
time 4	1:30:00	FL21-2006-014	-	-	-	-	-	-	-	-	
Run 3 - C											
time 1	0:10:00	FL21-2006-015	1.559	1.135	1.018	0.739	0.279	0.116	0.087	0.030	
time 2	0:40:00	FL21-2006-016	2.028	1.449	1.328	0.961	0.367	0.120	0.088	0.033	
time 3	1:00:00	FL21-2006-017	2.032	1.446	1.329	0.963	0.366	0.116	0.085	0.031	
time 4	1:30:00	FL21-2006-018	2.072	1.512	1.377	0.995	0.382	0.135	0.099	0.036	

Table 2. Summary of results of headspace PHC analyses by GC-FID, including sub-fractions, for flux rate experiments for coarse-grained soils.

Note: Data omitted from analysis from cells highlighted grey based on timing or sampling error
The average and standard deviation of the saturate and aromatic portions of each sub-fraction were calculated for the purpose of evaluating consistency. Table 3 provides a ratio of each sub-fraction to the total PHC F2 components. Results presented in Table 3 are ratios (as opposed to concentrations). Saturates and aromatics are ratios of corresponding fractions, and fractions are ratios of total F2. Data indicated as "incomplete dataset" and "time-based outlier" in Run 1-C were due to challenges in establishing sampling time points based on inconsistent PID readings; standard timing for samples was established in Runs 2 and 3. In Run 2-C, the syringe malfunctioned and a sample could not be obtained.

Replicated			nC ₁₀ apex-	nC ₁₂ apex-	nC ₁₀ apex	-nC ₁₂ apex	nC ₁₂ ape	x-nC ₁₆ apex		
experiment and sample point	Sample Lab ID	Sample Time on PID Curve	nC12apex : Total PHC F2	nC ₁₆ apex : Total PHC F2	Saturates to Total PHC F2	Aromatic to Total PHC F2	Saturates to Total PHC F2	Aromatic to Total PHC F2		
on PID Curve					Ra	tios				
Run 1-C										
time 1	FL21-2006-006	00:51			Data omitted - in	complete dataset	:			
time 2	FL21-2006-007	03:00		Data omitted - time-based outlier						
time 3	FL21-2006-008	06:00			Data omitted - ti	me-based outlier				
time 4	FL21-2006-009	08:30	0.876	0.124	0.745	0.255	0.761	0.239		
time 5	FL21-2006-010	45:00	0.912	0.088	0.755	0.245	0.761	0.239		
Run 2-C		- -		·	•					
time 1	FL21-2006-011	0:10:00	0.891	0.109	0.733	0.267	0.766	0.234		
time 2	FL21-2006-012	0:40:00	0.905	0.095	0.722	0.278	0.711	0.289		
time 3	FL21-2006-013	1:00:00	0.909	0.091	0.724	0.276	0.714	0.286		
time 4	FL21-2006-014	1:30:00	-	-	-	-	-	-		
Run 3-C		- -		·	•					
time 1	FL21-2006-015	0:10:00	0.897	0.103	0.726	0.274	0.746	0.254		
time 2	FL21-2006-016	0:40:00	0.917 0.083 0.723 0.277 0.729 0.271							
time 3	FL21-2006-017	1:00:00	0.919 0.081 0.725 0.275 0.731 0.269							
time 4	FL21-2006-018	1:30:00	0.911	0.089	0.722	0.278	0.732	0.268		
Average (PRSD*)			0.904 (1.53) 0.096 (14.4) 0.731 (1.60) 0.269 (4.3) 0.739 (2.8) 0.261 (7.82							

Table 3. Average and standard deviation of saturate and aromatic portions of $nC_{10}-C_{12}$ and $nC_{12}-C_{16}$ in headspace with coarse-grained soil samples. (Note: Data omitted from analysis from cells highlighted grey based on timing or sampling error).

* Percent relative standard deviation

Based on the averages and relative percent deviation of proportions of each sub-fraction within the headspace hydrocarbon vapour components, it is interpreted that ratios of saturate to aromatic sub-fractions are sufficiently consistent between replicates to negate the need for analysis of each replicate when samples are taken over a time of relatively consistent flux (*i.e.*, flat part of curve).

3.1.2.2 Fine-grained soil

Total PHC concentrations as measured by GC-FID were plotted with a representative curve of headspace vapour concentrations as measured by PID (Figure 6). Orange circles represent the average total PHC concentration in headspace of the 3 replicate runs at or around the sample time indicated. Error bars represent standard deviation of replicates.



Figure 6. Representative coarse-grained soil monitored via photoionization detector (PID) plotted with average total PHC vapour in headspace +/- standard deviation (n=3).

The PID and GC-FID curves are more aligned in experiments with fine-grained soils, and the curve and timing points differed from those in experiments with coarse-grained soils. The flux rate from each soil type is therefore unique and warrants exploration in any future experiments. It is notable that total headspace VOCs were higher in coarse-grained soil experiments (max. $\sim 2.0 \ \mu g/mL$) compared with those with fine-grained soils (max. $\sim 1.5 \ \mu g/mL$); in addition, the time to equilibrium was not reached at 1:30:00 with coarse-grained soils but reached a maximum concentration in fine-grained soils after approximately 20:00. This may be associated with migration of PHC F2 components through the soil to headspace, with greater retardation and retention in fine-grained soils.

A full dataset of headspace analyses for fine-grained soil samples is provided in Table 4.

Replicated					nC	10apex-nC12a	pex	ex nC12apex-nC16ape		ex
experiment and sample point on PID Curve		Sample Lab ID	Total PHC	PHC F2	Total	Saturate sub- fraction	Aromatic sub- fraction	Total	Saturate sub- fraction	Aromatic sub- fraction
Curve			μg/mL	µg/mL	µg/mL	μg/mL	µg/mL	μg/mL	µg/mL	μg/mL
Run 1 - F	1	1			1	1			1	
time 1	05:00	FL21-2006-019	0.902	0.683	0.614	0.443	0.170	0.069	0.056	0.012
time 2	20:00	FL21-2006-020	1.210	0.915	0.830	0.603	0.227	0.085	0.067	0.018
time 3	30:00	FL21-2006-021	1.270	0.964	0.880	0.640	0.240	0.084	0.067	0.017
time 4	40:00	FL21-2006-022	1.37	1.077	0.978	0.714	0.263	0.099	0.076	0.022
Run 2 - F	1	1			1	1			1	
time 1	0:05:00	FL21-2006-023	0.653	0.521	0.465	0.3445	0.1202	0.057	0.043	0.014
time 2	0:20:00	FL21-2006-024	1.208	0.955	0.867	0.639	0.228	0.088	0.067	0.021
time 3	0:30:00	FL21-2006-025	1.427	1.132	1.020	0.744	0.275	0.112	0.085	0.027
time 4	0:40:00	FL21-2006-026	-	-	-	-	-	-	-	-
Run 3 - F			•							
time 1	0:05:00	FL21-2006-027	0.779	0.631	0.557	0.4021	0.1544	0.075	0.056	0.019
time 2	0:20:00	FL21-2006-028	1.261	1.008	0.908	0.657	0.251	0.100	0.076	0.024
time 3	0:30:00	FL21-2006-029	1.321	1.050	0.952	0.689	0.263	0.098	0.075	0.024
time 4	0:40:00	FL21-2006-030	1.451	1.148	1.040	0.755	0.284	0.108	0.082	0.026

Table 4. Summary of results of headspace PHC analyses by GC-FID, including sub-fractions, for flux rate experiments with fine-grained soils.

The average and standard deviation of the saturate and aromatic portions of each sub-fraction were calculated for the purpose of evaluating consistency. Table 5 provides a ratio of each sub-fraction to the total PHC F2 components (as opposed to concentrations). Saturates and aromatics are ratios of corresponding fraction, and fractions are ratios of total F2.

Replicated			nC10apex-	nC12apex-	nC10apex	-nC12apex	nC ₁₂ apex-nC ₁₆ apex	
experiment and sample point on	Sample Lab ID	Sample Time on PID Curve	nC12apex: total PHC F2	nC16apex: total PHC F2	Saturates to total PHC F2	Aromatic to total PHC F2	Saturates to total PHC F2	Aromatic to total PHC F2
PID Curve					Ratio		·	
Run 1-F								
time 1	FL21-2006-019	05:00	0.899	0.101	0.722	0.278	0.820	0.180
time 2	FL21-2006-020	20:00	0.907	0.093	0.726	0.274	0.784	0.216
time 3	FL21-2006-021	30:00	0.913	0.087	0.727	0.273	0.793	0.207
time 4	FL21-2006-022	40:00	0.908	0.092	0.731	0.269	0.774	0.226
Run 2-F								
time 1	FL21-2006-023	0:05:00	0.891	0.109	0.741	0.259	0.753	0.247
time 2	FL21-2006-024	0:20:00	0.908	0.092	0.737	0.263	0.758	0.242
time 3	FL21-2006-025	0:30:00	0.901	0.099	0.730	0.270	0.756	0.244
time 4	FL21-2006-026	0:40:00						
Run 3-F		•		•			•	1
time 1	FL21-2006-027	0:05:00	0.882	0.118	0.723	0.277	0.751	0.249
time 2	FL21-2006-028	0:20:00	0.901	0.099	0.724	0.276	0.757	0.243
time 3	FL21-2006-029	0:30:00	0.906	0.094	0.724	0.276	0.758	0.242
time 4	FL21-2006-030	0:40:00	0.906	0.094	0.726	0.274	0.757	0.243
Average (PRSD*)			0.902 (0.98)	0.098 (9.1)	0.728 (0.82)	0.272 (2.2)	0.769 (2.8)	0.231 (9.4)

Table 5. Average and standard deviation of saturate and aromatic portions of $nC_{10}-C_{12}$ and $nC_{12}-C_{16}$ in headspace with coarse-grained soil samples.

* Percent relative standard deviation

Based on the averages and relative percent deviation of proportions of each sub-fraction within the headspace hydrocarbon vapour components, it is interpreted that ratios of saturate to aromatic sub-fractions are sufficiently consistent between replicates to negate the need for analysis of each replicate when samples are taken over a time of relatively consistent flux (*i.e.*, flat part of curve).

3.2 Part 2 – Controlled Air Exchange

3.2.1 Soil spiking and preparation

Results of spiking fine-grained soils with PHC F2 distillate are provided in Table 6, including composite samples taken directly following spiking (post-spike composite) and samples representative of soil at the start time of each run (Run 4/5/6 - C or - F). An additional composite sample was taken of the coarse-grained soil spiked in Part 1. All coarse runs were done on the same day and were set up at the same time. The analytical sample was collected during the setup of those 3 runs. The fine runs were done over the course of 2 days and each was set up before their respective runs. A fine soil sample was collected for analysis during the set up of each run. A composite sample was collected during the runs to confirm F2 concentration.

Sample	Moisture	Soil Bulk	Benzene	Tol- uene	Ethyl- benzene	Xylenes	PHC F1	PHC F2	PHC F3	PHC F4		
Description	(%)	Densit y		(mg/kg)								
				Fine-grained soil								
Post-spike composite	13.2	n/a	<0.005	<0.02	0.019	0.26	454	7,690	135	<100		
Run 4 - F	12.6		<0.005	<0.02	0.02	0.24	313	7,090	125	<100		
Run 5 - F	11.9		<0.005	<0.02	0.014	0.2	310	8,550	137	<100		
Run 6 - f	12.5		<0.005	<0.02	0.013	0.2	348	8,530	150	<100		
Run 4-6 Average (PRSD*)	12.33		<0.005	<0.02	0.0157 (19.73)	0.21 (8.84)	324 (17.25)	8057 (684)	137 (10)	<100		
	Coarse-grained soil											
Run 7-9 – C (composite)	2.23		0.011	0.45	0.043	0.48	622	5,660	94	<100		

Table 6. Laboratory analytical results for PHC F2-spiked soil samples; average and composite results forPHC F2 highlighted in blue as the parameter of interest.

* Percent relative standard deviation

In fine-grained soils, concentrations of PHC F2 showed some variability with a percent relative standard deviation (%RSD) of 10%. There was not sufficient coarse-grained soil available to run replicated analyses, and they were previously run in Part 1. A composite sample was taken of remaining coarse-grained soil, which was similar in concentration to the original concentration (10,100 mg/kg) but slightly higher than the average of three composites taken in Part 1 (7,146 mg/kg).

3.2.2 Flux Rate Experiments

Results for time to equilibrium and flux rates experiments are provided in the following sections. These include PID versus GC-FID results; hydrocarbon compounds in headspace over time in three replicated experiments for each soil type; and an evaluation of the ratio of saturate and aromatic sub-fractions to evaluate consistency (Table 7).

3.2.2.1 Coarse-grained soil

Total PHC concentrations as measured by GC-FID were plotted with a representative curve of headspace vapour concentrations as measured by PID in ppm (mol/mol; Figure 7). Orange circles represent the average total PHC concentration in headspace of the 3 replicate runs at the sample time indicated. Error bars represent standard deviation of replicates.



Figure 7. Representative coarse-grained soil monitored via photoionization detector (PID) plotted with average total PHC vapour in headspace +/- standard deviation (n=3).

Analytical results from the GC-FID seem to oppose those measured by PID. Units of PID (ppmv or mol/mol) and GC-FID (μ g/mL) concentrations differ, and it was determined that conversion of PID data to match FID units may introduce error based on assumptions around molecular weights of the compounds in headspace. It is therefore not recommended that concentrations from the PID be used as the basis for reporting.

Replicated				nC	C10apex-nC1	₂apex	nC	C12apex-nC10	₅apex
experiment and sample time from	Sample Lab ID	Total PHC	PHC F2	Total	Saturate sub- fraction	Aromatic sub- fraction	Total	Saturate sub- fraction	Aromatic sub- fraction
inception		µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	µg/mL	μg/mL
Run 4 - C									
15 min	FL22-0554-004	1.008	0.789	0.704	-	-	0.085	-	-
30 min	FL22-0554-005	0.962	0.749	0.645	-	-	0.104	-	-
60 min	FL22-0554-006	0.865	0.690	0.571	0.419	0.152	0.119	0.086	0.034
Run 5 - C									
15 min	FL22-0554-007	1.061	0.806	0.690	-	-	0.116	-	-
30 min	FL22-0554-008	1.056	0.842	0.687	-	-	0.155	-	-
60 min	FL22-0554-009	0.913	0.731	0.601	0.441	0.160	0.130	0.094	0.036
Run 6 - C									
15 min	FL22-0554-010	1.221	0.934	0.796	-	-	0.138	-	-
30 min	FL22-0554-011	1.070	0.845	0.696	-	-	0.149	-	-
60 min	FL22-0554-012	0.942	0.756	0.618	0.452	0.166	0.139	0.100	0.038

Table 7. Summary of results of headspace PHC analyses by GC-FID, including sub-fractions, for flux rate experiments with coarse-grained soils.

3.2.2.2 Fine-grained soil

Total PHC concentrations as measured by GC-FID were plotted with a representative curve of headspace vapour concentrations as measured by PID. Orange circles represent the average total PHC concentration in headspace of the 3 replicate runs at or around the sample time indicated. Error bars represent standard deviation of replicates.



Figure 8. Representative fine-grained soil monitored via photoionization detector (PID) plotted with average total PHC vapour in headspace +/- standard deviation (n=3).

The PID and GC-FID curves followed a similar pattern to those shown in Figure 5, where results from the GC-FID seem to oppose those measured by PID. It is therefore not recommended that concentrations from the PID be used as the basis for reporting.

A full dataset of headspace analyses for fine-grained soil samples is provided in Table 8. Results of the saturate and aromatic sub-fractions for nC_{10} apex- nC_{12} apex and nC_{12} apex- nC_{16} apex are relatively consistent between fine-grained soil replicates, with the exception of the saturate sub-fraction in run 4-F.

Replicated	Sample Lab ID	Total PHC	PHC F2		nC ₁₀ apex-nC ₁₂ ap	ex		nC ₁₂ apex-nC ₁₆ ap	ex
experiment and sample				Total	Saturate sub- fraction	Aromatic sub- fraction	Total	Saturate sub- fraction	Aromatic sub- fraction
inception		µg/mL	µg/mL	µg/mL	μg/mL	µg/mL	µg/mL	µg/mL	μg/mL
Run 4 - F									
15 min	FL22-0554-013	0.286	0.212	0.174	Not requested	Not requested	0.037	Not requested	Not requested
30 min	FL22-0554-014	0.298	0.228	0.172	Not requested	Not requested	0.056	Not requested	Not requested
60 min	FL22-0554-015	0.235	0.178	0.132	0.067	0.064	0.046	0.026	0.020
Run 5 - F								•	
15 min	FL22-0554-016	0.434	0.334	0.276	Not requested	Not requested	0.058	Not requested	Not requested
30 min	FL22-0554-017	0.393	0.314	0.245	Not requested	Not requested	0.069	Not requested	Not requested
60 min	FL22-0554-018	0.347	0.264	0.198	0.113	0.085	0.066	0.041	0.025
Run 6 - F								·	
15 min	FL22-0554-019	0.447	0.366	0.281	Not requested	Not requested	0.085	Not requested	Not requested
30 min	FL22-0554-020	0.430	0.310	0.256	Not requested	Not requested	0.053	Not requested	Not requested
60 min	FL22-0554-021	0.395	0.292	0.227	0.128	0.100	0.065	0.039	0.026

Table 8. Summary of results of headspace PHC analyses by GC-FID, including sub-fractions, for flux rate experiments with fine-grained soils.

4.0 CONCLUSIONS

In both parts 1 and 2 of the experiments, headspace concentrations reported by the PID were not consistent with those collected for analysis by flame ionization detector. The reason for this discrepancy could not be determined. The FID results were deemed to me more dependable than the PID values, and therefore only the FID results are recommended to be carried forward for guideline development.

In parts 1 and 2, flux rate experiments were conducted in two different ways: within a closed system using Tedlar[®] bags, and an open air exchange system more closely emulating a trench scenario. The data will be used as empirical input parameters for guideline derivation and the results from the two experimental designs will be compared to establish optimal experimental setup for the needs of this project.

5.0 REFERENCES

- ASTM D2786-91 (ASTM, 2016; modified). Standard Test Method for Hydrocarbon Types Analysis of Gas-Oil Saturates Fractions by High Ionizing Voltage Mass Spectrometry. ASTM International, West Conshohocken, PA, 2016, www.astm.org
- InnoTech Alberta, 2020. Petroleum Hydrocarbon Fractions 2 and 3: Evaluation of Management Limits. Report Prepared for Millennium EMS Solutions Ltd., Calgary, AB. May 2020. 57 pp.
- MEMS (Millennium EMS Solutions Ltd.) 2020. Re-Evaluation of F2 and F3 Petroleum Hydrocarbon Management Limits – Phase 2. Prepared for Petroleum Technology Alliance Canada. Millennium EMS Solutions Ltd.. Calgary, AB. 93 pp.
- Robinson, C.J., 1971. Low-resolution mass spectrometric determination of aromatics and saturates in petroleum fractions, Analytical Chemistry Vol. 43, No. 11: 1425-1434.

APPENDIX A. Soil Characterization

Fine- and coarse-grained soils used in the experiments are classified as loam and sand, respectively, with additional characterization information provided in Tables A1 and A2. Fine-grained soils were obtained from an uncontaminated field site and represent typical topsoil in Alberta. Coarse-grained soil was pure silica sand, representing a soil type with minimal potential retention of volatile hydrocarbon compounds.

Table A1.	. Soil texture	e and water	holding	capacity.
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		Sand Silt Clay			Water Holding Capacity			
	Texture	50mm-2mm	2mm-50mm	>2mm	Field Capacity	Wilting Point	AWHC	
		%	by weight		vol %			
Fine	Loam	30	48	22	28.43	12.24	16.19	
Coarse	Sand	97	<0.1	3	4.33	1.39	2.94	

Table A2. Detailed salinity.

	Salinity													Total
						Saturat	ted Paste E	Extract Dat	a					Carbon
	рН	EC	SAR	Sat'n	C	Ca Mg Na K							Carbon	
	H ₂ O	dS/m		%	meq/L	mg/kg	meq/L	mg/kg	meq/L	mg/kg	meq/L	mg/kg	TGR	%
Fine	7.5	1.625	0.5	48	13.465	129.4	6.785	39.35	1.725	19	0.235	4.5	<0.1	0.36
Coarse	6.55	0.075	0.4	27.5	0.205	1.1	0.105	0.35	0.15	1	0.06	<1	<0.1	<0.05

APPENDIX B. Original Laboratory Reports

Coarse-grained soil

					nC ₁₀ apex-nC ₁₂ apex		nC ₁₂ apex-nC ₁₆ apex			
Replicated		Total VOC	PHC F2	Total	Saturate sub-fraction	Aromatic sub-fraction	Total	Saturate sub-fraction	Aromatic sub-fraction	
experiment and sample time from inception	Sample Lab ID	µg/mL	μg/mL	μg/mL	μg/mL	µg/mL	μg/mL	μg/mL	μg/mL	
Run 4 - C										
15 min	FL22-0554-004	1.008	0.789	0.704	Not requested	Not requested	0.085	Not requested	Not requested	
30 min	FL22-0554-005	0.962	0.749	0.645	Not requested	Not requested	0.104	Not requested	Not requested	
60 min	FL22-0554-006	0.865	0.690	0.571	Pending	Pending	0.119	Pending	Pending	
Run 5 - C										
15 min	FL22-0554-007	1.061	0.806	0.690	Not requested	Not requested	0.116	Not requested	Not requested	
30 min	FL22-0554-008	1.056	0.842	0.687	Not requested	Not requested	0.155	Not requested	Not requested	
60 min	FL22-0554-009	0.913	0.731	0.601	Pending	Pending	0.130	Pending	Pending	
Run 6 - C										
15 min	FL22-0554-010	1.221	0.934	0.796	Not requested	Not requested	0.138	Not requested	Not requested	
30 min	FL22-0554-011	1.070	0.845	0.696	Not requested	Not requested	0.149	Not requested	Not requested	
60 min	FL22-0554-012	0.942	0.756	0.618	Pending	Pending	0.139	Pending	Pending	

Fine-grained soil

Replicated		Total VOC	PHC F2		nC ₁₀ apex-nC ₁₂ apex		nC ₁₂ apex-nC ₁₆ apex			
experiment and sample time from	Sample Lab ID	Total VUC	PHC F2	Total	Saturate sub-fraction	Aromatic sub-fraction	Total	Saturate sub-fraction	Aromatic sub-fraction	
inception		μg/mL	μg/mL	μg/mL	μg/mL	μg/mL	μg/mL	μg/mL	μg/mL	
Run 4 - F										
15 min	FL22-0554-013	0.286	0.212	0.174	Not requested	Not requested	0.037	Not requested	Not requested	
30 min	FL22-0554-014	0.298	0.228	0.172	Not requested	Not requested	0.056	Not requested	Not requested	
60 min	FL22-0554-015	0.235	0.178	0.132			0.046			
Run 5 - F										
15 min	FL22-0554-016	0.434	0.334	0.276	Not requested	Not requested	0.058	Not requested	Not requested	
30 min	FL22-0554-017	0.393	0.314	0.245	Not requested	Not requested	0.069	Not requested	Not requested	
60 min	FL22-0554-018	0.347	0.264	0.198			0.066			
Run 6 - F										
15 min	FL22-0554-019	0.447	0.366	0.281	Not requested	Not requested	0.085	Not requested	Not requested	
30 min	FL22-0554-020	0.43	0.31	0.256	Not requested	Not requested	0.053	Not requested	Not requested	
60 min	FL22-0554-021	0.395	0.292	0.227			0.065			

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					Lot Id	1532501	1532501
				Lo	t Reference Number	1	2
					Sample Id	7742660	7742661
				CI	ient Project Number		
					Client Project Name		
				Clie	ent Project Location		
				Client Pro	oject Legal Location		
					Client Project PO	64381	64381
				Client Pr	oject Account Code	43110161.3001	43110161.3001
					Received Date	2021-11-01	2021-11-01
					Sample Location		
					Sampling Method	Comp	Comp
					Sampled By	Victor Bachmann	Victor Bachmann
					Sampling Company	Innotech Alberta	Innotech Alberta
					Site Id		
					Sample Description	Coarse F2	Fine F2
					Sample Date	2021-10-28	2021-10-28
					Sampled Time	00:00:00.0000000	00:00:00.0000000
					Sample Depth		
					Sample Depth Unit		
					Legal Location		
					Description 2		
					Completed Date	2021-11-02	2021-11-02
					Matrix	Soil	Soil
Report Restults Group	Method Reference	Analyte	Descriptor	Unit	Detection Limit	Result Text	Result Text
Extractable Petroleum		Extraction Date	Total Extractables			1-Nov-21	1-Nov-21
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F2c C10-C16	Dry Weight	mg/kg	25	10100	13200
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F3c C16-C34	Dry Weight	mg/kg	50	225	253
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4c C34-C50	Dry Weight	mg/kg	100	<100	<100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4HTGCc C34-	Dry Weight	mg/kg	100	<100	<100
Extractable Petroleum		% C50+		%		<5	<5
Silica Gel Cleanup		Silica Gel Cleanup				Done	Done
Soil % Moisture		Moisture	Soil % Moisture	% by weight		3.29	12.20

Course	Sand	Total	F2 fraction
		ppm (ug/mL)	ppm (ug/mL)
EXP 1			
time 1	FL21-2006-006	0.631	0.484
time 2	FL21-2006-007	0.872	0.665
time 3	FL21-2006-008	1.23	0.942
time 4	FL21-2006-009	1.57	1.202
time 5	FL21-2006-010	2.02	1.502
EXP 2			
time 1	FL21-2006-011	1.328	0.970
time 2	FL21-2006-012	2.027	1.472
time 3	FL21-2006-013	2.055	1.498
time 4	FL21-2006-014	No sample was pulled	error in sampling
EXP 3			
time 1	FL21-2006-015	1.559	1.135
time 2	FL21-2006-016	2.028	1.449
time 3	FL21-2006-017	2.032	1.446
time 4	FL21-2006-018	2.072	1.512
Fine	Sand	Total	F2 fraction
		nnm (ug/ml.)	nnm (ug/ml.)
		ppin (ug/inc)	ppin (ug/inc)
EXP 1			
time 1	FL21-2006-019	0.902	0.683
time 2	FL21-2006-020	1.210	0.915
time 3	FL21-2006-021	1.270	0.964
time 4	FL21-2006-022	1.37	1.077
EXP 2			
time 1	FL21-2006-023	0.653	0.521
time 2	FL21-2006-024	1.208	0.955
time 3	FL21-2006-025	1.427	1.132
time 4	FL21-2006-026	No sample was pulled	error in sampling
EXP 3			
time 1	FL21-2006-027	0.779	0.631
time 2	FL21-2006-028	1.261	1.008
time 3	FL21-2006-029	1.321	1.050
time 4	FL21-2006-030	1.451	1.148

nC10apex-nC12apex	nC10apex-nC12apex	nC10apex-nC12apex
total	saturates	aromatic
ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
0.417	Not requested	Not requested
0.581	0.436	0.145
0.797	0.596	0.201
1.053	0.785	0.268
1.370	1.034	0.336
0.864	0.634	0.231
1.332		
1.362	0.987	0.375
1.018	0.739	0.279
1.328	0.961	0.367
1.329	0.963	0.366
1.377	0.995	0.382
nC10apex-nC12apex	nC10apex-nC12apex	nC10apex-nC12apex
total	saturates	aromatic
ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
0.614		
0.830		
0.880		
0.978		
0.465	0.3445	0.1202
0.867	0.639	0.228
1.020	0.744	0.275
0.557	0.4021	0.1544
0.908	0.657	0.251
0.952	0.689	0.263
1.040	0.755	0.284

nC12apex-nC16apex	nC12apex-nC16apex	nC12apex-nC16apex
total	saturates	aromatic
ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
0.067	Not requested	Not requested
0.084	0.065	0.019
0.145	0.111	0.033
0.149	0.113	0.036
0.132	0.100	0.032
0.106	0.081	0.025
0.139		
0.136	0.097	0.039
0.116	0.087	0.030
0.120	0.088	0.033
0.116	0.085	0.031
0.135	0.099	0.036
nC12apex-nC16apex	nC12apex-nC16apex	nC12apex-nC16apex
total	saturates	aromatic
ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
0.069		
0.085		
0.084		
0.099		
0.057	0.043	0.014
0.088	0.067	0.021
0.112	0.085	0.027
0.075	0.056	0.019
0.100	0.076	0.024
0.098	0.075	0.024
0.108	0.082	0.026

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				Lot	t Reference Number	1	2	3
					Sample Id	7835614	7835615	7835616
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					Client Project Name			
				Clie	ent Project Location			
				Client Pro	oject Legal Location			
					Client Project PO	64381	64381	64381
					Received Date	2021-12-16	2021-12-16	2021-12-16
					Sample Location			
					Sampling Method	Comp	Comp	Comp
					Sampled By	Victor Bachmann	Victor Bachmann	Victor Bachmann
					Sampling Company	Innotech	Innotech	Innotech
					Site Id			
					Sample Description	PTAC F2 Coarse R1	PTAC F2 Coarse R2	PTAC F2 Coarse R3
					Sample Date	2021-12-08	2021-12-10	2021-12-10
					Sampled Time	11:00:00.000000	10:00:00.000000	14:30:00.000000
					Sample Depth			
					Sample Depth Unit			
					Legal Location			
					Description 2			
					Completed Date	2021-12-18	2021-12-18	2021-12-18
					Matrix	Soil	Soil	Soil
Report Restults Group	Method Reference	Analyte	Descriptor	Unit	Detection Limit	Result Text	Result Text	Result Text
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Benzene	Dry Weight	mg/kg	0.005	<0.005	<0.005	<0.005
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Toluene	Dry Weight	mg/kg	0.02	0.53	0.46	0.47
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Ethylbenzene	Dry Weight	mg/kg	0.005	0.029	0.025	0.026
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Total Xylenes	Dry Weight	mg/kg	0.03	0.32	0.30	0.32
Mono-Aromatic Hydrocarbons -		Methanol Field				Yes	Yes	Yes
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 C6-C10	Dry Weight	mg/kg	10	231	279	302
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 -BTEX	Dry Weight	mg/kg	10	230	278	301
Extractable Petroleum		Extraction Date	Total Extractables			17-Dec-21	17-Dec-21	17-Dec-21
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F2c C10-C16	Dry Weight	mg/kg	25	6820	7300	7320
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F3c C16-C34	Dry Weight	mg/kg	50	146	157	153
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4c C34-C50	Dry Weight	mg/kg	100	<100	<100	<100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4HTGCc C34-	Dry Weight	mg/kg	100	<100	<100	<100
Extractable Petroleum		% C50+		%		<5	<5	<5
Silica Gel Cleanup		Silica Gel Cleanup				Done	Done	Done
Soil % Moisture		Moisture	Soil % Moisture	% by weight		2.14	2.34	2.32

					Report Id	2710281	2710281	2710281
					Lot Id	1546784	1546784	1546784
				Lot	t Reference Number	1	2	3
					Sample Id	7859156	7859157	7859158
				Cli	ient Project Number			
					Client Project Name			
				Clie	ent Project Location			
				Client Pro	oject Legal Location			
					Client Project PO	64381	64381	64381
				Client Pr	oject Account Code			
					Received Date	2022-01-13	2022-01-13	2022-01-13
					Sample Location			
					Sampling Method	Comp	Comp	Comp
					Sampled By	Victor Bachmann	Victor Bachmann	Victor Bachmann
					Sampling Company	Innotech Alberta	Innotech Alberta	Innotech Alberta
					Site Id			
					Sample Description	PTAC F2 Fine R1 Jan 22	PTAC F2 Fine HS R2	PTAC F2 Fine HS R3
					Sample Date	2022-01-06	2022-01-10	2022-01-10
					Sampled Time	00:00:00.0000000	00:00:00.0000000	00:00:00.000000
					Sample Depth			
					Sample Depth Unit			
					Legal Location			
					Description 2			
					Completed Date	2022-01-15	2022-01-15	2022-01-15
					Matrix	Soil	Soil	Soil
Report Restults Group	Method Reference	Analyte	Descriptor	Unit	Detection Limit	Result Text	Result Text	Result Text
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Benzene	Dry Weight	mg/kg	0.005	<0.005	<0.005	<0.005
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Toluene	Dry Weight	mg/kg	0.02	0.27	0.33	0.16
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Ethylbenzene	Dry Weight	mg/kg	0.005	<0.005	0.015	0.008
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Total Xylenes	Dry Weight	mg/kg	0.03	0.17	0.25	0.14
Mono-Aromatic Hydrocarbons -		Methanol Field				Yes	Yes	Yes
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 C6-C10	Dry Weight	mg/kg	10	230	342	126
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 -BTEX	Dry Weight	mg/kg	10	230	341	126
Extractable Petroleum		Extraction Date	Total Extractables			14-Jan-22	14-Jan-22	14-Jan-22
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F2c C10-C16	Dry Weight	mg/kg	25	8100	8020	12200
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F3c C16-C34	Dry Weight	mg/kg	50	364	358	537
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4c C34-C50	Dry Weight	mg/kg	100	<100	<100	<100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4HTGCc C34-	Dry Weight	mg/kg	100	<100	<100	<100
Extractable Petroleum		% C50+		%		<5	<5	<5
Silica Gel Cleanup		Silica Gel Cleanup	-			Done	Done	Done
Soil % Moisture		Moisture	Soil % Moisture	% by weight		12.40	12.30	12.30



	Operation			nC10apex-	nC10apex-	nC10apex-	nC12apex-	nC12apex-	nC12apex-
course	Sand	Total	F2 fraction	nC12apex	nC12apex	nC12apex	nC16apex	nC16apex	nC16apex
				total	saturates	aromatic	total	saturates	aromatic
		ppm							
		(ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
EXP 1									
15 min	FL22-0554-004	1.008	0.789	0.704	Not requested	Not requested	0.085	Not requested	Not requested
30 min	FL22-0554-005	0.962	0.749	0.645	Not requested	Not requested	0.104	Not requested	Not requested
60 min	FL22-0554-006	0.865	0.690	0.571	0.419	0.152	0.119	0.086	0.034
EXP 2									
15 min	FL22-0554-007	1.061	0.806	0.690	Not requested	Not requested	0.116	Not requested	Not requested
30 min	FL22-0554-008	1.056	0.842	0.687	Not requested	Not requested	0.155	Not requested	Not requested
60 min	FL22-0554-009	0.913	0.731	0.601	0.441	0.160	0.130	0.094	0.036
EXP 3									
15 min	FL22-0554-010	1.221	0.934	0.796	Not requested	Not requested	0.138	Not requested	Not requested
30 min	FL22-0554-011	1.070	0.845	0.696	Not requested	Not requested	0.149	Not requested	Not requested
60 min	FL22-0554-012	0.942	0.756	0.618	0.452	0.166	0.139	0.100	0.038
C *	Sand	Tatal	EQ (montion	nC10apex-	nC10apex-	nC10apex-	nC12apex-	nC12apex-	nC12apex-
Tine	Sanu	Iotai	F2 fraction	nC12apex	nC12apex	nC12apex	nC16apex	nC16apex	nC16apex
				total	saturates	aromatic	total	saturates	aromatic
		ppm							
		(ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
EXP 1									
15 min	FL22-0554-013	0.286	0.212	0.174	Not requested	Not requested	0.037	Not requested	Not requested
30 min	FL22-0554-014	0.298	0.228	0.172	Not requested	Not requested	0.056	Not requested	Not requested
60 min	FL22-0554-015	0.235	0.178	0.132	0.067	0.064	0.046	0.026	0.020
EXP 2									
15 min	FL22-0554-016	0.434	0.334	0.276	Not requested	Not requested	0.058	Not requested	Not requested
30 min	FL22-0554-017	0.393	0.314	0.245	Not requested	Not requested	0.069	Not requested	Not requested
60 min	FL22-0554-018	0.347	0.264	0.198	0.113	0.085	0.066	0.041	0.025
EXP 3									
15 min	FL22-0554-019	0.447	0.366	0.281	Not requested	Not requested	0.085	Not requested	Not requested
30 min	FL22-0554-020	0.430	0.310	0.256	Not requested	Not requested	0.053	Not requested	Not requested
60 min	FL22-0554-021	0.395	0.292	0.227	0.128	0.100	0.065	0.039	0.026

					Report Id	2697766
					Lot Id	1539063
				Lo	t Reference Number	1
					Sample Id	7796159
				CI	ient Project Number	
					Client Project Name	
				Cli	ent Project Location	
				Client Pr	oject Legal Location	
					Client Project PO	64381
				Client Pr	roject Account Code	
					Received Date	2021-11-26
					Sample Location	
					Sampling Method	Comp
					Sampled By	Victor Bachmann
					Sampling Company	Innotech Alberta
					Site Id	
					Sample Description	PTAC F2 Fine HS R1
					Sample Date	2021-11-26
					Sampled Time	00:00:00.000000
					Sample Depth	
					Sample Depth Unit	
					Legal Location	
					Description 2	
					Completed Date	2021-11-29
					Matrix	Soil
Report Restults Group	Method Reference	Analyte	Descriptor	Unit	Detection Limit	Result Text
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Benzene	Dry Weight	mg/kg	0.005	<0.005
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Toluene	Dry Weight	mg/kg	0.02	0.14
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Ethylbenzene	Dry Weight	mg/kg	0.005	0.015
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Total Xylenes	Dry Weight	mg/kg	0.03	0.30
Mono-Aromatic Hydrocarbons -		Methanol Field				Yes
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 C6-C10	Dry Weight	mg/kg	10	207
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 -BTEX	Dry Weight	mg/kg	10	207
Extractable Petroleum		Extraction Date	Total Extractables			27-Nov-21
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F2c C10-C16	Dry Weight	mg/kg	25	10100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F3c C16-C34	Dry Weight	mg/kg	50	411
Extractable Petroleum	COME [OWS PHOS TIER 1]	F4c C34-C50	Dry Weight	mg/kg	100	<100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4HTGCc C34-	Dry Weight	mg/kg	100	<100
Extractable Petroleum		% C50+		%		<5
Silica Gel Cleanup		Silica Gel Cleanup				Done
Soil % Moisture		Moisture	Soil % Moisture	% by weight		12.40

					Report Id	2704855	2704855	2704855
					Lot Id	1543347	1543347	1543347
				Lot	t Reference Number	1	2	3
					Sample Id	7835614	7835615	7835616
				Cli	ient Project Number			
					Client Project Name			
				Clie	ent Project Location			
				Client Pro	oject Legal Location			
					Client Project PO	64381	64381	64381
					Received Date	2021-12-16	2021-12-16	2021-12-16
					Sample Location			
					Sampling Method	Comp	Comp	Comp
					Sampled By	Victor Bachmann	Victor Bachmann	Victor Bachmann
					Sampling Company	Innotech	Innotech	Innotech
					Site Id			
					Sample Description	PTAC F2 Coarse R1	PTAC F2 Coarse R2	PTAC F2 Coarse R3
					Sample Date	2021-12-08	2021-12-10	2021-12-10
					Sampled Time	11:00:00.000000	10:00:00.000000	14:30:00.000000
					Sample Depth			
					Sample Depth Unit			
					Legal Location			
					Description 2			
					Completed Date	2021-12-18	2021-12-18	2021-12-18
					Matrix	Soil	Soil	Soil
Report Restults Group	Method Reference	Analyte	Descriptor	Unit	Detection Limit	Result Text	Result Text	Result Text
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Benzene	Dry Weight	mg/kg	0.005	<0.005	<0.005	<0.005
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Toluene	Dry Weight	mg/kg	0.02	0.53	0.46	0.47
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Ethylbenzene	Dry Weight	mg/kg	0.005	0.029	0.025	0.026
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Total Xylenes	Dry Weight	mg/kg	0.03	0.32	0.30	0.32
Mono-Aromatic Hydrocarbons -		Methanol Field				Yes	Yes	Yes
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 C6-C10	Dry Weight	mg/kg	10	231	279	302
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 -BTEX	Dry Weight	mg/kg	10	230	278	301
Extractable Petroleum		Extraction Date	Total Extractables			17-Dec-21	17-Dec-21	17-Dec-21
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F2c C10-C16	Dry Weight	mg/kg	25	6820	7300	7320
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F3c C16-C34	Dry Weight	mg/kg	50	146	157	153
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4c C34-C50	Dry Weight	mg/kg	100	<100	<100	<100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4HTGCc C34-	Dry Weight	mg/kg	100	<100	<100	<100
Extractable Petroleum		% C50+		%		<5	<5	<5
Silica Gel Cleanup		Silica Gel Cleanup				Done	Done	Done
Soil % Moisture		Moisture	Soil % Moisture	% by weight		2.14	2.34	2.32

					Report Id	2738126		2738126		2738126	2738126	2738126
					Lot Id	1565211		1565211		1565211	1565211	1565211
				Lot	t Reference Number	1		2		3	4	5
					Sample Id	8011302		8011303		8011304	8011305	8011306
				Cli	ient Project Number	PTAC F2 Flux		PTAC F2 Flux		PTAC F2 Flux	PTAC F2 Flux	PTAC F2 Flux
				(Client Project Name							
				Clie	ent Project Location							
				Client Pro	ject Legal Location							
					Client Project PO							
				Client Pro	oject Account Code	43110161.3001		43110161.3001		43110161.3001	43110161.3001	43110161.3001
					Received Date	2022-04-14		2022-04-14		2022-04-14	2022-04-14	2022-04-14
					Sample Location							
					Sampling Method							
					Sampled By							
					Sampling Company	,						
					Site Id							
				:	Sample Description	PTAC F2 Flux Coarse		PTAC F2 Flux Fine R1		PTAC F2 Flux Fine R2	PTAC F2 Flux Fine R3	PTAC F2 Flux Comp
					Sample Date	2022-04-06		2022-04-11		2022-04-11	2022-04-12	2022-04-12
					Sampled Time	00:00:00.0000000		00:00:00.000000		00:00:00.0000000	00:00:00.000000	00:00:00.000000
					Sample Depth							
					Sample Depth Unit							
					Legal Location							
					Description 2							
					Completed Date	2022-04-17		2022-04-17		2022-04-17	2022-04-17	2022-04-17
					Matrix	Soil		Soil		Soil	Soil	Soil
Report Restults Group	Method Reference	Analyte	Descriptor	Unit	Detection Limit	Result Text		Result Text		Result Text	Result Text	Result Text
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Benzene	Dry Weight	mg/kg	0.005		0.011		<0.005	<0.005	<0.005	<0.005
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Toluene	Dry Weight	mg/kg	0.02		0.45		<0.02	<0.02	<0.02	<0.02
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Ethylbenzene	Dry Weight	mg/kg	0.005		0.043		0.020	0.014	0.013	0.019
Mono-Aromatic Hydrocarbons -	US EPA [5021/8260]	Total Xylenes	Dry Weight	mg/kg	0.03		0.48		0.24	0.20	0.20	0.26
Mono-Aromatic Hydrocarbons -		Methanol Field					Yes		Yes	Yes	Yes	Yes
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 C6-C10	Dry Weight	mg/kg	10		623		313	310	348	454
Volatile Petroleum	CCME [CWS PHCS TIER 1]	F1 -BTEX	Dry Weight	mg/kg	10		622		313	310	348	454
Extractable Petroleum		Extraction Date	Total Extractables				15-Apr-22		15-Apr-22	15-Apr-22	15-Apr-22	15-Apr-22
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F2c C10-C16	Dry Weight	mg/kg	25		5660		7090	8550	8530	7690
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F3c C16-C34	Dry Weight	mg/kg	50		94		125	137	150	135
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4c C34-C50	Dry Weight	mg/kg	100		<100		<100	<100	<100	<100
Extractable Petroleum	CCME [CWS PHCS TIER 1]	F4HTGCc C34-	Dry Weight	mg/kg	100		<100		<100	<100	<100	<100
Extractable Petroleum		% C50+		%			<5		<5	<5	<5	<5
Silica Gel Cleanup		Silica Gel Cleanup					Done		Done	Done	Done	Done
Soil % Moisture		Moisture	Soil % Moisture	% by weight			2.23		12.60	11.90	12.50	13.20

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Enter tests above (✓ relevant samples below)	+ ↓		Matrix Sampling	Date/Time sampled	Depth start end in cm m	escription	Sample D	Site I.D.	
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Signature:			YES	Copy of Invoice:		port: No	Copy of Re	iote #:	Quo
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I authorize Element to proceed with	perta.ca		mone.Levy@Innotect	E-mail 1: Si	novates.ca	Allan.Mah@albertain	381 E-mail 1:	VAFE#: 64:	PO/
Company: Innotech Alberta				Fax:			Fax:	gal Location:	Leg:
Sampled by: Victor Bachmann				Cell:		780.450.5170	Cell:	oject Location:	Proj
Sample Custody			780-450-5210	Phone:			Phone:	oject Name:	Proj.
E-mail:			Simone Levy	Attention:		Allan Mah	Attention:	oject ID:	Proj
2) Name:			Edmonton, AB			Edmonton, AB	n	Project Informatic	
E-mail:		d.	250 Karl Clark R	Address:	INW	250 Karl Clark Rc	n Address:	www.Element.cor	
1) Name:		a	Innotech Alberta	Company:	les	Alberta Innovat	LT Company:	elemei	
Additional Reports to			Report To			Invoice To)	

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Confirmation of Service Request

 Element
 T: +1 (403) 291-2022

 Bay #5, 2712-37 Avenue N.E.
 F: +1 (403) 291-2021

 Calgary, Alberta
 F: info.Calgary@elen

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Page 1 of 2 T: +1 (403) 291-2022 F: +1 (403) 291-2021 E: info.Calgary@element.com W: www.element.com

Lot ID: 1539063

Number of Samples: 1

Printed Date: Dec 02, 2021

Please verify the following service request. If you have corrections or questions, please contact Client Services. No response to this confirmation of analysis will signify all services listed below are accurate.

Report To:		Invoice To:		Bill Paid by:	
Attn: Simone Levy		Attn: Allan Mah		Attn: Accounts Payable	
InnoTech Alberta Inc.		InnoTech Alberta Inc.		InnoTech Alberta Inc.	
Bag 4000		1 Oil Path Dr.		PO Box 8330	
Hwy 16A & 75 Street		Devon, AB T9G 1A8		250 Karl Clark Road	
Vegreville, AB T9C 1T4		Phone: (780) 987-8635		Edmonton, AB T6N 1E4	
Phone: (780) 450-5474		Fax: (780) 450-5333		Phone: (780) 450-5133	
Fax: (780) 913-0178				Fax: (780) 450-5542	
Agreement Id	92141		Control Id		
Project Id			Report Due Date	Dec 02, 2021	DUCU
Project Name			Received Date	Nov 26, 2021	KUJU
Project Location			Sampled By	Victor Bachmann	
Legal Location			Sampling Company	Innotech Alberta	
PO#	64381		Est. Disposal Date	Jan 01, 2022	
Proj. Acct. Code					

Service Information

Sample Id	1	Service	Service Name
	7796159	CCMEC	CCME Hydrocarbons: BTEX, F1-F4 in Soil
Date Sampled	Nov 26, 2021 12:00	DISP	by Cold Extraction
Priority	Rush 50%		Environmental Disposal Fee
Sample Description Temp: Received	PTAC F2 Fine HS R1 3.3 °C		

Other Billable Services	Service	Service Name	Quantity
	SUP-	VOC Coring Device	1.00
	SUP-MEOH	MEOH Vial	2.00

Service Count						
Service Name	Service Code	Quantity				
CCME Hydrocarbons: BTEX, F1-F4 in Soil by Cold Extraction	CCMEC	1				
Environmental Disposal Fee	DISP	1				
MEOH Vial	SUP-MEOH	2				
VOC Coring Device	SUP-CORER	1				

Notes

If required for invoice approval, please sign and return to the address indicated at the top of the page.

(Signature)



 Element
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Page 2 of 2 T: +1 (403) 291-2022 F: +1 (403) 291-2021 E: info.Calgary@element.com W: www.element.com

Confirmation of Service Request

Lot ID: 1539063

Number of Samples: 1

Printed Date: Dec 02, 2021

Please verify the following service request. If you have corrections or questions, please contact Client Services. No response to this confirmation of analysis will signify all services listed below are accurate.

Report Delivery Plan

Contact	Company	Address			
Simone Levy	InnoTech Alberta Inc.	Bag 4000, Hwy 16A & 75 Street			
		Vegreville, AB T9C 1T4			
		Phone: (780) 450-5474 Fax: (780) 913-0178			
		Email: Simone.levy@innotechalberta.ca			
Delivery	<u>Format</u>	Deliverables			
Email - Merge Reports	PDF	COA / COC			
Email - Merge Reports	PDF	COC / Test Report			
Email - Single Report	Standard Crosstab Without Tabs	Test Report			



T: +1 (780) 438-5522 F: +1 (780) 434-8586 E: info.Edmonton@element.com W: www.element.com

Report Trans Bill To: Attn: Sampled By: Company:	smission Cover Page InnoTech Alberta Inc. PO Box 8330 250 Karl Clark Road Edmonton, AB, Canada T6N 1E4 Les Spink	Project ID: Project Name: Project Location: LSD: P.O.: Proj. Acct. code:	PTAC F2 Flux 43110161.3001	Lot ID: Control Number: Date Received: Date Reported: Report Number:	1565211 Apr 14, 2022 Apr 21, 2022 2738126	
Contact	Company		Address			

oompany	Address				
InnoTech Alberta Inc.	PO Box 8330, 250 Karl Clark Road				
	Edmonton, AB T6N 1E4				
	Phone: (780) 450-5133 Fax:	(780) 450-5542			
	Email: innotech-finance@albertainnovates.				
<u>Format</u>	Deliverables				
PDF	COC / Invoice				
InnoTech Alberta Inc.	PO Box 8330, 250 Karl Clark Road				
	Edmonton, AB T6N 1E4				
	Phone: (780) 450-5033 Fax:	(780) 450-5083			
	Email: leslie.spink@albertainnovates.ca				
<u>Format</u>	Deliverables				
PDF	COC / Invoice				
InnoTech Alberta Inc.	Bag 4000, Hwy 16A & 75 Street				
	Vegreville, AB T9C 1T4				
	Phone: (780) 450-5474 Fax:	(780) 913-0178			
	Email: Simone.levy@innotechalberta.ca				
<u>Format</u>	Deliverables				
PDF	COC / COA				
PDF	COC / Test Report				
PDF	COC / Test Report / Invoice				
Standard Crosstab Without Tabs	Test Report				
	InnoTech Alberta Inc. Eormat PDF InnoTech Alberta Inc. Eormat PDF InnoTech Alberta Inc. Eormat PDF PDF PDF PDF PDF PDF Standard Crosstab Without Tabs	InnoTech Alberta Inc. PO Box 8330, 250 Karl Clark Road Edmonton, AB T6N 1E4 Phone: (780) 450-5133 Fax: Email: innotech-finance@albertainnovates. Eormat Deliverables PDF COC / Invoice InnoTech Alberta Inc. PO Box 8330, 250 Karl Clark Road Edmonton, AB T6N 1E4 Phone: (780) 450-5033 Fax: InnoTech Alberta Inc. PO Box 8330, 250 Karl Clark Road Edmonton, AB T6N 1E4 Phone: (780) 450-5033 Fax: Email: leslie.spink@albertainnovates.ca Eormat Deliverables COC / Invoice PDF COC / Invoice InnoTech Alberta Inc. Bag 4000, Hwy 16A & 75 Street Vegreville, AB T9C 1T4 Phone: (780) 450-5474 Fax: Email: Simone.levy@innotechalberta.ca Eormat Deliverables PDF COC / COA PDF COC / COA PDF COC / COA PDF PDF COC / Test Report PDF PDF COC / Test Report / Invoice Standard Crosstab Without Tabs Test Report			

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Analytical Re	eport						
Bill To: Attn: Sampled By: Company:	InnoTech A PO Box 83 250 Karl Cl Edmonton, T6N 1E4 Les Spink	Alberta Inc. 30 lark Road AB, Canada	Project ID: Project Name: Project Location: LSD: P.O.: Proj. Acct. code:	PTAC F2 Flux 43110161.3001	Lo Control Nun Date Rece Date Repo Report Nun	1565211 nber: ived: Apr 14, 2022 vrted: Apr 21, 2022 nber: 2738126	
Company			Reference Number	1565211-1	1565211-2	1565211-3	
			Sample Date	Apr 06, 2022	Apr 11, 2022	Apr 11, 2022	
			Sample Time	e NA	NA	NA	
			Sample Location				
			Sample Description	PTAC F2 Flux Coarse	PTAC F2 Flux Fine R1	PTAC F2 Flux Fine R2	
			Matrix	c Soil	Soil	Soil	
Analyte			Units	Results	Results	Results	Nominal Detection Limit
Mono-Aromatic	Hydrocarb	ons - Soil					
Benzene		Dry Weight	mg/kg	0.011	<0.005	<0.005	0.005
Toluene		Dry Weight	mg/kg	0.45	<0.02	<0.02	0.02
Ethylbenzene		Dry Weight	mg/kg	0.043	0.020	0.014	0.005
Total Xylenes (r	m,p,o)	Dry Weight	mg/kg	0.48	0.24	0.20	0.03
Methanol Field	Preservation	1		Yes	Yes	Yes	
Volatile Petrole	um Hydroca	arbons - Soil					
F1 C6-C10		Dry Weight	mg/kg	623	313	310	10
F1 -BTEX		Dry Weight	mg/kg	622	313	310	10
Extractable Pet	roleum Hyd	rocarbons - Soil					
Extraction Date		Total Extractables		15-Apr-22	15-Apr-22	15-Apr-22	
F2c C10-C16		Dry Weight	mg/kg	5660	7090	8550	25
F3c C16-C34		Dry Weight	mg/kg	94	125	137	50
F4c C34-C50		Dry Weight	mg/kg	<100	<100	<100	100
F4HTGCc C34-	-C50+	Dry Weight	mg/kg	<100	<100	<100	100
% C50+			%	<5	<5	<5	
Silica Gel Clear	nup						
Silica Gel Clear	nup			Done	Done	Done	
Soil % Moisture	9						

2.23

12.60

11.90

% by weight

Soil % Moisture

Moisture



Analytical Report

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c. Project ID: Project Name:	PTAC F2 Flux	Lot ID: Control Number:	1565211
d Project Location: hada LSD: P.O.: Proj. Acct. code:	43110161.3001	Date Received: Date Reported: Report Number:	Apr 14, 2022 Apr 21, 2022 2738126
	c. Project ID: Project Name: Project Location: ada LSD: P.O.: Proj. Acct. code:	c. Project ID: PTAC F2 Flux Project Name: g Project Location: ada LSD: P.O.: Proj. Acct. code: 43110161.3001	c. Project ID: PTAC F2 Flux Lot ID: Project Name: Control Number: Project Location: Date Received: ada LSD: Date Reported: P.O.: Report Number: Proj. Acct. code: 43110161.3001

		Reference Number	1565211-4	1565211-5		
		Sample Date	Apr 12, 2022	Apr 12, 2022		
		Sample Time	NA	NA		
		Sample Location				
		Sample Description	PTAC F2 Flux Fine R3	PTAC F2 Flux Comp		
		Matrix	Soil	Soil		
Analyte		Units	Results	Results	Results	Nominal Detection Limit
Mono-Aromatic Hydrocart	oons - Soil					
Benzene	Dry Weight	mg/kg	<0.005	<0.005		0.005
Toluene	Dry Weight	mg/kg	<0.02	<0.02		0.02
Ethylbenzene	Dry Weight	mg/kg	0.013	0.019		0.005
Total Xylenes (m,p,o)	Dry Weight	mg/kg	0.20	0.26		0.03
Methanol Field Preservatio	n		Yes	Yes		
Volatile Petroleum Hydroc	arbons - Soil					
F1 C6-C10	Dry Weight	mg/kg	348	454		10
F1 -BTEX	Dry Weight	mg/kg	348	454		10
Extractable Petroleum Hyd	drocarbons - Soil					
Extraction Date	Total Extractables		15-Apr-22	15-Apr-22		
F2c C10-C16	Dry Weight	mg/kg	8530	7690		25
F3c C16-C34	Dry Weight	mg/kg	150	135		50
F4c C34-C50	Dry Weight	mg/kg	<100	<100		100
F4HTGCc C34-C50+	Dry Weight	mg/kg	<100	<100		100
% C50+		%	<5	<5		
Silica Gel Cleanup						
Silica Gel Cleanup			Done	Done		
Soil % Moisture						
Moisture	Soil % Moisture	% by weight	12.50	13.20		

RhSeunem

Approved by:

Randy Neumann, BSc **Division Director**

Data have been validated by Analytical Quality Control and Element's Integrated Data Validation System (IDVS). Generation and distribution of the report, and approval by the digitized signature above, are performed through a secure and controlled automatic process.



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Quality Cont	rol							
Bill To:	To: InnoTech Alberta Inc. Project ID: PTAC F2 Flux PO Box 8330 Project Name: 250 Karl Clark Road Project Location: Edmonton, AB, Canada LSD: T6N 1E4 P.O.: thr: Los Spink Project code: 43110161.3001		F2 Flux	Lot ID: Control Number: Date Received: Date Reported: Report Number:	1565211 Apr 14, 2022 Apr 21, 2022 2738126			
Sampled By:	Les of	ULIK.	1 10]. / 1001. 0000.	10110				
Company:								
Extractable I Soil	Petrole	eum Hydrocarbo	ns -					
Blanks		Units	Measur	ed	Lower Limit	Upper Limit		Passed (
F2c C10-C1	6	µg/mL		0	-10	10		}
F3c C16-C3	4	µg/mL		0	-30	30		y
F4c C34-C5	0	µg/mL		0	-20	20		}
F4HTGCc C	34-C50-	+ μg/mL		0	-20	20		}
Date Acqui	red: A	pril 15, 2022						
Calibration Cl	heck	Units	% Recove	ery	Lower Limit	Upper Limit		Passed
F2c C10-C1	6	µg/mL	100	.10	80	120)

E

S

Blanks	Units	Measured	Lower Limit	Upper Limit	Passed QC
F2c C10-C16	µg/mL	0	-10	10	yes
F3c C16-C34	µg/mL	0	-30	30	yes
F4c C34-C50	µg/mL	0	-20	20	yes
F4HTGCc C34-C50+	µg/mL	0	-20	20	yes
Date Acquired: April	15, 2022				
Calibration Check	Units	% Recovery	Lower Limit	Upper Limit	Passed QC
F2c C10-C16	µg/mL	100.10	80	120	yes
F3c C16-C34	µg/mL	101.02	80	120	yes
F4c C34-C50	µg/mL	98.96	80	120	yes
F4HTGCc C34-C50+	µg/mL	95.11	80	120	yes
Date Acquired: April	15, 2022				
Matrix Spike	Units	% Recovery	Lower Limit	Upper Limit	Passed QC
F2c C10-C16	mg/kg	81	70	130	yes
F3c C16-C34	mg/kg	87	70	130	yes
F4c C34-C50	mg/kg	85	70	130	yes
F4HTGCc C34-C50+	mg/kg	84	70	130	yes
Date Acquired: April	15, 2022				

Mono-Aromatic Hydrocarbons - Soil

Blanks	Units	Measured	Lower Limit	Upper Limit	Passed QC
Benzene	ng	0	-0.005	0.005	yes
Toluene	ng	0	-0.06	0.06	yes
Ethylbenzene	ng	0	-0.030	0.030	yes
Total Xylenes (m,p,o)	ng	0	-0.09	0.09	yes
Styrene	ng	0	-0.030	0.030	yes
Date Acquired: April 1	5, 2022				
Calibration Check	Units	% Recovery	Lower Limit	Upper Limit	Passed QC
Benzene	ng	104.50	80	120	yes
Toluene	ng	98.17	80	120	yes
Ethylbenzene	ng	109.80	80	120	yes
m,p-Xylene	ng	115.56	80	120	yes
Total Xylenes (m,p,o)	ng	114.91	80	120	yes
Churana		00 I T		100	

Volatile Petroleum Hydrocarbons - Soil

Date Acquired: April 15, 2022

Blanks	Units	Measured	Lower Limit	Upper Limit	Passed QC
F1 C6-C10	ng	0	-10	10	yes
Date Acquired:	April 15, 2022				



Bill To: InnoTech Alberta Inc.

T6N 1E4

Attn: Les Spink

PO Box 8330

250 Karl Clark Road

Edmonton, AB, Canada

PTAC F2 Flux

43110161.3001

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Lot ID: **1565211** Control Number: Date Received: Apr 14, 2022 Date Reported: Apr 21, 2022 Report Number: 2738126

Method of Analysis

Sampled By: Company:

Methodology and Notes

Method Name	Reference	Method	Date Analysis Started	Location
BTEX-CCME - Soil	CCME	 * Reference Method for Canada-Wide Standard for PHC in Soil, CWS PHCS TIER 1 	Apr 15, 2022	Element Calgary
BTEX-CCME - Soil	US EPA	 Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis/Gas Chromatography Mass Spectrometry, 5021/8260 	Apr 15, 2022	Element Calgary
TEH-CCME-Soil (Shake)	CCME	 * Reference Method for Canada-Wide Standard for PHC in Soil, CWS PHCS TIER 1 	Apr 15, 2022	Element Calgary
References		* Reference Method Modified		

Project ID:

LSD:

P.O.:

Project Name:

Project Location:

Proj. Acct. code:

CCME	Canadian Council of Ministers of the Environment
US EPA	US Environmental Protection Agency Test Methods

Please direct any inquiries regarding this report to our Client Services group. Results relate only to samples as submitted. The test report shall not be reproduced except in full, without the written approval of the laboratory.

		Invoice To				Report ⁻	Го				Α	dditi	ona	l Rep	orte	s to	
eleme 🌔	company:	Alberta Innovat	es	Company:		Innot	ech Alberta			1) Name: Victor Bachmann							
www.Element.c	om Address:	250 Karl Clark Rd	NW	Address:		250 Ka	arl Clark Rd.			E-mail: <u>:or.Bachmann@innotechalbert</u>							
Project Informa	tion	Edmonton, AB				Edmonton,	AB			2) N	ame:						
Project ID: PTA	C F2 Flux Attention:	Les Spink		Attention:		Sim	one Levy			E-m	ail:						
Project Name:	Phone:			Phone:		780-	450-5210					Sar	nple	Cus	tody	/	
Project Location:	Cell:	780.450.5033	3	Cell:						Sampled by:							
Legal Location:	Fax:			Fax:	Fax:						Company:						
PO/AFE#:	E-mail 1:	les.spink@albertainr	novates.ca	<u>a</u> E-mail 1:	one.Levy@	Innotech/	Albert	<u>a.ca</u>	a I authorize Element to proceed with								
Proj. Acct. Code: 4311	0161.3001 Agreement	ID: 9214	1	E-mail 2:					the v	vork i	ndica	ited o	n thi	s forr	n:		
Quote #:	2141 Copy of Re	port: No		Copy of Inv	voice:	mone.Levy@InnotechAlberta.ca Sampled by: YES I authorize Element to the work indicated of Signature: PADWORG SPIGEC AB Tier 1 BCCSR Other (list below) Setting of the top of top of the top of to											
	RUSH Priority		Repo	rt Results		Requirem	ents			Date	e/Time	:					
Same Day (200% Next Day/Two Da Three or Four Day ✓ 5 to 7 Days (Regu Date Required) When "AS, default to a and turn aro (s (50%) the lab prio ilar TAT) not all samp in	 ✓ Ema Onli Fax 	il V QA/QC ne V PDF V Excel		CDWORG [B Tier 1 [Other (list b	SPIGEC BCCSR Delow)	of Containers										
Special Instruc	ctions/Comments (please incl	ng phone ni	umber if differen	t from ab	ove).	-	Number (MaOH Ei	COMEC									
Site I.D.	Sample D	escription	Depth start e in cm m	nd Date/1	Time Iled	Matrix	Sampling method	# v		(v	E rel	inter evan	tes t sa	ts ab mple	ove s be	low)
1	PTAC F2 F	lux Coarse		2022/4/	6 0:00			3 x	х								
2	PTAC F2 F	lux Fine R1		2022/4/1	1 0:00			3 x	X								
3	PTAC F2 F	lux Fine R2		2022/4/1	1 0:00			Зx	х								
4	PTAC F2 F	lux Fine R3		2022/4/1	2 0:00		3 x	X								_	
5	PTAC F2	Flux Comp		2022/4/1	2 0:00	2 0:00			X						_		
6											_	_	\square				
7							-		-	┥┥			\square		_		
8	<u> </u>							┝╌┠╴		┥┨			$\left \cdot \cdot \right $	_	+	$\left \right $	
9			 					┝─╂╴	╋	╂╴┨		+	┝─┤		+	┝╌┤	
10							-		╋	+			\square		—	$\left \right $	
						r							\vdash			┝─┤	
12														-+		$\left \right $	
13									_				\square				
14										+					-		
Please indicate Submission of this form ac terms and conditions (http:	Lo	Indicate lot # ot: 15652				Ter rece Delive	np. ived: ery Me	-3 ethod:	3	Dat P	te/Tin ≷_j4 Q∧ (ie stai PM12	np: :(:()	<u>. </u>			
Page <u>1</u> of <u>1</u> ED 120-005	Control #			Waybill: Received by:							. Om						

Coarse	Lab ID	Total	F2 fraction	nC10apex-nC12apex	nC10apex-nC12apex	nC10apex-nC12apex	nC12apex-nC16apex	nC12apex-nC16apex	nC12apex-nC16apex
	T			total	saturates	aromatic	total	saturates	aromatic
		ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
EXP 1									
time 1	FL21-2006-006	0.631	0.484	0.417	Not requested	Not requested	0.067	Not requested	Not requested
time 2	FL21-2006-007	0.872	0.665	0.581	0.436	0.145	0.084	0.065	0.019
time 3	FL21-2006-008	1.23	0.942	0.797	0.596	0.201	0.145	0.111	0.033
time 4	FL21-2006-009	1.57	1.202	1.053	0.785	0.268	0.149	0.113	0.036
time 5	FL21-2006-010	2.02	1.502	1.370	1.034	0.336	0.132	0.100	0.032
EXP 2									
time 1	FL21-2006-011	1.328	0.970	0.864	0.634	0.231	0.106	0.081	0.025
time 2	FL21-2006-012	2.027	1.472	1.332	0.962	0.370	0.139	0.099	0.040
time 3	FL21-2006-013	2.055	1.498	1.362	0.987	0.375	0.136	0.097	0.039
time 4	FL21-2006-014	No sample was pulled	error in sampling						
EXP 3									
time 1	FL21-2006-015	1.559	1.135	1.018	0.739	0.279	0.116	0.087	0.030
time 2	FL21-2006-016	2.028	1.449	1.328	0.961	0.367	0.120	0.088	0.033
time 3	FL21-2006-017	2.032	1.446	1.329	0.963	0.366	0.116	0.085	0.031
time 4	FL21-2006-018	2.072	1.512	1.377	0.995	0.382	0.135	0.099	0.036
Fine	Lab ID	Total	F2 fraction	nC10apex-nC12apex	nC10apex-nC12apex	nC10apex-nC12apex	nC12apex-nC16apex	nC12apex-nC16apex	nC12apex-nC16apex
				total	saturates	aromatic	total	saturates	aromatic
		ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)	ppm (ug/mL)
EXP 1									
time 1	FL21-2006-019	0.902	0.683	0.614	0.443	0.170	0.069	0.056	0.012
time 2	FL21-2006-020	1.210	0.915	0.830	0.603	0.227	0.085	0.067	0.018
time 3	FL21-2006-021	1.270	0.964	0.880	0.640	0.240	0.084	0.067	0.017
time 4	FL21-2006-022	1.37	1.077	0.978	0.714	0.263	0.099	0.076	0.022
EXP 2									
time 1	FL21-2006-023	0.653	0.521	0.465	0.3445	0.1202	0.057	0.043	0.014
time 2	FL21-2006-024	1.208	0.955	0.867	0.639	0.228	0.088	0.067	0.021
time 3	FL21-2006-025	1.427	1.132	1.020	0.744	0.275	0.112	0.085	0.027
time 4	FL21-2006-026	No sample was pulled	error in sampling						
EXP 3									
time 1	FL21-2006-027	0.779	0.631	0.557	0.4021	0.1544	0.075	0.056	0.019
time 2	FL21-2006-028	1.261	1.008	0.908	0.657	0.251	0.100	0.076	0.024
time 3	FL21-2006-029	1.321	1.050	0.952	0.689	0.263	0.098	0.075	0.024
time 4	FL21-2006-030	1.451	1.148	1.040	0.755	0.284	0.108	0.082	0.026

APPENDIX C. Summary of Headspace Analytical Results

Average and standard deviation for headspace results by time point are provided in Tables C1 to C4.

Table C1. Part 1 Coarse-grained soil experiment – average and standard deviation by sampling time.

Replicated experiment and sample time from inception							nC ₁₀ ape	ex-nC ₁₂ ap	ex				nC ₁₂ ap	Aromatic sub-fraction Aromatic sub-fraction nL stdev μg/mL stdev					
	Total PHC		РНС	C F2	То	tal	Saturate su	b-fraction	Aromatic s	ub-fraction	Tot	tal	Saturate s	ub-fraction	raction Aromatic sub-fra				
	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev			
10 min	1.484	0.135	1.102	0.119	0.979	0.101	0.719263	0.07766	0.259314	0.025493	0.124	0.022	0.093754	0.016956	0.029972	0.005399			
40 min	2.024	0.005	1.474	0.026	1.343	0.023	0.985698	0.0418	0.357783	0.019094	0.131	0.010	0.095747	0.0069	0.03482	0.004737			
60 min	2.043	0.016	1.472	0.037	1.346	0.023	0.975	0.017	0.371	0.007	0.126	0.014	0.091	0.008	0.035	0.005			
90 min	2.043 0.016 1 2.072 n/a 1		1.512	n/a	1.377	n/a	0.995	n/a	0.382 n/a		0.135 n/a		0.099	n/a	0.036	n/a			

 Table C2.
 Part 1 Fine-grained soil experiment – average and standard deviation by sampling time.

Replicated experiment and sample time from inception							nC ₁₀ ape	x-nC ₁₂ ape	C				nC ₁₂ apex-nC ₁₆ apex						
	Total PHC		РНС	C F2	То	tal	Saturate s	ub-fraction	Aromatic s	ub-fraction	То	tal	Saturate s	ub-fraction	Aromatic s	ub-fraction			
	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	μg/mL	stdev	µg/mL	stdev	µg/mL	stdev	μg/mL	stdev	µg/mL	stdev			
5 min	1.484	0.124	0.612	0.082	0.545	0.075	0.396621	0.049618	0.148375	0.025667	0.067	0.009	0.051727	0.007897	0.014969	0.003236			
20 min	2.024	0.030	0.959	0.047	0.868	0.039	0.632842	0.027728	0.235408	0.013114	0.091	0.008	0.069652	0.005342	0.021286	0.003003			
30 min	2.043	0.080	1.048	0.084	0.950	0.070	0.691	0.052	0.259	0.018	0.098	0.014	0.075	0.009	0.023	0.005			
40 min	2.072	0.055	1.112	0.050	1.009	0.044	0.735	0.029	0.274	0.274 0.015		0.103 0.007		0.079 0.004		0.003			

Replicated				PHC F2 nC ₁₀ apex-nC ₁₂ apex Total Saturate sub-fraction			nC ₁₀ a	apex-nC ₁₂ ap	ex		nC ₁₂ apex-nC ₁₆ apex									
experiment and sample	Total VOC		PHC				Aromatic s	ub-fraction	Tot	al	Saturate si	ub-fraction	Aromatic sub-fraction							
time from inception	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	μg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev				
Coarse Average																				
15 min	1.097	0.091	0.843	0.065	0.730	0.047	Not re	quested	Not requested		0.113 0.022		Not requested		Not rec	uested				
30 min	1.030	0.048	0.812	0.045	0.676	0.022	Not requested		Not requested		ot requested Not requested (0.136	0.023	Not requested		Not requested		d Not reque	
60 min	0.907	0.032	0.726	0.027	0.596	0.019	0.437	0.014	0.159	0.006	0.129	0.008	0.093	0.006	0.036	0.002				

Table C3. Part 2 Coarse-grained soil experiment – average and standard deviation by sampling time.

Table C4. Part 2 Fine-grained soil experiment – average and standard deviation by sampling time.

Replicated experiment	Total VOC		DHC	. 53			nC ₁₀ a	pex-nC ₁₂ ap	ex			nC ₁₂ apex-nC ₁₆ apex				
and sample					Total		Saturate sub-fraction		Aromatic sub-fraction		Total		Saturate sub-fraction		Aromatic sub-fraction	
inception	µg/mL	stdev	µg/mL	stdev	µg/mL	µg/mL stdev		stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev	µg/mL	stdev
Fine Average																
15 min	0.389	0.073	0.304	0.067	0.244	0.049	Not re	quested	Not requested		0.060 0.020		Not requested		Not req	uested
30 min	0.374	0.056	0.284	0.039	0.224	0.037	Not requested		Not requested		0.059	0.007	Not requested		Not req	uested
60 min	0.326	0.067	0.244	0.049	0.186	0.040	0.103	0.026	0.083	0.015	0.059	0.009	0.035	0.006	0.023	0.003


APPENDIX C: F2 MANAGEMENT LIMITS FOR TRENCH WORKER SCENARIO CALCULATIONS



Appendix C – F2 Management Limit Calculations - Trench Worker Scenario

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2.2	Trench Air Exchange Rate	C-5
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1.0 PURPOSE AND OVERVIEW

This Appendix provides details of the calculations for the PHC F2 management limit component for the trench worker scenario.

The PHC F2 management limit for the trench worker scenario is the concentration of F2 in coarse or fine soil that will not result in unacceptable exposure for hypothetical workers in a trench cut through F2 impacted soil.

Previous work (CCME, 2008) calculated this management limit using:

- 1. A diffusion model (VDEQ, 2005) to estimate the flux rate of F2 at the trench face.
- 2. Assumed air exchange rates for the trench.
- 3. Occupational exposure limits for kerosene/jet fuel.
- 4. A simple box model to describe the mixing of F2 vapours and trench air.

There was significant uncertainty in the management limits calculated by CCME (2008) primarily related to elements 1 and 2 above. Both these elements have been refined in the current work and the current calculations are based on measured data rather than assumed or modelled values. The current work calculates the management limit using:

- 1. Measured, rather than modelled F2 flux rates.
- 2. Trench air exchange rates based on actual measurements in physical trenches.
- 3. Occupational exposure limits for kerosene/jet fuel.
- 4. A simple box model to describe the mixing of F2 vapours and trench air.

For these reasons, the management limit values calculated in this Appendix have significantly less uncertainty than the values calculated by CCME (2008).

2.0 MANAGEMENT LIMIT CALCULATION

The four elements noted above are required in order to calculate the PHC F2 management limit component for the trench worker scenario. These elements are discussed in the following sections.

2.1 Determining F2 Flux Rates from Flow-Through Test Results

InnoTech (2020, 2022) completed three phases of lab-based investigations related to measuring the flux rate of F2 from the surface of F2-spiked soils.



- Phase 1 measured the equilibrium headspace vapour concentrations of F2 in equilibrium with F2 spiked soils.
- Phase 2 measured how the transient headspace vapour concentrations of F2 increased over time in a static headspace in contact with F2 spiked soils.
- Phase 3 measured the F2 vapour concentration in flow-through cells containing F2 spiked soils with an air exchange rate of 46/hour.

Full experimental details are available in the InnoTech reports included in Appendix A (Phase 1) and Appendix B (Phases 2 and 3).

Phase 1 was conducted as a range finding experiment. Phase 2 showed how the F2 concentration changed in headspace air over time. However, Phase 3, which measured the F2 vapour concentration in flow-through cells containing F2 spiked soils provides the most direct measurement of F2 flux rates at the soil surface and the results of Phase 3 are used here to calculate representative F2 flux rates at the surface of fine- and coarse-grained soils spiked with F2.

In Phase 3, fine- and coarse-grained soils were spiked with F2 at a nominal concentration of 10,000 mg/kg. Soil samples were taken immediately prior to conducting the Phase 3 flux rate experiments and analyzed for F2 hydrocarbons. Mean measured F2 concentrations at the start of the Phase 3 flux rate experiments were:

- Fine Soil: 8,057 mg/kg (average of 3 replicates, InnoTech, 2022, Appendix B, Table 6).
- Coarse Soil: 5,660 mg/kg (composite of 3 replicates, InnoTech, 2022, Appendix B, Table 6).

Spiked soils were compacted into the base of 500 ml flow-through containers (3 replicates each for fine- and coarse-grained soils). At time 0, air flow was started through the containers at 23 L/hour (46 air exchanges/hour). Air samples were withdrawn at 15 min, 30 min and 60 min and analyzed for F2 by GC-FID. Analytical results are summarized in Tables 7 and 8 of Appendix B and illustrated graphically in Figure 1 below.

As seen in Figure 1, the F2 concentration is the flow-through cells is higher for the coarse- than the fine-grained soil, and the concentrations for both fine- and coarse-grained soils decrease slightly over the 60 minutes of the experiment. This is interpreted as being related to depletion of F2 in the surficial soil layers.

For the purposes of the current report, it is assumed that workers would not enter a freshly dug trench for at least the first hour due to the necessity of providing shoring to ensure trench stability and therefore the data from 60 minutes are used as the departure point for calculating management limits.



Based on the data provided in Tables 7 and 8 of Appendix B, the mean F2 vapour concentrations in the flow-through cells at 60 minutes in this experiment are:

- Fine Soil: mean 60-minute vapour concentration = 0.245 ug/ml (n=3).
- Coarse Soil: mean 60-minute vapour concentration = 0.726 ug/ml (n=3).



Figure C-1 F2 Concentration vs. Time in Flow-Through Cells

The air in the flow-through cell is constantly being changed, and therefore the concentration of F2 in the cell is proportional to the flux rate at which F2 is entering the airspace from the soil surface. Knowing the air exchange rate in the cell (ACHc) and the surface area of the spiked soil in the cell (Ac) allows the F2 flux rate from the soil surface into the airspace to be calculated from first principles as follows. Note that in the following equations and definitions, the subscript c is used throughout to identify parameter values that relate to the flow-through cell. This is done to avoid confusion with similar parameters and equations that will be used for similar processes in the trench box model in Section 2.4, for which the subscript τ will be used.



The time (T_c) taken for the air to be changed once in the flow-through cell is given by:

$$T_C = \frac{1}{ACH_C}$$
 Equation 1

Where ACH_C is the air exchange rate. If the F2 flux rate at the soil surface is $F_C mg/m^2$ /hour, then the mass (M_C) of F2 entering the cell over the period of one air exchange is given by

$$M_C = F_C \cdot A_C \cdot T_C \qquad \qquad \text{Equation 2}$$

where A_C is the surface area of the spiked soil in the flow-through cell. The F2 vapour concentration, C_{VC} , in the cell is therefore given by:

$$C_{VC} = \frac{M_C}{V_C} = \frac{F_C \cdot A_C \cdot T_C}{V_C}$$
 Equation 3

Re-arranging this equation to solve for the flux rate, Fc gives:

$$F_C = \frac{C_{VC} \cdot V_C \cdot ACH_C}{A_C}$$
 Equation 4

Where:

 F_c = flux rate of F2 from soil surface into the air of the flow-through cell (mg/m²/hour)

 C_{VC} = vapour concentration of F2 in the airspace of the flow-though cell (mg/m³)

Vc = volume of the flow-though cell (m³)

ACHc= air exchange rate in the flow-though cell (exchanges per hour)

Ac = surface area of spiked F2 soil in the flow-though cell (m^2)

Parameter values used in the flux rate calculation are summarized in Table C-1. Values are provided in the units in which they were measured (to allow comparison with the data in Appendix B) as well as being converted to the appropriate units for the above equation.



Table C-1 Parameter Values Relating to the Flow-Through Cell				
	Fine Soil		Coarse Soil	
Parameter	Measurement Units	Calculation Units	Measurement Units	Calculation Units
Mean F2 vapour concentration in flow-through cell (Cc)	0.245 ug/ml	245 mg/m ³	0.726 ug/ml	726 mg/m ³
Volume of flow-through cell (Vc)	500 ml	0.0005 m ³	500 ml	0.0005 m ³
Air exchange rate (ACHc)	46 exch/hour	46 exch/hour	46 exch/hour	46 exch/hour
Soil surface area (Ac)	50.3 cm ²	0.00503 m ³	50.3 cm ²	0.00503 m ³

Substituting these values in the above equation yields the F2 flux rates for the flow-through experiments at the 60-minute time point:

- Fine soil: F2 flux rate in flow-through cell = 1,120 mg/m²/hour
- Coarse soil: F2 flux rate in flow-through cell = 3,320 mg/m²/hour

2.2 Trench Air Exchange Rate

The CCME (2008) calculations adopt the recommended trench air exchange rates in the VDEQ model, which are 360/hour where trench width is greater than depth and 2/hour otherwise. Neither of these air exchange rates were based on measured values in actual trenches. Since that time, some high-quality research has been done measuring actual air exchange rates in trenches constructed for the purpose. Thompson et al. (2017) conducted an empirical field study specifically to investigate the validity of the default air exchange rates of 360/hour and 2/hour in the VDEQ model. These authors measured the air exchange rate in five trenches all 3 feet wide and 8 feet deep in various orientations to the prevailing wind direction and under various conditions. Their methodology involved filling each trench with carbon dioxide and measuring the decrease of carbon dioxide concentration over time under various conditions of prevailing wind. Trench air exchange rates during these field studies ranged from 34 to 79 per hour with an average of 46 per hour for trenches with a ratio of width to depth that is less than one. Their average air exchange rate of 46/hour is adopted here.

2.3 Occupational Exposure Limits for Kerosene/Jet Fuel

In the absence of an established acute/sub-chronic inhalation reference value for F2, the 2008 PHC CWS used occupational exposure limits as a reference point to set management limits for the worker in a trench scenario (Meridian, 2006). This approach is retained here.



Kerosene is a petroleum hydrocarbon distillate which is obtained from the fractional distillation of petroleum between 150 and 275 °C. Kerosene predominantly consists of molecules containing between 9 and 16 carbon atoms (Shepherd et al., 2000) and thus closely resembles the range of F2. Accordingly, occupational exposure limits for kerosene are adopted here for setting management limits for the trench worker scenario.

Available occupational inhalation limits for kerosene (summarized in OSHA, 2022) include:

- NIOSH (National Institute for Occupational Safety and Health): The recommended airborne exposure limit (REL) for kerosene is 100 mg/m³ averaged over a 10-hour work shift.
- ACGIH (American Conference of Governmental Industrial Hygienists): The threshold limit value (TLV) for kerosene and jet fuels is 200 mg/m³ averaged over an 8-hour work shift.
- US EPA (United States Environmental Protection Agency): The US EPA has set an Acute Exposure Guideline Level-1 (AEGL-1) of 290 mg/m³ for military jet fuels JP-5 and JP-8 (similar in composition to kerosene and F2).
- In addition, NRC (2003) set a permissible exposure level (PEL) of 350 mg/m³ for the military jet fuel JP-8 (similar in composition to kerosene and F2).

In this document, the lowest of the above values, the NIOSH REL of 100 mg/m³ was selected as the reference point for setting management limits for the trench worker inhalation scenario.

2.4 Box Model – Mixing F2 Vapours and Trench Air

In the current work, a simple box model is used to describe the mixing of F2 vapours with air in the trench. CCME (2008) indicated that vapour/air mixing in the trench was addressed using a simple box model, however the equation for that model was not provided in CCME (2008) and therefore the equation for a simple box model is developed from first principles here.

The primary assumptions of the box model are that the concentration of vapours, in this case F2, in the trench is controlled by:

- the flux of F2 from the trench walls;
- complete mixing of the F2 vapours with trench air; and,
- trench air being regularly replaced based on the air exchange rate for the trench.

These processes are illustrated in Figure 2 below.





Figure C-2 Trench Box Model

The equation for the trench box model has a similar basis to the model developed for the flowthrough cell in Section 2.1 above. Several of the parameters are similar and the subscript τ is used in this section to identify parameters that relate to the trench model rather than the cell model.

The time (T_T) taken for the air to be changed once in the trench is given by

$$T_T = \frac{1}{ACH_T}$$
 Equation 5

Where ACH_T is the air exchange rate in the trench. If the F2 flux rate at the trench face is F_T mg/m²/hour, then the mass (M_T) of F2 entering the trench in the time taken for one air exchange is given by

$$M_T = F_T \cdot A_T \cdot T_T$$
 Equation 6

where A_T is the trench surface area with F2 impacted soil. For the purposes of these calculations, it is assumed that the F2 soil impacts extend over the full length (L) and depth (D) of both sidewalls of the trench but not the base or ends of the trench. Accordingly, the trench surface area with F2 impacted soil (A_T) is given by:

$$A_T = 2 \cdot L \cdot D$$
 Equation 7



The volume of the trench (V_T) is given by:

$$V_T = L \cdot D \cdot W$$
 Equation 8

Where L, D, and W are the length, depth, and width of the trench, respectively.

The F2 flux rate in the flow-though cells was determined in Section 2.1 above, and just needs to be scaled by the ratio of the F2 concentration in soil surrounding the trench (CsT) to the spiked F2 soil concentration used in the flow-through cell experiments (CsC):

$$F_T = F_C \cdot \frac{c_{ST}}{c_{SC}}$$
 Equation 9

The F2 vapour concentration in the trench, CvT, is therefore given by:

$$C_{VT} = \frac{M_T}{V_T} = \frac{F_T \cdot A_T \cdot T_T}{V_T}$$
 Equation 10

This can be re-organized by combining equations 5, 6, 7, 8 and 9 to give:

$$C_{VT} = \frac{2 \cdot F_C \cdot C_{ST}}{C_{SC} \cdot W \cdot A C H_T}$$
 Equation 11

And re-arranged to give the soil concentration surrounding the trench C_{ST} in terms of parameters who:

$$C_{ST} = \frac{C_{VT} \cdot C_{SC} \cdot W \cdot ACH_T}{2 \cdot F_C}$$
 Equation 12

Where:

Cst	=	F2 soil concentration surrounding the trench (mg/kg)
CVT	=	F2 vapour concentration in the trench (mg/m ³)
Csc	=	spiked F2 soil concentration in the flow-through cell (mg/kg)
W	=	width of trench (m)
ACHT	=	air exchange rate in the trench (exchanges per hour)
Fc	=	F2 flux rate determined in the tests in the flow-through cell, and corresponding
		to the soil concentration Csc noted above (mg/m ² /hour)
Cst	=	volume of the flow-though cell (m ³)



2.5 Calculation of Management Limit

The management limits for fine- and coarse-grained soils can now be calculated from equation 12 by setting the F2 vapour concentration in the trench to the occupational exposure limit identified in Section 2.3 and substituting appropriate values for the other parameters as indicated in Table C-2 below.

Table C-2Parameter Values Relating to the Trench				
Parameter and Corresponding Symbol in	Paramet	Carrier		
Equation 12	Fine Soil	Coarse Soil	Source	
Occupational exposure limit (CvT)	100 mg/m ³	100 mg/m ³	Section 2.3	
Spiked F2 soil concentration in flow-through cell (Csc)	8,057 mg/kg	5,660 mg/kg	Section 2.1	
Trench width (W)	1 m	1 m	See below	
Air exchange rate in trench (ACHT)	46 exchanges/hour	46 exchanges/hour	Section 2.2	
F2 flux rate from flow-through tests (Fc)	1,120 mg/m²/hour	3,320 mg/m²/hour	Section 2.1	

The trench width (W) was set at 1 m which was assumed to be the narrowest trench that it was realistic for a worker to enter. Greater values for W in equation 12 would result in higher values for management limit, and thus using the smallest realistic value for W is conservative.

Substituting the values from Table C-2 into equation 12 allows the following values to be calculated for the F2 management limits protective of the worker in trench scenario (rounded to the nearest 1,000 mg/kg):

- Fine Soil: Management limit = 17,000 mg/kg
- Coarse Soil: Management limit = 4,000 mg/kg



3.0 REFERENCES

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