NewFields

September 30, 2014

Mr. Tom Graan
Weston Solutions, Inc.
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Vernon Hills, IL 60061

RE: Technical Memorandum – Determination of Line 6B Oil Concentration in Kalamazoo River Sediments.

Dear Mr. Graan,

1.0 Introduction

Line 6B oil is a high viscosity oil sands based bitumen product that is diluted with a gas condensate solvent pipeline flow improver. As such, it exhibits unique physical and chemical properties when released to the river environment. These physical changes include globule and droplet formation in the water column as the lighter condensate evaporates after the release. Re-suspended sediment particles adsorb onto the oil and the oil sinks to the sediment surface. Activities such as oil recovery efforts, natural river turbulence, and recreational activities (e.g., boating) mix the oil laden surface sediments deeper in the sediment core diluting the original surface Line 6B chemical signal within a complex river sediment background hydrocarbon (BH)[1] signature. The ability to forensically identify and quantify the presence of the Line 6B oil in these sediments becomes more difficult as oil/sediment dilution increases.

Chemical analysis of Line 6B oil has identified a unique chemical feature which provides a means to distinguish it from the BH present in the Kalamazoo River sediment. Line 6B oil is enriched in a class of biomarker compounds called triaromatic steroids (TAS)[2] relative to sediment background. Ratios of these compounds to other stable yet less discriminating biomarker compounds (triterpanes) are called source/quantification ratios (QR) and are used to assist in the identification and quantification of the Line 6B oil in the sediment.[3] Of the many source/quantification ratios that have been evaluated, the TAS2[4]/Hopane[5,6] QR exhibits the highest stability, Line 6B resolving power, and the least false positive issues within Kalamazoo River spill zone sediments.

[1]Background hydrocarbons (BH) represent the hydrocarbons present in the sediment from sources such as coal tar, atmospheric deposition of combustion PAHs, road runoff, and leaks/losses from non-Line 6B oils.
[2]Peters, K. E., Walters, C. C., Moldowan, J.M. 2005. The Biomarker Guide, Volumes1&2. Biomarker and Isotopes in Petroleum Exploration and Earth History. **2005**, Cambridge University Press. Cambridge, UK.
[3]Douglas, G.S. and Hallebone, B.P. 2012. Forensic Identification And Quantification of Oil Sands-Based Diluted Bitumen Released Into a Complex River Environment – The Kalamazoo River Oil Spill. SETAC North America 33rd Annual Meeting.
[4] TAS2 = C28,20S-triaromatic steroid.
[5]Wang, Z, and Soutt, S. 2007. Oil Spill Environmental Forensics – Fingerprinting And Source Identification. Academic Press, Burlington, MA. 2007.
[6] Hopane (C_{30}17α21β(H)-hopane) is also referred to as T19 in this text.

NewFields has been requested by the United States Environmental Protection Agency (USEPA) to develop a scientifically sound protocol for the identification and quantification of Line 6B oil in the Kalamazoo River sediments. Multiple approaches were evaluated during the method development process, including reliance on TPH and gravimetric weight measurements, Line 6B calibrated quantification relative to a dominant Line 6B compound (e.g., C3-DBT), source double ratio mixing models,[7] and sediment calibration using representative river sediment Line 6B spiking studies (Range Finding Study, RFS).

The primary problem identified in these initial studies was the abundance and variability of BH concentrations in the sediments relative to the Line 6B oil chemical fingerprint signal. This problem was first observed in the Toxicity Study[8] sediment samples where Line 6B quantification estimates were either lower/higher than could be justified by the TPH, gravimetric and forensic chemistry data (e.g., sediment sample MP10.75).[9] To address this issue, sediments representing different background signatures were spiked with Line 6B oil at concentrations ranging from approximately 10 ppm Line 6B to 17,000 ppm Line 6B. These Line 6B calibration samples were then applied geographically from MP2-MP15.75 (SEKR0000R024S092112D004, R024), and from the Battle Creek convergence (MP16.5) to MP39.75 (SEKR3510R018S092112D004, MP35.1) respectively. The primary assumption for this approach was that a similar BH signature *and* concentration existed within each of these two sections of the Kalamazoo River.[10] The results for each Range Finding Study (RFS) were reduced to a mathematical equation using accepted curve fitting programs, and directly applied to the respective sediments.[9] These "*Reference*" sediments were selected because they contained moderate amounts of BH and but did not contain Line 6B oil. Quantified Line 6B values using these calibration mixtures produced highly variable and generally biased low Line 6B results relative to TPH, Gravimetric, and source ratio measurements.[9]

The Submerged Oil Quantification Study was designed to collect representative sediment samples both geographically and with sediment depth in the Line 6B spill zone. Sediment cores were collected and processed at selected depths and shipped to the laboratory for analysis. The RFS Line 6B calibration was applied to these samples with limited success. Application of the RFS calibrations to the Submerged Oil Quantification Study sediments exhibited a wide range of Line 6B sensitivity (spatially and vertically). Line 6B sensitivity is defined as the change in Line 6B oil concentration/change in quantification ratio (e.g., TAS2/Hopane). Sediments with low Line 6B sensitivity generally have high concentrations of BH or Line 6B oil. In these sediments, it may take substantially more Line 6B oil to even detect a change in the Quantification Ratio relative to a sediment sample with low BH (e.g., R024). *Only sediments with the same Line 6B sensitivity as the reference samples produce reliable Line 6B quantification results using the RFS Line 6B calibration method.*

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[7]Douglas, G.S., Stout, S.A., Uhler, A.D., McCarthy, K.J., Emsbo-Mattingly, S.D. 2007. Advantages of quantitative chemical fingerprinting in oil spill source identification. *In:* Oil Spill Environmental Forensics: Fingerprinting and Source Identification. Z. Wang and S.A. Stout, Eds. Elsevier Publishing Co., Boston, MA.
[8]G.M. DeGraeve. 2012. Final Report. *Chironomus dilutus* and *Hyalella Azteca*, 10-day Whole Sediment Toxicity Testing Results, Kalamazoo River Sediment Sampling Line 6B Oil Spill, Marshall, Michigan. Prepared for: Enbridge Energy. June 10, 2012.
[9] Technical Review Of Enbridge Report "Supplement To The Response Plan for Downstream Impacted Areas Commonly Referred As The "Quantification of Submerged Oil Report" NewFields May 7, 2013.
[10] River sediments down-stream from the Battle Creek convergence would contain Kalamazoo River BH and Battle Creek BH.

Figure 1 is an illustration of the change in QR versus multiples of sediment BH concentrations for a given concentration of Line 6B oil (100 mg/Kg, 200 mg/Kg, 500 mg/Kg and 1,000 mg/Kg). Assuming that the identified Critical Value (CV) reflects the background signature,[11] and given that background hydrocarbon concentrations vary in the spill zone, this illustration explains why the single RFS-type calibration method failed. The Line 6B QR is not only dependent on the amount of line 6B in the sample, but the concentration of BH (e.g., TAS2 and Hopane)[12] as well.

Source Ratio Binary Mixing Model Theory

This problem was resolved with the development of a two end member mixing model for each sediment sample. Using this approach, the *calculation of Line 6B oil includes the impact of sample specific BH within each sample on the behavior of the quantification ratio (QR).* This sediment sample specific approach (discussed below) is based on the accurate measurements[13] of quantification ratio compounds (e.g., TAS2, Hopane, TAS1, T30) in each sediment sample and the spilled Line 6B oil (e.g., CL-6B-072223-092710-JPS-KA-001-33_TOPPED, Topped Line 6B Oil).[14] Line 6B oil is added or removed mathematically from the field sample and plotted versus the QR. The Critical Value (CV) is defined by the QR of reference samples R024 and MP35.1 above which Line 6B is detected and below which it is not (Figure 2).

Mixing of two or more components or end-members is of fundamental importance in several branches of geosciences,[15] and the mathematics of this process are well understood.[16,17] The mixing process is fundamentally controlled by the conservation of mass. The two end mixing model for a single constituent (e.g., TAS2) is given by a simple algebraic expression based on the fraction (or concentration) of each end-member in a given mixture of Line 6B and BH in sediment.

$$C_{mix} = (C_1 * f_1) + (C_2 * f_2) \qquad (1)$$

Where: C_{mix} = contaminant concentration in the mixture
C_1 = contaminant concentration in end-member 1 (e.g., BH)
C_2 = contaminant concentration in end-member 2 (e.g., Line 6B)
f_1 = fraction (concentration) of end-member 1 in the mixture
f_2 = fraction (concentration) of end-member 2 in the mixture
And $f_1 + f_2 = 1$

[11] The Critical Value (CV) is defined by the QR of reference samples R024 and MP35.1 above which Line 6B is detected and below which it is not.
[12] The y-axis represents increasing concentrations of R024 background in the sediment and the associated increases in TAS2 and Hopane at constant CV (0.34).
[13] As defined by the Enbridge Kalamazoo River Analytical Quality Assurance Plan V2.2.
[14] Topping is a process by which the volatile component of the fresh oil is removed by heating in the laboratory to more accurately reflect the chemical composition of the weathered oil present in the sediment and sheen samples. Approximately 21% of the fresh Line 6B oil was removed by topping.
[15] Vollmer, R. 1976. Rb-Sr and U-Th-Pb systematics of alkaline rocks: the alkaline rocks from Italy. Geochim. Cosmochim. Acta **40**, 283-29l5.
[16] Langmuir, C.H., Vocke, R.D., Hanson, G.H., Hart, S. 1978. A general mixing equation with applications to Icelandic basalts. Earth and Planetary Science Letters, 37, 380-392.
[17] Faure, G. 1986. Principles of Isotope Geochemistry. John Wiley & Sons Inc.

Equation (1) describes a simple linear relationship between the two end-members such that the values for C_{mix} fall on a straight line when plotting concentration against the fraction of one of the end-members in the mixture.

Within the Kalamazoo River sediment spill zone, the two measured variables are 1) the concentration of the contaminant in end-member 2 (Line 6B), and 2) the concentration of the contaminants in the sediment mixture C_{mix}. Given that the fraction (concentration) of the end-member 1 (BH) varies in the sediment, there is insufficient data to calculate the fraction (concentration) of Line 6B oil in the sediment.

To account for variation in the background hydrocarbon concentration, a two end-member mixing model was developed for the quantification of Line 6B oil in the spill zone sediment using a representative and stable diagnostic biomarker ratio (TAS2/Hopane). This approach has been used by others to de-convolute oil mixtures in environmental samples.[18] Although the absolute concentrations of TAS2 and Hopane in non-impacted or background sediments within the spill zone are variable, the ratio of TAS2/Hopane is both environmentally stable and diagnostic of Line 6B inputs. TAS2/Hopane ratios of 0.34 and 0.41 are considered representative (as discussed below) of the upper and lower Kalamazoo sediment BH signatures respectively. A new binary mixing model based on the conservation of mass and the stable BH TAS2/Hopane CV was then developed.

$$CV = (A - (D * C_{L6B})) / (B - (E * C_{L6B})) \qquad (2)$$

Where: A = measured concentration of TAS2 in the sediment sample
B = measured concentration of Hopane in the sediment sample
D = measured concentration of TAS2 in the topped Line 6B oil
E = measured concentration of Hopane in the topped line 6B oil

Solving for C_{L6B}:

$$C_{L6b} = ((B * CV) - A) / ((E * CV) - D) \qquad (3)$$

The curvature of these mixing model plots (Figure 2) provided a measure of sediment sample-specific Line 6B Detectability (LOD) and a means to evaluate how much Line 6B oil is required to achieve the sample specific QR relative to the reference sample CV. This approach is more accurate than the reference sample curve fitting estimates because it incorporates the impact of differential Line 6B sensitivities within the oil quantification result. The validity of this approach has been verified in the Line 6B spiked RFS and Method Detection Limit (MDL) sediments, where known amounts of Line 6B oil were added to reference sediments R024, MP35.1 and Battle Creek (BC).[19]

[18] Douglas, G.S., Stout, S.A., Uhler, A.D., McCarthy, K.J., Emsbo-Mattingly, S.D. 2006. Advantages of quantitative chemical fingerprinting in oil spill source identification. *In:* Oil Spill Environmental Forensics: Fingerprinting and Source Identification. Z. Wang and S.A. Stout, Eds. Elsevier Publishing Co., Boston, MA.
[19] Battle Creek RFS and MDL results are included in this report to document the utility of the mixing model method in different field sediment matrices.

This Technical Memorandum describes the USEPA Line 6B methodology for the quantification of Line 6B oil within the Kalamazoo River Line 6B oil spill zone.

2.0 Analytical Methods

Range Finding and Quantification Study sediment samples were extracted and analyzed according to Enbridge Kalamazoo River Analytical Quality Assurance Plan V2.2 by Alpha Analytical located in Mansfield, Massachusetts. Sediment samples were dried with sodium sulfate, spiked with surrogate compounds, serially extracted with methylene chloride, concentrated to 1 mL and analyzed for extract gravimetric residue weight. A portion of the extract was treated with activated copper and processed through a silica gel column to remove polar interferences. The sample extract was then analyzed for alkanes, Total Petroleum Hydrocarbons (TPH), and Total Resolved Hydrocarbons (TRH) by gas chromatography with flame ionization detection (GC-FID). A second aliquot of the extract was analyzed by gas chromatography with mass spectrometer detector (GC-MS) for polycyclic aromatic hydrocarbons (e.g., phenanthrenes), sulfur heterocyclics (e.g., dibenzothiophenes) and their associated alkylated homologs (e.g., C3-dibenzothiophenes). Triterpane, sterane and triaromatic sterane biomarker compounds are also analyzed and reported during this procedure.

Due to mass discrimination[20] variability in the TAS1, TAS2, TAS3, and TAS4 analysis (TAS), all samples were additionally calibrated with the Line 6B control oil analyzed with each analytical batch relative to the Line 6B control oil rolling average. The analysis of a Cold Lake Control Oil (Cold Lake) with each analytical batch allows for the data user to correct for over or under response of TAS compounds. The TAS correction factor was calculated as follows:

1. Average TAS concentrations were calculated using Line 6B oil analyzed over a 1 year period (n=42)
2. The TAS concentration in the Line 6B oil analyzed with each batch of field samples was divided by the average TAS reference oil concentration calculated in Step 1.
3. The TAS concentrations in the field samples are corrected by dividing the field sample concentration by the correction factor.

Although Hopane and T30 were affected to lesser degree by instrument mass discrimination, these were also calibrated and corrected using same procedure illustrated above.

A multi-tiered interpretive approach was used to identify the presence or absence of Line 6B oil in the Quantification Study sediment samples. These included the following interpretive analyses:

1. Comparison of the Line 6B oiled sediment (e.g., MP10.75) GC/FID hydrocarbon signatures to Line 6B oil (Figure 3).
2. Comparison of reference and oiled sediment PAH distributions to Line 6B oil (Figures 4 and 6).
3. Analysis of bar plot and extracted ion plot outputs of reference and oiled sediment Triterpane, Sterane, and Tri-Aromatic Sterane compound distributions to Line 6B oil (Figures 5 and 6).

[20]Douglas, G.S., Emsbo-Mattingly, S.D., Stout, S.A., Uhler, A.D., McCarthy, K.J. 2007. Chemical fingerprinting methods. *In:* B. Murphy and R. Morrison, Eds., Introduction to Environmental Forensics 2nd Edition. Elsevier Academic Press, Burlington, MA.

4. Comparison of field sample QR relative to CV with subsequent quantification of Line 6B inputs (Figure 2).

3.0 Line 6B Quantification Method

The analytical methods used for Line 6B oil spill provide reliable measurements of key diagnostic hydrocarbons from which interpretive methods were developed to quantify Line 6B oil in the river sediment: The method used for Line 6B quantification in Kalamazoo River sediment samples is as follows:

3.1 Define Site Specific Line 6B Quantification Ratios.

Line 6B oil chemical fingerprint is unique when compared to sediment background because it contains elevated triaromatic steroids (TAS1, TAS2) relative to the triterpanes (Hopane, T30, Figure 5). Based on extensive analysis and testing, the TAS2/Hopane biomarker source/quantification ratio was selected as the primary Line 6B quantification ratio because it provided adequate source specificity, was present in the spilled oil in proportions distinct from site background BH[21], and exhibited minimal matrix interferences.[22] Other ratios were calculated and used for interpretive purposes such as TAS1/Hopane and TAS1/T30.

3.2 Define the Mixing Model Critical Values.

The critical value (CV) is defined as the TAS2/Hopane ratio that best describes the Kalamazoo reference sediment BH. Spill zone samples with TAS2/Hopane values greater than the defined critical value contain Line 6B oil. Samples with TAS2/Hopane values less than the CV are reported as not detected (ND). These sub CV values most likely reflect relatively high experimental error associated with the lower BH concentrations, or the predominance of a single oil component in the background mixture that was not identified in the RFS studies. This may explain the dominant non Line 6B oil signatures observed in the deeper sediments within Morrow Lake.

The CV was first determined for specific reference R024 (upper Kalamazoo River) and MP35.1 (Lower Kalamazoo River) sediment samples (USEPA, Table 1). A second approach (Enbridge) was based on the average of multiple reference sediment samples collected above the Talmadge Creek convergence (upper Kalamazoo River) and the reference samples from the Battle Creek river (lower Kalamazoo River) (Table 1). A third approach (USEPA) was evaluated for reference Site R024 where sediment core samples were also collected. The TAS2/Hopane CV results for all three approaches were similar.

These R024 core sediment samples documented low CV variability (RSD = 14%) to a depth of 1.3 feet (SEKR0000R024AS121312D013) which includes the depth range of most of the sediment quantification study samples. TAS1/T30 CV variance for the same samples was double the TAS2/Hopane CV variance and was one factor considered for not using this ratio as a primary quantification ratio.[23] The single sample CVs were adopted for this method given they were slightly

[21] Specifically the proportion of TAS compounds to Triterpanes (e.g., Hopane).

[22] Reference sediment core R024 TAS2/Hopane variability is low and the historical BH signature does not generate false positive results for Line 6B oil in Morrow Lake.

[23] The TAS1/T30 ratio is used for data interpretation purposes only, as discussed later in this report, this ratio is prone to false positive Line 6B results in the Lower Kalamazoo River spill zone.

higher than the average Enbridge values and were considered conservative (i.e., they would generate slightly lower Line 6B results).

3.3 Calculate Line 6B Concentration Within Each Sediment Sample

The basic premise of the Line 6B mixing model method is that the background reference values are reasonably well defined. Hydrocarbons are most often associated with fine grain particles in riverine systems, therefore absolute concentration data for pyrogenic and petrogenic background will be variable due to sedimentation processes.

To solve this problem, TAS2/Hopane source ratios are used because they retain their characteristic ratio independent of sedimentation. The concentrations of TAS2 and Hopane are quantitatively measured in each sample and incremental amounts of topped Line 6B oil are mathematically added or subtracted from the sediment sample and new TAS2/Hopane ratio is calculated. These new ratios are then plotted versus Line 6B oil added/subtracted and the amount of oil required to reduce the QR to the CV is determined (Figure 2). This concentration represents the amount of Line 6B present in the field sample.

The curvature[24] of these mixing model plots are indicative of the amount of background hydrocarbons in the sample. For example a flattened curvature means that very little Line 6B oil is required to increase or decrease the source ratio. This would be typical of clean sand/gravel sediment (e.g., SEKR1950C501S042612DX)[25] A steep curvature is indicative of a sediment sample with substantial background hydrocarbons or that the sample is heavily oiled. These curvatures vary from sample to sample, and is the reason that direct calibration methods using a single sediment type (e.g., Range Finding Study R024) do not produce reliable results (Figure 1).[25] Given that the TAS2 and Hopane concentrations are carefully measured in the sediment and Line 6B source oil samples according to the Enbridge Kalamazoo River Analytical Quality Assurance Plan V2.2, the amount of oil present in the sediment sample is defensibly quantified. As noted above, the major assumption for this analysis is that the selected CV for the Kalamazoo River Sediments is representative of actual conditions (Table 1).

The mass-balance equation used to calculate Line 6B oil concentration for TAS2/Hopane values > CV is as follows:

For Positive detections where QR > CV:

$$CV = (A-D*C_{L6B})/(B-E*C_{L6B})$$

$$C_{L6B} = (B*CV-A)/(E*CV-D)$$ **where:**

C_{L6B} = Line 6B concentration (mg/Kg sediment),
A = Sample TAS2 concentration (µg/Kg)
B = Sample Hopane concentration (µg/Kg)

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[24] Or slope.
[25] Technical Review Of Enbridge Report "Supplement To The Response Plan for Downstream Impacted Areas Commonly Referred As The "Quantification of Submerged Oil Report" NewFields May 7, 2013.

CV = Critical Value of TAS2/Hopane ratio,
D = TAS2 concentration in topped L6B oil (g/Kg) [26]
E = Hopane concentration in topped L6B oil (g/Kg)

For sediment samples where TAS2/Hopane < CV, calculate and report the sample specific quantification limit (SQL).[27]

3.4 Method Validation
To validate the method performance, three sediments representing different background types[28] were spiked with topped[29] Line 6B oil from 10 mg/kg to approximately 17,000 mg/kg. The results for each study are provided in Table 2. The results indicate good agreement between the spiked and calculated Line 6B sediment concentrations. The R024 RFS study spiked concentrations versus measured TAS2/Hopane and the associated mixing model results are provided in Table 2 and is presented graphically in Figure 2.

3.5 Data Reasonableness
Reported sediment Line 6B data are also evaluated for reasonableness as follows.

1. Comparison to Line 6B GC/FID chromatograms to Line 6B oil.
2. Comparison of PAH distributions to Line 6B oil.
    a. Petrogenic versus Pyrogenic PAH distributions.
    b. High pyrogenic background interferes with 2-4 ring alkylated PAH Line 6B oil patterns.
    c. High pyrogenic background also interferes with secondary source ratios[30] such as C2-dibenzothiophene/C2-phenanthrene and C3-dibenzothiophene/C3-phenanthrene ratios. Caution is recommended when using these ratios as quantification ratios if pyrogenic signature dominates the oil PAH distribution. The reason is that the pyrogenic inputs have high phenenthrenes and low dibenzothiophenes and will bias the oil related D2/P2 and D3/P3 ratios low, resulting in an underestimate of Line 6B oil in the sample.
3. Comparison to Line 6B TPH = TPH/fraction of Line 6B detected by GC/FID (approximately 0.43).
4. Comparison of maximum Line 6B based on concentrations of C3-dibenzothiophenes, C4-dibenzothiophenes, C3-naphthobenzothiophenes, and C4-naphtobenzothiophenes.
5. Biomarker patterns – Comparison of triterpanes and aromatic steranes relative to Line 6B oil.
    a. 18a-22,29,30-Trisnorneohopane-TS/17a(H)-22,29,30-Trisnorhopane-TM
    b. TAS1/T30

[26] Substitution of the Topped Line 6B source oil representative globule samples (mean of globule samples SEKR0550C603S120612PX, SEKR0550C611S120612PX, SEKR0900I3004D041912P005, SEKR1950C501S042612PX, SEKR3455C501S042612PX) provided negligible Line 6B quantification differences.
[27] SQL is the sediment MDL value based on the R024 MDL study, adjusted for differences in sample weight, percent water, dilutions and pre-injection volume.
[28] Upper Kalamazoo = R024, Lower Kalamazoo = MP35.1, and SEBC = Battle Creek River.
[29] Topped refers to the removal of light end hydrocarbons by evaporation to generate a Line 6B oil chemically more similar to that which was deposited in the sediment after the oil spill.
[30] Douglas, G.S., Bence, A.E., Prince, R.C., McMillen, S.J. and Butler, E.L. 1996. Environmental stability of selected petroleum hydrocarbon source and weathering ratios. *Environ. Sci. Technology*, 30(7):2332-2339.

c. TAS1/Hopane
d. T30/T30+T31 = 0.6 at equilibrium
e. Distribution of TAS compounds

6. Note: At low Line 6B sediment concentrations (<100 mg/kg), the CV may be the primary factor used to report Line 6B. In these cases, the sample is qualified based on professional judgment including analysis of the 191 (triterpane) and 231 (triaromatic sterane) extracted ion plots (EICPs). Samples with QR values greater than Line 6B were evaluated and identified as not detected (qualified with a U) due to the trace concentrations present and incomplete patterns observed in the biomarker signatures.

3.6 Data Reporting

For each sample a sample quantification limit (SQL) is calculated. Although three separate MDL studies were performed,[31] the R024 MDL was considered as the primary MDL study because it had the lowest level of background hydrocarbon contamination and more precisely reflected the sediment matrix rather than the BH. MDL studies are generally performed on clean sediment matrices and use of the MP35.1 and BC MDL studies would likely overestimate the MDL and SQLs for the respective samples.

The quantification and identification of Line 6B oil in Kalamazoo Sediments is a complex analytical process. Equally complex is the determination of method detection limits of the analyses when Line 6B is not detected in the sediment (e.g., QR < CV). However, given that the BH within the spill zone sediments can vary from as low as 30 ppm to as high as 23,000 ppm, the reported MDL is not the single reference sample MDL but the sample quantification limit (SQL) adjusted to reflect differences in sample weight, percent solids, splits, dilutions and final extract volumes.[32] These SQL values are reported by NewFields for each spill zone sediment sample result in Table 3 according to the following criteria:

1. The sample specific Line 6B MDL (SQL) was calculated for each spill zone sediment sample by applying the total µg of Line 6B based on TAS2/Hopane from the R024 MDL Study to the sample specific conditions;

2. SQL = Total µg x Final Volume / Sample Dry Weight

3. Example Calculation: SEKR0325C701S072512DX (1209019-18) **SQL = 200µg x 2 / 5.77g = 69 mg/kg Line 6B SQL;**

The following data qualifiers are used to report the final Line 6B concentration results.

U = The analyte was analyzed for, but was not detected above the level of the reported sample quantification limit. This qualifier was also used when sample concentrations and biomarker

[31] R024 MDL, MP35.1 MDL and SEBC MDL.

[32] Guidance for Data Usability in Risk Assessment (Part A): 3.2.4 Detection and Quantification Limits and Range of Linearity (USEPA, 1990).

signatures indicated that one or both of the target analyte results (e.g., TAS2, Hopane) were not reliable.

J = The result is an estimated quantity and is applied when TAS2 and/or Hopane, and/or Line 6B oil concentration is present at < the associated SQLs. The associated numerical value is the approximate concentration of the analyte in this sample.

I = Inconsistent with Line 6B oil.

UJ = The analyte was analyzed for, but was not detected above the level of the reported sample quantification limit or one of the values is unreliable based on professional judgment. The associated numerical value is an approximation of the SQL.

These qualifiers are applied to individual samples as follows:

1. No Qualifier Required
   a. TAS2/T19 > CV
   b. And $TAS2/T19_{L6B}$ > SQL
   c. And TAS1/T19 L6B result > CV
   d. And TAS2 > SQL
   e. And T19 > SQL
   f. And Forensic Reasonableness (Max L6B, TAS1/T19, biomarker EICPs results are reasonable)
2. J Qualified
   a. $TAS2/T19_{L6B}$ < SQL
   b. Or TAS2 < SQL
   c. Or T19 < SQL
   d. And $TAS1/T19_{L6B}$ > CV
3. U Qualified at the TAS2/T19 SQL
   a. $TAS2/T19_{L6B}$ < CV
   b. Or $TAS2/T19_{L6B}$ > CV
      i. And $TAS2/T19_{L6B}$ < SQL
      ii. And TAS1/T19 < CV
   c. Or forensic analysis of the biomarker bar plots and EICPs indicate that the TAS2 and Hopane measurements are unreliable.
4. I Qualified. Forensic Analysis Indicates It Is Not L6B Oil.
   a. TAS2/T19 > CV
   b. And Forensic Analysis ≠ L6B
      i. GC/FID chromatogram
      ii. TS/TM
      iii. Triaromatic Sterane and Triterpane Patterns

3.7 Method Improvements

The analytical and interpretive methods developed for the quantification of Line 6B in Kalamazoo River sediments have evolved over time as new information and new samples (e.g., sediment core samples) were evaluated. The two important method modifications were; 1) Elimination of TAS1/T30 as a quantification ratio, and 2) Replacement of the Limit of Detectability (LOD) with sample quantification limits (SQLs). Both of these method modifications, improved the accuracy of the Line 6B quantification analysis.

3.7.1 Elimination of the TAS1/T30 Quantification Ratio.

The initial Line 6B quantification method employed the use of two different quantification ratios TAS2/Hopane and TAS1/T30. The Line 6B results derived from these two QRs were averaged and reported. This approach worked reasonably well for surface sediments and sediments with high concentrations of Line 6B. However, as more sediment cores were evaluated, it was learned that the BH in these deeper Lower Kalamazoo River sediments generated substantial false positive Line 6B concentrations when the TAS1/T30 QR was applied. Further analysis of these deeper sediment samples identified the presence of a BH heavy oil with depleted T30 triterpane relative to hopane and Line 6B oil (Figure 6). As a result, the relatively depleted T30[33] drove the TAS1/T30 above its CV resulting in a quantified Line 6B result.[34] The presence of reportable amounts of Line 6B in these sediments is contrary to the biomarker signatures in the samples and the TAS2/Hopane Line 6B quantification results. The TAS2/Hopane QR was robust enough to accurately report the absence of Line 6B oil in these samples (e.g., deep morrow lake sediment samples). As a result, in this report the TAS1/T30 QR was only used as a secondary Line 6B indicator for the evaluation of data reasonableness.

3.7.2 Replacement of Limits of Detectability (LOD) with Sample Quantification Limits (SQL).

BH varies significantly among different sediment sample locations within the Kalamazoo River spill zone. To address the impact of the BH on the Line 6B detection limit, NewFields previously proposed a novel approach referred to as the "Limit of Detectability"[35,36] (LOD) to define varying non-detect levels. Further analysis of the data indicated that this approach would overestimate the detection limits within a given sediment sample.

For the current data set, a more accurate approach using USEPA –approved methodology that adjusts the MDL to a sample quantification limit (SQL)[37] is used. This SQL is based upon sample-specific details of the laboratory extraction, including mass, pre-injection volume, and percent solid differences between the MDL samples and the field samples and accounts for varying levels of BH in the sediment samples to generate a more defensible sample specific quantification limit.

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[33] Relative to TAS1

[34] The CV exceedance was not due to an increase in triaromatic steroids in the sample (e.g., from Line 6B oil) but from a relative depletion of the T30 from a background oil source.

[35] Defined as the amount of Line 6B that must be added to a sediment sample to exceed the Line 6B critical value (CV).

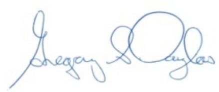
[36] NewFields Technical Memorandum – Determination of Line 6B Oil Concentration in Kalamazoo River Sediments. March 1, 2014.

[37] Guidance for Data Usability in Risk Assessment (Part A): 3.2.4 Detection and Quantification Limits and Range of Linearity (USEPA, 1990).

4.0 Results and Discussion
Final results for the Line 6B Sediment Quantification Study, and associated MDL and Range Finding Studies are provided in Table 3.

The conclusions in this report are based on currently available data. Should additional data or information become available to me, or if the analytical data is modified as a result of the on-going quality assurance reviews, I reserve the right to update this report as needed.
Please let me know if you have any additional questions concerning the identification and quantification of Line 6B oil in Kalamazoo River sediment samples.

Sincerely,



Gregory S. Douglas, Ph.D.
Sr. Consultant.

Table 1. Reference critical values used for sediment mixing model Line 6B calculations.

River Zone To Be Applied	Single Sample Method	Multiple Sample Method[38]
Upper Kalamzaoo River MP2 to MP15.75	Sample R024 CV = 0.34[39]	Average CV = 0.29[40]
Lower Kalamazoo River MP16.5 to MP 39.75	Sample MP35.1 CV = 0.41[41]	Average CV = 0.38[42]
Upper Kalamazoo River MP2 to MP15.75	Sample R024 CV = 0.34[43]	R024 Site Sediment Core Average CV = 0.33±.05 (SD)

Table 2. Spiked and calculated concentrations of Line 6B oil in Kalamazoo Sediment Range Finding Study R024, MP35.1 and SEBC (Battle Creek River sediment).

Field Sample ID	Lab ID	Spiked L6B	L6B Calc	RPD
		mg/kg	mg/kg	Percent
SEKR0000R024S092112D004	1210002-01	0	0	0
SEKR0000R024S092112D004_RFS1	1210002-02X	14	11.1	23
SEKR0000R024S092112D004_RFS2	1210002-03	131	134	2
SEKR0000R024S092112D004_RFS3	1210002-04	268	248	8
SEKR0000R024S092112D004_RFS4	1210002-05	677	681	1
SEKR0000R024S092112D004_RFS5	1210002-06	1328	1260	5
SEKR0000R024S092112D004_RFS6	1210002-07	13394	12600	6
SEBC0000L012S092112D004	1210003-01	0	0	0
SEBC0000L012S092112D004_RFS1	1210003-02	16	18	12
SEBC0000L012S092112D004_RFS2	1210003-03	159	129	21
SEBC0000L012S092112D004_RFS3	1210003-04	318	400	23
SEBC0000L012S092112D004_RFS4	1210003-05	795	835	5
SEBC0000L012S092112D004_RFS5	1210003-06	1569	1603	2
SEBC0000L012S092112D004_RFS6	1210003-07	15349	15963	4
SEKR3510R018S092112D004	1210004-01	0	0	0
SEKR3510R018S092112D004_RFS1	1210004-02	17	76	127
SEKR3510R018S092112D004_RFS2	1210004-03	167	238	35
SEKR3510R018S092112D004_RFS3	1210004-04	345	438	24
SEKR3510R018S092112D004_RFS4	1210004-05	843	1056	22
SEKR3510R018S092112D004_RFS5	1210004-06	1654	2054	22
SEKR3510R018S092112D004_RFS6	1210004-07	17094	17011	0

[38] Enbridge_Mixing_Model_Example_Working Draft_5_29_2014
[39] SEKR0000R024S092112D004
[40] SEKR0000C019S022312D004,SEKR0000C023S022212D004, SEKR0000L020S022312D004, SEKR0000L021S022312D004,SEKR0000R022D022212D004, SEKR0000R022S022212D004, SEKR0000R024S022012D004, SEKR0000R024S092112D004, SEKR0000R024AS121312D006, SEKR0000R024AD121312D010, SEKR0000R024AS121312D010, SEKR0000R024AS121312D013.
[41] SEKR3510R018S092112D004
[42] SE1111C703S120712DX, SE1111C704S121012DX, SE2222C702S120712DX, SE2222C702D120712D006, SE2222C702S120712D006, SE2222C702S120712D011, SE2222C702S120712D015, SE2222C702S120712D018, SE3333C701S120712DX, SE3333C701D120712D006, SE3333C701S120712D006, SEBC0000L010S022412D004, SEBC0000L012S022412D004, SEBC0000L012S092112D004, SEBC0000R011S022412D004.
[43] SEKR0000R024S092112D004

Table 3. Line 6B Oil Concentration and SQL Based on R024.

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	3.25	SEKR0325C701S072512DX	1209019-18	Sediment		69.4	U
Oil Quant	3.25	SEKR0325C701S072512D002	1211038-07	Sediment		49.7	U
	3.35	SEKR0335R001S022112D004	1202070-07	Sediment	65.3	60.7	
Oil Quant	4	SEKR0400C701S072512DX	1209019-19	Sediment		59.2	U
Oil Quant	4	SEKR0400C701S072512D003	1211038-08	Sediment	399	49.6	
	4	SEKR0400C701S072512D008	1406040-01	Sediment	22.4	36.3	UJ
Oil Quant	4.25	SEKR0425C701S072512DX	1208009-14	Sediment	681	230	
Oil Quant	4.25	SEKR0425C701S072512D007	1208009-15	Sediment	309	106	
Oil Quant	4.25	SEKR0425C702S112812DX	1212031-11	Sediment		52.3	U
Oil Quant	4.25	SEKR0425C702D112812D005	1212031-13	Sediment		56.7	U
Oil Quant	4.25	SEKR0425C702S112812D005	1212031-12	Sediment		54.6	U
Oil Quant	4.25	SEKR0425C702S112812D009	1212031-14	Sediment		33.5	U
Oil Quant	4.25	SEKR0425C702S112812D019	1212052-03	Sediment		67.7	U
	4.25	SEKR0425C701S072512D011	1406040-02	Sediment	129	67.3	U
	4.25	SEKR0425C701S072512D016	1406040-03	Sediment	41	58.7	UJ
	4.25	SEKR0425C701S072512D020	1406040-04	Sediment	27.5	43.3	UJ
	4.25	SEKR0425C701S072512D020	1406040-04D	Sediment	30.1	43.1	UJ
	4.25	SEKR0425C701S072512D022	1406040-05	Sediment	41.4	38	J
Oil Quant	4.75	SEKR0475C701S072612DX	1209020-06R	Sediment		102	UJ
Oil Quant	4.75	SEKR0475C701S072612D004	1211039-03	Sediment	18.5	54.5	UJ
Oil Quant	4.75	SEKR0475C701S072612D008	1211039-04	Sediment	22.4	59.6	UJ
Oil Quant	4.75	SEKR0475C701S072612D017	1212052-11	Sediment	12.5	46.4	UJ
Oil Quant	4.75	SEKR0475C702S072612DX	1209020-09R	Sediment		91.3	U
Oil Quant	4.75	SEKR0475C702S072612D005	1211039-09	Sediment		31.2	U
Oil Quant	4.75	SEKR0475C702S072612D009	1211039-10	Sediment		30.1	U
Oil Quant	4.75	SEKR0475C702S072612D013	1211039-11	Sediment		34.3	U
Oil Quant	5	SEKR0500C701S112912DX	1212034-03	Sediment		120	U
Oil Quant	5	SEKR0500C701D112912D006	1212034-05	Sediment		52	U
Oil Quant	5	SEKR0500C701S112912D006	1212034-04	Sediment		55.2	U
Oil Quant	5	SEKR0500C701S112912D012	1212034-06	Sediment		90.2	U
Oil Quant	5	SEKR0500C702S112812DX	1212031-02	Sediment		74.4	U
Oil Quant	5	SEKR0500C702S112812D007	1212031-03	Sediment	14	60.4	UJ
Oil Quant	5	SEKR0500C702D112812D013	1212031-05	Sediment		48.8	UJ
Oil Quant	5	SEKR0500C702S112812D013	1212031-04	Sediment		70.7	UJ
	5.10	SEKR0510C001D022112D004	1202070-09	Sediment		123	U
	5.1	SEKR0510C001S022112D004	1202070-06	Sediment	470	117	
Oil Quant	5.25	SEKR0525C702S112812DX	1212032-02	Sediment	1650	235	
Oil Quant	5.25	SEKR0525C702S112812D006	1212032-03	Sediment	295	142	
Oil Quant	5.25	SEKR0525C702S112812D011	1212032-04	Sediment		149	U
Oil Quant	5.25	SEKR0525C702S112812D016	1212032-05	Sediment		159	U
Oil Quant	5.25	SEKR0525C702S112812D020	1212032-06	Sediment		165	U
Oil Quant	5.25	SEKR0525C702S112812D024	1212032-07	Sediment	1190	151	I
Oil Quant	5.5	SEKR0550C701D072612DX	NA	Sediment	N/A	N/A	N/A
Oil Quant	5.5	SEKR0550C701S072612DX	1212028-19	Sediment		160	U
Oil Quant	5.5	SEKR0550C701S072612DX	1209020-02R	Sediment		277	U
Oil Quant	5.5	SEKR0550C701S072612D005	1211038-11	Sediment		77.5	U
Oil Quant	5.5	SEKR0550C701S072612D010	1211038-12	Sediment	62.9	65.2	J
Oil Quant	5.5	SEKR0550C702S112912DX	1212034-07	Sediment		125	U
Oil Quant	5.5	SEKR0550C702D112912D006	1212034-09	Sediment	11.4	77.9	J
Oil Quant	5.5	SEKR0550C702S112912D006	1212034-08	Sediment		71.1	U
Oil Quant	5.5	SEKR0550C702S112912D011	1212034-10	Sediment		76.3	U
Oil Quant	5.5	SEKR0550C702S112912D015	1212034-11	Sediment		225	U
Oil Quant	5.5	SEKR0550C702S112912D019	1212034-12	Sediment		78.6	U
Oil Quant	5.5	SEKR0550C703S112712DX	1212028-09	Sediment	130	85.3	
Oil Quant	5.5	SEKR0550C703S112712D006	1212028-10	Sediment		60.4	U
Oil Quant	5.5	SEKR0550C703S112712D011	1212028-11	Sediment		81.3	U
Oil Quant	5.5	SEKR0550C703S112712D016	1212028-12	Sediment		171	U

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	5.5	SEKR0550C703S112712D019	1212028-13	Sediment		106	U
Oil Quant	5.5	SEKR0550C703S112712D022	1212028-14	Sediment		73.9	U
Oil Quant	5.5	SEKR0550C704S112812D006	1212031-07	Sediment		108	U
Oil Quant	5.5	SEKR0550C704S112812DX	1212031-06	Sediment	373	90.9	
Oil Quant	5.5	SEKR0550C704S112812D011	1212031-08	Sediment		180	U
Oil Quant	5.5	SEKR0550C704S112812D011	1212031-08D	Sediment		181	U
Oil Quant	5.5	SEKR0550C704S112812D016	1212031-09	Sediment		329	U
Oil Quant	5.5	SEKR0550C704S112812D020	1212031-10	Sediment		162	U
Oil Quant	5.5	SEKR0550C705D112712D016	1212030-08	Sediment	27.5	135	UJ
Oil Quant	5.5	SEKR0550C705S112712DX	1212030-04	Sediment		120	U
Oil Quant	5.5	SEKR0550C705S112712D006	1212030-05	Sediment	0.036	131	U
Oil Quant	5.5	SEKR0550C705S112712D011	1212030-06	Sediment	79.9	130	J
Oil Quant	5.5	SEKR0550C705S112712D016	1212030-07	Sediment	25.1	142	UJ
Oil Quant	5.5	SEKR0550C705S112712D020	1212030-09	Sediment	6.68	176	UJ
Oil Quant	5.5	SEKR0550C705S112712D025	1212030-10	Sediment	19.3	147	UJ
Oil Quant	5.5	SEKR0550C705S112712D030	1212052-01	Sediment	40.1	54.9	UJ
	5.65	SEKR0565R003S022012D004	1202070-03	Sediment	894	130	
Oil Quant	5.75	SEKR0575C701S072612DX	1209020-04R	Sediment		402	U
Oil Quant	5.75	SEKR0575C701S072612D007	1211038-16	Sediment		152	U
Oil Quant	5.75	SEKR0575C701S072612D013	1211038-17	Sediment		94.5	U
Oil Quant	5.75	SEKR0575C701S072612D019	1211038-18	Sediment		53.2	U
Oil Quant	5.75	SEKR0575C702S112712DX	1212028-01	Sediment		107	U
Oil Quant	5.75	SEKR0575C702S112712D006	1212028-02	Sediment		89.8	U
Oil Quant	5.75	SEKR0575C702S112712D011	1212028-03	Sediment		71.9	U
Oil Quant	5.75	SEKR0575C702S112712D016	1212028-04	Sediment		82.4	U
Oil Quant	5.75	SEKR0575C702S112712D016	1212028-04D	Sediment		82.7	U
Oil Quant	5.75	SEKR0575C702S112712D020	1212028-05	Sediment		102	U
	8.86	SEKR0886R004S022112D004	1202070-10	Sediment	1240	212	
Oil Quant	9	SEKR0900C701S112912DX	1212037-02	Sediment	57.3	67.7	
Oil Quant	9	SEKR0900C701S112912D006	1212037-03	Sediment	N/A	N/A	
Oil Quant	9	SEKR0900C701S112912D006	1212037-03X	Sediment		54.7	U
Oil Quant	9	SEKR0900C701S112912D011	1212037-04	Sediment		18	U
Oil Quant	9	SEKR0900C701S112912D016	1212037-05	Sediment	N/A	N/A	N/A
Oil Quant	9	SEKR0900C701S112912D016	1212037-05X	Sediment		36.5	UJ
Oil Quant	9	SEKR0900C702S112712DX	1212028-15	Sediment	1350	296	
Oil Quant	9	SEKR0900C702S112712D006	1212028-16	Sediment	23.6	96.7	J
Oil Quant	9	SEKR0900C702D112712D011	1212028-18	Sediment		88.6	U
Oil Quant	9	SEKR0900C702S112712D011	1212028-17	Sediment		80.2	U
Oil Quant	9	SEKR0900C702S112712D014	1212029-01	Sediment		91.9	U
Oil Quant	9	SEKR0900C702S112712D017	1212029-02	Sediment		62.9	U
	9	SEKR0900I3004S041912D005	1204031-02	Sediment	1310	119	
Oil Quant	10.5	SEKR1050C701S072512DX	1209019-14	Sediment		126	U
Oil Quant	10.5	SEKR1050C701S072512D003	1211038-02	Sediment	43.8	53.2	J
	10.6	SEKR1061L005S022012D004	1202070-02	Sediment	1820	121	
Oil Quant	10.75	SEKR1075C701S112812DX	1212030-18	Sediment		155	U
Oil Quant	10.75	SEKR1075C701S112812D005	1212030-19	Sediment	276	44	
Oil Quant	10.75	SEKR1075C701S112812D009	1212031-01	Sediment	8160	253	
Oil Quant	10.75	SEKR1075C701S112812D014	1212052-02	Sediment	266	36.7	
Oil Quant	10.75	SEKR1075C702S113012DX	1212038-02	Sediment	2150	109	
Oil Quant	10.75	SEKR1075C702S113012D006	1212038-03	Sediment	2920	65.8	
	10.75	SEKR1075L201S042512DX	1204035-05	Sediment	7100	222	
	10.75	SEKR1075C701S112812D019	1406040-06	Sediment		29.6	U
Oil Quant	11	SEKR1100C701S112912DX	1212035-17	Sediment	621	82.9	
Oil Quant	11	SEKR1100C701S112912D005	1212035-18	Sediment	241	54.2	
	11	SEKR1100C701S112912D009	1406040-07	Sediment	18.2	50.5	J
	11	SEKR1100C701S112912D013	1406040-08	Sediment	8.64	26	UJ
	11.32	SEKR1132L006D022112D004	1202070-08	Sediment	59.4	74.8	J
	11.32	SEKR1132L006S022112D004	1202070-04	Sediment	186	84.5	
Oil Quant	12	SEKR1200C701S072512DX	1209019-13	Sediment	181	176	
Oil Quant	12	SEKR1200C701S072512D006	1211037-18	Sediment	240	88.7	

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	12	SEKR1200C701S072512D011	1211038-01	Sediment	537	53.1	
	12	SEKR1200C701S072512D016	1406040-09	Sediment	14.6	22.9	UJ
Oil Quant	14.25	SEKR1425C701S113012DX	1212037-15	Sediment	257	46.6	
Oil Quant	14.25	SEKR1425C701S113012D004	1212037-16	Sediment	134	17.8	
	14.25	SEKR1425C701S113012D010	1406040-10	Sediment	1130	50.8	
	14.25	SEKR1425C701S113012D013	1406040-11	Sediment	50.3	23	
	14.25	SEKR1425C701S113012D016	1406040-12	Sediment	6.78	15.3	UJ
	14.25	SEKR1425C701S113012D018	1406040-13	Sediment	6.09	15.4	UJ
Oil Quant	14.75	SEKR1475C701S072612DX	1209020-11R	Sediment	265	192	
Oil Quant	14.75	SEKR1475C701S072612D006	1211039-13	Sediment	840	57.2	
Oil Quant	14.75	SEKR1475C701S072612D011	1211039-14	Sediment	1170	70.1	
Oil Quant	14.75	SEKR1475C702S072612DX	1209020-14R	Sediment	940	159	
Oil Quant	14.75	SEKR1475C702S072612D004	1211039-17	Sediment		72	U
Oil Quant	14.75	SEKR1475C702S072612D007	1211039-18	Sediment		72.4	U
	14.75	SEKR1475C701S072612D014	1406041-01	Sediment	1290	35.1	
	14.77	SEKR1477R007S022212D004	1202077-12	Sediment	539	40.6	
	14.95	SEKR1495L008S022212D004	1202077-09	Sediment	694	62.1	
Oil Quant	15	SEKR1500C701S072612DX	1209020-16R	Sediment	477	271	
Oil Quant	15	SEKR1500C701S072612D003	1211040-02	Sediment	419	87	
Oil Quant	15	SEKR1500C701S072612D006	1211040-03	Sediment	1750	80.7	
Oil Quant	15	SEKR1500C701S072612D009	1211040-04	Sediment	808	52.7	
Oil Quant	15	SEKR1500C701S072612D013	1211040-05	Sediment		41.3	U
Oil Quant	15	SEKR1500C701S072612D028	1212052-12	Sediment	71.4	33.9	
	15.22	SEKR1522R009S022212D004	1202077-11	Sediment		103	U
	15.22	SEKR1522R009D022212D004	1202070-12	Sediment	1580	146	
Oil Quant	15.5	SEKR1550C701S112812DX	1212033-07	Sediment	761	173	
Oil Quant	15.5	SEKR1550C701S112812D006	1212033-08	Sediment	1960	166	
Oil Quant	15.5	SEKR1550C701S112812D011	1212033-09	Sediment	229	103	
Oil Quant	15.5	SEKR1550C701S112812D016	1212033-10	Sediment		83.4	U
Oil Quant	15.5	SEKR1550C701S112812D021	1212033-11	Sediment	153	97.7	
Oil Quant	15.5	SEKR1550C701S112812D026	1212033-12	Sediment	203	123	
Oil Quant	15.75	SEKR1575C701S072612DX	1208010-05	Sediment	621	204	
Oil Quant	15.75	SEKR1575C701S072612D007	1208010-06	Sediment	754	169	
Oil Quant	15.75	SEKR1575C701S072612D013	1208010-07	Sediment		158	U
Oil Quant	15.75	SEKR1575C701S072612D019	1208010-08	Sediment		90.2	U
Oil Quant	15.75	SEKR1575C701S072612D019	1208010-08D	Sediment		90.6	U
Oil Quant	15.75	SEKR1575C702S072612DX	1208010-02	Sediment	698	217	
Oil Quant	15.75	SEKR1575C702S072612D005	1208010-03	Sediment	820	158	
Oil Quant	15.75	SEKR1575C702S072612D010	1208010-04	Sediment		76.7	U
Oil Quant	15.75	SEKR1575C703S112812DX	1212031-15	Sediment	53.4	24.7	
Oil Quant	15.75	SEKR1575C703S112812D006	1212031-16	Sediment	53.7	15.3	
Oil Quant	15.75	SEKR1575C703S112812D012	1212031-17	Sediment	48.3	17.2	
Oil Quant	15.75	SEKR1575C703S112812D022	1212052-04	Sediment	106	65.8	UJ
Oil Quant	18.5	SEKR1850C701S072412D003	1211037-05	Sediment	105	13.8	
Oil Quant	18.5	SEKR1850C701S072412DX	1209019-03	Sediment	109	43	
Oil Quant	18.75	SEKR1875C701S072512D003	1211037-15	Sediment	105	14.6	
Oil Quant	18.75	SEKR1875C701S072512DX	1209019-11	Sediment	95.3	46.8	
Oil Quant	19	SEKR1900C701S072512DX	1208009-05	Sediment	159	59.9	
Oil Quant	19	SEKR1900C701S072512D005	1208009-06	Sediment	167	33.5	
Oil Quant	19	SEKR1900C701S072512D005	1208009-06D	Sediment	117	33.5	
Oil Quant	19	SEKR1900C701S072512D009	1208009-07	Sediment		61.6	U
	19.34	SEKR1934L013S022312D004	1202077-03	Sediment		159	U
	19.34	SEKR1934L013S041912D005	1204031-01	Sediment		252	U
	19.5	SEKR1950C501S042612DX	1204035-07	Sediment	625	71.3	
Oil Quant	19.5	SEKR1950C701S072412DX	1209019-04	Sediment		149	U
Oil Quant	19.5	SEKR1950C701S072412D006	1211037-06	Sediment		46.3	U
Oil Quant	20	SEKR2000C701S072412D005	1211037-02	Sediment		66.8	U
Oil Quant	20	SEKR2000C701S072412DX	1209019-02	Sediment		170	U
Oil Quant	20	SEKR2000C701S072412D010	1211037-03	Sediment		65	U
Oil Quant	20	SEKR2000C701S072412D010	1211037-03D	Sediment		65.1	U

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	20	SEKR2000C701S072412D014	1211037-04	Sediment		47.2	U
Oil Quant	20	SEKR2000C701S072412D020	1212052-08	Sediment		75.4	U
Oil Quant	20	SEKR2000C701S072412D023	1212052-09	Sediment	486	79	
Oil Quant	20	SEKR2000C702S072512DX	1209019-09	Sediment	49.7	151	J
Oil Quant	20	SEKR2000C702S072512D002	1211037-13	Sediment	11.5	72.6	J
	20	SEKR2000C701S072412D028	1406041-02	Sediment	26.1	15.7	U
Oil Quant	20.25	SEKR2025C701S072412DX	1209019-01	Sediment	275	85.7	
Oil Quant	20.25	SEKR2025C701S072412D007	1211037-01	Sediment	19.7	52.9	J
Oil Quant	20.25	SEKR2025C702S072412DX	1208009-01	Sediment	1310	113	
Oil Quant	20.25	SEKR2025C702S072412D005	1208009-02	Sediment		75	U
Oil Quant	20.25	SEKR2025C703S072412DX	1209019-05	Sediment		76.4	U
Oil Quant	20.25	SEKR2025C703S072412D004	1211037-07	Sediment	97.1	44.5	
Oil Quant	21.25	SEKR2125C701S072412DX	1209019-06	Sediment	552	124	
Oil Quant	21.25	SEKR2125C701S072412D006	1211037-08	Sediment	436	45.4	
	21.25	SEKR2125C701S072412D010	1406041-03	Sediment	49.4	21.3	I
	21.25	SEKR2125C701S072412D010	1406041-03D	Sediment	50.2	21.3	I
	21.31	SEKR2131R014S022312D004	1202077-04	Sediment	451	134	
Oil Quant	21.5	SEKR2150C701S072512D004	1211038-03	Sediment		44.5	U
Oil Quant	21.5	SEKR2150C701S072512DX	1209019-16	Sediment	13.6	89.2	J
Oil Quant	21.5	SEKR2150C701S072512D008	1211038-04	Sediment		39.1	U
Oil Quant	21.5	SEKR2150C702S072712DX	1209021-04	Sediment		77.4	U
Oil Quant	21.5	SEKR2150C702S072712D005	1211040-11	Sediment	154	14.9	UJ
Oil Quant	21.5	SEKR2150C702S072712D010	1211040-12	Sediment	43.6	15.2	UJ
Oil Quant	21.5	SEKR2150C702S072712D015	1211040-13	Sediment	16.1	15.4	UJ
Oil Quant	21.5	SEKR2150C703S072712DX	1209021-06	Sediment	332	225	
Oil Quant	21.5	SEKR2150C703S072712D005	1211040-16	Sediment	295	72.2	
Oil Quant	21.5	SEKR2150C704S072712DX	1209021-08	Sediment	706	180	
Oil Quant	21.5	SEKR2150C704S072712D003	1211041-02	Sediment	65.9	38.8	
Oil Quant	21.5	SEKR2150C704S072712D005	1211041-03	Sediment	85.7	43.4	
Oil Quant	21.5	SEKR2150C704S072712D010	1211041-04	Sediment		44.2	U
Oil Quant	21.5	SEKR2150C705S112912DX	1212035-04	Sediment		44.5	U
Oil Quant	21.5	SEKR2150C705S112912D006	1212035-05	Sediment		37.6	U
Oil Quant	21.5	SEKR2150C705S112912D011	1212035-06	Sediment		16.7	U
Oil Quant	21.75	SEKR2175C701S072712DX	1209021-12	Sediment	205	145	
Oil Quant	21.75	SEKR2175C701S072712D002	1211041-12	Sediment	605	60.5	
Oil Quant	21.75	SEKR2175C701S072712D005	1211041-13	Sediment		35.1	U
Oil Quant	21.75	SEKR2175C701S072712D006	1211041-14X	Sediment		88.5	U
Oil Quant	21.75	SEKR2175C702S112912DX	1212036-06	Sediment	N/A	N/A	N/A
Oil Quant	21.75	SEKR2175C702S112912DX	1212036-06X	Sediment		86.3	U
Oil Quant	21.75	SEKR2175C702S112912D004	1212036-07	Sediment		42.2	U
Oil Quant	21.75	SEKR2175C702S112912D007	1212036-08	Sediment		43.7	U
Oil Quant	22	SEKR2200C701S072412DX	1209019-07	Sediment	619	123	
Oil Quant	22	SEKR2200C701S072412D005	1211037-09	Sediment	299	87.4	
Oil Quant	22	SEKR2200C701S072412D008	1211037-10	Sediment	151	41.4	
	22	SEKR2200C701S072412D013	1406041-04	Sediment	15.7	18.8	UJ
Oil Quant	22.75	SEKR2275C701S072512DX	1209019-10	Sediment	420	72.2	
Oil Quant	22.75	SEKR2275C701S072512D002	1211037-14	Sediment	117	43	
	22.75	SEKR2275C701S072512D007	1406041-05	Sediment	48.9	21.5	
Oil Quant	23	SEKR2300C701S113012DX	1212037-17	Sediment	1150	135	
Oil Quant	23	SEKR2300C701D113012D003	1212038-01	Sediment	1200	53.7	
Oil Quant	23	SEKR2300C701S113012D003	1212037-18	Sediment	1580	83.2	
	23	SEKR2300C701S113012D007	1406041-06	Sediment	26.1	20.5	
	23	SEKR2300C701S113012D011	1406041-07	Sediment	16.5	15.9	J
Oil Quant	24	SEKR2400C701S072512DX	1209019-15	Sediment	80.1	125	J
	24	SEKR2400C701S072512D004	1406041-08	Sediment	140	36.3	
Oil Quant	24.75	SEKR2475C701S072512DX	1209019-17	Sediment	236	170	
Oil Quant	24.75	SEKR2475C701S072512D004	1211038-05	Sediment	549	61.1	
Oil Quant	24.75	SEKR2475C701S072512D007	1211038-06	Sediment	5.82	51.1	J
Oil Quant	25.25	SEKR2525C701S112912DX	1212036-09	Sediment	N/A	N/A	N/A
Oil Quant	25.25	SEKR2525C701S112912DX	1212036-09X	Sediment	66.1	135	UJ

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	25.25	SEKR2525C701D112912D006	1212036-11	Sediment		64.6	U
Oil Quant	25.25	SEKR2525C701S112912D006	1212036-10	Sediment		59.9	U
Oil Quant	25.25	SEKR2525C701S112912D012	1212036-12	Sediment		38	U
	26.21	SEKR2621R015S022312D004	1202077-01	Sediment	540	119	
Oil Quant	26.25	SEKR2625C701S112912DX	1212035-01	Sediment	302	118	
Oil Quant	26.25	SEKR2625C701S112912D006	1212035-02	Sediment	580	95.5	
Oil Quant	26.25	SEKR2625C701S112912D011	1212035-03	Sediment		61.5	U
Oil Quant	26.25	SEKR2625C702S112912DX	1212036-13	Sediment	665	108	
Oil Quant	26.25	SEKR2625C702S112912D005	1212036-14	Sediment	768	115	
Oil Quant	26.25	SEKR2625C702S112912D010	1212036-15	Sediment		56.4	U
Oil Quant	27.25	SEKR2725C701S072712DX	1209021-13	Sediment		33.8	U
Oil Quant	27.25	SEKR2725C701S072712D007	1211041-15X	Sediment	10.1	22.4	UJ
Oil Quant	27.5	SEKR2750C701S112912DX	1212036-16	Sediment		129	U
Oil Quant	27.5	SEKR2750C701S112912D006	1212036-17	Sediment		76.9	U
Oil Quant	27.5	SEKR2750C701S112912D011	1212036-18	Sediment		57.2	U
Oil Quant	27.5	SEKR2750C701S112912D011	1212036-18D	Sediment	2.93	57.1	UJ
Oil Quant	27.5	SEKR2750C701S112912D016	1212037-01X	Sediment	77	30.3	UJ
Oil Quant	27.5	SEKR2750C702S113012DX	1212037-09	Sediment		161	U
Oil Quant	27.5	SEKR2750C702S113012D005	1212037-10	Sediment		53.8	U
Oil Quant	28	SEKR2800C701S072412DX	1209019-08	Sediment	998	232	
Oil Quant	28	SEKR2800C701S072412D007	1211037-11	Sediment	84.6	47.3	
Oil Quant	28	SEKR2800C701S072412D012	1211037-12	Sediment		39.2	U
	28.16	SEKR2816R016S022312D004	1202077-15	Sediment	872	89.7	
Oil Quant	28.5	SEKR2850C701S072412DX	1208009-03	Sediment		43.4	U
Oil Quant	28.5	SEKR2850C701S072412D003	1208009-04	Sediment	38.5	40.8	J
Oil Quant	28.75	SEKR2875C701S112812DX	1212033-03	Sediment		147	U
Oil Quant	28.75	SEKR2875C701D112812D004	1212033-05	Sediment		91.1	U
Oil Quant	28.75	SEKR2875C701S112812D004	1212033-04	Sediment		136	U
Oil Quant	28.75	SEKR2875C701S112812D008	1212033-06	Sediment		53.4	U
Oil Quant	28.75	SEKR2875C701S112812D016	1212052-06	Sediment	16.5	16.4	UJ
Oil Quant	30.75	SEKR3075C701S072712DX	1209021-02	Sediment		200	U
Oil Quant	30.75	SEKR3075C701S072712D005	1211040-07	Sediment		59.4	U
Oil Quant	30.75	SEKR3075C701S072712D012	1212052-13	Sediment		45.4	U
Oil Quant	30.75	SEKR3075C702S112712DX	1212029-08	Sediment	5.42	33.4	UJ
Oil Quant	30.75	SEKR3075C702S112712D006	1212029-09	Sediment	7.44	14.4	UJ
Oil Quant	30.75	SEKR3075C702S112712D011	1212029-10	Sediment	39	15	
Oil Quant	30.75	SEKR3075C702S112712D016	1212029-11	Sediment	16.4	17.2	UJ
Oil Quant	30.75	SEKR3075C702D112712D020	1212029-13	Sediment	5.44	15.2	UJ
Oil Quant	30.75	SEKR3075C702D112712D020	1212029-13D	Sediment		15.1	U
Oil Quant	30.75	SEKR3075C702S112712D020	1212029-12	Sediment	30.9	18.1	
Oil Quant	30.75	SEKR3075C702S112712D025	1212029-14	Sediment		17	U
Oil Quant	32.5	SEKR3250C701S112912DX	1212033-18	Sediment	7.33	36.2	UJ
Oil Quant	32.5	SEKR3250C701S112912D006	1212033-19	Sediment	1.28	14.3	UJ
Oil Quant	32.5	SEKR3250C701S112912D010	1212034-01	Sediment	10.8	16	UJ
Oil Quant	32.5	SEKR3250C701S112912D014	1212034-02	Sediment	7.3	18.9	UJ
Oil Quant	32.5	SEKR3250C701S112912D014	1212034-02D	Sediment	8.93	19	UJ
	33.01	SEKR3301R017S022312D004	1202077-02	Sediment		52.8	U
	34.55	SEKR3455C501S042612DX	1204035-06	Sediment	2160	115	
	35.10	SEKR3510R018D022412D004	1202077-20	Sediment	286	90.8	
	35.10	SEKR3510R018S022412D004	1202077-17	Sediment	25.3	40.4	UJ
Range Finding	35.1	SEKR3510R018S092112D004	1210004-01	Sediment	0.262	45	UJ
	36.44	SEKR3644R025S022112D004	1202070-05	Sediment		203	U
Oil Quant	36.5	SEKR3650C701S072512DX	1208009-08	Sediment		133	U
Oil Quant	36.5	SEKR3650C701S072512D006	1208009-09	Sediment		140	U
Oil Quant	36.5	SEKR3650C701S072512D010	1208009-10	Sediment		47.9	U
Oil Quant	36.5	SEKR3650C701S072512D019	1212052-10	Sediment	12.3	31.5	UJ
	36.73	SEKR3673L026S02212D004	1202070-13	Sediment	311	83.8	
	36.73	SEKR3673L026D022212D004	1202077-10	Sediment		76.7	U
Oil Quant	36.75	SEKR3675C701S112712DX	1212028-06	Sediment		78	U
Oil Quant	36.75	SEKR3675C701D112712D006	1212028-08	Sediment		55.8	U

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	36.75	SEKR3675C701S112712D006	1212028-07	Sediment		58.1	U
Oil Quant	37	SEKR3700C701S112712DX	1212030-15	Sediment	1010	76.2	
Oil Quant	37	SEKR3700C701S112712D005	1212030-16	Sediment	79.3	40	
Oil Quant	37	SEKR3700C701S112712D008	1212030-17	Sediment		62	U
	37	SEKR3700L027S022212D004	1202070-11	Sediment	282	99	
Oil Quant	37.25	SEKR3725C701S072512DX	1209019-12	Sediment		148	U
Oil Quant	37.25	SEKR3725C701S072512D007	1211037-16	Sediment	5.88	105	J
Oil Quant	37.25	SEKR3725C701S072512D013	1211037-17	Sediment	60.9	132	UJ
Oil Quant	37.25	SEKR3725C702S072712DX	1209021-11	Sediment	361	185	
Oil Quant	37.25	SEKR3725C702S072712D004	1211041-09	Sediment	515	111	
Oil Quant	37.25	SEKR3725C702S072712D010	1211041-10	Sediment	0.583	101	UJ
Oil Quant	37.25	SEKR3725C702S072712D015	1211041-11	Sediment	28	46.5	UJ
Oil Quant	37.25	SEKR3725C704S112812DX	1212032-18	Sediment	42.3	30.4	
Oil Quant	37.25	SEKR3725C704S112812D006	1212032-19	Sediment	32	15.9	
Oil Quant	37.25	SEKR3725C704S112812D011	1212033-01	Sediment	43	15.9	UJ
Oil Quant	37.25	SEKR3725C704S112812D017	1212033-02	Sediment	19.7	16.1	UJ
Oil Quant	37.25	SEKR3725C705S112812DX	1212033-13	Sediment		83.2	U
Oil Quant	37.25	SEKR3725C705D112812D006	1212033-15	Sediment		76.3	U
Oil Quant	37.25	SEKR3725C705S112812D006	1212033-14	Sediment		72	U
Oil Quant	37.25	SEKR3725C705S112812D006	1212033-14D	Sediment	10	71.8	J
Oil Quant	37.25	SEKR3725C705S112812D010	1212033-16	Sediment		42.1	U
Oil Quant	37.25	SEKR3725C705S112812D013	1212033-17	Sediment		66.2	U
Oil Quant	37.25	SEKR3725C706S112912DX	1212034-13	Sediment		23.7	U
Oil Quant	37.25	SEKR3725C706S112912D002	1212034-14	Sediment		18.6	U
Oil Quant	37.25	SEKR3725C706S112912D004	1212034-15	Sediment	640	38.7	
Oil Quant	37.25	SEKR3725C706S112912D005	1212034-16	Sediment	73.2	35.4	
Oil Quant	37.25	SEKR3725C706S112912D010	1212034-17	Sediment		52.8	U
Oil Quant	37.25	SEKR3725C706S112912D013	1212034-18	Sediment		258	U
Oil Quant	37.25	SEKR3725C706S112912D015	1212052-07	Sediment	256	88.9	
Oil Quant	37.25	SEKR3725C707S112712DX	1212029-15	Sediment		64.2	U
Oil Quant	37.25	SEKR3725C707S112712D006	1212029-16	Sediment	32.8	13.8	
Oil Quant	37.25	SEKR3725C707S112712D010	1212029-17	Sediment	68.3	14	UJ
Oil Quant	37.25	SEKR3725C707S112712D013	1212029-18	Sediment	59.9	14.2	UJ
Oil Quant	37.25	SEKR3725C708S112912DX	1212035-07	Sediment		124	U
Oil Quant	37.25	SEKR3725C708D112912D005	1212035-09	Sediment	12.1	66.3	J
Oil Quant	37.25	SEKR3725C708S112912D005	1212035-08	Sediment	27.1	62.3	UJ
Oil Quant	37.25	SEKR3725C709S113012DX	1212037-11	Sediment		84.9	U
Oil Quant	37.25	SEKR3725C709S113012D005	1212037-12	Sediment		74.4	U
Oil Quant	37.25	SEKR3725C709D113012D008	1212037-14	Sediment		56.5	U
Oil Quant	37.25	SEKR3725C709S113012D008	1212037-13	Sediment	652	58.8	
Oil Quant	37.25	SEKR3725C709S113012D008	1212037-13D	Sediment	665	58.3	
	37.25	SEKR3725C709S113012D011	1406041-09	Sediment		64.1	U
	37.25	SEKR3725C709S113012D015	1406041-10	Sediment		55.9	U
	37.25	SEKR3725C709S113012D018	1406041-11	Sediment	15.2	16.2	UJ
	37.36	SEKR3736L028S022212D004	1202077-14	Sediment		75.7	U
Oil Quant	37.5	SEKR3750C701S072512DX	1208009-11	Sediment	280	319	J
Oil Quant	37.5	SEKR3750C701S072512D006	1208009-12	Sediment		157	U
Oil Quant	37.5	SEKR3750C701S072512D010	1208009-13	Sediment		129	U
Oil Quant	37.5	SEKR3750C703D112812D012	1212032-01	Sediment		14.9	U
Oil Quant	37.5	SEKR3750C703S112812DX	1212031-18	Sediment		153	U
Oil Quant	37.5	SEKR3750C703S112812D007	1212031-19	Sediment		15.3	U
Oil Quant	37.5	SEKR3750C703S112812D012	1212038-18	Sediment	10.5	15	UJ
Oil Quant	37.5	SEKR3750C703S112812D022	1212052-05	Sediment	4.89	17.8	UJ
Oil Quant	37.5	SEKR3750C703S112812D022	1212052-05D	Sediment	5.65	17.7	UJ
Oil Quant	37.5	SEKR3750C704S112712DX	1212029-03	Sediment	1040	166	
Oil Quant	37.5	SEKR3750C704S112712D006	1212029-04	Sediment	221	74.9	
Oil Quant	37.5	SEKR3750C705S112812DX	1212032-13	Sediment	393	144	
Oil Quant	37.5	SEKR3750C705D112812D006	1212032-15	Sediment		155	U
Oil Quant	37.5	SEKR3750C705S112812D006	1212032-14	Sediment		153	U
Oil Quant	37.5	SEKR3750C705S112812D011	1212032-16	Sediment		150	U

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	37.5	SEKR3750C705S112812D016	1212032-17	Sediment		61.7	U
Oil Quant	37.5	SEKR3750C706S112912DX	1212036-01	Sediment		88.8	U
Oil Quant	37.5	SEKR3750C706S112912D006	1212036-02	Sediment		60	U
Oil Quant	37.5	SEKR3750C706D112912D009	1212036-04	Sediment	218	68.8	
Oil Quant	37.5	SEKR3750C706S112912D009	1212036-03	Sediment		57	U
Oil Quant	37.5	SEKR3750C706S112912D012	1212036-05	Sediment		79.4	U
Oil Quant	37.5	SEKR3750C707S112812DX	1212030-11	Sediment	123	110	
Oil Quant	37.5	SEKR3750C707D112812D006	1212030-13	Sediment	117	72.8	
Oil Quant	37.5	SEKR3750C707S112812D006	1212030-12	Sediment	183	131	
Oil Quant	37.5	SEKR3750C707S112812D010	1212030-14	Sediment		63.7	U
Oil Quant	37.5	SEKR3750C708S112912DX	1212035-10	Sediment	326	112	
Oil Quant	37.5	SEKR3750C708S112912D006	1212035-11	Sediment	42	39	
Oil Quant	37.5	SEKR3750C708D112912D011	1212035-13	Sediment		32.4	U
Oil Quant	37.5	SEKR3750C708S112912D011	1212035-12	Sediment		37.4	U
Oil Quant	37.5	SEKR3750C708S112912D011	1212035-12D	Sediment		37.3	U
Oil Quant	37.5	SEKR3750C708S112912D016	1212035-14	Sediment	16.3	33.4	UJ
Oil Quant	37.5	SEKR3750C708S112912D021	1212035-15	Sediment	17.2	34.4	UJ
Oil Quant	37.5	SEKR3750C708S112912D026	1212035-16	Sediment	20.5	23.2	UJ
Oil Quant	37.5	SEKR3750C709S112712DX	1212029-19	Sediment		90	U
Oil Quant	37.5	SEKR3750C709D112712D006	1212030-02	Sediment	240	86.1	
Oil Quant	37.5	SEKR3750C709D112712D006	1212030-02D	Sediment	228	86.4	
Oil Quant	37.5	SEKR3750C709S112712D006	1212030-01	Sediment	185	93.2	
Oil Quant	37.5	SEKR3750C709S112712D011	1212030-03	Sediment	23.2	53	J
Oil Quant	37.5	SEKR3750C710S112812DX	1212037-06	Sediment	90.2	88.8	
Oil Quant	37.5	SEKR3750C710S112812D005	1212037-07	Sediment	183	79.3	
Oil Quant	37.5	SEKR3750C710S112812D008	1212037-08	Sediment	542	75.7	
	37.5	SEKR3750C704S112712D011	1406041-12	Sediment	23.3	18.4	
	37.71	SEKR3771C029S022412D004	1202077-18	Sediment		45.9	U
Oil Quant	37.75	SEKR3775C701S072712DX	1209021-14	Sediment		844	U
Oil Quant	37.75	SEKR3775C701S072712D006	1211041-16	Sediment	460	92.6	
Oil Quant	37.75	SEKR3775C701S072712D007	1211041-17	Sediment		75.2	U
Oil Quant	37.75	SEKR3775C702S072712DX	1208010-16	Sediment	63.5	267	J
Oil Quant	37.75	SEKR3775C702S072712D005	1208010-17	Sediment	594	120	
Oil Quant	37.75	SEKR3775C702S072712D009	1208010-18	Sediment		49.4	U
Oil Quant	37.75	SEKR3775C703S112812DX	1212032-08	Sediment		122	U
Oil Quant	37.75	SEKR3775C703D112812D006	1212032-10	Sediment	221	102	
Oil Quant	37.75	SEKR3775C703D112812D006	1212032-10D	Sediment	36.4	103	J
Oil Quant	37.75	SEKR3775C703S112812D006	1212032-09	Sediment		141	U
Oil Quant	37.75	SEKR3775C703S112812D011	1212032-11	Sediment		80.5	U
Oil Quant	37.75	SEKR3775C703S112812D015	1212032-12	Sediment		27	U
Oil Quant	37.75	SEKR3775C704S112712DX	1212029-05	Sediment	972	63	
Oil Quant	37.75	SEKR3775C704S112712D007	1212029-06	Sediment	9.86	36.8	UJ
Oil Quant	37.75	SEKR3775C704S112712D012	1212029-07	Sediment	17.7	42.3	UJ
Oil Quant	38	SEKR3800C701S072612DX	1209020-10R	Sediment		182	U
Oil Quant	38	SEKR3800C701S072612D003	1211039-12	Sediment		77.4	U
Oil Quant	38	SEKR3800C702D072612DX	1209020-13R	Sediment	4000	1170	
Oil Quant	38	SEKR3800C702S072612DX	1209020-12R	Sediment	5650	1340	
Oil Quant	38	SEKR3800C702S072612D005	1211039-15	Sediment		170	U
Oil Quant	38	SEKR3800C702S072612D009	1211039-16	Sediment		141	U
Oil Quant	38	SEKR3800C703S072612DX	1209020-15R	Sediment		513	U
Oil Quant	38	SEKR3800C703S072612D007	1211040-01	Sediment		107	U
Oil Quant	38	SEKR3800C704S072712DX	1209021-01	Sediment		148	U
Oil Quant	38	SEKR3800C704S072712D006	1211040-06	Sediment		58.3	U
Oil Quant	38	SEKR3800C705S072712DX	1209021-05	Sediment	419	176	
Oil Quant	38	SEKR3800C705S072712D002	1211040-14	Sediment	55.6	145	J
Oil Quant	38	SEKR3800C705S072712D004	1211040-15	Sediment		68.9	U
Oil Quant	38	SEKR3800C706S072712DX	1209021-09	Sediment	20.3	112	J
Oil Quant	38	SEKR3800C706S072712D004	1211041-05	Sediment	41.6	39.7	
Oil Quant	38	SEKR3800C707S072712DX	1208010-09	Sediment	207	186	
Oil Quant	38	SEKR3800C707S072712D004	1208010-10	Sediment		86.2	U

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
Oil Quant	38	SEKR3800C707S072712D009	1208010-11	Sediment		110	U
Oil Quant	38	SEKR3800C707S072712D014	1208010-12	Sediment		104	U
Oil Quant	38	SEKR3800C708S072712DX	1209021-10	Sediment		306	U
Oil Quant	38	SEKR3800C708S072712D005	1211041-06	Sediment		115	U
Oil Quant	38	SEKR3800C708S072712D005	1211041-06D	Sediment		115	U
Oil Quant	38	SEKR3800C708S072712D010	1211041-07	Sediment		98.3	U
Oil Quant	38	SEKR3800C708S072712D014	1211041-08	Sediment		76.6	U
Oil Quant	38	SEKR3800C709S072712DX	1208010-13	Sediment		127	U
Oil Quant	38	SEKR3800C709S072712D006	1208010-14	Sediment		113	U
Oil Quant	38	SEKR3800C709S072712D011	1208010-15	Sediment		104	U
Oil Quant	38.25	SEKR3825C701S072712DX	1209021-07	Sediment		152	U
Oil Quant	38.25	SEKR3825C701S072712D003	1211040-17	Sediment		91.2	U
Oil Quant	38.25	SEKR3825C701S072712D009	1211040-18	Sediment		84	U
Oil Quant	38.25	SEKR3825C701S072712D015	1211041-01	Sediment		142	U
Oil Quant	38.5	SEKR3850C701S072612DX	1209020-01R	Sediment		294	U
Oil Quant	38.5	SEKR3850C701S072612D007	1211038-09	Sediment		72.2	U
Oil Quant	38.5	SEKR3850C701S072612D013	1211038-10	Sediment		89.9	U
Oil Quant	38.5	SEKR3850C702S072612DX	1209020-05R	Sediment		292	U
Oil Quant	38.5	SEKR3850C702S072612D006	1211039-01	Sediment		71	U
Oil Quant	38.5	SEKR3850C702S072612D010	1211039-02	Sediment		40.1	U
Oil Quant	38.5	SEKR3850C702S072612D010	1211039-02D	Sediment		40.2	U
Oil Quant	38.5	SEKR3850C703S072712DX	1209021-03	Sediment		199	U
Oil Quant	38.5	SEKR3850C703S072712D002	1211040-08	Sediment		89.3	U
Oil Quant	38.5	SEKR3850C703S072712D005	1211040-09	Sediment		87.4	U
Oil Quant	38.5	SEKR3850C703S072712D005	1211040-09D	Sediment		87	U
Oil Quant	38.5	SEKR3850C703S072712D009	1211040-10	Sediment		75.4	U
Oil Quant	38.5	SEKR3850C703S072712D015	1212052-14	Sediment		106	U
Oil Quant	38.75	SEKR3875C701S072612DX	1209020-03R	Sediment		309	U
Oil Quant	38.75	SEKR3875C701S072612D006	1211038-13	Sediment		102	U
Oil Quant	38.75	SEKR3875C701S072612D006	1211038-13D	Sediment		102	U
Oil Quant	38.75	SEKR3875C701S072612D012	1211038-14	Sediment		102	U
Oil Quant	38.75	SEKR3875C701S072612D017	1211038-15	Sediment		80.5	U
Oil Quant	39.25	SEKR3925C701S072612DX	1209020-08R	Sediment		305	U
Oil Quant	39.25	SEKR3925C701S072612D006	1211039-08	Sediment		54.1	U
Oil Quant	39.5	SEKR3950C701S072612DX	1208009-16	Sediment		323	U
Oil Quant	39.5	SEKR3950C701S072612D007	1208009-17	Sediment		223	U
Oil Quant	39.5	SEKR3950C701S072612D013	1208010-01	Sediment		177	U
Poling	39.75	SEKR3975C001S052212D005	1205027-05	Sediment		178	U
Poling	39.75	SEKR3975C002S052212D005	1205027-06	Sediment		171	U
Oil Quant	39.75	SEKR3975C701S072612DX	1209020-07R	Sediment		508	U
Oil Quant	39.75	SEKR3975C701S072612D005	1211039-05	Sediment		106	U
Oil Quant	39.75	SEKR3975C701S072612D010	1211039-06	Sediment		90	U
Oil Quant	39.75	SEKR3975C701S072612D015	1211039-07	Sediment		98.8	U
Poling	39.9	SEKR3990L001S052212D005	1205027-08	Sediment		109	U
Poling	40.1	SEKR4010L001S052212D003	1205027-07	Sediment	1.09	63.8	UJ
Oil Quant	11.11	SE1111C703S120712DX	1212038-07	Sediment	175	82	I
Oil Quant	11.11	SE1111C704S121012DX	1212038-14	Sediment		70.5	U
Oil Quant	11.11	SE1111C704S121012D005	1212038-15	Sediment	842	37.8	I
Oil Quant	11.11	SE1111C704S121012D009	1212038-16	Sediment	2370	30.8	I
Oil Quant	11.11	SE1111C704S121012D014	1212038-17	Sediment	158	17.1	I
Oil Quant	22.22	SE2222C702S120712DX	1212038-08	Sediment		117	U
Oil Quant	22.22	SE2222C702D120712D006	1212038-10	Sediment		72.7	U
Oil Quant	22.22	SE2222C702S120712D006	1212038-09	Sediment		72.5	U
Oil Quant	22.22	SE2222C702S120712D011	1212038-11	Sediment		54.1	U
Oil Quant	22.22	SE2222C702S120712D015	1212038-12	Sediment		101	U
Oil Quant	22.22	SE2222C702S120712D018	1212038-13	Sediment		164	U
Oil Quant	33.33	SE3333C701S120712DX	1212038-04	Sediment		178	U
Oil Quant	33.33	SE3333C701D120712D006	1212038-06	Sediment		63.9	U
Oil Quant	33.33	SE3333C701S120712D006	1212038-05	Sediment		74.1	U
	SEBC	SEBC0000L010S022412D004	1202077-08	Sediment		127	U

Study	Mile Post	Client ID	Lab ID	Matrix	Sample Line 6B oil conc TAS2/Hopane (Topped)	Line 6B SQL (Based on R024) (TAS2/Hopane)	Qualifier
	SEBC	SEBC0000R011S022412D004	1202077-19	Sediment		105	U
	SEBC	SEBC0000L012S022412D004	1202077-21	Sediment	91	136	I
Range Finding	SEBC	SEBC0000L012S092112D004	1210003-01	Sediment	1.18	41.7	I
	SEKR	SEKR0000C019S022312D004	1202077-05	Sediment		289	U
	SEKR	SEKR0000L020S022312D004	1202077-16	Sediment		151	U
	SEKR	SEKR0000L021S022312D004	1202077-06	Sediment		221	U
	SEKR	SEKR0000R022D022212D004	1202070-15	Sediment	241	110	I
	SEKR	SEKR0000R022S022212D004	1202077-13	Sediment	207	152	I
	SEKR	SEKR0000C023S022212D004	1202070-14	Sediment		165	U
	R024	SEKR0000R024S022012D004	1202070-01	Sediment		65	U
Deep Core	R024	SEKR0000R024AS121312D006	1212022-01	Sediment		32.1	U
Deep Core	R024	SEKR0000R024AD121312D010	1212022-03	Sediment	7.91	32.8	I
Deep Core	R024	SEKR0000R024AS121312D010	1212022-02	Sediment	16.3	33.3	I
Deep Core	R024	SEKR0000R024AS121312D013	1212022-04	Sediment		35.3	U
Range Finding	R024	SEKR0000R024S092112D004	1210002-01	Sediment		35.7	U
	SETC	SETC0000C210S022412D004	1202077-07	Sediment	99.1	60.1	I
Range Finding	R024	SEKR0000R024S092112D004_RFS1	1210002-02X	Sediment	11.1	35.8	
Range Finding	R024	SEKR0000R024S092112D004_RFS2	1210002-03	Sediment	134	34.6	
Range Finding	R024	SEKR0000R024S092112D004_RFS3	1210002-04	Sediment	248	35.3	
Range Finding	R024	SEKR0000R024S092112D004_RFS4	1210002-05	Sediment	681	35.6	
Range Finding	R024	SEKR0000R024S092112D004_RFS5	1210002-06	Sediment	1260	35.4	
Range Finding	R024	SEKR0000R024S092112D004_RFS6	1210002-07	Sediment	12600	178	
Range Finding	SEBC	SEBC0000L012S092112D004_RFS1	1210003-02	Sediment	18.1	41.7	J
Range Finding	SEBC	SEBC0000L012S092112D004_RFS2	1210003-03	Sediment	129	41.7	
Range Finding	SEBC	SEBC0000L012S092112D004_RFS3	1210003-04	Sediment	400	41.8	
Range Finding	SEBC	SEBC0000L012S092112D004_RFS4	1210003-05	Sediment	836	41.8	
Range Finding	SEBC	SEBC0000L012S092112D004_RFS5	1210003-06	Sediment	1610	41.8	
Range Finding	SEBC	SEBC0000L012S092112D004_RFS6	1210003-07	Sediment	16000	210	
Range Finding	35.1	SEKR3510R018S092112D004_RFS1	1210004-02	Sediment	75.9	45.1	
Range Finding	35.1	SEKR3510R018S092112D004_RFS2	1210004-03	Sediment	238	44.1	
Range Finding	35.1	SEKR3510R018S092112D004_RFS3	1210004-04	Sediment	439	45.4	
Range Finding	35.1	SEKR3510R018S092112D004_RFS4	1210004-05	Sediment	1060	44.3	
Range Finding	35.1	SEKR3510R018S092112D004_RFS5	1210004-06	Sediment	2060	44	
Range Finding	35.1	SEKR3510R018S092112D004_RFS6	1210004-07	Sediment	17000	228	
MDL Study	R024	SEKR0000R024S092112D004_MDL1	1210081-01	Sediment	238	35.8	
MDL Study	R024	SEKR0000R024S092112D004_MDL2	1210081-02	Sediment	250	35.8	
MDL Study	R024	SEKR0000R024S092112D004_MDL3	1210081-03	Sediment	237	35.7	
MDL Study	R024	SEKR0000R024S092112D004_MDL4	1210081-04	Sediment	369	35.7	
MDL Study	R024	SEKR0000R024S092112D004_MDL5	1210081-05	Sediment	237	35.7	
MDL Study	R024	SEKR0000R024S092112D004_MDL6	1210081-06	Sediment	254	35.8	
MDL Study	R024	SEKR0000R024S092112D004_MDL7	1210081-07	Sediment	262	35.8	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL1	1210082-01	Sediment	347	42	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL2	1210082-02	Sediment	462	41.9	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL3	1210082-03	Sediment	461	42	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL4	1210082-04	Sediment	476	41.9	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL5	1210082-05	Sediment	492	41.9	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL6	1210082-06	Sediment	644	41.9	
MDL Study	SEBC	SEBC0000L012S092112D004_MDL7	1210082-07	Sediment	568	42	
MDL Study	35.1	SEKR3510R018S092112D004_MDL1	1210083-01	Sediment	177	45.4	
MDL Study	35.1	SEKR3510R018S092112D004_MDL2	1210083-02	Sediment	357	45.5	
MDL Study	35.1	SEKR3510R018S092112D004_MDL3	1210083-03	Sediment	424	45.5	
MDL Study	35.1	SEKR3510R018S092112D004_MDL4	1210083-04	Sediment	404	45.4	
MDL Study	35.1	SEKR3510R018S092112D004_MDL5	1210083-05	Sediment	375	45.4	
MDL Study	35.1	SEKR3510R018S092112D004_MDL6	1210083-06	Sediment	422	45.5	
MDL Study	35.1	SEKR3510R018S092112D004_MDL7	1210083-07	Sediment	250	45.5	

Figure 1. Illustration of how the TAS2/Hopane QR changes with increasing concentrations of background hydrocarbons for four different Line 6B input levels. This figure explains why direct RFS calibration for spill zone sediments did not produce accurate Line 6B quantification results given that one TAS2/Hopane QR can reflect multiple Line 6B inputs as a function of the background hydrocarbon concentration.

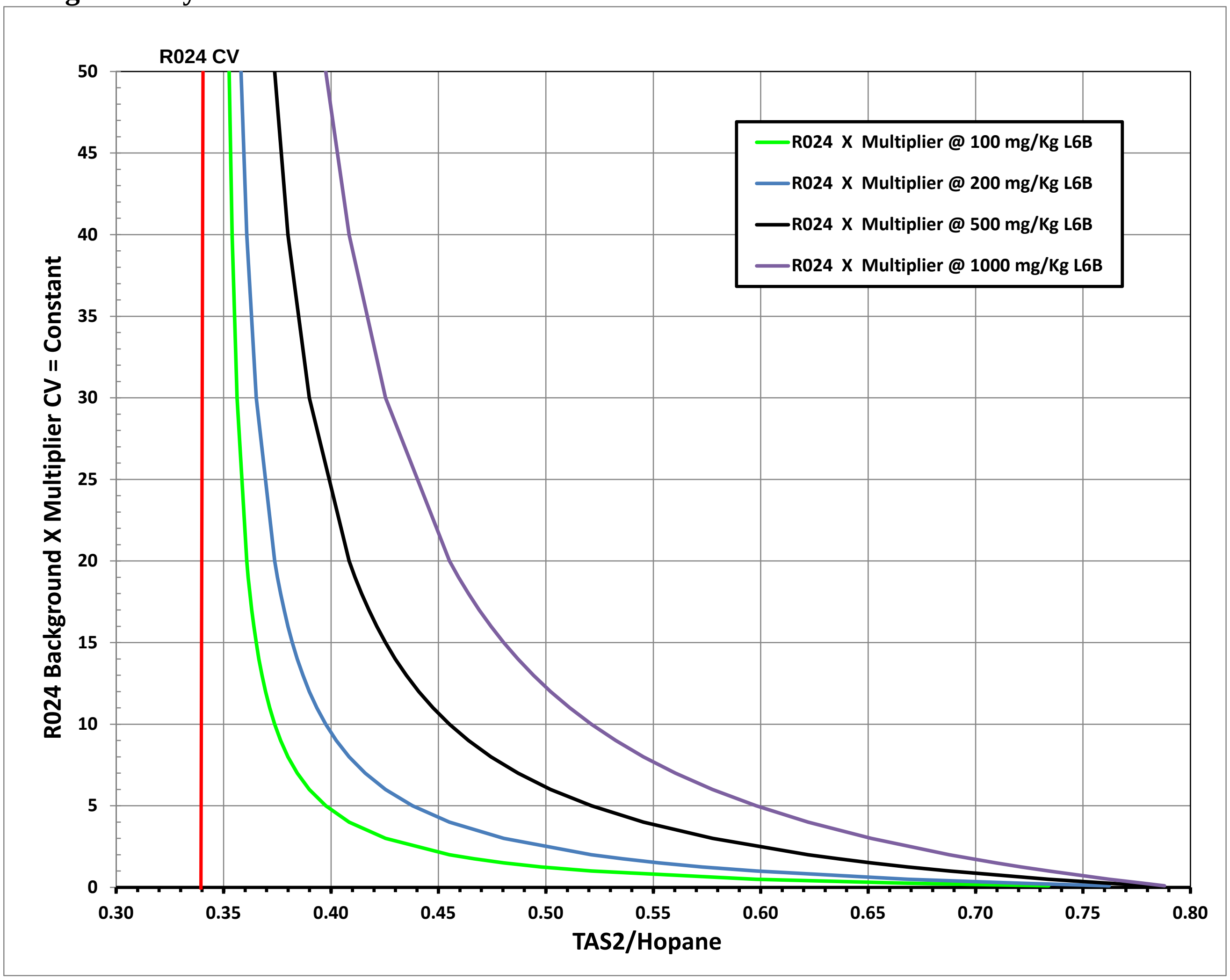


Figure 2. Examples of Mixing Model calculations for Kalamazoo River sediment samples. The R024 Range Finding Study results are also provided with the corresponding R024 mixing model curve.

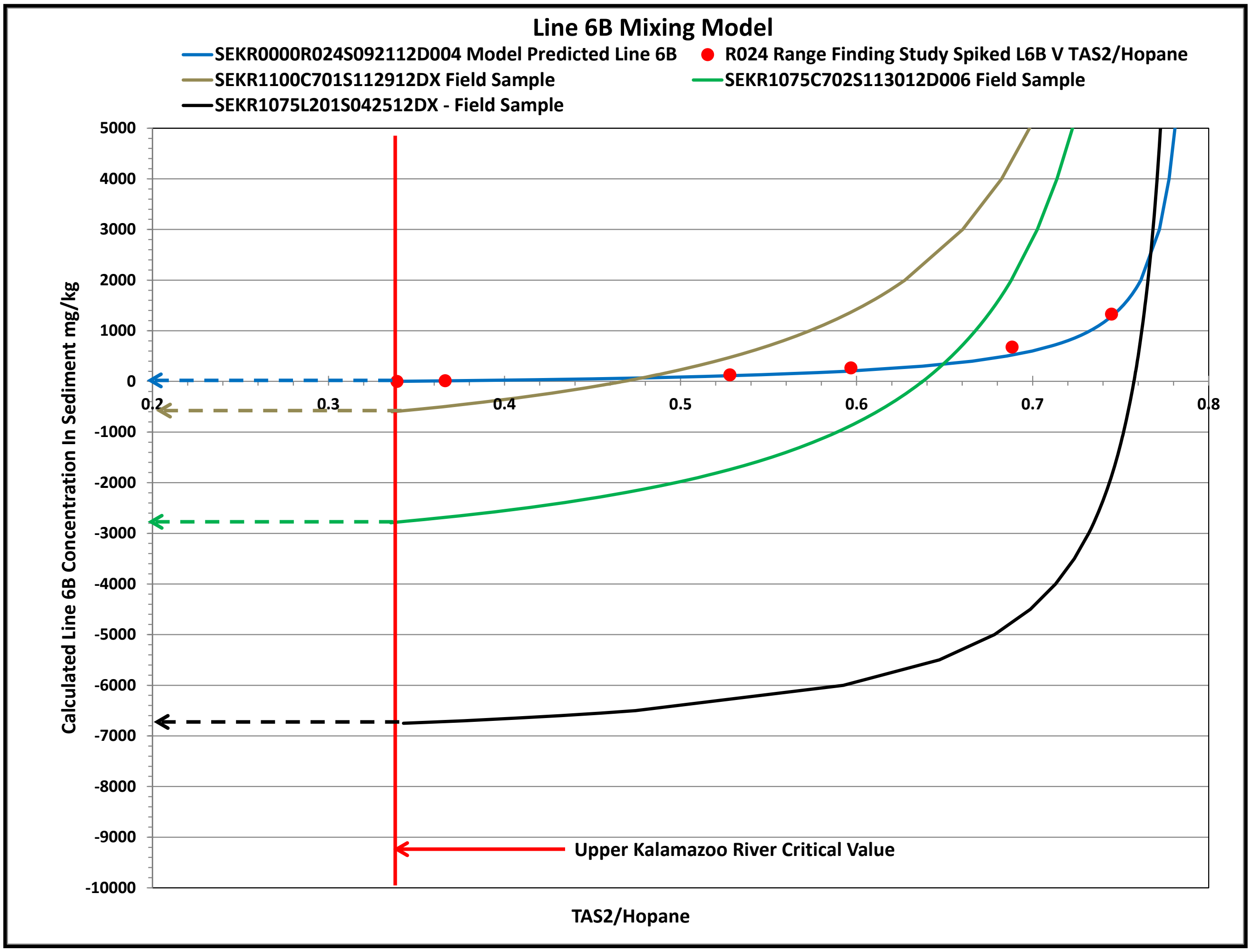


Figure 3. GC/FID chromatograms of A) Topped Cold Lake Oil sample SO092812CL01 CL-6B-072223-092710-JPS-KA-001-33_TOPPED, B) Line 6B impacted sediment sample SEKR1075L201S042512DX, and C) Non Line 6B oil impacted sediment sample SEKR3750C705S112812D011.

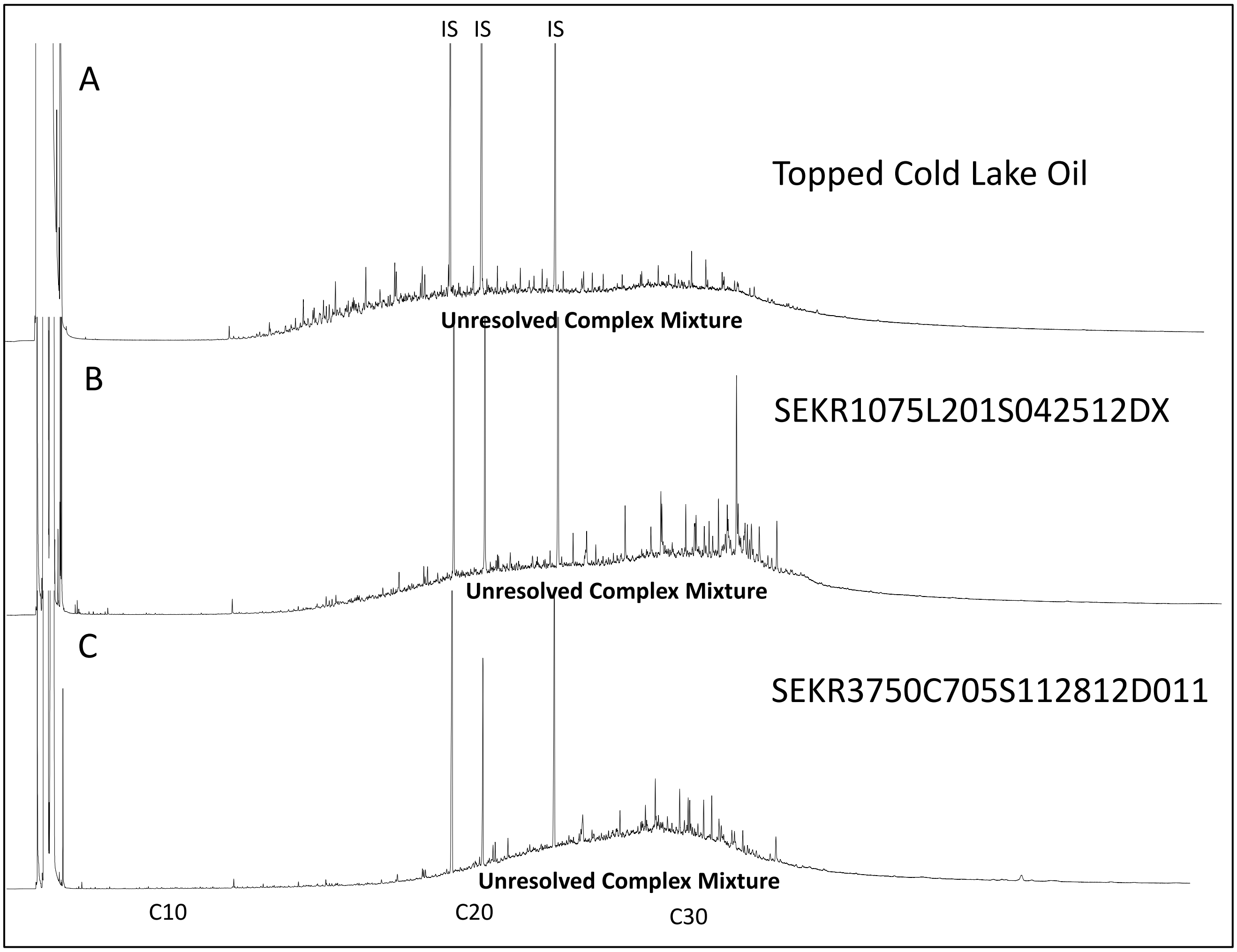


Figure 4. PAH and sulfur heterocyclic distribution plot of topped Cold Lake oil (blue bars) versus A) RFS sediment sample R024 (red bars) collected above the Talmadge Creek - Kalamazoo River convergence and B) MP10.75 sediment sample containing 7,100 mg/kg Line 6B oil. The Y axis for each sample has been visually adjusted (to C4-chrysenes) to compare the PAH distributions between the two samples. The purple-red gradient bars represent the sulfur heterocyclic compound distributions in the sediment samples.

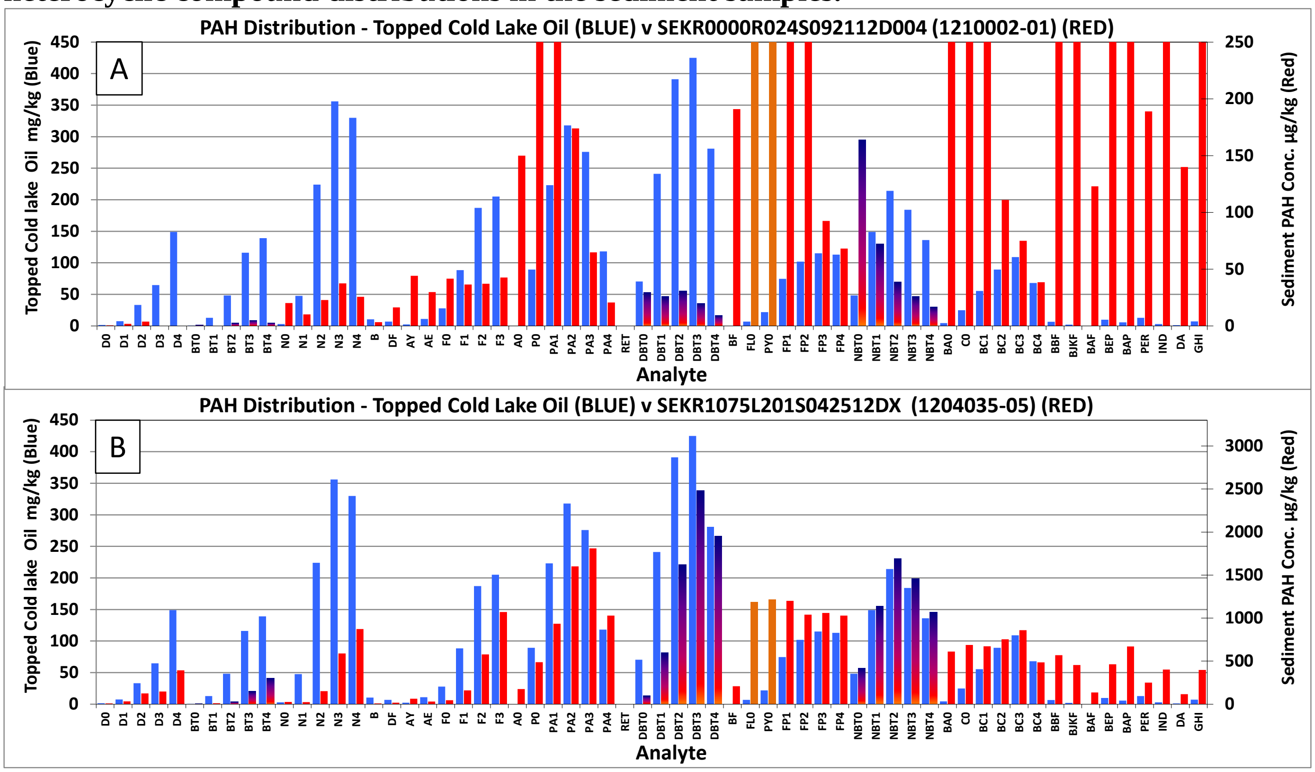


Figure 5. Triterpane, sterane and tri-aromatic sterane (TAS) biomarker distribution of topped Cold Lake oil (blue bars) versus A) RFS sediment sample R024 (red bars) collected above the Talmadge Creek - Kalamazoo River convergence and B) a surface sediment sample from MP10.75 containing 7,100 mg/kg Line 6B oil. The differences in the TAS distributions between the topped Cold Lake Oil and the reference sediment sample provide a means to distinguish between the two sources of hydrocarbons in the Kalamazoo River sediments. B)The biomarker distribution in sediment MP10.75 relative to the topped Line 6B oil documents the presence of Line 6B in the sample. The Y axis for each sample has been visually adjusted (to Hopane (T19) to compare the biomarker distributions between the two samples.

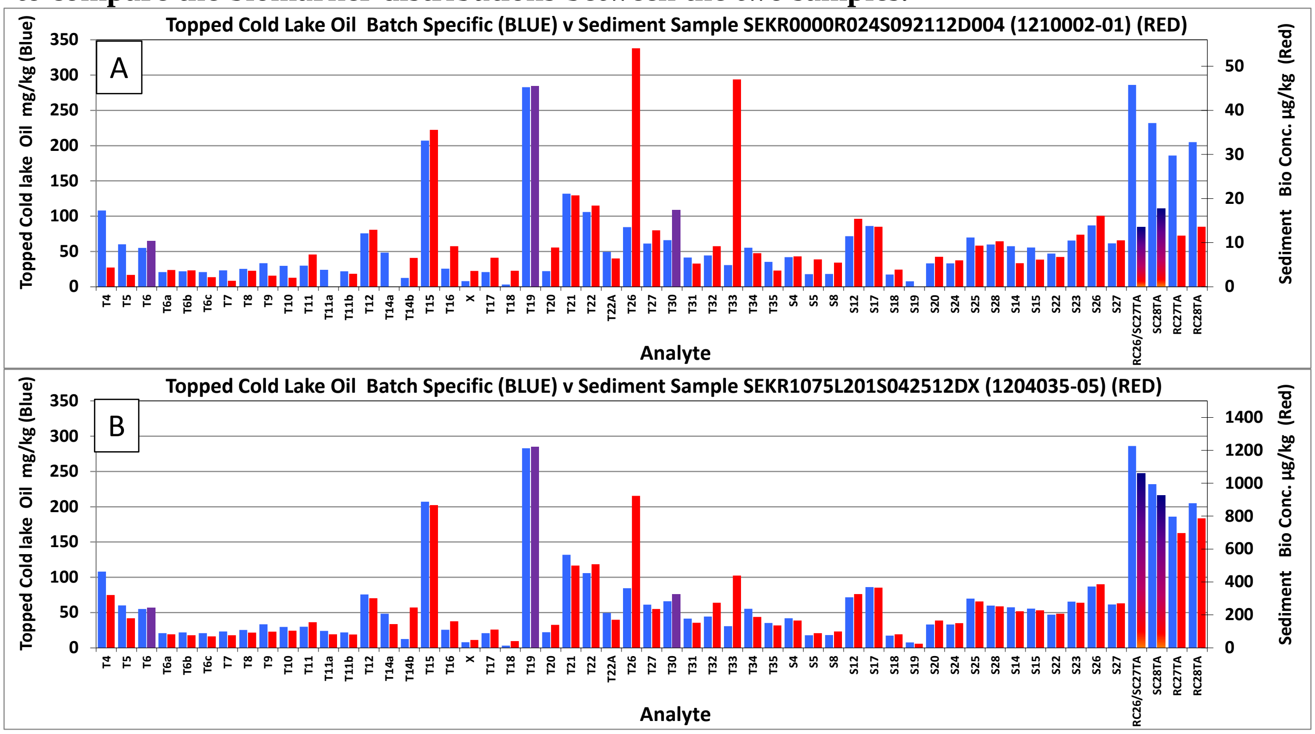


Figure 6. PAH and biomarker distributions for Lower Kalamazoo River sediment sample SEKR3750C705S112812D011 relative to topped Cold Lake Crude Oil. A) The PAH distribution represents a mixture of pyrogenic/petrogenic PAHs derived from a mixture of combustion source inputs and BH oil unrelated to Line 6B oil. B) Note that the T30 (purple bar) biomarker is depressed relative to the major triterpanes (e.g., Hopane, T19) in the sample. The depressed T30 biomarker concentration, a source characteristic of the BH oil, artificially increases the TAS1/T30 QR above its CV resulting in a false positive response for Line 6B oil.