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Paragon Soil and Environmental Consulting Inc. 2018. *Literature Review of Naturally-Occurring Petroleum Hydrocarbons in Oil Sands Mining Reclamation Practice*. Canada's Oil Sands Innovation Alliance, 2020, <u>www.cosia.ca</u>.



Literature Review of Naturally-Occurring Petroleum Hydrocarbons in Oil Sands Mining Reclamation Practice

Prepared for:

Canada's Oil Sands Innovation Alliance Joint Industry Project Partners

Syncrude Canada Limited Imperial Oil Resources Limited Canadian Natural Resources Limited

Prepared by:

Paragon Soil and Environmental Consulting Inc.

August 2018

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ABBREVIATIONS

AER	Alberta Energy Regulator
ASCS	Aurora Soil Capping Study
AWHC	available water holding capacity
BTEX	benzene, toluene, ethylene and xylene
CCME	Canadian Council of Ministers of the Environment
CEMA	Cumulative Environmental Management Association
cm	centimetre
CNRL	Canadian Natural Resources Ltd.
COSIA	Canadian Oil Sands Innovation Alliance
ECN	equivalent carbon number
EMF	ecotomycorrhizal fungi
EPEA	Environmental Protection and Enhancement Act
FFM	forest floor material
Imperial	Imperial Oil Resources Ltd.
JIP	Joint Industry Project
Ks	saturated hydraulic conductivity
LOS	lean oil sand
m	metre
m/s	metres per second
mg/kg	milligrams per kilogram
mg/L	milligrams per litre
NA	naphthenic acid
NorthWind	NorthWind Land Resources Inc.
NP	net percolation
NPHC	naturally-occurring petroleum hydrocarbon
OSPW	oil-sands-affected process water
PAH	polyaromatic hydrocarbon
Paragon	Paragon Soil and Environmental Consulting Inc.
PHC	petroleum hydrocarbon
SFR	sand to fines ratio
Syncrude	Syncrude Canada Ltd.
Tr	actual transpiration

1 INTRODUCTION

Paragon Soil and Environmental Consulting Inc. (Paragon) was contracted to complete a literature review of naturally-occurring petroleum hydrocarbons (NPHCs) in oil sands mining reclamation practice for the Canadian Oil Sands Innovation Alliance (COSIA) Joint Industry Project (JIP) partners Syncrude Canada Ltd. (Syncrude), Imperial Oil Resources Ltd. (Imperial), and Canadian Natural Resources Ltd. (CNRL).

The main objective of the COSIA JIP on PHCs in oil sands mining reclamation practice (the Project) is to provide capping thickness and cover design guidance to industry members. Recent (2018) *Environmental Protection and Enhancement Act* (EPEA) approval clauses received by Canadian Natural Upgrading Ltd. require a risk assessment of elevated NPHC levels found in overburden and interburden, located on reclaimed landforms and covered by reclamation materials. This literature review is intended to collate existing information related to reclamation performance on soil materials, interburden and overburden containing NPHCs, in preparation for a risk identification and assessment process that will be undertaken by the JIP partners, AER specialists, and additional review experts.

Prior context for the literature review and risk assessment work includes the Aurora Soil Capping Study (ASCS) at the Syncrude Aurora North Mine (which has focused on NPHCs in overburden material), and a literature review by Paragon for Imperial (Paragon, 2017) that focused on the behaviour and fate of gravimetrically-heavy NPHCs in overburden and interburden containing lean oil sand. This literature review is a bibliographical summary that builds on previous review work from peer-reviewed sources, and applies the results of internally-developed studies from the JIP partners and other "gray" literature sources.

The focus areas of the literature review include:

- Geologic history of the region and its relevance to NPHCs in the local region.
- The occurrence of NPHCs in the region, based largely on the work completed by the Cumulative Environmental Management Association (CEMA):
 - distribution and variability in the natural landscape.
 - distinction between forms and types of NPHCs (e.g., interburden, lean oil sand, as well as aggregates and bands of NPHCs in surface Pleistocene soil reclamation material).
- NPHC composition characteristics and degradation potential characteristics *in situ* (predisturbance) and *ex situ* (after redistribution into the closure landscape).
- In situ and ex situ soil chemical and physical properties of soil reclamation materials, interburden and overburden containing NPHCs in the closure landscape.
- Current operator overburden landform design approaches (e.g., strategic placement of interburden and overburden materials or no separation of overburden), surface hydrologic considerations and controls, and soil cover designs.
- Environmental performance of soil reclamation materials, interburden and overburden containing NPHCs on the closure landscape (e.g., biological, movement and degradation potential).
- Existing regulatory guidance regarding assessment of the risk posed by NPHCs in soil reclamation materials, interburden and overburden.

2 REGIONAL GEOLOGIC HISTORY

Geologic activity during the Lower Cretaceous played a prominent role in the formation of the oil sand bitumen resource, while geologic activities during the last continental glacial advance and retreat shaped the landscape observed today in the Athabasca Oil Sands region. A description of the geologic setting and the resulting sediments that overlie the oil sand bitumen resource are described in this section.

The Mannville Group McMurray Formation and Wabiskaw Member stratigraphic intervals of the Lower Cretaceous Age comprise the oil sand bitumen resource (Wightman and Pemberton, 1997; Fox, 1988). They are overlain by other Mannville Groups, Clearwater and Grand Rapids Formations. Their presence and depth varies across the region depending on post-deposition erosion and folding processes. The Clearwater Formation is a saline-sodic marine clayshale layer, while Grand Rapids Formation is a shoreline and incised valley fill sandstone (Muwais, 1983).

Cretaceous deposits are overlain by Quaternary Period (*i.e.*, Pleistocene and Holocene Epoch) deposits. Pleistocene and Holocene deposits comprise most of the surficial geologic materials of the region and are the result processes that took place during the glacial advance and retreat of approximately 25 to 9.9 thousand (k) years ago before present (ya BP) (Bruce Geotechnical Consultants Inc., 1998). During the Late Wisconsin glacial advance (~25 to 20k ya BP) of the Laurentide ice sheet, lodgement till blanketed the McMurray Formation. A gradual and general retreat of Laurentide ice then followed from approximately 20 to 9.9k ya BP and in some parts of the region deposited ablation till. More importantly the weight of the ice sheet caused an isostatic depression, tilting the land surface in a northeast direction (into the ice sheet). Large proglacial lakes and meltwater channels formed at the margin of the ice sheet. The glacial sediments were re-sorted and deposited as glaciolacustrine sediments (predominantly fine-textured silt and clay).

The continued retreat of Laurentide ice in the region culminated in a catastrophic flood of glacial Lake Agassiz through the region approximately 9.9k ya BP (Bruce Geotechnical Consultants Inc., 1998). An elevation low was reached in moraine deposits in northwest Saskatchewan and the lake rapidly drained, incising and forming the Clearwater River valley. The floodwaters drained through the Clearwater River valley to the Athabasca River and then turned north. North of the Suncor and Syncrude's base mines the spillway widens and shallows, then bifurcates around the Fort Hills along the present course of the Athabasca and Muskeg River valleys. During this flood, Pleistocene and McMurray Formation sediments were eroded in the spillway and then deposited throughout the Fort Hills area and to the north as glaciofluvial sediments. These sediments range from boulders to delta sand deposits, depending on the energy of water flow.

Following the flood, wind and water erosion further modified the landscape to create aeolian deposits and fluvial channels until slope gradients, compaction and vegetation stabilized the sediments (Bruce Geotechnical Consultants Inc., 1998). Water-saturated areas, where organic matter accumulation exceeds the rate of decomposition, resulted in surface peat accumulation and creation of the bogs and fens present in the region.

2.1 Relevance of Geologic Events to NPHCs

The geologic events of the region have resulted in a range of proportions and concentrations of NPHCs in the sediments overlying the oil sand deposit. Assessment of the McMurray Formation to map oil sand reservoir bodies has also determined that the oil sand deposit is not entirely homogeneous and contains a range of oil sand concentrations, some of which do not contain a great enough concentration to be classified as an ore resource (Mattison, 1987). Even though they are not suitable as an ore resource, they still contain a significant amount of NPHC that has the potential to negatively impact the closure

reclamation landscape. Pleistocene lodgement till deposits overlying McMurray Formation sediments can also contain NPHCs, as some of the debris was originally McMurray Formation.

The Holocene flood event resulted in the erosion of sediments into the Cretaceous Formation, some of which were oil sand-bearing layers. These NPHC-containing materials were mixed with non-oil sand containing materials (e.g., Pleistocene sediments) in various forms and proportions in the surface sediments in the region. The forms consist of bands or layers that were sorted during by the floodwaters, to discrete conglomerates that range from the size of pebbles to large boulders.

Localized areas have also been eroded/scoured to the McMurray Formation, exposing oil sand-bearing deposits with variable proportions of NPHC concentrations and fractions. At these locations, soil development and vegetation establishment has taken place directly in oil sand deposits. NPHC analyses of the lower solum depths of these soils (approximately 50 to 100 cm), where soil development and NPHC degradation has been limited compared to shallower depths, suggest that the NPHC concentrations were at one time similar to approximate bitumen content for processing (\geq 7 or 8%).

3 OCCURRENCE OF NATURALLY-OCCURRING PHCS IN SOILS OF THE REGION

A soil survey of the occurrence of NPHCs in the region was conducted for CEMA in 2006 (Paragon, 2006). Regional soils information from the Alberta Oil Sands Environmental Research Program Study Area (AOSERP Report 122) (Alberta Environment, 1982) and surveys conducted for previous Environmental Impact Assessments (Albian [then Shell] and Imperial Kearl) were used to provide initial mapping for a field survey that covered an area from Townships 93 to 99 and Ranges 7 to 11, W4 in the Athabasca Oil Sands Region. Maps showing surficial deposits, terrain units and soil series extent with sample point locations are provided in Appendix A.

Both the Leskiw (2005) and Paragon (2006) studies concluded that the main soil series with NPHCs are the Firebag, Fort and Mildred Series, which are all derived from glaciofluvial parent materials (see Appendix A). In depths between 0 and 3 metres (m), 90 % of the sites with NPHCs sites were characterized by glaciofluvial parent material, with the remaining sites being Aeolian or Till (Paragon, 2006). Soil textures of the series were sands to loamy sands for the Mildred soil series, sandy loams and loamy sands for the Firebag series, and sandy to loamy stratified deposits for the Fort series. Stone and gravel content was highly variable in Firebag and Fort series, and tended to be low in the Mildred series. Organic soils were not the focus of the Paragon (2006) study, however organic soil sites that Paragon inspected and several sites from Kearl and Albian data indicated that NPHCs are widespread in the underlying glacial fluvial deposits.

The study found the following key findings related to the presence of NPHCs in soils of the region (Paragon, 2006):

- Approximately half of soils to a 3 m depth contained at least a trace of NPHCs.
- NPHCs comprised >25 % of the soil volume in approximately 10 % of the sites in the top 1 m of the soil profile, and 16 % in the 1 to 3 m soil depth.
- NPHCs comprised 5 to 25 % of the soil volume in approximately 14 % of the sites in the top 3 m of the soil profile.

These soil attributes indicate that most of the hydrocarbon was likely deposited by erosion and deposition processes during the draining of glacial Lake Agassiz, by meltwater, or as ice contact glaciofluvial material (Paragon, 2006). The remaining hydrocarbon-affected material is the result of erosion/scouring from the flood or post-flood fluvial processes forming McMurray Formation outcrops.

From the soil maps shown in Appendix A, it is evident that the Firebag soils generally correspond to ice contact deposit areas, and Mildred soils match the glacial fluvial plain unit (Paragon, 2006). Firebag soils usually have some textural stratification (e.g., presence of sand layers ranging from fine to coarse, or occurrence of clay bands in the profile, or underlying till). Mildred soils are usually uniformly very fine to fine sand textured. Both Leskiw (2005) and Paragon (2006) noted that internal soil drainage may be impeded by textural variations and/or presence of NPHCs in these soil series, but that the relative contribution of each was difficult to determine.

Investigations of the T-Pit (gravel pit) area at the Mildred Lake Mine (NorthWind, 2005) identified two types of surficial geologic materials containing NPHCs: Pleistocene origin, glaciofluvial coarse sand and gravel material and weathered Cretaceous oil sand material. Soils ranged from Eluviated Eutric Brunisols for upland soils to Orthic Luvic Gleysols in lower slope positions. The oil in soil concentrations for samples taken in undisturbed areas surrounding the T-Pit ranged from 3.2 to 8.8 % within 1 m of the surface (NorthWind, 2005), with the oil in soil content of one soil profile that appeared to contain no NPHCs being 0.2 % and 0.1 % for upper and lower subsoil, respectively. There was a general trend of increasing NPHC content with depth, with this trend more evident in soils developed on lean oil sand deposits than in gravel deposits containing NPHCs (NorthWind, 2005). Further information about the T-Pit hydrocarbon fractions is provided in Section 5.

3.1 NPHC-associated Ecosites

The dominant ecosites associated with inspection sites in Paragon (2006) were a and b, with b ecosites being located in areas with more widespread and stronger hydrocarbon concentration. Shallow strongly-concentrated NPHC sites tended to support c ecosites, and the density of the vegetative cover was noted to be adversely affected in these sites compared to surrounding areas in which shallow NPHCS were not noted (Paragon, 2006). NPHC concentration for 'strong' and 'medium' values (see Figure 1) was highest in the c ecosite in both (60 to 90 %), followed by b ecosites with 34 and 36 % respectively (Paragon, 2006). In contrast, a ecosites typically had a much lower ('trace') concentration of NPHC.

NorthWind's (2005) investigation of the T-Pit gravel pit identified a range of ecosite types identified on NPHC deposits including *a* and *c* ecosites on upper slopes, and *b* and *d* ecosites typically on level or lower slopes. Treed bogs (*i*1) ecosites were noted in depressional areas overlying weathered NPHCs.

4 FORMS OF NPHCS IN SOIL RECLAMATION MATERIALS AND LANDFORM SUBSTRATES

Oil sands mining reclamation requires the salvage of an appropriate volume of suitable surface coversoil (topsoil) and subsoil horizons. Following the salvage of soils for reclamation, the remaining materials are classified as overburden and removed to expose the oil sands ore body. Most of the overburden is placed in dedicated disposal areas as large, above-grade landform structures, but may also be used in structures such as dams, berms and road building.

Mining operations where the occurrence of NPHCs in surface soils is widespread require these soils to be used as soil reclamation material over landform substrates. As discussed in the geologic history of the region, the soil reclamation materials are predominantly coarser-textured, glaciofluvial and aeolian sediments of Pleistocene and Holocene origin, and Holocene peat. The presence of NPHCs in the soils salvaged for reclamation are carried forward, and remain in the reclaimed soil profile.

The remaining overburden varies across and within each mining operation in the region, depending on the depths of the surficial Pleistocene and Holocene sediments, as well as the underlying Pleistocene till and Cretaceous Formations above the oil sand ore body. NPHCs can comprise a significant proportion in these materials and they are reconstituted and will remain in the closure reclaimed landscape.

Over the life of oil sands mining and reclamation, a range of soil and material types containing NPHCs have been identified and classified.

- Interburden material immediately overlying or interspersed through the layers of bituminous sand that does not meet a cut-off bitumen content required for processing (7 % or 8 % depending on the operator).
- Lean oil sand (LOS) EPEA approvals pre-dating 2017 and AEW (2012b) defined lean oil sand as having a bitumen content by weight of ≤ 8 % (Pernitsky et al., 2016; Visser, 2008). Recent (2017 and 2018) EPEA approvals have removed this definition (e.g. CNRL-Albian Muskeg River Mine, Imperial Kearl) from the glossary.

The particle size distribution of interburden and LOS is also variable as shown in the LOS characterization of the Aurora Soil Capping Study (ASCS - see Section 7). Organic carbon content (or NPHC concentration) and sand, silt, and clay content was variable across the ASCS research site (Pernitsky et al., 2016). Texture analysis of LOS samples from the ASCS returned an average texture of sandy loam, with proportions of 55 %-60 % sand, 30 % silt, and 10 %-15 % clay (NorthWind, 2013).

There are a wide range of NPHC forms that have been identified in surface soils of the region which include the following:

- Lenses or layers of NPHCs appearing as continuous or discontinuous bands within soil horizons
- Outcrops or slabs of NPHCs appearing as a solid block
- Aggregates of NPHCs in sand or gravel (e.g., 'tarballs'), that can range in size from pebbles to rafted materials as large as a small vehicle. These are lumps of oil-impregnated sand encased in a layer of coarse sand and/or small gravel that is similar to the soil matrix (Visser, 2008a; Paragon 2006). Paragon (2006) noted that it often appeared that the sand was impregnated with an oil "blob" or slick rather than being rolled around during fluvial deposition. In some inspection sites (Paragon, 2006) tar balls appeared in a layer, in which it was difficult to determine whether the NPHCs were deposited in a ball format, or degraded in a band with the remaining NPHCs coalescing to form a ball shape. In a few sample sites it appeared that the oil moved in the soil following iron staining patterns.
- Mineral soil profiles beneath a bog or fen (where saturated and reducing conditions prevail), may become gray and mottled and be classified as a Cg horizon (Visser, 2011). Soils from the Cg horizon can contain NPHCs, especially where they are close to deeper interburden or overburden containing NPHCs, but are not readily visible like most other NPHCs forms. Soil saturation and reducing conditions can alter the patterns of PHC weathering so that the hydrocarbons associated with the mineral soil profile underlying the peat deposit are potentially more poorly weathered than PHCs associated with C horizon soil that is aerobic (Visser, 2011).

5 NPHC COMPOSITION CHARACTERISTICS AND DEGRADATION POTENTIAL

The characteristics of NPHCs both *in situ* (pre-disturbance) and *ex situ* (after redistribution into the closure landscape) are described in this section.

5.1 NPHC Composition

The compounds comprising NPHCs include (Cermak et al, 2010, 2013; Visser 2008a; Turle et al, 2007):

- saturates (straight-chain, branched-chain and cyclic alkanes, alkenes, and alkynes)
- aromatics (compounds containing one or more benzene rings including polyaromatic hydrocarbons [PAHs])
- polar nitrogen and sulphur heterocyclic aromatic compounds
- low-molecular-weight condensed polar compounds such as alcohols, acids and hydroxyacids
- asphaltenes (mixture of aliphatic and aromatic hydrocarbons).

To address soil contamination by PHC release in the environment, many jurisdictions (see Section 9) such as the *Canadian Council of Ministers of the Environment* (CCME), have developed criteria based on groupings of PHC constituents with similar environmental fate, behavior, and toxicity (Figure 1). These groupings are typically based on boiling point ranges, presented as equivalent carbon number (ECN) ranges (Cermak et al, 2013). The ECN normalizes the number of carbon atoms in a compound to that of alkanes, so that compounds with the same boiling point are assigned the same ECN. In Canada, the *Canada-Wide Standards for Petroleum Hydrocarbons in Soil* (CCME, 2008; 2001) regulate PHCs within four ECN ranges (or fractions [F]): F1 ($C_6 - C_{10}$), F2 (> $C_{10} - C_{16}$), F3 (> $C_{16} - C_{34}$), and F4 (> C_{34}). The F1 fraction is typically combined with the benzene-toluene-ethylbenzene-xylene (BTEX) constituents for analysis, due to their toxicity at low concentrations (CCME, 2008; Visser, 2008a). An additional fraction used is F4 gravimetric (F4G) to describe NPHCs with an ECN in excess of C_{50} (Paragon, 2017). F4G NPHCs are notoriously difficult to accurately analyze, with the gravimetric method generally being replaced by high temperature gas chromatography methods.





PETROLEUM HYDROCARBONS

To prepare NPHC samples for analysis, a clean-up step using silica gel is performed (CCME, 2008; Brassington et al., 2007). Theoretically, polar compounds typical of biological organic compounds (BOCs)

are retained in the silica matrix (Brassington et al., 2007) and removed from being erroneously measured as an NPHC fraction (usually F3, F4 and F4G). However, it is likely that any moderately polar compounds will also be retained in the silica matrix, including any NPHCs that increase in polarity as a result of degradation through weathering or in some cases remediation (Brassington et al., 2007). Wang et al. (2012), Kelly-Hooper et al. (2013) and Kelly-Hooper et al. (2014) also identified that naturally-occurring BOCs in soils are frequently extracted along with NPHCs, and are often mistakenly identified and regulated as NPHC, leading to a larger apparent total loading. Organic soils are often naturally rich in BOCs in the C_{16} to C_{34} (F3) fraction carbon-number, and complicated non-polar plant alkanes, large lipids, terpenoids, chlorophyll, porphyrin, and soil humic molecules will not be removed by the silica gel clean-up step (Kelly-Hooper et al., 2013).

The contribution from naturally-occurring BOCs is demonstrated in data from Hunter (2011). This study was carried out on eight sites in northern Alberta. Four of these were undisturbed jack pine (*Pinus banksiana*) stands with a lichen-covered forest floor on coarse textured, nutrient poor soils, classified as *a* ecosites (Beckingham and Archibald, 1996), including *a* ecosite 1 (AE1) and Soil Vegetation Plots 10, 26 and 27 (SV10, SV26 and SV27) (Table C.0.1 presented as Figure 2). Three were reclaimed from open pit oil sands mining including the Shallow-Stripping Trial (SS) at CNRL-Albian, the Coke Cover Capping Study (CC) at Suncor Base Mine, and the Aurora LFH Capping Study (ALFH). The ALFH study was located at Aurora North Mine, in an area that later became part of the Fort Hills dump). It was a soil cover trial, testing coarse textured coversoil and subsoil (Brunisol soils of loamy to sand texture) over lean oil sand overburden. It no longer is present, as the dump in this area was extended into the Suncor's Fort Hill operation. These soils materials were in the same general area as the soil reclamation materials of the Aurora Soil Capping Study. The influence of peat and biogenic BOCs can be seen in the LFH and Peat results, where one F3 concentration (SV10) exceeds *Alberta Tier 1 Guidelines* (AEP, 2016a), and where all F4 concentrations exceed guidelines once the F4G concentrations are added.

	F2	F3	F4	EATTCIC COAL COOL		E 40
Sample	C10-C16	C16-C34	C34-C50	F4H1GC C34-C50+	%C50+	F4G
	(mg kg ⁻¹)	(%)	(mg kg ⁻¹			
		R	eclaimed Mineral	Soil		
SS	20	187.4	144.4	375.6	35.9	2216
CC	20	424.4	344.6	1013.6	44.2	4454
ALFH	20	20.8	30	30	5	966.4
			<i>LFH</i>			
AE1	28	1732	1235	2562	28.02	25220
AE2	21.6	911.2	1213	2920	43.56	36960
SV10	40.6	1559.4	1062.4	3576	49.04	29880
SV26	30.4	645.8	834.4	1534	30.8	32560
SV27	37.6	688.6	538.4	1202.6	35.62	38580
			Peat			
Fibric	26.4	597.4	230.8	1226.4	55	20680
Mesic	27.8	482.0	188	1173.6	57.5	22260
Humic	37.2	425.4	216	912.8	53.82	9973.8

Figure 2. Hydrocarbon Analysis from Hunter (2011)

Table C.0.1. Hydrocarbon analysis of reclaimed mineral soil, LFH and peat.

5.2 NPHC Content by Fraction Present in Pre-disturbance Soils

NPHC content by fraction for pre-disturbance and natural soils in various studies is presented in Table 1. The level of detail as to form of NPHC and depth of sampling (or horizon) varies by study. Analytical results indicate that the F3, F4 and F4G fractions are responsible for exceedances of coarse-textured, natural area land use CCME guidelines (shaded cells). The F1 and BTEX fraction does not exceed guidelines, and the F2 fraction does not generally exceed except for one sample collected at depth (1.1 m) in the subsoil, where the presence of unweathered, unoxidized NPHCs in anaerobic conditions is likely. Rapid oxidation and breakdown after exposure and during sampling for this sample was likely coupled with conditions that did not allow any F2 fraction to volatilize prior to sampling. The F4G fraction above C_{50} is half, or close to half of the NPHC content in most samples (Table 1).

Studies by Pernitsky et al. (2016) and Visser (2008b) reported that LOS contained trace amounts of NPHC in the F1 and F2 fractions, and proportions of approximately 38 % and 54 % in the F3 and F4 fractions, respectively. The tar balls used in the Visser (2008a) study contained 5 % F2 and 95 % F3 and F4 NPHCs (>2,500 mg/kg for F2, >20,000 mg/kg for F3 and >25,000 mg/kg for F4). Visser (2008a) reported that F1/BTEX NPHCs were negligible in the tar balls, and the distribution of F2, F3 and F4 NPHCs was highly variable amongst tar balls used in the study. Available nutrients in the tar balls included high levels of sulphate-S (sulphur) and trace amounts of ammonium-N (nitrogen) (Visser, 2008a).

Table 1. NPHC Concentrations from Pre-disturbance and Natural Sites (Compiled from Leskiw, 2005;NorthWind, 2005; Paragon, 2006 and Fleming et al., 2011)

Sample Identification	Sample Depth/Horizon	Material	F1 (C6-C10) - BTEX (mg/kg)	F2 (C10-C16) (mg/kg)	F3 (C16-C34) (mg/kg)	F4 (C34-C50) (mg/kg)	F4HTGC (C34-C50+) (mg/kg)	% C50+
From Leskiw, 2005				•				
Tar sand	С	subsoil	1	72	2,540	3,040	13,900	65.9
Bog	Om	organic	1	10	91	64	107	22
Fen	Om	organic	1	10	139	116	218	28.1
Site 29	С	subsoil	1	10	325	428	959	41.7
Site 65	С	subsoil	1	10	42	70	216	56.8
From NorthWind, 2005								
S05-1	0.0-0.2 m	sand/gravel	<1	<10	360	460	920	35.9
S05-1	0.2-0.5 m	sand/gravel	<1	30	3,810	3,540	7,920	37.2
S05-1	0.5-1.0 m	sand/gravel	<1	20	4,460	3,760	9,280	40.1
S05-1	1.1 m	sand/gravel	11	6,940	29,100	13,200	30,800	26.3
S05-07	0.2-0.5 m	lean oil sand	<1	70	5,060	6,700	17,900	48.6
S05-07	0.5-1.0 m	lean oil sand	<1	20	2,090	3,560	8,860	48.3
From Paragon, 2006								
Site 05	0.5-1.0 m	subsoil		16	83	168	554	59.1
Site 07	1.5-2.0 m	subsoil		<10	76	80	115	18.3
Site 07	2.5-3.0 m	subsoil		<10	125	162	504	54.4
Site 03	0.5-1.0 m	subsoil		<10	13,900	19,200	55,000	52.0
Site 15	0.5-1.0 m	subsoil		<10	11,000	11,300	39,400	55.8
Site 10	0.8-0.9 m	subsoil		<10	82,000	13,700	44,500	24.3
From Fleming et al, 20	011							
Samples	Number of			moor	(ma/ka) + cta	ndard daviati	<u></u>	
anaryseu	detects	to she th	2 ± 12	mear	i (iiig/kg) ± sta		011	
168	13	tarball	3 I 13					
276	68	tarball		37 ± 125	1 720 + 1 120			
2/0	272	tarball			1,730 ± 1,120			
147	147	tarball				0,070 ± 2,550	24 700 ± 16 700	
total samples - 276	220	lainall					34,700 ± 10,700	
101a1 3ampies - 270								

Note: Shaded values are in exceedance of Alberta Tier 1 Guidelines (AEP, 2016a) for Natural Area, Coarse Soil Type: F1- 210 mg/kg; F2 – 150 mg/kg; F3 – 300 mg/kg; F4 (and F4G) – 2,800 mg/kg.

-- denotes parameter not analysed or not applicable.

5.2.1 Surface Soil and Overburden Analysis from CNRL-Albian Sands

Paragon (2006) noted that in a synthesis of three Albian salvage soil surveys for hydrocarbons in the 0 to 3 m depths, 299 of 453 sites were found to be positive for NPHCs. The sites investigated contained tar balls and LOS in a natural soil matrix. Of these 254 sites for which parent material and depositional mode were documented in the study, 75 were classified as organic (29%), and 213 contained NPHCs (84%). The proportion of organic soils affected by underlying NPHCs is similar to that in uplands in this locality. From data collected as part of Muskeg River Mine overburden and mine advance sampling and Jackpine Mine overburden sampling, overburden attributes are summarized below for NPHC content, salinity and CCME-regulated metals in Table 2.

5.2.2 Surface Soil and Overburden Analysis from Imperial Kearl

Paragon (2006) noted that of 725 plots sampled for hydrocarbons in the 0 to 3m depths, 133 or 16 % were found to have NPHCs as fragments or bands of LOS within the soil matrix. Of these 113 affected sites, 15 or 13 % were overlain by organic materials indicating that wetlands may also contain shallow NPHCs in the underlying mineral profile.

From data collected as part of overburden and mine advance sampling overburden attributes are summarized for NPHC content, salinity and CCME-regulated metals in Table 2.

5.2.3 NPHC Analysis of LOS from Syncrude Aurora Soil Capping Study

Korbas (2013) found the NPHC fractions present in LOS at the ASCS contained small amounts of volatile and easy degraded F1 (0.1 % excluding undetectable BTEX) and F2 (8.3%). On this basis, only the F1 fraction of the LOS was below *the Alberta Tier 1 Guidelines* (AEP, 2016a) limit (210 mg/kg for F1) for Natural Areas and coarse-grained soils. Korbas' (2013) initial characterisation of LOS material showed that LOS was comprised mostly of F3 (47%) and F4 (45%) (see Table 3 below [Table 5.1 from Korbas 2013]).

ASCS substrate attributes are summarized below for NPHC content, salinity and CCME-regulated metals (collected by O'Kane [2015]) and provided in Table 3.

	Oil Sand Mine Development												
		Syncrude	e ASCS (O'Kane,	, 2015)	CNRL-All	CNRL-Albian Muskeg River Mine			-Albian Jackpir	ne Mine	Imperial Kearl		
Parameter	Units	Range	Mean	Standard Deviation	Range	Mean	Standard Deviation	Range	Mean	Standard Deviation	Range	Mean	Standard Deviation
pН		6.8 - 8.6			3.9 - 7.4	6.3	1.6	7.37			7.7 - 7.9		
EC	(dS/m)	0.4 - 3.1	1.5	0.6	1.8 - 4.5	3	1.2	1.7			0.8 - 2.1	1.5	0.47
SAR		0.3 - 38.6	12.6	11.4	1.4 - 11.3	6.3	4.2	2			0.2 - 1.2	0.5	0.38
Texture					Sandy Loam			Sandy Loam			Sandy Loam		
F1-BTEX	(mg/kg)	<4 - 176	69.4	45.9	ND			ND			ND		
F2	(mg/kg)	178 - 4,650	2,335	1,221	847 - 3,600	1,794	1,247	1,700			160 - 1,400	624	479
F3	(mg/kg)	1,050 - 18,500	10,597	5,388	6,710 - 16,000	9,567	4,378	11,000			1,500 - 9,900	4,668	3,552
F4	(mg/kg)	382 - 7,270	4,152	2,189	2,910 - 7,200	4,145	2,055	5,100			900 - 5,000	2,418	1,800
Boron (hotwater)	(mg/kg)				2.6			0.64			0.17 - 1.7	0.9	0.67
Hexavalent Chromium (Cr6+)	(mg/kg)				<0.080			<0.080			<0.08		
Aluminum (Al)	(mg/kg)				1,090 - 2,320	1,856	668						
Antimony (Sb)	(mg/kg)				<0.20 - <0.50			<0.50			<0.5		
Arsenic (As)	(mg/kg)	<0.2 - 2.3	1.1	0.4	0.6 - 13.5	4.5	6.1	1.1			<1.0 - 1.8	1.6	0.25
Barium (Ba)	(mg/kg)				16.0- 47.0	32.3	12.7	16			7.5 - 20	14.1	3.72
Beryllium (Be)	(mg/kg)				0.1 - 0.5	0.3	0.2	<0.40			<0.40		
Cadmium (Cd)	(mg/kg)				0.02 - 0.1			<0.05			<0.05		
Chromium (Cr)	(mg/kg)				5.1 - 9.2	7.4	1.9	7.7			1.8 - 13	6.3	4.4
Cobalt (Co)	(mg/kg)				1.5 - 12.0	6.2	4.6	3.9			0.83 - 2.7	1.9	0.64
Copper (Cu)	(mg/kg)				4.0 - 14.0	7.7	4.5	3.8			2.0 - 3.9	3.3	0.6
Iron (Fe)	(mg/kg)				2,860 - 15,600	9,953	6,492	`					
Lead (Pb)	(mg/kg)				3.9 - 25.6	10.3	10.3	3.3			1.1 - 3.1	2.1	0.66
Mercury (Hg)	(mg/kg)										<0.05		
Molybdenum (Mo)	(mg/kg)				0.7 - 1.0	0.8	0.2	0.77			<0.4 - 0.62	0.6	0.76
Nickel (Ni)	(mg/kg)				9.7 - 46.5	22.5	16.8	9.8			1.6 - 9.9	5.2	2.5
Selenium (Se)	(mg/kg)				<0.3 - 0.4	0.35	0.05	<0.50			<0.50		
Silver (Ag)	(mg/kg)				0.1 - <0.2	0.1	0.1	<0.20			<0.20		
Thallium (Tl)	(mg/kg)				0.1 - 0.2	0.15	0.1	<0.10			<0.10		
Tin (Sn)	(mg/kg)				<1.0 - 3.0	2	1	<1.0			<1.0		
Uranium (U)	(mg/kg)				0.47			0.5			0.28 - 0.34	0.3	0.02
Vanadium (V)	(mg/kg)	9.0 - 25.6	19.8	3.2	15.8- 25.7	20.5	4.2	13			2.2 - 10	6.6	2.6
Zinc (Zn)	(mg/kg)				10.0 - 29.0	20	8	13			10 - 13	11.5	1.29

Table 2. Summary of NPHC, Salinity and Metal Analysis Data for Overburden Containing Lean Oil Sand at Syncrude Aurora, CNRL-Albian and Imperial Kearl.

Notes: ND - not detected; -- no data

PHC Fractions		Regulatory	gulatory Columns 1-3 Columns		ns 4-6	
	Units	Guidelines	Mean	STDEV [#]	Mean	STDEV
BTEX	mg/kg	N/A	non-detect	N/A	non-detect	N/A
F1 C6-C10	mg/kg	210	37	17	29	8
F2 C10-C16	mg/kg	150	2894	767	1678	351
F3 C16-C34	mg/kg	310	16328	5671	9442	1831
F4HTGC C34-50+	mg/kg	2800	15549	6665	9288	2179
Total F4G	mg/kg	2800	53313	14998	23383	3696

Table 3. Summary of NPHC Data from the Aurora Soil Capping Study by Korbas (2013).

*Alberta Tier 1 guidelines - Natural area land use for coarse grained soils

*STDEV - Standard Deviation

5.3 NPHC Degradation Potential

Degradation is one of the key processes determining the fate of NPHCs in the environment, as it results in their breakdown or transformation to metabolites, and possible transfer to different parts of the ecosystem (soil, water, vegetation and rhizosphere, fauna or air). In the soil, mixtures of NPHCs can change through weathering (including volatilization, biodegradation, partitioning, oxidation, hydrolysis, and photodegradation). More soluble or volatile compounds will migrate to other locations, and the mostly non-mobile components will remain in the soil matrix. In soils, NPHCs are affected by various chemical and biological processes conditioning their biotic and abiotic degradation; retention (adsorption, desorption); transfer toward groundwater, surface water and atmosphere; and consequent bioavailability and potential effects on organisms (Mamy et al., 2015; Dobbins et al., 1992).

5.3.1 Abiotic Degradation

The mechanism of breakdown of NPHCs in soil depends on their location within the soil stratum, and the soil attributes. On the surface of the soil, NPHCs are subject to photodegradation, where the energy of sunlight initially cleaves branched and cyclo-alkanes, and then a portion of linear chain NPHCs (Gennadiev et al., 2015; Prince et al., 2003). In photodegradation, larger polycyclic aromatic PHCs are broken down before smaller ones, and more alkylated compounds are broken down before less alkylated compounds (Gennadiev et al., 2015; Tsibart and Gennadiev, 2013).

Volatilization is governed by NPHC compound size, with F1/BTEX and lower end F2 fractions typically departing soils as vapour. AEW (2012a) noted that tar balls present in surface soil profiles are composed primarily of heavier, complex fractions (F3 and F4), with a small amount of F2 hydrocarbons, and the content of BTEX and F1 hydrocarbons was negligible. The heavier fractions tend to be less bioavailable and hence more restricted in terms of exposure pathways, but also less amenable to degradation (AEW, 2012a).

Any F1/BTEX and F2 content present in NPHC fragments or created as a product of the degradation of larger fraction compounds is quickly lost, such that samples analyzed for CNRL-Albian and Imperial (see Sections 5.2.1 and 5.2.2) by Paragon (2006) in natural sample sites and by Visser (2008a, 2008b), either

did not find these fractions at all, or they were close to the detection limit of the laboratory analysis. It could be expected that perturbation of soil samples during the sampling process would further expose NPHCs to oxidation and/or photodegradation, however this did not result in development of a significant amount of volatile fractions in the sampled material. Visser (2008a) noted that because tar ball PHCs are dominated by high molecular weight F3 and F4 hydrocarbons, degradation over the course of the experiments (0.5 to 8 months) was limited to approximately 7.5 % of the NPHC by weight. The pattern of degradation showed that less complex F2 PHCs are more readily available for microbial degradation, than F3 and F4 NPHCs.

5.3.2 Sorption

NPHCs are subject to sorption by mineral and organic components of soil, and sorption is enhanced in soils with high organic content (Pernitsky et al., 2016; Gennadiev et al., 2015; Tsibart and Gennadiev, 2013). Highly sorptive surfaces of some clay and organic matter fractions limit availability, especially for intensely-weathered soil where contaminants have had the time to partition into soil organic matter or diffuse into micro-pores (Marchand et al., 2016; Gennadiev et al., 2015; Huesemann et al., 2004). In highly weathered soils where NPHCs have become sequestered, degradation kinetics are then limited by mass transport (desorption, diffusion, and convection) processes (Pernitsky et al., 2016).

Brassington et al. (2007) noted that the PHC matrix is itself a strong partition medium, and weathering develops further hydrophobicity. Visser's study (2008a) of tar balls, and the field observations of Paragon (2006) support that the NPHCs coalesce into forms that produce hydrophobic and relatively inert surfaces in contact with surrounding soil. Risk-critical components (e.g., the higher ring polynuclear aromatic hydrocarbons [PAH]) in weathered PHCs are less bioavailable because they are effectively partitioned within the PHC matrix (Brassington et al., 2007). Brassington et al. (2007) noted that in contaminated sites with a concentration of residual PHCs, the PHC–soil matrix can act as a discrete partition medium at \sim 1,000 mg/kg, which is typically 10 times more effective a partition medium than soil organic matter for hydrophobic PHCs. Zemanek et al. (1997) showed that between 71–96 % by weight of PAH in weathered diesel-contaminated loam soils was partitioned to residual oil (at 2 – 6 % by weight of the total soil composition) in petroleum and weathered creosote-contaminated soils, with 84 % by weight of benzo[a]pyrene partitioned to the residual oil phase. Under these conditions, highly partitioned constituents in weathered PHC and NPHC matrices may be biologically inaccessible to microbial communities and resistant to degradation.

Although high organic content is typically associated with porosity and water retention in the soil, Pernitsky et al. (2016) showed that NPHCs in LOS differ from other types of soil organic content because they tend to be located in the interior of soil pore space and can therefore reduce the water content and hydraulic conductivity of soil. At a high bulk density (1.7 g/cm^3) and >3.25 % NPHC content, there was a significant reduction in saturated hydraulic conductivity of soil, and there was potential for using this as an attribute to reduce percolation and increase water retention time in the overlying coarse-textured reclamation subsoil. Research by Ren et al. (2009) also supports this conclusion as they found that weathering tends to force PHCs to bind into mineral particle void spaces, which makes the soil surface more hydrophobic.

5.3.3 Biodegradation

Microbial biodegradation is often considered as a major driving force of the fate of organic compounds in the environment (Mamy et al., 2015), and can be the result of aerobic or anaerobic enzymatic activities of microorganisms. The three factors affecting the type and rate of biodegradation process are the environmental conditions (e.g., pH, redox potential of the soil, presence of nutrients, availability of

oxygen, temperature and moisture content), the chemical and structural properties of the NPHC constituents, and the community of microorganisms present (Mamy et al., 2015; Korbas, 2013). The energy for microorganism growth obtained by biodegradation processes is largely due to the transfer of electrons from organic molecules to inorganic electron acceptors such as sulphur, nitrogen and oxygen (Mamy et al., 2015; Turle et al., 2007). Electron acceptors are not interchangeable for microorganisms, therefore the microbial community depends on what is available in the soil.

As noted by Korbas (2013) temperature has a significant effect on degradation of PHCs in LOS by its influence on physical nature and chemical composition of the NPHC, rate of NPHC metabolism by microorganisms and composition of the microbial community. The rates of NPHC degradation generally increase with increasing temperature, and at low temperatures the degradation process is slowed down due to reduction in volatilization of short chain alkanes and their increased solubility in water. Soil respiration results in Korbas (2013), showed that the total estimated NPHC degradation in LOS from respiration was 60 grams/year at 22°C and 15 grams/year at temperatures from 2 to 14°C. Korbas (2013) noted that these amounts constitute only a small portion of the approximately 2.8 kilograms (kg) of NPHCs present in columns used for his study, and the degradation of NPHCs from LOS material was therefore occurring at relatively slow rates. For comparison, Fleming and Fleming (2012) showed that degradation of NPHCs from surficial bitumen materials (at 20°C) was on average 8.5 grams/year, which is significantly slower than that measured for LOS in Korbas (2013). In evaluating tarball NPHC content and composition and leaching characteristics, Fleming (2012) found that heavier NPHCs (F3 and greater) were the primary hydrocarbon constituent fractions. Where found, lighter relatively mobile NPHC fractions were located at the core of larger fragments consisting of heavier F3 and F4 NPHC fractions. These results suggest that differences in degradation rates between LOS and surficial bitumen may reflect differences in degree of weathering associated with surficial bitumen compared to relatively unweathered LOS NPHCs.

The availability of oxygen in soils for the process of PHC biodegradation is critical, and depends on rates of microbial oxygen consumption, type and saturation of soil, and the presence of other substrates that can lead to oxygen depletion (Scale, 2017; Korbas, 2013). Microorganisms also require nitrogen and phosphorus for decomposition of PHCs, and these can be limiting factors for biodegradation rate (Mamy et al., 2015).

Extreme pH values can have negative effects on the function of microorganisms, and influence the community of microorganisms present. Korbas (2013) found pH in LOS at the ASCS ranged from 7.2 to 8.5 with a mean pH of 7.7. A soil pH range of 6.5 to 8.5 has been found to be to most optimal for microbial biodegradation efficiency (Gennadiev et al., 2015; Korbas, 2013).

Microbial activity in the soil increases with increasing water content, due to the greater availability of nutrients, biogenic carbon and other oxidizable compounds (Mamy et al., 2015; Turle et al., 2007). There is a threshold water content value above which microbial activity begins to decline, because at high levels of saturation water occupies most of the voids in the soil matrix leaving very little space for transport and availability of oxygen for microorganisms. The optimal soil moisture content for remediation is in a range 30 - 80 % by volume (Mamy et al., 2015; Korbas, 2013). Biodegradation is more intense in the presence of oxygen, therefore degradative processes are slowed in waterlogged soil, and at increasing depth in the soil profile (Gennadiev et al., 2015), notwithstanding the potential for increased aeration by plant roots. The redox potential of frozen ground changes with freeze-thaw processes and can also influence the rate of biodegradation.

Biodegradation activities have been harnessed in phytoremediation work for PHC-contaminated sites. Phytoremediation of PHC-contaminated soil is mainly based on biodegradation in the rhizosphere (Shirdam et al., 2008). In phytoremediation, a combination of processes are involved that result in increased remediation of PHCs, including soil stabilization within the matrix, plant uptake, transformation to storable nontoxic forms, and stimulation of soil microorganisms capable of degrading PHCs (Mamy et al., 2015; Shahsavari et al., 2013; Phillips et al., 2009; Shirdam et al., 2008). Plants can not only degrade PHCs directly via their own enzymatic activities (phytodegradation), but also stimulate the rhizosphere microbial community which may cause the degradation of PHCs by microorganisms (rhizoremediation or rhizodegradation) (Shahsavari et al., 2013). Addition of organic material (such as peat and compost) and fertilizer has shown a positive effect on phytoremediation efficiency in a variety of (mostly agricultural) plant species such as sorghum and common flax (Shirdam et al., 2008), maize and wheat (Shahsavari et al., 2013), barley (Visser 2008a; 2008b), and also in native plant species (Merkl et al., 2005 [Venezuela]; Visser, 2008a; 2008b [Oil Sands Region]).

While both the chemical extractability and bioavailability of NPHCs are known to decrease with aging or weathering (Phillips et al., 2009; Visser 2008a; 2008b; 2011) there is evidence that specific plants may increase the chemical extractability of strongly-sorbed compounds in aged or weathered soils containing PHCs. The concentration of 5- and 6-benzene-ring PAHs was increased in soil microcosms amended with artificial plant root exudates, and pea, cress, and pansy increased the extractability of PAHs by up to 60 % and of total PHCs by up to 16 % (Phillips et al., 2009). This has been attributed to both mobilization and movement of PHCs to the rhizosphere, and to desorption of previously unextractable compounds (by the action of microbial or plant surfactants, and perhaps the pattern of plant root exudations) (Phillips et al., 2009). Rochmann (2016) investigated specific bacterial species within oil sands process-affected water (OSPW) and tailings ponds that break down hydrocarbons (benzene and naphthalene were used as test compounds).

Within the soil stratum, NPHCs are subject to biodegradation which tends to degrade the linear chain portion of PHCs first (Gennadiev et al, 2015; Huesemann et al., 2004). Huesemann et al. (2004) found that biodegradation rates of hexadecane and all other *n*-alkanes were significantly greater than the abiotic release rates, indicating that *n*-alkanes do not have to be dissolved in an aqueous phase in order to be biodegraded by soil microorganisms. It was hypothesized that microorganisms are able to overcome the mass-transfer limitations by either excreting biosurfactants to solubilize *n*-alkanes, or by facilitating the direct uptake of these highly insoluble compounds across lipophilic membranes (Huesemann et al., 2004).

Visser (2008b) found that in both sand and peat/sand receiving soils, respiration rose rapidly after addition of LOS, and peaked within one to two weeks. A rapid rise in respiration in all LOS treatments indicated that there were bioavailable NPHCs in all LOS treatments, and that they were metabolized quickly by the microorganisms. Based on PHC analyses conducted before and after completion of the respiration study, the PHCs metabolized during the peak respiration period were primarily F2 PHCs and some F3 PHCs (Visser, 2008b). Once a stable respiration endpoint had been achieved (100 days), the NPHCs remaining in the soil were primarily F3 and F4 fractions. Across all LOS treatments, average NPHC removal in the sand receiving soil was 100, 75, 31 and 1.1 % for F1, F2, F3 and F4, respectively, with an overall NPHC loss of 24 %, compared with 100, 74, 39 and 0.2 % for F1, F2, F3, F4, respectively, in the peat/sand receiving soil, and an overall PHC removal of 25 %. However, the temporal pattern of NPHC loss indicated that weathering cannot be solely relied on to reduce concentrations of NPHCs to values less than applicable *Alberta Tier 1 Soil and Groundwater Remediation Guidelines* (AEP, 2016a). Nonetheless, weathering would be expected to reduce (but not eliminate) exposure to receptors (AEW, 2012a).

6 CHEMICAL AND PHYSICAL PROPERTIES OF SOIL RECLAMATION MATERIALS AND LANDFORM SUBSTRATES CONTAINING NPHCS

The chemical and physical characteristics of NPHCs both *in situ* (pre-disturbance) and *ex situ* (after redistribution into the closure landscape) are described in this section.

6.1 Movement and Leaching of NPHCs

The movement of NPHCs through soil materials and to ground and surface waters first depends on their availability to be leached through the soil profile or to be mobilized by runoff water. This availability changes with time, and with climatic and soil conditions that enhance or inhibit movement processes (Mamy et al., 2015), such as the frequency and duration of precipitation events, soil hydrodynamics and initial soil water profile, and soil physical and chemical characteristics.

As noted in Paragon (2017), melting snow, ice and rain can potentially disperse NPHCs laterally and horizontally in soils. During the infiltration of water through the soil towards the saturated zone, some of the NPHC may dissolve in water, be adsorbed on soil particles, vaporize or be trapped between soil particles in the vadose zone (Gennadiev et al., 2015; Korbas, 2013). Once the PHCs accumulate in the saturated zone, they may move further due to advective contaminant transport (Korbas, 2013).

Korbas (2013) divided the Aurora North mine Fort Hills dump soil into three zones: the unsaturated (vadose) zone, capillary fringe and saturated zone. NPHCs are present in soil either in aqueous or vapour phases that can migrate within the unsaturated zone (enhanced by infiltration of precipitation). In addition, freeze-thaw cycles encourage the radial migration of NPHCs that can form oil lenses in the soil (Gennadiev et al., 2015; Paragon, 2006). The distribution of NPHCs within the vadose zone can take place either in aqueous or vapour phases (Scale et al., 2015; Korbas, 2013). The direction of movement of the gas phase is independent and depends on the relative density and pressure gradients, while for the aqueous phase the direction is usually downward due to gravity.

Evaluation of the leaching potential of LOS material in Korbas (2013) suggest that there would be low environmental impacts to surface or groundwater quality due to leaching of NPHCs. The LOS/overburden material collected from the ASCS and used in the column study contained NPHCs predominantly composed of F3 (47 %) and F4 (45 %) hydrocarbon fractions (Scale et al., 2015). The F1 fraction (including BTEX) was not detected in the column leachate, while the F2 fraction slightly exceeded *Alberta Tier 1 Guidelines* (AEP, 2016a) on a few occasions at temperatures ≥ 22 °C (which does not reflect average field conditions), but generally remained below guidelines and decreased over time. At low temperatures (\leq 4°C), the concentration of F2 hydrocarbons was close to the 0.1 mg/L detection limit. The F3 hydrocarbons were present at concentrations below F2 regulatory limits, and are not regulated for groundwater (Korbas, 2013). Scale et al. (2015) noted that based on measurements made at the site, the LOS temperature beneath 0.3 to 1.5 m of reclamation cover ranged from 17 to -7 °C, with an average annual temperature of 4.2°C.

Preliminary seepage water analyses conducted by O'Kane (2010) at the ASCS (Fort Hills Dump) agree with the Korbas (2013) column studies. Concentrations of most toxic metals/metalloids, polycyclic aromatic hydrocarbons (PAHs), and F1 to F4 hydrocarbons were either less than the detection limits or slightly greater than the detection limit (maximum concentration of 1.0 mg/L) (O'Kane, 2010). Concentrations of naphthenic acids (NA) were much greater (23.9 and 26.3 mg/L) and O'Kane (2010) noted that these may be a dissolved constituent of interest.

Rogal et al. (2018) modeled the migration of PHCs into the soil cover of a set of sand-to-fines (SFR) ratio consolidated tailings products (i.e., the PHCs were contained in tailings rather than NPHCs from LOS). The

most important loading mechanism was advection as the tailings consolidated, rather than desorption of PHCs from the tailings material. Diffusion was the dominant transport mechanism in long-term model runs, but it was slow enough that it did not create significant PHC loading within the modeled soil cover in 20 years. Rogal et al. (2018) found that regardless of the cover type (coarse or fine-grained soil, or both, between 1.6 and 2.0 m deep), none of the model simulations resulted in F3 loading approaching *Alberta Tier 1 Guidelines* (AEP, 2016a) and only one model simulation resulted in F2 concentrations above the guidelines – coarse-grained soil over 2:1 SFR. Using a 4:1 SFR effectively diluted PHC concentrations and prevented PHC loading to the top 0.75 m of soil. Visser (2008a) noted that although NPHC concentrations in tar ball disks were extremely high (47,000 to 83,000 mg/kg), and the experimental systems were watered regularly, there was no detectable leaching of NPHCs into soil in the immediate vicinity (within 2 cm) of the tar balls. It appeared that the large molecular size and complexity of F3 and F4 NPHCs limits their mobility, and Visser (2008a) concluded that NPHC leaching from tar balls would be expected to be negligible. In comparison, sulphate contained in the tar balls was highly mobile in the soil solution and formed precipitated salts at the soil surface in all of the Visser (2008a; 2008b; 2011) studies.

6.2 Soil Physical Characteristics

In addition to the soil series characteristics noted in Section 3, studies by Pernitsky et al. (2016) have noted that there is limited information on the effect of NPHCs on soil hydraulic properties. While increased clay and organic carbon in soil can act as aggregating agents to increase the water holding capacity by improving soil structure and increasing porosity, increases in bulk density reduces soil porosity which in turn reduces the overall soil water storage and saturated hydraulic conductivity (Pernitsky et al., 2016). Mossop (1980, cited in Pernitsky et al., 2016) found that Athabasca oil sand is unique in the sense that the oil is located in the interior of the pore space rather than coating individual soil particles. As NPHC concentration increases, more of the soil micropore space (NPHCs preferentially filled micropores rather than meso- or macro-pores) is filled with NPHCs, leaving less pore space for water to occupy (Pernitsky et al., 2016).

Cermak et al. (2013) noted that as the boiling point range for PHCs increases, the water solubility decreases leading to greater sorption to soil particles (especially organic carbon) and lower porewater concentrations. A decrease in bioavailability with time may result from the movement of PHCs to nonbioavailable sites within the soil (Cermak et al., 2013). Visser (2011) commented that the negative effects of NPHCs on plants and other soil receptors should be considered in conjunction with other soil physical/chemical properties that may inhibit receptor responses.

Pernitsky et al (2016) also found that an increase in NPHC concentration reduces the overall water holding capacity of LOS, which may be a problem for plants needing to access the water in the LOS layer. In contrast, the available water holding capacity (AWHC) in LOS increases as bulk density increases, and the reduced hydraulic conductivity in higher NPHC concentration LOS will limit the downward movement of water. The higher water storage and reduced hydraulic conductivity of the LOS will increase the amount and duration of root zone water access, and potentially nutrient access (Pernitsky et al., 2016). Paragon (2006) observed that in natural soil profiles with a significant proportion of the subsoil volume consisting of NPHCs, the soil just above the NPHC fragment or layer was consistently wetter than the surrounding soil. Where NPHC content was shallow and solid in form, rooting depth and moisture storage appeared to be impaired, resulting in reduced tree growth (Paragon, 2006). The implications of these observations are that if the NPHCs are covering or near the soil surface, this could result in reduced water entry into the profile because of its physical sealing as well as potentially hydrophobic and root restricting effects. However, if the NPHCs occur within a suitable depth in the subsoil (adequate rooting zone depth), there

is an opportunity for improved soil moisture availability via retention (Pernitsky et al., 2016; Paragon, 2006).

The conjunction of hydraulic conductivity and NPHC content was demonstrated in high bulk density (1.7 g/cm³) LOS with a low (1.63 %) PHC concentration, where less NPHC was required to reduce hydraulic conductivity than in lower bulk density (1.3 g/cm³) LOS with a higher PHC concentration (3.25 %). Pernitsky et al. (2016) hypothesized that as NPHC content reaches a threshold concentration (around 3.25 %), the water storage-reducing effects of NPHCs within the LOS becomes significant.

Evaluating LOS and weathered hydrocarbon contaminated surface soil, respectively, Huang et al. (2016) and Phillips et al. (2009) both noted that the hydraulic properties of reclamation soil covers also evolve over time as a result of changes in secondary structure brought about by processes such as freeze—thaw or wet–dry cycles, or bioturbation processes caused by rooting or burrowing animals. Hydraulic conductivity can increase due to the formation of macropores and fractures over time. The sand content and NPHC concentration had the most impact on the estimation of saturated hydraulic conductivity in overburden at the ASCS.

6.3 The Effect of Peat

Visser (2011) investigated Cg horizon soils (water-saturated mineral C horizon (gleyed) overlain by organic materials such as those found in bogs and fens) containing NPHCs. The pattern of toxicity to plant growth corresponded with NPHC concentrations in Cg soils, but this pattern was not maintained when the Cg NPHC soils were mixed with peat. Poor soil structure (resulting in reduced aeration and low porosity), high salinity, low fertility and poor water infiltration likely interacted with the effects of NPHCs in the Cg soils tested, but added peat not only reduced NPHC concentrations through dilution but alleviated other adverse soil properties. A similar effect of peat in soils containing tar balls (Visser, 2008a) and LOS (Visser, 2008b) was noted.

7 CURRENT OPERATOR OVERBURDEN LANDFORM DESIGN APPROACHES

7.1 Strategic Placement of Interburden and Overburden Materials

CNRL-Albian, Imperial and Syncrude have all constructed overburden disposal areas in which lean oil sands or other formats of NPHCs have been detected in soil sampling (see Section 4). While it is comparatively early in the construction and application of coversoils to these overburden disposal areas, Syncrude has undertaken a multi-year study with a dedicated research facility built on the Aurora North Mine Fort Hills Dump - the Aurora Soil Capping Study (ASCS). The following sections summarize research information compiled from the ASCS.

7.1.1 ASCS Overburden

Syncrude Canada Ltd.'s ASCS is a long-term, instrumented watershed research site on a predominantly LOS overburden dump (Appendix B). The research site is located on the Fort Hills dump at Syncrude's Aurora North mine, approximately 50 km north of Fort McMurray, Alberta (57°20'03" N, 111°32'04" W), and is referred to as the ASCS (Pernitsky et al, 2016). The purpose of the study is to test the efficacy of various reclamation material types, placement configurations, and depths over LOS overburden, to return the disturbed land to equivalent land capability (Pernitsky et al., 2016; NorthWind, 2013a).

The Fort Hills dump was graded to create a landform that is geotechnically stable and has integrated surface water drainage. It consists of approximately 40 m to 60 m of backfilled LOS and oilsands-affected overburden (surficial Pleistocene and Holocene) material located above natural ground (O'Kane, 2015).

The upper 30 m in the ASCS area is predominantly LOS and the bottom ~20 m (~30 m – 50 m below ground surface) is predominantly oilsand-affected overburden (sand with some bitumen) (O'Kane, 2015). Due to inherent variability of NPHC concentration in LOS and the use of large equipment to haul and spread the material to its designated location, there is a spatial variability of NPHC concentration and compaction throughout the entire area of LOS on the ASCS (Pernitsky et al., 2016).

NorthWind (2013a) collected overburden samples from each cell located within the ASCS to characterize the substrate within the research area. The design consists of 36 cells, the majority (33) of which were sampled in October 2010, followed by the remaining 3 cells in November 2011. The results of the sampling analysis are provided in Figure 3.

Particle Size Distribution (%) Soil Chemical Analyses SAR Oil in soil (%) Silt Soil pH EC Sand Clay 28.8 5.5 2.5 mean 58.7 12.5 mean 7.7 mean mean mean 2.7 72 0.3 min 59 0.1 0.1 0 min 0 min min min 56.3 37.8 100 8.5 44.9 7.6 max max max max max 7.7 % Dist % Dist. % Dist. /alue (%) % Dist. Count Value Count Value Count Value Count /alue (% Count 147 <10 27 <7.3 0% <4 351 55% <1 21 71 12% 3 3% <1 10-20 98 455 7.3-7.5 42 7% 4-8 149 23% 1-2 247 39% 1-2 120 21% 8 209 34 7.5-7.8 295 46% 8-12 51 8% 2-3 248 39% 2-3 24% 20-30 3-4 30-40 34 217 7.8-8.0 224 35% 12-20 67 11% 50 8% 3-4 144 25% 1 102 76 20-30 4-5 25 4-5 62 40-50 0 8.0-8.2 57 9% 16 3% 4% 11% 50-60 200 10 0 8.2-8.4 13 2% 30-40 0% 5-6 25 4% 5-6 21 4% 1 17 17 60-70 176 0 2 6-7 3% 6-7 3% 0 8.4-8.6 3 0% 40-50 0% 70-80 80 0 0 7-8 4 1% 7-8 3 1% 80-90 26 0 0 90-100 0 0 9 637 637 637 total 637 100% total 637 100% total 637 100% total 100% total

Figure 3. ASCS Overburden Sampling Analysis (from NorthWind, 2013a)

Appendix 5. Distribution of overburden substrate analytical data.

Drozdowski et al. (2010) characterized the dry bulk density and field water contents within each of the 36 ASCS cells to provide baseline data for the overburden surface. The surface 20 cm of overburden was the primary focus for characterization, as this is the interface between the overburden and overlying capping material. Average bulk density for the CPN® MC-1 DR-P Portaprobe for all depths (0-5, 5-10 and 10-20 cm) and cells was 1.62 Mg/m³, which was comparable to the average bulk density for the ring samples of 1.66 Mg/m³. For most subplots, bulk density decreased with depth, indicating that there is a compaction effect as a result of placing and contouring the overburden.

Scale (2017) studied the rate of NPHC degradation as indicated by gas flux (carbon dioxide and methane) from the ASCS cells. Factors affecting gas transfer as a function of depth were soil moisture, soil temperature, gas pressures, and in situ bulk density of the overburden. Gas flux rates in the field varied spatially and temporally, and it was also observed that higher levels of soil moisture caused by rainfall events might have temporarily reduced carbon dioxide flux rates (Scale et al., 2015). Occasionally, elevated concentrations of soil carbon dioxide (up to 21 %) and methane (up to 12 %) were detected. These high concentrations of carbon dioxide and methane might have been caused by the presence of low permeability layers of bitumen in the subsurface inhibiting the free flow of gas to the surface. High concentrations of carbon dioxide and methane at these depths in the soil may have some negative impact on plant growth (Scale et al., 2015). Methane entering a well-aerated cover soil at concentrations greater than 10 % can potentially consume all oxygen in the soil atmosphere due to the stoichiometry of methane oxidation (CH4 + 2O2 \rightarrow CO2 + 2H2O). Carbon dioxide concentrations greater than 15 – 20 % can potentially impact nutrient and water absorption and be lethal to plants (Scale et al., 2015).

The construction of the overburden landform appeared to be more impactful to pore-gas dynamics than the design of the soil covers (Scale, 2017). To simultaneously facilitate CH₄ oxidation in the uppermost

horizon of the overburden, while maintaining sufficient pore-gas O₂ in the plant-rooting zone of the soil covers (to facilitate growth and survivability of reclamation vegetation), Scale (2017) recommended that overburden be placed to a bulk density of <1.5 Mg/m³ (at a relatively lower void ratio) for soil covers thicker than 1 m and placed to 1.6-1.8 Mg/m³ (at a relatively higher void ratio) for soil covers thinner than 1 m. Scale (2017) also suggested that extreme moisture conditions in the soil covers and uppermost overburden have the potential to restrict gas exchange and CH₄ oxidation.

7.1.2 ASCS Capping and Cover Layers

The ASCS was designed such that scientifically rigorous comparisons could be made across soil treatments (O'Kane, 2018). Twelve treatment options were constructed in triplicate (Figure 4) with the following reclamation materials salvaged within the mine disturbance footprint (O'Kane, 2018; NorthWind, 2013b):

- **Peat** Surface organic horizons collected from peatland bog and fens
- LFH Surface litter layer, A horizon and potentially a portion of the B horizon salvaged from coarse-textured, upland a and b ecosites (Beckingham and Archibald, 1996)
- Bm Coarse-textured (sandy loam and coarser) subsoil material salvaged at a depth of approximately 15 to 50 cm from upland a and b ecosites, containing NPHCs and from stockpile
- Center Pit Bm Coarse-grained sandy loam to loamy sand subsoil material, differing in lithology from other subsoils of the study, salvaged at a depth of approximately 15 to 50 cm from upland a and b ecosites, containing NPHCs and from stockpile
- Blended B/C Coarse-textured subsoil material salvaged at a depth of approximately 50 to 100 cm from upland a and b ecosites, containing NPHCs and from stockpile
- **Subsoil** Coarse-textured subsoil material salvaged at a depth of approximately 15 to 250 cm from upland a and b ecosites, containing NPHCs

The subsoils (Bm, Blended B/C and Subsoil) and LFH were salvaged from the same location, while Center Pit Bm and Peat were salvaged from other areas of the mine.

Initial planning of the study included two components to create contrasting treatments (O'Kane, 2018):

- Constructing various soil cover system designs using reclamation material types available at the Aurora North mine
- Varying the total reclamation capping thickness of select soil cover system designs

Revegetation added a third component to the study to test the effect of different tree species and planting density on aspects including soil-water demand, understory development and growth performance. Revegetation included planting a mix of tree and understory species. Trembling aspen (*Populus tremuloides*), white spruce (*Picea glauca*) and jack pine (*Pinus banksiana*) were planted to an even density of approximately 1,800 stems per ha across the study plots. Understory species of green alder (*Alnus crispa*), pin cherry (*Prunus pensylvanica*) and Saskatoon (*Amelanchier alnifolia*) were planted to a density of approximately 800 stems per ha. Within each study plot (cell), vegetation subplots of 25 x 25 m were also created, and tree planting species and density was controlled. Each cell contains 4 vegetation subplots of either trembling aspen, white spruce, jack pine or a mix of each species at a density of 10,000 stems per ha. Some cells also have an additional 4 subplots planted to 2,000 stems per ha. No understory species were planted in the vegetation subplots.



Figure 4. Reclamation Material and Subsoil Treatments Applied at the ASCS (from O'Kane, 2018)

Note: All treatments are underlain with LOS overburden (not shown), however only soil designs with LOS within the top 150 cm show overburden in the soil profile depictions.

Bulk density of the surface reclamation capping coversoil and subsoil materials (and LOS for shallow (30 cm) capping treatment) to a maximum depth of approximately 60 cm was collected by Drozdowski et al. (2013) using a bulk density collar sampling technique and a CPN® MC-1 DR-P Portaprobe Moisture/Density Gauge. Materials evaluated consisted of Peat, LFH, Subsoil, Bm, Blended B/C, and Center Pit Bm (See above for descriptions). Dry bulk density did not differ significantly with depth for the majority of the materials within the same treatment (Drozdowski et al., 2013). However, there was a trend of increasing density with depth for most of the materials evaluated. Similar dry bulk densities were found for Subsoil and Bm, which had overall dry bulk densities (probe measured) of 1.55 ± 0.07 g/cm³ (range 1.38 to 1.69 g/cm^3) and 1.59 ± 0.07 g/cm^3 (range from 1.35 to 1.71 g/cm^3) respectively, and Blended B/C and Center Pit Bm which had overall dry bulk densities of 1.73 ± 0.07 g/cm³ (range 1.54 to 1.91 g/cm³) and 1.71 ± 0.07 g/cm³ (range 1.57 to 1.85 g/cm³) (Drozdowski et al., 2013). The coversoil materials (Peat and LFH) were less homogenous and had higher variability than the underlying materials. Drozdowksi et al. (2013) noted that although several of these materials reached bulk densities which could be considered limiting for plant root development and water infiltration and movement, the sandy texture of the materials needs to be taken into consideration (which will have a significantly different effect on plant root development at a high bulk density than a much finer-textured material such as the underlying LOS which has an overall dry bulk density of $1.62 \pm 0.21 \text{ g/cm}^3$) (Drozdowski et al., 2010).

Drozdowski et al. (2013) noted that the substantial difference in bulk densities and texture between coversoil, subsoil and underlying LOS overburden may influence the infiltration, storage and movement of water through the soil profile. For example, the LFH and Peat coversoil materials have relatively low bulk densities (1.11 ± 0.14 g/cm3 and 0.59 ± 0.13 g/cm3, respectively) and are typically underlain by materials with higher bulk densities. The abrupt change in bulk density at the interface could influence the soil's capillary, lateral and preferential water movement as well as available water and water holding capacity in the rooting zone.

7.2 Surface Hydrologic Considerations and Controls

7.2.1 Five-Year Hydrological Monitoring Report

The conceptual hydrological model of performance at the ASCS was developed in 2013 and includes higher net percolation rates following spring snowmelt and autumn rainfall events when evaporation rates are lower and water storage capacity of the cover system treatments is exceeded (O'Kane, 2018). Lower net percolation rates were expected during the summer months, as there is adequate water storage capacity to store rainfall and then release it back to the atmosphere through surface evaporation and plant transpiration. Surface runoff is expected to be low for test plots with Peat or LFH as a surface material, due to the high hydraulic conductivity of cover system materials and the relatively flat topography. Surface runoff was expected to be higher for test plots with no cover soil, due to the hydrophobicity of the coarse textured subsoil material (O'Kane, 2018). Actual evapotranspiration (AET) rates will be driven by the productivity, density, and health of the vegetation cover.

O'Kane (2018) found that the hydrologic performance of the five monitoring years generally followed the conceptual model. Net percolation rates were highest in response to the spring snowmelt and large rainfall events in spring and fall that exceeded the field capacity of the cover system materials. Cover system treatments with Peat surface materials had the lowest net percolation rates. Net percolation was higher for treatments with LFH surface materials and no cover treatment. Water storage capacity for treatments with Peat was higher than other treatments, and the LFH treatments reached wilting point sooner and stayed in the condition longer than Peat treatments.

2015 and 2017 were the warmest and driest growing seasons of the 5-year period, and rainfall was lower than the 1944 to 2017 Fort McMurray average of 326 mm for those years (O'Kane, 2018). The remaining years were all significantly higher than the average. Snow water equivalent as measured by snow survey ranged from 27 mm in 2017 to 93 mm in 2013. Average total reference evapotranspiration for the 5-year monitoring period was calculated as 598 mm (O'Kane, 2018).

Soil temperatures were examined for treatment types to determine freezing patterns and the number of active growing days (O'Kane, 2018). Freezing was more gradual, and temperatures were not as cold at depth for test plots with Peat surface material, as the lower thermal conductivity of peat caused insulation of subsurface materials. However, the presence of peat generally did not minimize frost penetration depth. Depth of freezing was examined across the capping study and was found to be deeper in the perimeter plots, possibly due to an exposed crest resulting in an increased temperature gradient between the cover system material and the surrounding air (O'Kane, 2018). The number of days soil temperatures at 10 cm depth exceeded 5°C was used as criteria for when vegetation was active. Plots with peat as a surface material had a significantly lower number of days with active vegetation than plots with LFH or no coversoil (O'Kane, 2018).

Volumetric water content and water storage data were examined to determine dynamics within profiles, and to identify periods of possible vegetation water stress (O'Kane, 2018). For surface materials, Peat had higher volumetric water contents than test plots with LFH or no coversoil. Water storage for plots with

LFH as a surface material more frequently fell below wilting point during the water year and stayed in that state longer, indicating water stress for vegetation (O'Kane, 2018). The presence of overburden underlying the cover systems was found to restrict drainage of excess water from the soil profile, indicated by higher volumetric water content (VWC) above the cover system / overburden material interfaces relative to shallower soil depths. Overburden materials generally did not exhibit volumetric water content values below field capacity (O'Kane, 2018).

Although vegetation growth is still in the early years of growth and demand will likely change as they reach maximum water demand, water storage data were examined for tree plot locations to determine differences in vegetation treatments and planting densities. To date there is no consistent pattern of cover system storage with vegetation type. The high-density vegetation plots generally used more water stored in the soil profile compared to the low-density plots, which is expected given a higher demand for water with denser vegetation (O'Kane, 2018). Water balance fluxes for one test plot of each of the cover system treatments were calculated for each of the five monitoring years. Net percolation for individual years was found to range from 3 % (10 mm) and 27 % (80 mm) of annual precipitation over all treatments over the 5-year monitoring period. Average net percolation for each treatment over the 5-year monitoring period ranged from 8 % to 22 % of annual precipitation (O'Kane, 2018). In general, net percolation was least in treatments with peat as a surface material and greater in treatments with LFH and no coversoil treatments.

Groundwater levels in standpipe piezometers at the ASCS were measured and samples taken for chemistry analysis during the 5-year monitoring period (O'Kane, 2018). A deep and shallow water table was identified by water level measurements and a steady decline in both water tables was observed over the 5-year period.

7.2.2 Hydraulic Conductivity

The saturated hydraulic conductivity (Ks) is a key property governing the performance of the ASCS covers in that it influences key aspects of the water balance including infiltration and net percolation (Huang et al., 2016). *In situ* testing is required to both characterize the cover soil and to evaluate the spatial distribution of Ks following placement of the soil reclamation cover. The hydraulic properties of the soil reclamation covers can also evolve over time as a result of changes in secondary structure brought about by processes such as freeze–thaw or wet–dry cycles, or bioturbation processes caused by rooting or burrowing animals. All of these processes can lead to the formation of macropores and fractures, which increase the hydraulic conductivity over time.

O'Kane (2013) conducted 42 Guelph permeameter tests in October, 2010. The range of saturated hydraulic conductivity of the LOS study area found was between 6.0E-09 m/s and 4.3E-05 m/s with a geometric mean of 2.1E-07 m/s. Zettl et al. (2013) conducted 145 Air-K tests in October 2010 and the range of saturated hydraulic conductivity of the LOS study area found using the Air-K between 8.0E-08 m/s and 7.7E-04 m/s with a geometric mean of 4.2E-06 m/s. (Barbour and Zettl (2016) subsequently noted an updated range of Ks in a letter for Syncrude, to adjust for updated information about the source of the LOS. The average Ks for the Existing LOS and New Source LOS were 1.9E-06 m/s and 8.1E-06 m/s, respectively. The higher Ks value corresponds with the slightly higher average sand content in the New Source LOS, 55.8% as compared to 54.7% for the Existing LOS.)

On average the Air-K saturated hydraulic conductivity values were less than a half an order of magnitude lower than the Guelph values and this discrepancy is not thought to be due to differences in material properties at different sub-plot locations (Zettl et al., 2013). Both Guelph and Air-K showed similar normalized probability distributions for the K values obtained using each instrument. Zettl et al. (2013)

suggested that rapid testing with the air-K device can capture the variability in K with time or area effectively and should be able to be correlated to the K values obtained from GP testing.

O'Kane (2013) found that the Ks of all the cover materials are comparable. The Ks values were the same order of magnitude, but the air-K results had a greater range (2.0E-05 m/s to 9.0E-m/s) than Guelph permeameter (GP) results (3.0E-05 m/s to 5.0E-m/s). The air-K spatial pattern is dependent on material type, while the GP K spatial pattern is not. O'Kane (2013) noted that the Peat had the lowest Ks of all the material types, as measured by air-K. They also noted that for subsurface materials, spatial patterns do not appear to be dependent upon material type, but this may be due to the limited number of data collection locations.

Huang et al. (2016) found that geostatistical analysis showed a weak spatial dependency of Ks values with an effective range of 158 m for overburden material at the ASCS. Multiple regression of Ks calculated from Ks at field capacity showed a positive relationship between Ks and sand content, and a negative relationship between Ks and clay and silt content. The model used by Huang et al. (2016) indicated that the sand and bitumen content of LOS overburden played the dominant roles in the estimation of Ks for overburden material at the ASCS, and that increasing bitumen content in the overburden could result in a significant reduction in Ks value.

Alam et al. (2018) completed inverse modeling using HYDRUS-1D of five soil hydraulic parameters and used the results to simulate long-term water balances for ASCS covers. They found that peat coversoil had the highest uncertainty in water retention curves compared to subsoil and LOS layers, and the greatest uncertainty amongst all hydraulic parameters and soil types (coversoil, subsoil and LOS) was the Ks of LOS. Alam et al. (2018) found a wide range of simulation performance when the uncertainty of soil hydraulic parameters was combined with climate variability (e.g., the maximum difference between annual actual transpiration and net percolation when compared to the base case (median) was 14 % and 212 % respectively). It was noted that the parameter uncertainty exerted the greatest control over the long-term water balance variability. This range of performance resulted in a recommendation that further research was needed to evaluate the impact of reclamation cover thicknesses, to resolve some of the parameter uncertainty.

7.2.3 The Effect of NPHCs

Pernitsky et al. (2016) showed that increasing NPHC concentration reduced the water retention of LOS due to the presence of NPHCs in soil micropores. Due to the reduced Ks with increased PHCs and bulk density, Pernitsky et al. (2016) recommended that LOS used as the base material in reclamation would reduce percolation and retain more soil-water in the reclamation soil cover.

Hunter (2011) and Rosso (2016) investigated whether reclamation practices increase soil water repellency. Hunter (2011) found that soil water repellency was highly spatially variable in mineral soils salvaged in close proximity to the soils salvaged for the ASCS. The severity of water repellency of materials examined was Mineral Soil > Peat \approx LFH > Coarse-textured tarball-affected materials (aggregate oil sand material [AOSM] in the Rosso [2016] study).

Rosso (2016) completed water retention and hydraulic conductivity studies that indicated that although the AOSM was hydrophobic, its placement at concentrations typical of soils in the regions did not create consistent differences in the amount of moisture retained or transported. Results from column studies showed that under steady state and transient conditions, AOSM could result in decreasing infiltration rates and increasing chloride retention. The integration of soil layers further slowed the infiltration rate and delayed chloride transport. Overall, it can be concluded that with appropriate material placement, the addition of layering schemes and hydrocarbon material, the potential exists to increase soil water content in the upper layers of the soil, thereby increasing soil water storage for plant use.

Rosso (2016) concluded that the subsoil containing NPHCs has higher retained moisture levels, slower flux rates and lower hydraulic conductivity than similar textured soils that have no NPHCs, which increases soil water storage for plant use. The ability of the subsoil material to exhibit these characteristics may be attributed to the greater susceptibility of the coarse material to water repellency due to smaller specific particle surface area and therefore reduced organic matter, or hydrocarbon material, required to coat the surface. The increased moisture may also potentially be attributed to the presence of a greater fine sand fraction within the subsoil material where present. Although there may be concerns about contamination from hydrocarbon leaching from AOSM integration into reclamation designs, low levels of hydrocarbon fractions were observed in the outflow solution of the columns used (Rosso, 2016).

Hunter (2011) made the following recommendations, based on the water repellency of materials in an LFH study situated on the Fort Hills Dump (prior to development of the ASCS):

- Wettable reclamation materials are suitable for use on highly exposed surfaces and steep slopes.
- Water repellent materials are best used on protected, north facing slopes, on flat surfaces, in the subsurface or in mixes with highly wettable materials.
- Mineral soils are generally wettable above gravimetric water content 5-10%.
- Water repellency increases with decomposition level of peat.
- Organic reclamation materials may be wettable, but can be at risk of becoming water repellent.
 Slope and structure are important considerations for these materials.

Neil (2018) found that a correlation exists between AOSM salvage depth (soil containing NPHCs) and extent of weathering, where near-surface deposits contain fewer NPHCs in more degraded forms than at depth. Neil (2018) commented that this relationship confirms onion-skin weathering, where exposure to weathering and degradation is greater in the near-surface of the profile and on the surface of individual aggregates. As with Hunter (2011) and Rosso (2016), Neil (2018) noted that the water repellency of AOSM was significantly greater than the surrounding soils, indicating potential for reduced absorption and conductivity. Neil (2018) noted that as salvage depth of AOSM increased (and weathering decreased), NPHC content and water repellency increased while infiltration rate decreased. AOSM may slow the flow of water through the profile, increasing water storage and providing additional plant-available water, potentially modifying the soil water regime.

Applying a 20 cm peat coversoil depth (as is found in the most recent operating approvals for oil sands mine operations), Huang et al. (2017) studied whether there is an optimal depth of sandy subsoil which will meet the desired revegetation requirements. The increases in the mean annual T_r (actual transpiration) and the subsequent decreases in net percolation (NP), over the 60 year climate cycle as a result of increasing the subsoil thickness from 50 cm to 150 cm, were relatively small (Huang et al., 2017). The main reason is that the subsoil is very sandy and has a very low field capacity of approximately 5 %, so the maximum volume of plant available water would only increase by 50 mm for a 100 cm increase in subsoil thickness (50 versus 150 cm).

Huang et al. (2017) found that the LOS K_s had the greatest influence on annual mean T_r and NP. The LOS K_s had a large range of optimized values, from 6.0e-9 to 4.3e-5 m/s (approximately 0.5 to 4000 mm/day). At the low end of this range, the LOS K_s could restrict drainage sufficiently to increase the water availability to plants following a large infiltration event. The reclamation design should consider the effect of spatial

variability of LOS hydraulic properties on the performance of reclamation covers. The peat K_s and water storage both played an important, albeit secondary, role in affecting Tr. The higher values of peat K_s did appear to increase soil evaporation and consequently reduce the available water for plant transpiration.

7.3 Soil Cover Design

7.3.1 Pore-Gas Effects

Scale (2017) and Scale et al. (in prep[a], in prep[b] and in prep[c]) analysed pore-gas O_2 , CO_2 , and CH_4 concentrations in the soil covers and LOS of the ASCS following the placement of single and multi-layered soil covers. Concentrations of O_2 and CO_2 within the soil covers and within the uppermost 1 m of LOS were generally at levels considered safe for plant growth (e.g. >10 % O_2 and <15 % CO_2). Pore-gasses deeper than 1 m within the LOS surpassed the threshold considered safe for plant growth, where minimum O_2 concentrations were measured at 0 %, peak CO_2 at 16 %, and peak CH_4 at 36 %. Frozen pore-water in the 30 cm Peat overlying LOS remained frozen until late June. It is believed the frozen layer blocked soil-atmosphere gas exchange and led to pore-gas CH_4 accumulating to concentrations greater than 36 % deeper than 2 m in the LOS.

Since pore-gases within the 30 cm soil covers and in the uppermost LOS horizon did not surpass the threshold that is considered to pose a risk to plant growth and survivability, the thickness of soil covers can likely be reduced from the 1.2 to 1.5 m thickness currently used by some mine operators to 30 cm without risking the development of conditions of low- O_2 stress or high- CO_2 toxicity (Scale, 2017). Pore-gas concentrations of CH₄ within the soil covers and the uppermost LOS horizon were typically undetectable or present at low concentrations (<1 %), indicating that the soil covers and uppermost LOS horizon are passively oxidizing CH₄ *in situ* (except in the case of the 30 cm peat cover mentioned above). Soil cover placement thickness was positively related to CH₄ removal efficiency.

Scale et al. (in prep [c]) noted a series of operational considerations for the outcomes of the pore-gas concentration and flux studies noted above. Minimizing the bulk density of the LOS and coversoil(s) was recommended to support CH_4 oxidation with a maximized void ratio. Balancing oxidation of CH_4 while maintaining sufficient pore-gas O_2 concentrations within the plant rooting zone was found to depend on the soil reclamation capping thickness. A capping thickness of 1 m and LOS bulk density less than 1.5 Mg/m³ achieved this balance, and a LOS bulk density of between 1.6 and 1.8 Mg/m³ was recommended for capping depths thinner than 1 m.

7.3.2 Vegetation Performance on Soil Covers

Landhausser and Karst (2018) evaluated tree, vegetation, and mycorrhizal growth and development over the first five growing seasons at the ASCS. Trembling aspen and jack pine grew the tallest in treatments with LFH as the coversoil material, while the treatments with no coversoil or 30 cm peat over LOS generally resulted in the shortest aspen and pine tree heights (Figure 5). Treatments with 30 cm peat over a reclamation subsoil generally resulted in intermediate tree heights of aspen and pine. There was less variability in the white spruce tree heights among the treatments (Figure XX), however the treatments with no coversoil and 30 cm peat placed directly LOS resulted in the shortest spruce tree heights. All tree species responded poorly to the 30 cm of peat directly over LOS overburden, but the placement of a subsoil layer under the peat coversoil, regardless of thickness, influenced soil water and chemical dynamics of the peat layer and increased seedling growth.

They concluded that coversoil type influenced tree and vegetation growth primarily through resource availability and soil temperatures. Early successional species (aspen and jack pine) responded best on FFM coversoils, presumably through high N, P, and NH₄+ availability which met the higher nutrient

requirements of these early successional species. Tree growth on peat coversoils was less responsive when compared to FFM, likely through high S concentrations and lower P and K availability, which was potentially limited through complexation with higher concentrations of Ca and Mg present.

Landhausser and Karst (2018) found that peat coversoil soil temperatures remained cooler for longer periods during the growing season due to increased water content and greater insulating characteristics compared to mineral coversoils, which may have limited seedling growth. White spruce, a late successional species, was generally more tolerant of resource variability between coversoils. Although not statistically significant, seedlings on Bm (coarse-textured from stockpile) subsoil material exhibited better growth with peat coversoils, possibly due to increased nutrient availability in the subsoil.



Figure 5. Average and Standard Error of Tree Seedling Height for Trembling Aspen, Jack Pine and White Spruce in 2016 for Soil Treatments at the Aurora Soil Capping Study (from Landhäusser Research and Karst Lab 2018)

Note: Uppercase letters indicate significant differences within the heights of each species (α =0.1) following pair-wise comparisons that were adjusted using the Holms-Bonferonni method.

Soil materials colors are the following: Green - LFH, Brown - Peat, Red - Bm, Blue – Center Pit Bm, Pink – Blended B/C, Yellow – Subsoil and Black – lean oil sand. (see Section 7.2.2 for a description of each soil reclamation material type).

The effect of total depth of capping material (coversoil and subsoil) was tested, with treatments consisting of 30 cm peat directly over LOS (Treatment 5), 60 cm (30 cm peat and 30 cm Blended B/C; Treatment 4), 100 cm (30 cm peat and 70 cm Blended B/C; Treatment 11) and 150 cm (30 cm peat and 120 cm Blended B/C; Treatment 10). For aspen, pine and spruce species, the tree height after five growing seasons was significantly less in the 30 cm peat directly over LOS than all other treatments, except for pine growth in the 60 cm total depth treatment (Figures 6 to 8). There was no significant difference in tree height for all species between the 60, 100 and 150 cm treatments.

Figure 6. Average and Standard Error of Trembling Aspen Growth in the First Five Growing Seasons and Total Height in 2016 in Treatments with Different Total Capping Depths (from Landhäusser Research and Karst Lab 2018)



Note: Uppercase letters indicate significant differences in the 2016 heights (α =0.1) following pair-wise comparisons that were adjusted using the Holms-Bonferonni method.

Figure 7. Average and Standard Error of Jack Pine Growth in the First Five Growing Seasons and Total Height in 2016 in Treatments with Different Total Capping Depths (from Landhäusser Research and Karst Lab 2018)



Note: Uppercase letters indicate significant differences in the 2016 heights (α =0.1) following pair-wise comparisons that were adjusted using the Holms-Bonferonni method.

Figure 8. Average and Standard Error of White Spruce Growth in the First Five Growing Seasons and Total Height in 2016 in Treatments with Different Total Capping Depths (from Landhäusser Research and Karst Lab 2018)


Paragon (2018) completed analysis of soil samples and tree heights collected from reclaimed sites with an overburden substrate at the Suncor Base Mine. The concentrations of NPHCs analysed from substrate material varied over the sample sites, suggesting that the inclusion of LOS in overburden materials used to construct the reclamation areas at Suncor was variable. The sample size of tree heights collected to calculate site index was small (due to the focused selection of sample sites to ensure LOS within the substrate material), but despite this the performance of both white spruce and aspen appeared to show that there is no substantial negative effect of NPHC content on tree growth of these species, even at sample sites with high NPHC content.

The site index values of white spruce and trembling aspen from Paragon (2018) were comparable to a reclaimed d ecosite site index from Syncrude's (2016) Performance Surveys and Permanent Sample Plots, and a reclaimed d ecosite from Suncor's (2018) Performance Surveys. The site index values of white spruce and trembling aspen from Paragon (2018) were also comparable to Archibald and Beckingham (2006) site indices for native b and d ecosites of the region.

Hu et al. (in preparation) investigated the effect of coversoil type on soil temperature (ST) and soil water storage (SWS), and found that peat coversoils increased the soil water storage and buffered against the sensitivity of SWS and ST to seasonal conditions. FFM as a coversoil corresponded to higher soil temperature during the growing season when compared to a peat coversoil, and experienced increased SWS with layered subsoil types. For both coversoil types, the presence of LOS increased the SWS of subsoil which was attributed to the low hydraulic conductivity of clay material and hydrophobicity of NPHCs in the LOS.

Jones (2016) investigated the vegetation cover and community composition of ASCS plots. There was no significant difference in total cover, species richness or community composition due to the depth of FFM (forest floor material) at either 10 cm or 20 cm (referred to as LFH in Figure 4). The placement depth of peat did not affect species richness, but the vegetation cover was higher in 30 cm depth of cover compared to 10 cm (see Figure 5) and the community composition was significantly different. The type of subsoil under the FFM was important – species richness and cover were higher over the Bm subsoil than other subsoil treatments (B/C blended subsoil or C subsoil). Jones (2016) also found that vegetation cover and community composition did not differ between treatments with 30, 60, 100 and 150 cm of total capping depth (see Figure 4). Species richness was significantly higher in the 30 and 60 cm capping depths than deeper depths. Landhausser and Karst (2018) suggested that lack of species richness and cover in the early stages of vegetation development on peat and subsoil treatments resulted from the limited propagule bank in those materials. They further state that species richness on all coversoil treatments will increasingly be influenced by tree and colonizing vegetation dispersal over time, potentially limiting early developmental differences in species richness between coversoils.

Hogberg's (2017) study found that based on nutrient levels, the reclamation treatment of B/C blended subsoil (BCB) capped with FFM was most similar to reference upland forest sites. Among treatments capped with peat-mineral mix, a subsoil BCB horizon thickness of 70 cm was most similar to the reference site, followed by the 120 cm BCB and 30 cm BCB treatments. Sites capped with FFM materials were found to be more similar to natural reference sites. Peat-mineral mix is still capable of supporting desired tree and understory species on the sites, as evidenced by the fact that aspen, pine, spruce, and other species grow on those soils and that foliar nutrient concentrations are similar to or exceed those found in the natural reference sites.

Gaster (2015) examined the influence of aspen seedling nutrient and carbon reserve factors on the community development of ectomycorrhizal fungi (EMF) on trembling aspen seedling feed types (high and standard), which had differing tissue carbon storage and nutrient levels, planted into two types of

cover soils, a reclaimed forest floor and a peat-mineral soil mix. The abundance of EMF was influenced by feed type but not cover soil type, with high feed seedlings having increased colonization by EMF than standard feed seedlings. Gaster (2015) proposed that seedlings should undergo nutrient loading prior to outplanting to improve EMF recovery and that the mechanism behind increased EMF colonization of high feed seedlings may be related to exudation.

Hankin (2015) and Hankin et al. (2015) found that the species of tree seedling was more important in determining EMF community composition rather than reclamation soil type. FFM, peat, and subsoil contain EMF and after one growing season EMF communities generally display host preference, regardless of cover soil type. Based on these results, Hankin (2015) suggested that using a diversity of tree seedlings for outplanting onto reclamation areas may recover a more diverse ectomycorrhizal fungal community. In the absence of EMF associations (which was tested through the removal of all soil biota by autoclave sterilization), jack pine seedlings experienced 40 % mortality, spruce seedlings experienced 10 % mortality, and aspen experienced no mortality, indicating that EM fungi are critical to the establishment of coniferous seedlings, particularly jack pine. Landhausser and Karst (2018) found that EMF community composition differed among white spruce, aspen, and jack pine host species. For a given host species, EMF composition did not differ among coversoils on one- and two-year old seedlings. However, the effect of coversoil changed with time as EMF community composition differed among FFM, Peat, and Subsoil C on four-year old seedlings. Additionally, mixed species plots had a higher diversity of EMF strictly related to the greater number of host species relative to single species plots, indicating that using mixed species plots may have an additive rather than a synergistic effect on EMF communities at early stages in forest development.

Pec et al. (in prep) compared the EMF community composition in ASCS coversoil treatments with that of disturbed reference ecosystems (mature jack pine stands with trees removed and forest floor left intact or removed). The severity of disturbance had a much greater effect on the EMF composition than the type of tree seedling, type of coversoil or time since planting, although the seedling type had the greatest effect in the first two years of the study. Pec et al. (in prep) comment that the effects of different coversoils may result in an altered EMF fungal community trajectory in comparison to natural areas recovering from disturbance.

7.3.3 Cover Soil Nutrients and Microorganism Activity

Quideau et al. (2017) compared carbon (C), nitrogen (N), and phosphorus (P) release rates from peat and FFM (LFH) coversoil used to cap reconstructed sandy soils at the ASCS. The two materials released comparable amounts of N on a per kilogram of soil basis (111-118 mg N kg–1). However, when results were normalized based on each material's organic C content, N release was six times greater for FFM than for peat. Overall C mineralization and P release rates were over one order of magnitude higher with FFM than with peat (Quideau et al., 2017). As opposed to N, however, P release seemed to be controlled more by abiotic processes than by organic matter mineralization.

Quideau et al. (2017) noted that while the FFM material overall released more N and P, it also degraded faster; in comparison, peat may provide a smaller but more stable release of N. The mineral component of both materials was sandy (>90 % sand), but peat samples contained five times more organic matter than the FFM and had a much greater water-holding capacity. Total N concentration was seven times greater for peat than for FFM (7.0 vs. 0.8 mg g-1), and although total P was similar in both materials extractable phosphate concentrations were 10 times greater in FFM than peat (25 ± 2 vs. 2.5 ± 0.7 mg g-1).

Barnes et al. (2017) and Barnes (2016) investigated the effect of the physical properties of Brunisolic soils developed on sandy deposits on the accumulation and distribution of soil nutrients. The same soils

comprise a significant portion of the land disturbed by surface mining in the Athabasca Oil Sands Region and on the ASCS. Under jack pine, relatively small decreases in sand content of the upper soil profiles, from 97 to 88 %, were associated with greater total nutrient stocks and lower C/N ratios in the forest floors. In soils under aspen, forest floor nutrient stocks related most strongly with texture of the B horizons, with finer B horizons (from 94 to 78 % sand) leading to larger forest floor nutrient stocks. These results indicate that textural characteristics play an important role in the vegetation species that are present and in nutrient cycling of these sandy soils, and thus, should be a primary focus in reclamation. Barnes et al. (2017) findings further indicate that during the reclamation of both jack pine and aspen stands on sandy soils, returning optimal soil physical characteristics may outweigh initial soil fertility considerations over the long-term. When all sites were considered together, differences in forest floor total and available nutrients were largely influenced by the texture of the upper soil profile (0 to 7.5 cm).

Dietrich et al. (2017) tested the effect of admixing subsoil with peat and peat biochar on bioavailable nutrients, foliar nutrient concentration, and aspen productivity in a greenhouse study. Seedling productivity increased with admixing subsoil in both experiments with and without biochar, and there was an overall positive effect of amendment with biochar when comparing all treatments of both experiments using multivariate statistics, with biochar being more similar to FFM. However, Dietrich et al. (2017) found that peat-subsoil mixes did not provide sufficient amounts of P and Cu to seedlings, and a lower K and Mn availability in peat-subsoil mixes was also identified. This might be of importance when targeting plant communities adapted to certain soil characteristics, prescribing amendments targeting nutrient deficiencies deemed critical or when an ideal admix ratio cannot be achieved due to availability of salvaged materials.

Howell and MacKenzie (2017) and Howell (2015) investigated soil microbial community activity and bioavailable nutrient supply in a coarse-textured FFM (LFH) and organic matter-rich peat at the ASCS. Shallow (10 cm) and deep (20–30 cm) placement depths of FFM and peat were compared to a control receiving no coversoil and a harvested jack pine site as a reference. The greatest similarity was between FFM and the reference site for bioavailable nutrient supply and other measures of microbial activity and community. Shallow peat placement depth was more similar to reference conditions than deep placement for all parameters measured, suggesting that shallow coversoil applications might be sufficient for the reclamation target. Soil respiration rates were greatest in FFM, followed by the reference site and peat treatments, with no difference attributable to placement depth. Peat had greater nitrogen and sulphur availability, but was lower in phosphorus and potassium when compared to FFM and the reference site. Ecosystem function was more similar in coversoils that mimicked the reference site conditions as much as possible, which in this case meant shallow placement and material salvaged from upland forests.

Howell (2015) found that FFM shares the greatest similarity with natural upland benchmarks with respect to nutrient availability and soil biology. FFM introduces a similar microbial community structure and function to reclaimed sites based on analyses, and these results support the concept of returning FFM and PM to similar topographic and drainage positions from which they were salvaged where practicable. Deep applications of FFM do not provide statistically different nutrient profiles from shallow applications, and microbial community function shares greater similarity to the harvested benchmark. Deep FFM did create greater similarity in microbial community structure than shallow, but microbial diversity introduced from the shallow application may translocate deeper into the soil profile.

Pre-disturbance soils within mine development footprints have a significant proportion of fen and bog peatland soils (in some cases exceeding 50 % of development area). The reclaimed landscape at closure will shift to a higher proportion of upland area relative to bog and fen area compared to pre-disturbance distribution, necessitating the use of peat as coversoil for upland soil reclamation. Similarly to FFM, shallow peat shared a greater similarity with the reference site than deep applications, especially in

microbial community function at 10 - 20 cm depth. Howell and MacKenzie (2017) suggested that minimum capping depths should be maintained to reduce potential contaminant migration from overburden to surficial horizons, but the results suggest that deep coversoil applications may provide minimal or no added benefits to nutrient availability and soil biology relative to target benchmark soils in the region.

Kirby (2017) investigated if peat and subsoil could be mixed to produce a FFM-like bacterial community. The overall bacterial diversity of the peat-containing admixtures was indistinguishable from that of FFM, while that of subsoil was lower than all other admixtures. However, this was not reflected in the soil respiration, as the respiration rate was indistinguishable for all peat-containing admixtures, which was lower than for the FFM and higher than for the pure subsoil. The trends seen in the soil respiration rate correlated to the community composition, where there were three distinct groups; peat-containing admixtures, FFM, and subsoil. Initial results (2 years of growth) suggest that peat cannot be used if the goal is to approach a FFM-like bacterial community within the first couple years of growth, and that subsoil can be used to "dilute" the peat microbial community without an effect on its composition. No amount of subsoil added to peat can shift the peat community to become more similar to the FFM community, at least up to a 1:4 peat:subsoil ratio. Eventually vegetation and LFH layer development may start to drive nutrient cycling and return a FFM-like bacterial community, if peat can provide the initial starter conditions for vegetation growth, However, because the subsoil appears to act as somewhat of a blank in peat mixtures (increasing soil mass while not altering the microbial community composition or bulk activity), Kirby suggests that testing this for FFM soils may be worthwhile.

8 POTENTIAL ENVIRONMENTAL EFFECTS OF ELEVATED NPHCS

This section reviews the potential environmental effects associated with soil reclamation materials, interburden and overburden containing elevated NPHCs, to biological receptors in the closure landscape. Note that information related to the effects of leaching to water is provided in Section 6.

Where NPHCs are present in soils in the Oil Sands Region, they may influence soil quality and function and consequent vegetation community establishment (AEW, 2012a). AEW (2012a) noted that:

"Based on the limited information available, it is not possible to provide a comprehensive description of the effects of NPHCs on soils and vegetation in their natural state and be certain that the effects of NPHCs in the natural setting will translate to the reclaimed environment. Redistribution of NPHCs during salvage, stockpiling and placement of reclamation materials may increase exposure to receptors. Hydrocarbons in the reclaimed environment may inhibit plant establishment and growth, and interfere with soil biota that mediate decomposition, nutrient release and in some instances (e.g., mycorrhizae) nutrient uptake."

Over time, environmental receptors can be exposed to a continually different mixture of NPHCs due to the effects of ageing and weathering (see Section 5). Factors including location, length of time of exposure, and media of exposure, can all contribute to changing environmental effects (Ohio EPA, 2010).

Information available from studies on PHC effects has been included in this section with any available NPHC-specific studies. While the pace of research work on the environmental effects of NPHCs is increasing, the context of other PHC studies has been included to provide as full a description as possible.

8.1 Bioavailability

Peijnenburg et al. (2012) defined three issues that determine the way biota are exposed to their local environment: the morphology of the organism, the physiology of the organism, and the behavior of the

organism. The relative contribution of each exposure pathway (Figure 5) varies for different organisms, and depends on factors such as the hydrophobicity of the chemical of concern, and variations in environmental conditions such as soil type and climate. Peijnenburg et al. (2012) therefore provided this definition of bioavailability:

"The fraction of a total amount of a chemical present in a specific environmental compartment that, within a given time span, is either available or can be made available for uptake by (micro)organisms or plants, from either the direct surroundings of the organism or the plant, or by ingestion of food."





8.2 Effects on Vegetation

Chemicals of concern present in soil, water and air may be taken up by plants in the following ways (Mamy et al., 2015):

- From the soil and/or water by uptake through the roots (symplastic route)
- From the atmosphere by aboveground portions (including shoots, adventitious roots, surficial rhizomes) after wet or dry deposition
- Following rain-splash in which soil particles are dispersed onto leaf surfaces following the impact of raindrops on the soil surface
- From atmospheric chemicals of concern through the plant cuticle

For NPHCs in reclamation soils, this list presents the order of likelihood of interaction with plant species at risk of exposure.

Phillips et al. (2009) found that plant uptake of PHCs is, in general, governed by the hydrophobicity of the PHC. Moderately hydrophobic hydrocarbons are readily taken up by plant roots, while highly hydrophobic hydrocarbons bind strongly to lipids found on the root surface and may not be taken up.

Visser (2008a, 2008b, 2011) has completed the most comprehensive work to date on the effects of NPHCs on plant species used in oil sands reclamation work. Visser's work (2008a) found that tar balls did not impede growth of barley (*Hordeum vulgare*), trembling aspen (*Populus tremuloides*) and white spruce (Picea glauca) in microcosms, but there was less apparent root growth of jack pine (*Pinus banksiana*) in the presence of tar balls. Tar balls did not impede colonization of roots of the three tree species by ectomycorrhizal fungi, although that does not necessarily mean that ectomycorrhizal fungi are insensitive.

Visser (2008b) also found that jack pine and white spruce appeared to be more tolerant to high NPHC concentrations in LOS than barley, based on both shoot length and dry mass measurements. Visser (2008b) theorized that tolerance to NPHCs by the conifers may be related to their slow growth rate, and a longer-term bioassay (i.e., > 3 months) may be required to obtain a more accurate assessment of NPHC effects. Addition of peat improved plant growth, and an improvement in soil physical, chemical and biological properties, including structure (aggregation), organic matter content, fertility, aeration and drainage is thought to account for the better performance of the receptors in the peat/sand mix receiving soil. On the basis of shoot, root and total dry mass measurements, the weathered NPHC level in peat/sand mix that exhibited little to no toxicity to the majority of plant species was 5,157 mg kg-1 (F2, F3 and F4 PHCs were 94; 1,333; 3,730 mg/kg, respectively). This level meets the Alberta Tier 1 Guidelines (AEP, 2016a) for PHCs in fine-textured surface soil, but does not meet the guidelines for F3 PHCs in coarsetextured surface soil (Visser, 2008b). Visser (2008b) therefore suggested that 5,000 mg/kg (0.5 %) of wellweathered PHCs in a peat/sand mix would not constrain plant productivity. Visser (2008b) also found that although trembling aspen was very sensitive to fresh NPHCs, it appeared to be more tolerant to NPHCs than barley, jack pine and white spruce after the LOS had been weathered (the level of weathered NPHCs that did not inhibit productivity by more than 25 % was 8,094 mg/kg). At this concentration, F2, F3 and F4 NPHCs exceed the Alberta Tier 1 Guidelines (AEP, 2016a) for both coarse- and fine-textured surface soils.

In 2011, Visser examined the response of the same plant species to Cg horizon soils (native soil with gleyed subsoil under peat) containing NPHCs. The pattern of toxicity to plant growth corresponded with NPHC concentrations in the soils, except when peat was mixed into the Cg material and the productivity of all four plant species was good in all Cg soil samples. As noted in Section 6, Visser (2011) noted that the inconsistency in the relationship between soil NPHC concentrations and plant productivity in the Peat:Cg treatments indicated that other factors were influencing plant growth (such as poor soil structure

(aeration, porosity), high salinity, low fertility and poor water infiltration). In addition, trembling aspen was significantly better when Cg soils were distributed as discrete islands in the peat, than if mixed homogeneously with the peat (Visser, 2011). Aspen roots did not colonize the Cg islands, but grew extensively throughout the peat matrix surrounding the island.

8.2.1 Effect on Plant Roots

Phillips et al. (2008) found that diverse plant species (*Lolium perenne* [perennial rye grass], *Medicago sativa* [alfalfa], *Festuca rubra* [creeping red fescue], *Agropyron elongatum* [tall wheat grass], *Elymus angustus* [Altai wild rye], *Puccinellia nuttalliana* [Nuttall's salt meadow grass]) growing in weathered-PHC contaminated soil maintained distinct, heterogeneously-distributed endophytic (i.e., growing within root cells) microbial populations. The types of endophytic degrader populations and degradation activity showed substantial inter-species variation, which was independent of that shown by the respective rhizosphere populations, and likely due to root morphology and physiology. PHC degradation is believed to occur through a rhizosphere effect where plants exude organic compounds through their roots, which increases the density, diversity and activity of specific microorganisms in the surrounding rhizosphere, which in turn degrades PHCs (Balasubramaniyam and Harvey, 2014; Phillips et al., 2008). Degradation was specific to PHC type – although aliphatic PHCs were effectively degraded, few endophytic communities were able to effectively degrade aromatic (benzene ring-containing) PHCs.

Balasubramaniyam and Harvey (2014) investigated the responses of plants with different root architectures (beetroot [*Beta vulgaris*] which has a central taproot and many narrower lateral roots, and tall fescue [*Festuca arundinacea*] which has a mass of threadlike fibrous roots) grown in crude oil-treated sand. There were structural changes in roots of both species, and an inhibition in root lengthening especially in beetroot. In addition, the development of fewer, shorter root hairs were interpreted as potentially being a way for these species to absorb PHCs but prevent entry to the remainder of the root by sloughing off (Merkl et al., 2005). In general, morphological differences in plant species exposed to PHCs shows that root and shoot biomass, root length and root elongation rate are reduced due to growth in contaminated soil, whereas the root diameter was found to have increased under these conditions (Balasubramaniyam and Harvey, 2014; Visser 2008a; 2008b; Merkl et al., 2005).

Visser (2008a) found that shoot and root productivity of barley, trembling aspen and white spruce was not significantly affected by the presence of tar ball bitumen, and there were no significant effects on shoot growth in jack pine but root growth was significantly reduced in the tar ball treatment. The behaviour of roots near the tar ball disk was similar for all species, with roots branching at the point of contact with the tar ball, and then growing around the disk. None of the species appeared to avoid the tar ball, and the root density of all of the tree species was significantly higher in soil immediately adjacent to the tar ball. Roots that penetrated the tar ball had extensive root hair development. The colonization and community development of ectomycorrhizal fungi on white spruce and trembling aspen did not seem to be affected by the tar ball material. However, there was a reduction in number of ectomycorrhizal species on jack pine.

8.3 Effects on Microorganisms

Plant species or even genotypes of a species produce different root exudates which influence microbial communities (Shahsavari et al., 2013). Plants tolerant to PHCs produced specific root exudates which led to the activation of specific microbes (including hydrocarbon-utilizing microorganisms, and these microorganisms were associated with a reduction in toxicity associated with PHCs (resulting in good plant growth) (Hassan et al., 2014; Shahsavari et al., 2013). However, soils exposed to long-term contamination with PHCs may present extreme challenges to maintenance of diverse structural and functional microbial

communities. Pessacq et al. (2015) analyzed soils historically contaminated by different levels of heavy metals and PHCs and observed that the contaminants had caused a genetic and metabolic alteration in soil microbial communities. The presence of hydrocarbon-degrading bacteria and fungi in PHC-uncontaminated soil confirms that soil naturally contains hydrocarbon-utilizing microorganisms (Hassan et al., 2014).

Blain et al. (2017) investigated the Bitumount Provincial Historic site in Alberta, where despite historic PHC levels from 330 to 24,700 mg/kg, natural revegetation has been successful. Root endophytic bacterial communities were characterized from plant species, and the population abundance of rhizosphere and root endosphere bacteria was influenced by both plant species and sample location. Members of the Actinomycetales, Rhizobiales, Pseudomonadales, Burkholderiales and Sphingomonadales were the most commonly identified orders, as is the case with most boreal forest soils.

Visser (2008b) measured the respiration rate of peat/sand/LOS treatments, and found it was 2 to 4 times greater than that measured in the sand/LOS treatments due to the microbial biomass present in the peat. Respiration of both the sand/LOS and peat/sand/LOS treatments stabilized after 100 days incubation. Microbial diversity and biomass associated with the peat in the peat/sand treatments would be expected to enhance PHC degradation relative to the sand treatments, which lacked peat. However, NPHC removal in the peat/sand was very similar to that in the sand alone, which suggests that the addition of peat to the receiving soil did not significantly influence NPHC removal processes.

Visser (2008b) found that both the composition and abundance of ectomycorrhizal fungi were altered by the presence of NPHCs in the root zone. Total % abundance of mycorrhizal fungi decreased with increasing concentrations of fresh LOS, and no colonization was detected at soil PHC concentrations of 4 % or greater. Colonization by ectomycorrhizal fungi with dark hyphae [*Mycelium radicis atrovirens* complex (MRA) and *Cenococcum*] increased with increasing NPHC concentrations, while *Cenococcum* was the only fungus able to form mycorrhizas at 2.5 % PHC. The data suggest that some fungi such as E-strain (*Wilcoxina*) and some basidiomycete species are sensitive to fresh NPHCs in the soil, while others such as *Cenococcum* are very tolerant. Inhibition of E-strain (*Wilcoxina*) by NPHCs may have negative repercussions on conifer establishment and growth, since this fungus has been shown to promote tree growth during the early stages of reclamation of oil sands sites.

8.4 Effects on Soil Fauna

Risk and hazard assessments for the soil environment are performed on the basis of the total content of a contaminant in the dry bulk soil. Presently, scientific evidence is emerging and indicating that pore water may be a more relevant exposure medium for uptake of chemicals by biota and plants in soil (Peijnenburg et al., 2012). The mode of exposure of soil invertebrates is determined by morphology, physiology, and behavior, as well as the mode of uptake of food, water, and oxygen (Peijnenburg et al., 2012; Saterbak et al., 1999). The contribution of oral uptake may vary within a specific taxon but for soil organisms in close contact with the soil solution, pore water—mediated uptake is in general the dominant pathway and it is commonly modified by soil specific ageing and speciation, and by specific factors of the organisms, such as nutrition status. Residual uptake appears to be the most important uptake route following pore water—mediated uptake. It is likely that in this case, too, pore water is involved as carrier in or at the surface of the soil in which the chemicals are dissolved.

There is a distinction between hard-bodied organisms (e.g., spiders, woodlice, and collembolans) where uptake of oxygen and water is through specialized organs, and soft-bodied organisms (e.g. earthworms) where water and oxygen are mainly taken up via the skin (Peijnenburg et al., 2012). Uptake of nutrients and chemicals is possible for all invertebrates via their food, and this may be an important route in the case of food sources in which high concentrations of chemicals are present.

Cermak et al (2013) found that the aromatic hydrocarbons, although present in lower concentrations in the PHCs and soil, are the main contributors to the toxicity of contaminant distillates. Aromatics always reached maximal tissue concentrations before aliphatics within the exposure duration of the toxicity test and were accumulated to the greatest degree by earthworms.

For toxicity to occur, a substance must first be taken up by the organism and reach the site of toxic action in sufficient quantity and for a sufficient length of time to elicit a toxic response (Cermak et al., 2010). There is also evidence that internal exposure can be transitory, in particular for aromatic fractions. Petroleum hydrocarbons are expected to cause toxicity via nonpolar narcosis, a reversible mode of action (Cermak et al., 2013).

Sites contaminated with lighter PHCs are more toxic to earthworms than those with heavier PHCs in standard, 14-d lethality tests (Cermak et al., 2013). Earthworm accumulation rates were greatest for the lowest-boiling-point distillate (F2) and progressively decreased as the boiling point range of the distillate increased. This likely is due to a decrease in the bioavailability of oil constituents resulting from changes in sorption and water solubility as the boiling point range for hydrocarbons increases (Cermak et al., 2013). Likewise, sites contaminated with petroleum products with higher aromatic content are more toxic than those with lower aromatic content. Varying accumulation patterns for PHCs in earthworms result from the complex interaction of loss of contaminant from soil and changes in bioavailability over time. The result is differences in the internal exposure scenarios for earthworms for different distillates and for aromatic versus aliphatic hydrocarbons.

Visser (2008a) found that tar ball NPHCs had no significant effects on the survival and growth of adult Eisenia andrei (red wiggler earthworm). Earthworm reproduction, as measured by numbers of juveniles generated by two adults after 4 weeks, also was not inhibited by tar ball bitumen. However, the growth of juvenile earthworms, based on body weight per individual, was significantly reduced in the presence of tar balls (Visser, 2008a). It is not clear how directly applicable the impact of tar balls on earthworms (intended as surrogates of the soil macrofauna) is to biota of regional soils (AEW, 2012a). Results of these toxicological trials with selected receptors were consistent with expectations based on the chemistry of tar balls and the predominance of heavier fractions that are less bioavailable. AEW (2012a) stated that when present as tar balls, NPHCs do not present a severe risk to ecological receptors. Hence the presence of NPHCs in this form does not warrant exclusion from the current requirements in Industrial Approvals for Oil Sands mines for salvage of upland soil. This general recommendation may be conditioned by other concerns and amended on a case-by-case basis (AEW, 2012a).

In Visser (2008b), the bioassay results for peat/sand indicated that the concentrations of weathered LOS PHCs that would not pose a risk to ecological receptors in the soil contact pathway would be in the range of 4,000 – 5,000 mg/kg for the plant species tested, and 2,500 to 4,500 mg/kg for the more sensitive soil fauna species. The amount of LOS that could be incorporated into reconstructed soil and not inhibit plants and soil macrofauna by more than 25 % would depend on the PHC content of the LOS, and whether or not it was well-weathered. The undiluted lean oil sand contained approximately 5 % PHCs, which was reduced by 25 % after weathering for four months in the laboratory. The amount of fresh LOS added to the peat/sand to achieve a weathered PHC endpoint of approximately 5,000 mg/kg was 6.3 %, while the amount required to achieve an endpoint of 2,500 mg/kg was approximately 3.3 %.

AEW (2012a) noted that Visser (2008b) attempted to interpret the results against *Alberta Tier 1 Soil Remediation Guidelines* (AEP, 2012a) and infer critical hydrocarbon concentrations that would not constrain plant productivity. However, AEW (2012a) noted that at this time it is difficult to extrapolate from a microcosm study in which receptors experienced a definite concentration of hydrocarbons to the field where concentration is a more ambiguous description of what receptors experience. Earthworms

were more sensitive to LOS than the plants. Most of the soil biota involved in decomposition and nutrient release would occur in surface litter, the A horizon or the rhizosphere/detritosphere. The total percentage abundance of ectomycorrhizal fungi decreased with increasing concentrations of unweathered LOS.

9 REGULATORY APPROACH TO ENVIRONMENTAL RISK

9.1 Global Approach to Risk-Based Frameworks

Several risk-based frameworks for PHCs in soil have been published under the auspices of the Total Petroleum Hydrocarbon Criteria Working Group (TPHCWG), the American Society for Testing and Materials (ASTM), the Massachusetts Department of Environmental Protection (MaDEP), the Environment Agency of England and Wales (EA), the American Petroleum Institute (API), and the Canadian Council of Ministers of the Environment (CCME), each reflecting national legislation and socioeconomic issues (Brassington et al. 2007)(see Appendix C). These frameworks, and the exposure assessment methods embedded within them, do not specifically address weathered hydrocarbons, although many acknowledge that petroleum products released to the environment will have undergone some degree of degradation (Brassington et al., 2007).

Total petroleum hydrocarbons (TPH) is a standard used to date in Europe (Pinedo et al., 2013; Mao et al., 2009), but there is increasing evidence supporting quantification of individual hydrocarbons that may cause risk. These guidelines, referred to as Intervention Values (Pinedo et a., 2013). Mao et al. (2009), confirmed that TPH concentrations in soil (obtained by conventional chemical analysis) are not indicative of the corresponding ecotoxicity and leaching behaviour. High performance liquid or gas chromatography methods provide more accurate details in the chemical composition of the TPH, enabling a better estimation of their ecotoxicity in the soil phase and leaching water.

Saterbak et al. (1999) commented that the use of benchmark values for PHC-contaminated soils at a Tier 1 or 2 level should be discouraged on the basis that there is insufficient data and understanding of the impact of most hydrocarbon contaminants on soils to allow for the development of soil benchmark screening values. Additional complexities arise because of differences in soil types and in the length of time the contaminant has been in the soil. Interspecies differences in the sensitivities of plants, microbes, and invertebrates that constitute the soil community make it difficult to develop soil-quality criteria or benchmark values that are not overly conservative.

Huesemann et al. (2004) and Marchand et al. (2017) noted that the belief that slow or incomplete biodegradation of hydrocarbons in aged soils is primarily caused by bioavailability limitations has significant implications for risk assessment. If the residual PAHs that remain in aged soils and sediments even after long and extensive biotreatment are recalcitrant to biodegradation because they are sequestered and therefore not available for uptake and metabolism by microorganisms, it can easily be argued that a lack of bioavailability implies also a lack of toxicity and risk to potential environmental environmental receptors. Numerous investigators have demonstrated a decrease not only in contaminant concentration but also bioavailability, leachability, and toxicity with increasing biotreatment time and concluded that 'environmentally acceptable endpoints' were reached at the end of a treatment period although residual contaminants were still present in the soil.

9.1.1 Canadian Context

The CCME reference method (CCME, 2001) for the Canada-wide standard (CWS) for PHC in soil (CCME, 2008) is based on a risk assessment approach to managing contaminated sites remediation. The standards can be applied at three risk assessment levels or "tiers." Tier 1 is based on generic numerical standards

corresponding to four land uses. Exceeding the Tier 1 soil guidelines may lead to detailed site-specific evaluations at the Tier 2 or Tier 3 levels. The CWS PHC soil standards are organized into the following four PHC carbon-range fractions: F1 (C6–C10), F2 (C10–C16), F3 (C16–C34), and F4 (> C34). The carbon-range fraction F1 consists of nonpolar aliphatic and volatile aromatic PHCs, and F2 consists primarily of nonpolar semivolatile PHCs. Both F2 and F3 contain nonpolar aromatic and aliphatic hydrocarbons. Fraction F4 has low aromaticity and contains small amounts of polar nitrogen, sulfur, and oxygen heteroatoms (CCME, 2008).

The Tier 1 CWS PHC generic soil toxicity guidelines for F1, F2, F3, and F4 carbon ranges are based on risk management of environmental and human health exposures to PHC concentrations for each of the four fractions. Tier 1 considers site-specific conditions such as land use, groundwater potability, and mineral soil coarse and fine textures. Highly organic peat soils have low mineral content and do not therefore apply to either the fine or coarse soil categories. Regulatory discretion is used in the selection of the most appropriate fine or coarse soil toxicity guidelines for peat soils, which may depend on site-specific conditions. For example, the most stringent coarse soil guideline might be applied to contaminated sites located in higher risk areas with potable drinking water and/or nearby surface water systems that could carry PHCs to off-site locations (CCME, 2008).

The definition of acceptable concentrations of NPHC in reconstructed soils is problematic (AEW, 2012b). The document that has been used to assess the reclamation suitability of salvaged soils (*Soil Quality Criteria Relative to Disturbance and Reclamation* [AAFRD, 1987]) does not include hydrocarbon content, and the criteria provided in *Alberta Tier 1 Soil and Groundwater Remediation Guidelines* (AEP, 2016a) are intended for substance release and hence not directly applicable. Furthermore, concentrations of NPHCs in native soils have been shown to exceed *Alberta Tier 1 Soil Remediation Guidelines* (see Section 5). Some Oil Sands mines (e.g., CNRL-Albian, Imperial Kearl and Syncrude) employ/have employed a precautionary approach that defines a 'cut off' depth for soils containing NPHCs. Hydrocarbon concentrations >1 % are used to identify unsuitable reclamation material and delineate the depth of soil salvage.

AEW (2012a) notes that when concentrations are reported in soil surveys it is unclear how to interpret them in respect of relevant exposure pathways and receptors. Likewise, while microcosm studies have provided important preliminary information on toxicology, they did not include all relevant receptors at all relevant scales. Secondly, as indicated previously, *Alberta Tier 1 Soil and Groundwater Remediation Guidelines* (AEP, 2016a) is intended for substance release, and applies to mineral soils as opposed to organic soils. However, the Tier 1 Guidelines provide objective criteria for interpretation of results of toxicological trials and some of the principles underlying their derivation are relevant and informative.

As summarized in AEW (2012a) "the occurrence of NPHCs in regional soils that support functioning ecosystems defines background soil quality at some locations on the pre-disturbance landscape. The *Alberta Tier 1 Soil and Groundwater Remediation Guidelines* (AEP, 2016a) state that *"in some situations, the background concentration of some substances can be a significant proportion of, or even exceed, the Tier 1 guidelines. In cases where the natural background is demonstrated to be greater than Tier 1 guidelines, the remediation level shall be to natural background or to guidelines developed using Tier 2 procedures"*. Application of this principle is constrained by the fact that NPHCs occur in some but not all regional soils, and disturbance of reclamation material in salvage, stockpiling and placement may redistribute NPHCs in a manner that aggravates their experience by some receptors. For example, tar balls may break open and expose a relatively less weathered surface. Furthermore, the argument for an elevated background concentration would not apply to situations where indigenous PHC are intentionally introduced into reclamation material that was originally free of it. Rather, *"soil or groundwater with naturally elevated substance concentrations may become a source of contamination if it is redistributed and causes the receiving soil or water to exceed Tier 1 or 2 remediation guidelines"* (AEP, 2016a). Finally,

where tar balls occur on the landscape, it may be unreasonable to discriminate between the presence of indigenous PHC in soil and the underlying parent geologic material from which it has developed."

10 CLOSURE

This literature review was collated to provide support for risk assessment activities related to the effects of NPHCs in oil sand mining reclamation areas, and to support capping thickness and cover design decisions by industry members. There has been a focus on information from NPHC-specific research in this literature review (including peer-reviewed, internally-developed and other "gray" literature sources available from the JIP industry partners), supplemented by research on PHCs where there is currently a research gap or research activities are relatively recent for oil sands mining.

Environmental Protection and Enhancement Act (EPEA) approval clauses received by Canadian Natural Upgrading Ltd. in 2018 require a risk assessment of elevated NPHC levels found in overburden and interburden, located on reclaimed landforms and covered by reclamation materials. The literature review is intended to support regulatory submissions and further risk assessment activities required by oil sands industry members, along with the other components developed for this COSIA Joint Industry Project.

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Appendix A Hydrocarbon and Ecosite Maps from Paragon (2006)











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Appendix B Aurora Soil Capping Study – Layout Diagram from NorthWind Land Resources Inc. (2013)



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Appendix C Summary of Global Regulatory Hydrocarbon Analysis Types from Brassington et al. (2007)

	Massachusetts Department of Environmental Protection ⁵⁴	Total Petroleum Hydrocarbon Criteria Working Group ^{85–89}	Canadian Council of Ministers of the Environment ¹⁸	New Zealand ⁶¹	New South Wales ⁶⁴
Description Use of two methods. Volatile petroleum hydrocarbon (VPH) method ⁵⁸ and extractable petroleum hydrocarbon (EPH) method ⁵⁷ developed by MaDEP. The EPH method refers to U.S. EPA methods for sample extraction. ^{57,99}		Use of "The Direct method" (AEHS) ¹ developed for the TPHCWG framework. Based upon U.S. EPA SW-864 test methods ⁹⁹ and MaDEP EPH method. ⁵⁷	Recommends the use of benchmarked methods ¹⁹ ; however, also allows the use of nonbenchmarked methods providing that validation data demonstrate that the substitute method provides data comparable to the benchmark method.	Permits the use of a variety of methods, including those prepared by the Oil Industry Environmental Working Group (1999), ⁶⁷ which outlines methods for several different petroleum products often referring the reader to U.S. EPA documentation. ^{67,99}	Recommends the use of methods specified in the National Environmental Protection Councils (NEPC) Schedule B(3): Guideline on Laboratory Analysis of Potentially Contaminated Soils (1999) ⁶⁴ . Where no suitable analytical method is available it recommends the use of U.S. EPA ⁹⁹ or equivalent methods. ⁶⁴ All chemical analysis should be carried out in laboratories currently accredited by the national association of testing authorities (NATA).
Reported range Sample collection	C ₅ to C ₃₆ EPH method uses amber glass wide mouth sample jars with Teflon lined screw caps. These are cooled immediately after collection and extracted within 14 days of receiving the sample. VPH method uses specially designed air tight collection vials with Teflon-lined septa screw caps stored at 4°C and preserved with methanol before analysis within a maximum of 28 days.	C ₆ to C ₃₅ Wide mouth glass jars with Teflon lined caps stored at 4°C. Analysis must be performed within 14 days of sample collection.	C_6 to C_{50} Wide-mouth glass jars with aluminum foil or Teflon-lined lids. Samples must completely fill the jars. Samples are not chemically preserved but are cooled to 4°C. Laboratory sample handling procedure is also outlined.	C ₆ to C ₃₆ 100 ml (volatiles) and 250 ml (semivolatiles) Borosilicate jars with Teflon-lined cap and completely filled. Stored at 4°C in the dark.	C7to C36 Use of U.S. EPA ⁹⁹ or equivalent methods
	-				(Continued on next page)

TABLE 1. Summary of the Analysis Methods Developed for Several Risk Assessment Frameworks
	Massachusetts Department of Environmental Protection ⁵⁴	Total Petroleum Hydrocarbon Criteria Working Group ^{85–89}	Canadian Council of Ministers of the Environment ¹⁸	New Zealand ⁶¹	New South Wales ⁶⁴
Extraction technique	VPH method uses purge and trap with methanol. EPH method uses DCM for extraction and solvent exchanges into hexane. Using U.S. EPA methods 3540C ⁹³ (Soxhlet), 3545A ⁹⁴ (pressurised fluid extraction (PFE)), 3541 ⁹¹ (automated Soxhlet extraction), 3546 ⁹⁶ (Microwave extraction) and 3570 ⁹⁷ (microscale solvent extraction (MSE)).	Vortex or shaker method using <i>n</i> -pentane.	Purge and trap for C_6 to C_{10} range using methanol. Soxhlet is the benchmarked method for the C_{10} to C_{50} range.	For the C_{10} to C_{36} range any method that can be demonstrated to meet the performance criteria can be used. For the C_6 to C_9 range purge and trap is used.	U.S. EPA methods 3540B ⁹⁹ or C ⁹³ (Soxhlet extraction), 3550B ⁹⁵ (sonication extraction) or sequential bath sonication and agitation described by NEPC. ⁶⁴
Evaporation	The EPH method uses those specified by the U.S. EPA. However, after fractionation the use of gentle stream of air or nitrogen is recommended to bring the sample to the required volume. Evaporation is not applicable to the VPH method	N/A	Uses an evaporation vessel after extraction for the C_{10} to C_{50} range. After silica gel cleanup rotary evaporator is the benchmarked method to reach the required sample volume.	Permits the use of any method that can be demonstrated to meet the performance criteria.	U.S. EPA methods specified for extraction using Kurderna–Danish (K-D) evaporation.
Cleanup/fractionation	Silica gel cleanup for EPH method. Not applicable to VPH method.	Extract fractionation using alumina or silica.	One of two specified clean up steps for C_{10} to C_{50} range, not fractionated.	Clean up steps and fractionation are optional as this may not be required for each sample/analytical approach.	Solvent exchange into hexane followed by K-D evaporation and treated with silica gel as described in U.S. EPA method 1664. ^{64,99}
Analysis technique	EPH uses GC/FID.* VPH may use either GC/FID* or GC/PID.*	GC/FID*	GC/FID*	For the C_{10} to C_{36} range GC/FID^* is used and for the C_6 to C_9 range $GC/MS^{\$}$ is used.	GC/MS ^{\$} , or GC/FID [*] , however the use of GC/MS ^{\$} to identify unusual mixtures is noted as being necessary when analysing by GC/FID.*

TABLE 1. Summary of the Analysis Methods Developed for Several Risk Assessment Frameworks (Continued)

*GC/FID refers to gas chromatography with flame ionization detection.

[#]GC/PID refers to gas chromatography with photoionization detection.

^{\$}GC/MS refers to gas chromatography with mass spectroscopy detection.

TABLE 2. Summary of Risk Assessment Used by Several Different Jurisdictions (Modified After Environment Agency, 2003 ³⁰)										
	American Society for Testing and Materials ⁵	Massachusetts Department of Environmental Protection ⁵⁴	Total Petroleum Hydrocarbon Criteria Working Group ^{85–89}	Agency for Toxic Substances and Disease Registry ⁶	Canadian Council of Ministers of the Environment ¹⁸	American Petroleum Institute ³	Environment Agency ³²	New Zealand ⁶¹	New South Wales ⁶⁴	National Institute for Public Health and the Environment ¹⁰
Indicator compounds	Uses "chemicals of concern" only.	Use most toxic and those most frequently tested for	Uses most toxic compounds only	Uses most toxic compounds only	Uses most toxic and those most frequently tested for	Uses most toxic compounds only	Most toxic and most prevalent in petroleum hydrocarbon- contaminated environment	Use of "contaminants of concern" to address most toxic substances and aromatics	Individual compounds identified	Uses most toxic and those most frequently tested for
Fractions Number	None	6 Analytical fractions (3 aromatic, 3 aliphatic), using 4 toxicity values(3 aliphatic, 1 aromatic.	13 Analytical fractions (6 aliphatic, 7 aromatic), using 7 toxicity values (3 aliphatic, 4 aromatic).	Similar to TPHCWG. Minor modification to aromatic groups to include BTEX compounds in same fraction	4 Fractions, based on TPHCWG, separate evaluation of aliphatic and aromatic compounds not required	14 Fractions based on TPHCWG (7 aromatic, 6 Aliphatic and 1 aliphatic and aromatic combined)	16 Fractions based on TPHCWG and API (7 Aliphatic, 8 Aromatic and 1 aliphatic and aromatic combined)	3 Aliphatic fractions	2 Petroleum hydrocarbon fractions	7 fractions based on toxicity values (3 aliphatic and 4 aromatic)
Basis Application of approach	N/A RBCA 3 tiered look-up tables for tier 1 and increasing use of site-specific information in tiers 2 and 3.	Carbon number Not tiered as appropriate method is selected prior to assessment. 3 methods can be used— increasing specificity with methods 1 generic 3 site-specific.	Equivalent carbon number RBCA 3 tiered look-up tables for tier 1 and increasing use of site-specific into in tiers 2 and 3.	RBCA 3 tiered look-up tables for tier 1 and increasing use of site-specific into in tiers 2 and 3.	Equivalent carbon number RBCA 3 tiered look-up tables for tier 1 and increasing use of site specific information in tiers 2 and 3.	Equivalent carbon number Modified TPHCWG approach.	Equivalent carbon number Modified TPHCWG approach within UK context.	Equivalent carbon number Use of a 3-tiered approach, moving from generic guidelines to less conservative values using site-specific information.	Not defined None specified	Equivalent carbon number Use of a tiered approach, moving from generic to less conservative values using site-specific information in tiers 2 and 3.

Analysis	No recommended method of analysis	Use of two methods developed by MaDEP for volatile petroleum hydrocarbons (VPH) ⁵⁸ and Extractable petroleum hydrocarbons (EPH) ⁵⁷	The "Direct Method," developed by AEHS. ¹	The "Direct Method" ¹	Benchmarked methods for the C ₆ to C ₁₀ and C ₁₀ to C ₅₀ ranges. ¹⁹	Modified "Direct Method" for C ₄₄ + range.	No specified methods, however, are to adopt performance criteria— MCERTS ³²	Use of method prepared by the Oil Industry Environment Working Group. ⁶⁷	Dependent on source of threshold concentration. Using NEPC methods. ⁶⁴	Single analytical method (NEN 5733) recommended.
Additivity effects	Not recommended	Precautionary based on addition of hazard quotients across fractions	Precautionary based on addition of hazard quotients across fractions	Precautionary. Developing index of concern based on addition of hazard quotients across fractions for compounds affecting same target organs of systems	Not advised due to different toxicological endpoints and exposure pathways of different fractions	Precautionary based on addition of hazard quotients across fractions	Assumes additivity of toxicological effects across all fractions, unless there are scientific data to the contrary.	Additivity of excess lifetime cancer risk for non-threshold substances. Precautionary approach, as for ATSDR	Not discussed in guidance document	Precautionary approach, based on addition of hazard quotients across fractions
Range		nC5–nC ₃₆ Aliphatics, nC9–nC ₂₂ Aromatics	EC ₅ -EC ₂₁ Aliphatics, EC ₅ -EC ₃₅ Arom1atics	EC ₅ -EC ₂₁ Aliphatics, EC ₅ -EC ₃₅ Aromatics	EC ₆ -EC ₅₀	EC_6 to EC_{44+}	EC ₅ to EC ₇₀	EC ₇ to EC ₃₆	EC_6 to EC_{40}	EC5 to EC35