Title:	BIC Scale for Delineating Petroleum Hydrocarbons in Organic Soils and Compost
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Purpose

This Information Letter describes the Biogenic Interference Calculation (BIC) Scale and regulatory requirements for its use in Alberta. The BIC Scale is a mathematical tool for identifying false exceedances of the Alberta Tier 1 soil guidelines (AEP, 2016) for petroleum hydrocarbons (PHCs) due to the presence of natural biogenic organic compounds (BOCs). However, the BIC Scale does not quantify true PHC concentrations. Plants and animals biosynthesize BOCs (e.g. tissues, wastes, etc.) which are integral components of organic soils and compost. By definition, organic soils contain greater than 17% total organic carbon (TOC), with peat soils containing greater than 40% TOC. Compost typically ranges from 50% to 60% TOC.

This Information Letter provides guidance on analytical and reporting requirements when using the BIC Scale for closure at sites regulated by Alberta Environment and Parks or the Alberta Energy Regulator.

Scope

Detailed instructions are provided for applying PHC F2 (C10-C16) and sub-fraction PHC F3b (C22-C34) concentrations to the BIC Scale, in order to determine if organic samples have falsely exceeded the Alberta Tier 1 soil guidelines for PHC F3 (C16-C34).

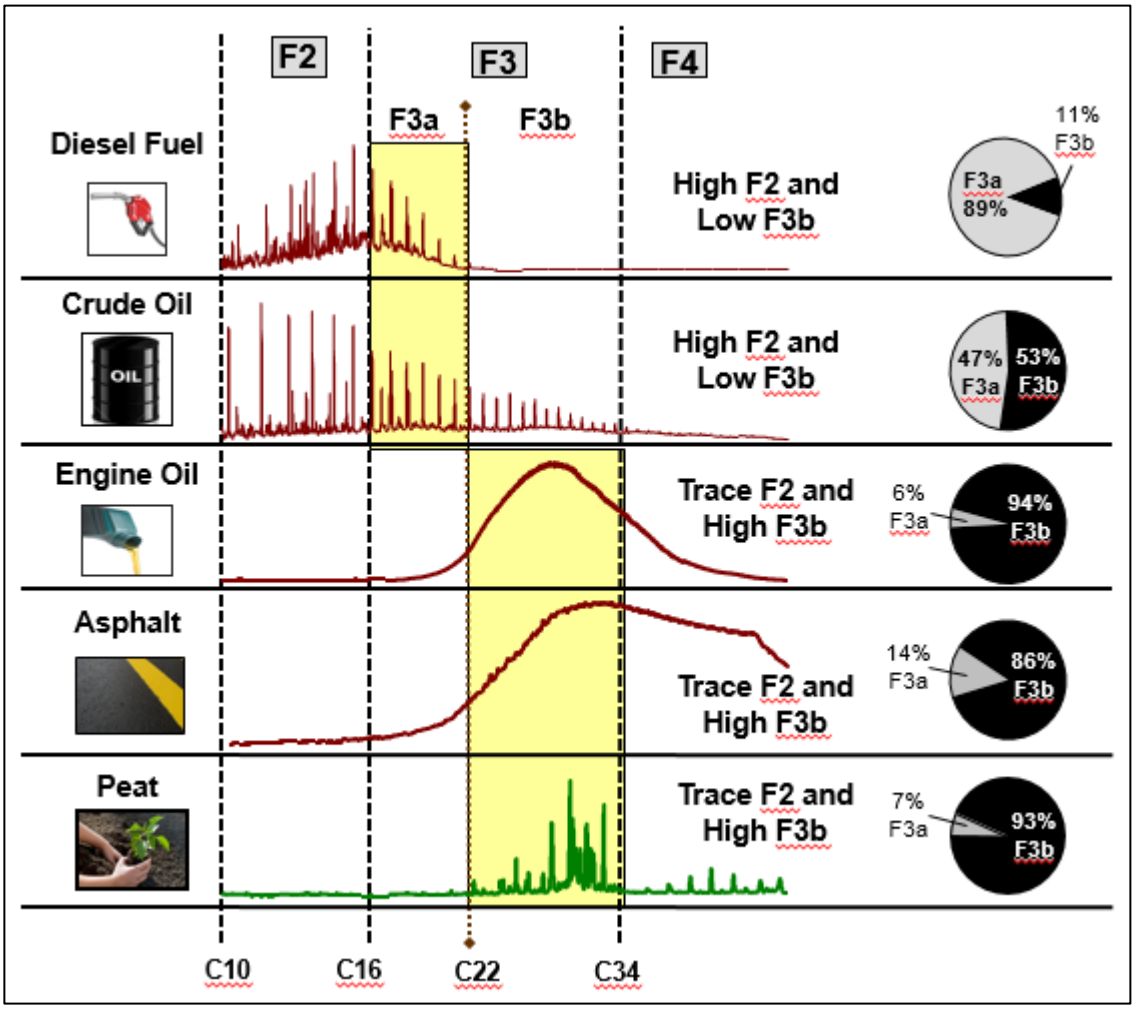
Introduction

The Canada-wide Standard (CWS) for PHCs in Soil (CCME, 2008) provides the protocols and primary technical basis for the Alberta Tier 1 PHC Soil and Groundwater Remediation Guidelines (AEP, 2016). These guidelines are established for the following four carbon ranges/fractions: F1 (C6–C10), F2 (C10–C16), F3 (C16–C34), and F4 (C34-C50). The reference method for the Canada-Wide Standard (CWS) for petroleum hydrocarbons (PHC) in soil provides laboratory methods for generating accurate and reproducible soil analysis results (CCME, 2001).

The CWS PHC analytical methods quantify PHC F1, F2, F3 and F4 concentrations for light to heavy PHC products, such as diesel, crude oil, bitumen, asphalt, motor oil, etc. However, BOCs can be falsely detected as PHCs in uncontaminated organic soils and compost materials. They can also cause slightly

contaminated soils to exceed the Tier 1 guidelines for F3 PHCs. Kelly-Hooper et al. (2013, 2014) found that almost all uncontaminated organic soils had PHC F2 concentrations of <30 mg/kg. In contrast these same soils had elevated PHC F3 concentrations (150 mg/kg to 1430 mg/kg), which frequently exceeded the 300 mg/kg PHC F3 Tier 1 coarse soil guideline[1]. The PHC F4 concentrations (150 mg/kg to 1580 mg/kg), did not exceed the Tier 1 guidelines. Most (>90%) of the F3 concentrations in the uncontaminated samples occurred at a carbon chain length ≥C22 (F3b). Figure 1 provides examples of Gas Chromatogram-Flame Ionization Flame Detector (GC-FID) chromatograms that show variable patterns and carbon ranges in clean peat and various PHC products.

Figure 1: GC-FID chromatograms and F2, F3, F4, F3a, F3b distributions for examples of clean peat and a range of light to heavy PHC products



Notes:

1. Asphalt and peat were analyzed as soil samples, which included insitu silica gel cleanups
2. Diesel, crude oil and motor were analyzed as direct injections without silica gel cleanups

[1] The Alberta Tier 1 guidelines (AEP 2016) specify the use of coarse texture remediation guidelines for organic contaminants in organic soil.

Based on these predictable trends, the BIC Scale provides a tool for identifying false exceedances of the Tier 1 PHC F3 guidelines caused by BOC interferences.

The Alberta Tier 1 Soil and Groundwater Remediation Guidelines (2016) recognizes background interference issues as follows:

> For the purpose of applying Alberta Tier 1 or Alberta Tier 2 Soil and Groundwater Remediation Guidelines, the background concentration of a substance in soil or groundwater is defined as:
>
> 1. The natural concentration of that substance in the absence of any input from anthropogenic activities or sources; or
> 2. The background concentration in the surrounding area as a result of generalized non-point anthropogenic sources.
>
> In some situations, the background concentrations of a substance can be a significant proportion of, or even exceed, the Alberta Tier 1 Guidelines. In cases when the background concentration is demonstrated to be greater than Alberta Tier 1 Guidelines, the remediation level shall be set to background or to guidelines developed using Tier 2 procedures. **Background concentrations will vary with soil parent material, soil depth, and hydrologic regime. These factors lead to spatial variations in background concentrations that may or may not be predictable.** To gain a good understanding of background conditions at a site, it is necessary to take sufficient representative samples from soils with similar characteristics to the affected site, but which are taken from outside the area affected by contamination. Sample depth and landscape position, soil profile characteristics and parent material should be recorded for all samples.

Weight-of-Evidence Methods for Identifying False PHC Exceedances

The BIC Scale is one of several options for identifying false PHC exceedances in organic soil and compost. Contaminated site managers must be familiar with the advantages and limitations of each method when choosing the appropriate one for their site.

i) **Silica Gel Cleanup** (CCME, 2001) – BOCs are generally more polar than PHCs. Silica gel is a polar substance that laboratories use to remove polar BOCs from PHC extracts. The CWS PHC (CCME, 2008) allows the use of a controlled amount of silica gel for the purpose of BOC removal. However, highly organic peat and compost can exceed silica gel saturation capacities, resulting in false exceedances of the AB Tier 1 F3 PHC soil guidelines. Relatively minor amounts of non-polar BOCs can contribute to false exceedances as well.

ii) **Subtraction of Background PHC Concentrations** (CCME, 2001) - Clean background soil concentrations can be subtracted from contaminated soil concentrations. Sufficient background samples are needed to adequately characterize background concentrations. However, this quantitative approach can generate highly variable concentrations due to the non-homogeneous consistency of peat.

iii) **Biomarker Forensics Analysis -** This semi-quantitative approach can identify the presence versus absence of PHCs, by highly specialized Gas Chromatography Mass Spectrometry (GC-MS) laboratory analysis methods. This approach can identify specific biogenic compounds that may cause false exceedances of PHC soil guidelines.

iv) **Biogenic Interference Calculation (BIC) Scale** –The BIC Scale is a semi-quantitative approach to determining if organic samples such as peat or compost have falsely exceeded the Tier 1 soil guidelines for PHC F3. This approach considers the standard PHC F2 and F3

carbon ranges, while dividing PHC F3 into two sub-fractions “PHC F3a” (C16-C22) and “PHC F3b” (C22-C34). The following sections of this Information Letter provide detailed information regarding the use of the BIC Scale for resolving false PHC detections and PHC F3 guideline exceedances in compost and organic soils such as peat.

BIC Scale Details

The BIC Scale provides a mathematical approach to identifying false PHC F3 detections and false exceedances of Tier 1 PHC F3 guideline in organic samples. The premise of the BIC Scale is that uncontaminated organic soils typically have four characteristics:

i) PHC F2 concentrations are less than 30 mg/kg and do not exceed the Tier 1 soil guidelines;
ii) PHC F3 concentrations are detectable and may exceed the Tier 1 soil guidelines;
iii) PHC F4 concentrations are detectable but do not exceed the Tier 1 soil guidelines; and
iv) Greater than 85% of the total PHC F3 range occurs within the F3b range.

The Biogenic Interference Calculation focuses on F2 and F3b concentrations and percentages, as shown in the following formula:

$$BIC = \frac{[PHC\ F2]}{[PHC\ F2] + [PHC\ F3b]} \times 100$$

Note:
When F2 concentrations are reported as less than the Laboratory Method Detection Limit (MDL), the PHC F2 concentrations must be calculated as half the MDL concentration. For example, 30 mg/kg MDL would be calculated as 15 mg/kg.

BIC values are compared to a threshold value of 10%, which was developed through empirical observations of PHC concentrations and carbon distributions in over 100 samples including:

- Clean compost (food compost, leaf compost and manure compost);
- Peat spiked with high and low concentrations of fresh and weathered crude oil;
- Manure compost spiked with high and low concentrations of diesel drilling waste;
- Peat samples collected from a 30-year old crude oil contaminated peatland; and
- Survey of soil samples collected from thirty-four background sites across Canada

Samples with BIC values of <10% indicate potentially false exceedances of the PHC F3 guideline, while samples with BIC values of $\geq$10% indicate potentially true PHC F3 exceedances. However, reaching correct conclusions requires skilled evaluations of chromatograms. It also critical that laboratory reports display the full patterns of each chromatogram. Figure 2 provides a decision tree and screening criteria for indicating if biogenic interferences have caused false exceedances of the 300 mg/kg PHC F3 coarse soil guideline.

Restrictions on Heavy PHCs

Figure 1 provides example GC-FID chromatograms, which illustrate carbon distribution patterns for clean peat and light to heavy PHC products. Since the BIC Scale relies on detectable PHC F2 concentrations to identify PHCs, it can only be applied to light PHC products with detectable PHC F2 concentrations (e.g. gasoline, condensate, diesel, crude oil, etc.). The BIC Scale cannot be applied to heavy PHC products that do not have detectable PHC F2 concentrations (e.g. asphalt, bitumen, motor oil, etc.). Figure 2 shows that clean peat and heavy PHCs share similar carbon ranges. For this reason, the BIC Scale would

wrongly indicate that a sample was clean even if it were contaminated by heavy PHCs. It is therefore essential for GC-FID chromatograms to be reviewed in conjunction with laboratory data to determine if high PHC F3b percentages are due to the presence of BOCs or heavy PHC products.

Analytical Requirements

Silica Gel Cleanup

Laboratories commonly use one of two silica gel cleanup (CCME, 2001) methods when measuring PHC F2 to F4 concentrations: in-situ and column. When analyzing samples for use with the BIC Scale, the in-situ method must be used. Column cleanup must not be used if the PHC data will be used to determine the BIC Scale. Repeated silica gel clean ups must not be used.

High Moisture Samples

Peat soils frequently have high moisture contents (typically ~ 85% by weight). As a result, a smaller equivalent dry weight is often obtained from peat than from mineral soils when routine laboratory methods are used. This can result in elevated MDLs of >30 mg/kg, which can cause the BIC Scale to misidentify clean samples as contaminated. When submitting high moisture samples for laboratory analysis and BIC Scale evaluation, it is recommended that the laboratory be requested to provide the lowest detection limit possible.

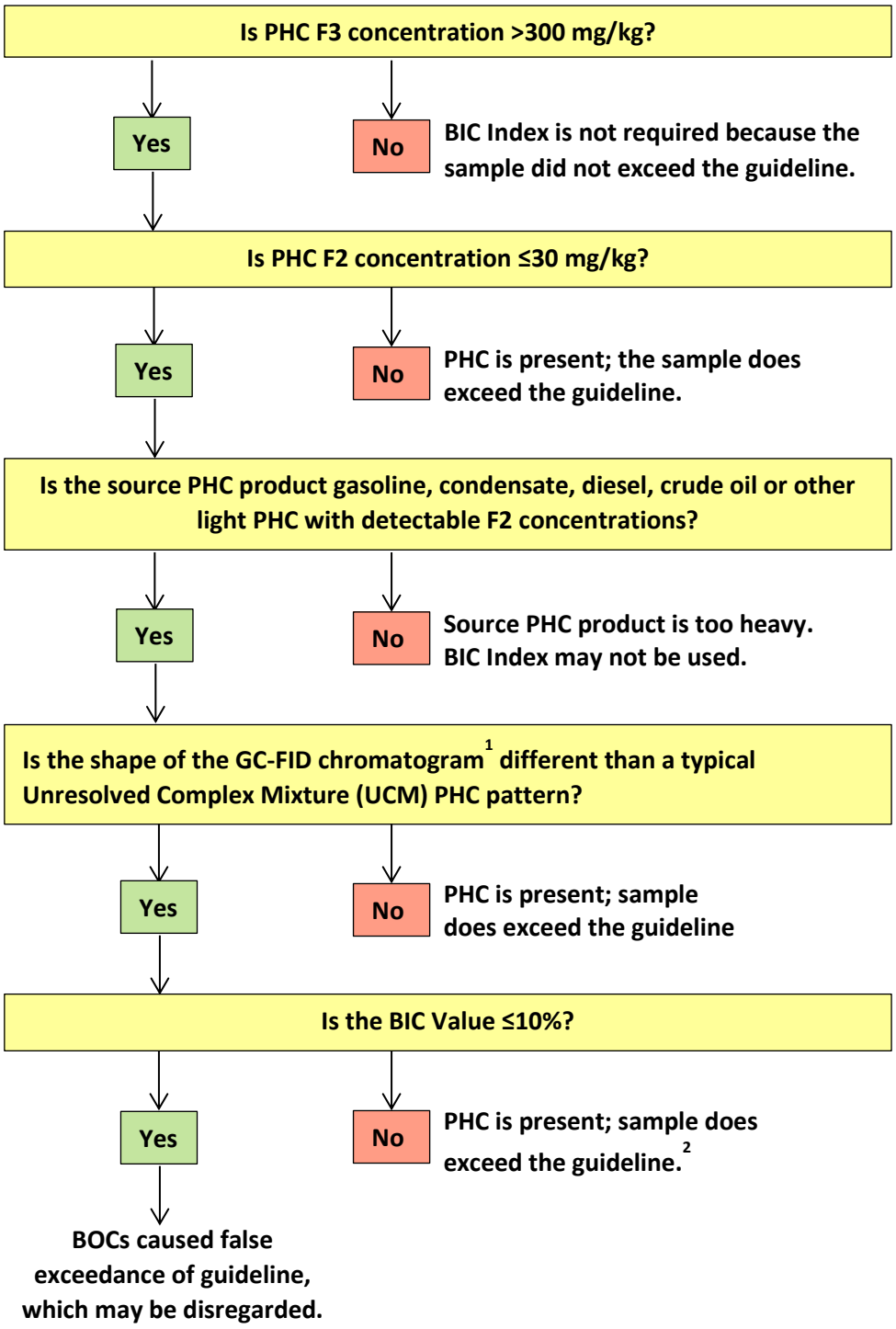
Using the BIC Scale

Reports submitted to Alberta Environment and Parks or the Alberta Energy Regulator must include the following information for all samples to which the BIC Scale has been applied:

i) Standard F2 (>C10-C16), F3 (>C16-C34) and F4 (>C34-C50) concentrations;
ii) Sub-fraction PHC F3a (>C16-C22) and PHC F3b (>C22-C34) concentrations; and
iii) GC-FID chromatograms for each sample.
iv) Total organic Carbon percentages (for large projects a minimum of 10 samples must be analyzed for TOC).
v) BIC Scale evaluation for each sample

The report must confirm that the material released was a light PHC product that extends into the PHC F2 range (e.g., gasoline, condensate, diesel, crude oil, etc.).

Figure 2: Decision Tree for Identifying False Exceedances of the Alberta Tier 1 F3 Guideline in Organic Soils and Compost



Notes:

[1]Chromatograms to be evaluated by a qualified person.

[2]Detailed GC-Mass Spectroscopy (GC-MS) analysis may be used to identify rare cases where unusual biogenic peaks in the F2 and/or F3b range cause clean sample to be misidentified as contaminated.

References

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Original signed by: _____ Date: March 28, 2018

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