

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

December 19, 2011

Enbridge Energy, Limited Partnership c/o Mr. Rich Adams Vice President, Operations Superior City Centre Second Floor 1409 Hammond Ave. Superior, Wisconsin 54880

Re: U.S. EPA Notice of Disapproval of an Enbridge Energy, Limited Partnership November 11, 2011 submittal in response to the Administrative Order issued by U.S. EPA on July 27, 2010, pursuant to §311(c) of the Clean Water Act (Docket No. CWA 1321-5-10-001) and Supplement to the Administrative Order issued by U.S. EPA on September 23, 2010.

Dear Mr. Adams:

The United States Environmental Protection Agency (U.S. EPA) has completed its review of the following document submitted by Enbridge Energy, Limited Partnership, Enbridge Pipelines (Lakehead) L.L.C., Enbridge Pipelines (Wisconsin), and Enbridge Energy Partners, L.P. (herein collectively referred to as "Enbridge") on November 11, 2011:

Enbridge Line 6B MP 608, Marshall, MI Pipeline Release, Report of Findings for Background Sediment Concentrations, Investigation for Upstream Sample Areas, Prepared for United States Environmental Protection Agency, Enbridge Energy, Limited Partnership, Submitted: November 11, 2011

U.S. EPA disapproves Enbridge's above referenced *Report of Findings for Background Sediment Concentrations, Investigation for Upstream Sample Areas* submitted on November 11, 2011 due to deficiencies described herein. Specific comments are set forth below and shall be incorporated into a revised report, pursuant to Paragraph 18 of the U.S. EPA Administrative Order.

- 1. Section 1, Page 1: Please define total petroleum hydrocarbon (TPH) ranges with carbon ranges (i.e., C_{10} - C_{20}) for each range reported.
- 2. Section 1.1:
 - a. Starting in this Section and continuing throughout the report: The term "bedload" is used incorrectly. The submerged oil is associated with fine-grained sediment, which if transported, is expected to be in the suspended sediment component.

Please consider replacing "bedload" with "streambed sediment" in most, but not all, situations throughout the report.

- b. Due to differing hydrodynamics and streambed sediment characteristics in the various waterways, the concentration of contaminants in streambed sediments in the tributaries/locations where background samples were collected are not necessarily the same concentrations that will be found in the affected portions of the Kalamazoo River. Please modify the text accordingly.
- c. Please describe how organic reference material concentrations will specifically be utilized.
- 3. Section 2.1.1:
 - a. Page 3 and Figures 1 through 4 (Pages 67 through 70): Please provide proposed sampling locations on the figures, if significantly different from actual sampling locations and describe what the significant changes were in the text.
 - b. Page 3:
 - i. The statement that the four target areas were not impacted by historical releases is contradictory to the statement in Section 1.1 "the concentration of PNAs in sediments upstream from the impacted areas are attributable to releases that are not related to the Line 6B crude oil spill...., additional historical sources have released hazardous substances into the bed load for the Kalamazoo River." Please revise the sections appropriately so they are accurate and consistent.
 - ii. Please cite references used to verify that four target areas have not been impacted by other historical spills.
 - iii. Please clarify whether any of the field-modified locations were at sites on Talmadge Creek. If so, please clarify that USGS (which was not present for background sampling in Talmadge Creek) did not consent to such modifications.
- 4. Section 2.1.2:
 - a. Page 3: Please Include Talmadge Creek sample collection procedures.
 - b. Page 4: Please identify what type of global positioning system (GPS) system was used for documenting sample locations.
 - c. Page 4: Please remove the last sentence since it is described in Section 2.1.1.

- d. It is not documented that two cores were collected at each sample location, and that subsamples of lithologic intervals were collected from one core, but the other core was analyzed as a whole, single sample for bulk density, etc. Please clarify and provide an accurate description of the cores.
- 5. Section 2.1.3:
 - a. Based on the description of sampling handling, it appears that samples to be analyzed for VOCs were collected immediately upon opening the core, before cores were photographed and logged. Please verify this is accurate, and revise as necessary.
 - b. Please add a description of the methods for selection of samples for duplicate analysis, type of replicate sampling techniques used, and any related details about processing of duplicate samples.
 - c. Please describe how "the remaining sample was homogenized" and clarify that this homogenization and sample collection was performed for a single lithologic unit in isolation from other units of the core.
 - d. Please clarify which laboratory(ies) performed which chemical analyses. This information would also be appropriate in Section 2.2.
- 6. Section 2.2:
 - a. Please specify analytical methods, reference published methods and corresponding tables.
 - b. Please list the detection limit for each analyte in a table.
- 7. Section 2.3:
 - a. Page 5 and Section 3.2 (Page 8):
 - i. Section 2.3 states that field duplicate samples were treated as discrete samples and included in the statistical analysis; however, analytical results are only summarized for the primary samples. Please provide a comprehensive list of and results for all samples, including duplicates.
 - ii. It is inappropriate to use both samples from paired duplicate samples as discrete samples as it unjustifiably biases statistical calculations and violates the assumption of independent observations for hypothesis tests. Therefore, please exclude analytical results for duplicate samples (where valid primary sample results exist) from statistical analyses.

- b. Page 5:
 - i. Because samples were collected of bed material only, then one can make inferences about bed material and bed load only, not suspended load. Please clarify.
 - ii. The inclusion of both the primary and replicate samples from each QC duplicate pair effectively double-weights the sampling location. Please recalculate each affected statistic using only one sample from each pair; the method of selection shall not allow subjectivity or bias to affect inclusion/exclusion.
 - iii. Please clarify which analyses are referenced for QC samples, as the included references are ambiguous.
 - iv. Please change "nonparametric prediction limit" to "nonparametric UTL."
- c. Page 6:
 - i. Second bullet:
 - 1. Please clarify methods used for "data validation" and what is meant by "excluded from UTL data set," as UTL is a statistic and not a data set.
 - 2. Please describe the objective criteria applied to ascertain whether an outlier value came from a natural or anthropogenic source.
- d. General: The stated purpose was to characterize the incoming bed-material load. However, methods described focus chiefly on the UTL, not the most characteristic or expected value. Therefore, please include methods for more fully characterizing the set of concentration values in each target area sampled for this evaluation.
- 8. Section 3: Visual and ultra-violet observations are not reported or discussed. These parameters are also absent from consideration in establishing an organic reference. Please provide the observations and a discussion of the results, as they relate to establishing an organic reference.
- 9. Section 3.1:
 - a. Section title: The material in this section is not primarily about sample locations; rather, it provides a general description of the appearance and lithology of sampled materials by target sampling area. Please rename the section as "General Descriptions of Samples by Targeted Area".

- b. Page 7:
 - i. Please provide range of core recoveries for each collection area.
 - ii. Please clarify whether the determination of sheen as biogenic was based on the lack of fluorescence under UV light.
- 10. Section 3.2:
 - a. General: The results from duplicate analyses shall be presented to convey the QA, uncertainty conclusions and interpretations. The sample size of QC pairs is likely too small to confidently report spatial patterns or relations with other variables unless they are strong and show little scatter. Please specific the minimum rate of QC sampling for bed material sample collections.
 - b. Page 8:
 - i. Please identify whether analytical results have been validated or if the results are unvalidated.
 - ii. On page 5 it was stated that field duplicates were treated as discrete samples and included in "the analysis" (although it is unclear which analysis), whereas on page 8, summaries are indicated to include only primary samples. Please clarify.
 - c. Pages 8 and 9:
 - i. Please present discussions about each analyte group in the same order for Talmadge, Kalamazoo, Battle Creek, and Marshall Impoundment. In addition, please separate discussions for TPH diesel range organics (DRO) and oil-range organics (ORO) by sampling area as well.
 - ii. Please present the background concentrations in the context of: (1) uncertainty (e.g., n-percent confidence intervals of means/geometric means, or medians & IQR), by target sampling area or C.I. of detection frequency by target sampling area; and (2) differences between target areas.
 - iii. Listings of individual concentration results do not help achieve the study objectives, and a graphical summary allowing readers to see the distribution would be more informative than the range alone. Therefore, please provide graphical representations of the concentrations.
 - iv. Please qualify the ranges listed to clarify that the range of detectable concentrations is listed, and not the total range in concentrations among the samples.

- v. Please provide the detection frequency (percentage) for each constituent.
- d. Page 9 and Section 3.3 (Page 12): The range of results for high bulk density and low TOC in samples collected from Talmadge Creek indicate that these cores are from bank or streambed environments and are not representative of the same type of settings that were sampled in the three samples collected from the Kalamazoo/riverine settings. In contrast, the sediment samples collected from the Kalamazoo River settings were more representative of fine-sediment deposition. Therefore, it is expected that statistical differences were observed between samples from the Talmadge Creek and the Kalamazoo River. Please revise the statistical analyses (Section 3.3) that examined relations between concentrations and grain-size categories to consider Talmadge Creek data separately from the data for the other three target sample areas.
- e. Page 9: Please provide an explanation for the presence of chloromethane in the sediment sample from immediately upstream of the Marshall impoundment.
- f. Pages 9 and 10: Neither the text nor Table 5 provide useful summaries by target sample area for the analytes determined from the "bulk density" core (second core collected at each sample site). Please provide an appropriate summary to understand the differences between the areas.
- 11. Section 3.3:
 - a. Page 10: Please provide correlation (continuous scales) or contingency table (categorical scales) to examine potential relations between pairs of variables. The box plots can be used to examine relations between concentration and grain size.
 - b. Metals, Page 11:
 - i. The report compares target sample areas in terms of medians; however, group medians are not listed in the table of summary statistics. Please add group medians to Table 6 for Talmadge Creek.
 - ii. Please add new tables to give group summary statistics for each of the other three target sample areas, and for each of the three grain-size groups.
 - iii. Please provide the actual values for beryllium, medians for molybdenum, TC median for DRO and BAP in order to support the given statistically significant differences.
 - iv. The report identifies "differences between grain size group," but summary statistics are not provided for grain size groups. Please provide a summary of statistics for grain size groups by adding a table(s).

- c. Other Parameters, Page 12:
 - i. Second to last paragraph:
 - 1. Please identify which analytes are the subject of the discussion regarding "regionally different" and "higher median results."
 - 2. The results presented clearly indicated that the Marshall Impoundment was significantly different than the Battle Creek and Kalamazoo River sample areas with respect to nickel concentrations, and given the higher concentrations of several analytes in samples from the fine-grained group, please examine whether grain size differed significantly among the target sample areas.
 - ii. Last paragraph:
 - 1. The inclusion of the Marshall Impoundment sample area with the Battle Creek and Kalamazoo River area samples to form a single group is not appropriate, particularly with respect to determining a reference condition for bed material entering the impacted area of the Kalamazoo River. The Marshall Impoundment area differed significantly in terms of one-fourth of the metals analyzed. Given its flat energy slope, it also is expected to differ in grain size distribution. The Marshall Impoundment sample area may provide a valid reference condition for comparison with bed material samples collected from the impoundments in the impacted area; however, it is unlikely that the Marshall Impoundment bed material is comparable generally to the bed material throughout the remainder of the impacted area. It is unlikely to be representative of the incoming bed load to the impacted reach, because impoundments generally trap a high percentage of the sand in the incoming sediment load, as well as all of the larger grain-size classes in the incoming sediment load. Further, the failure of multiple-comparison test to reject the null hypothesis of "no difference" does not provide conclusive evidence that the groups are from the same population. Please evaluate multivariate clustering techniques to determine how many statistically distinct groups are represented by the 4 targeted sample areas, and revise the text accordingly.
- 12. Section 3.3.1.1, Page 13: Using molybdenum in the Talmadge Creek sample area as an example, the Q-Q plot (Att. D, fig. 1) indicates that a concentration of 1 mg/kg was substituted for all 14 non-detections among the 29 total samples. Thus, the analysts apparently assumed and imposed a uniform distribution for approximately half of the data set. As a result, one cannot validly use that data set in a statistical test

for normality of the frequency distribution. Please reference the analysts to Helsel (2005, Non-detects and data analysis: Statistics for censored environmental data, Wiley, 250 p.). Serious errors are common when using simple substitution for censored values; and substitution yields the poorest estimates of statistical parameters when there are multiple detection limits present. It appears that the arithmetic mean (as indicated in margin of Att. D, fig. 1) from the data set is reported with value substitution, which is not a useful method to estimate the population mean. However, for the 95th percentile, they appear to report a Kaplan-Meier estimate. Using S-plus, a KM an estimate of the mean (2.2 mg/kg) and median (1.3 mg/kg) for molybdenum concentration for the TC data set, so for this analyte the KM estimate and "fabricated by substitution" estimate happen to be close; however, since Table 6 lists "nonparametric" as the frequency distribution type, then the median is the appropriate statistic to report as the "average" or expected value, and rather than reporting the standard deviation, the KM 95% confidence interval of the median should be reported. Although the above example is for molybdenum, this same evaluation shall be performed for all analytes.

- 13. Section 3.3.1 and 3.3.2, General: Background and reference concentrations calculated by MDEQ methods for the protection of aquatic life focus on the higher percentiles of the frequency distribution of concentrations of contaminants because of the objectives specific to their use of the calculated background or reference level. Reporting of those higher-percentile values in the report may support similar objectives for use. However, for the objective of identifying a characteristic expected concentration of an analyte in background or non-impacted bed sediment, the median of reported results is a more efficient estimator of central tendency. These sections appear to give significant emphasis to the 95% UTL, and inadequate emphasis to the expected concentration, or for nonparametric use, the median.
- 14. Section 3.3.2, General: This section shall be re-analyzed with separate analyses for: a) the Marshall Impoundment area; and b) the Kalamazoo River and Battle Creek combined as one analysis area. Also, as noted earlier, only one sample from each duplicate pair shall be included to represent a single sample location in the analyzed data set.
- 15. Section 3.3.2.1:
 - a. As expected, beryllium concentrations in streambed sediment were below reporting limits (i.e., <0.5 mg/kg). Given that beryllium was not detected above reporting limits, please explain: i) why the background concentration in the report was set to a concentration of 1.2 mg/kg, which is above the reporting limit; ii) if the use of 1.2 mg/kg as a background concentration for beryllium in sediment is in accordance with MDEQ guidance; and iii) how the beryllium concentration in oil-containing sediment (resulting from the Line 6B spill) compares to the proposed background concentration for beryllium.

- b. Molybdenum is a metal not commonly detected in sediment. However, it was detected at above-expected concentrations in samples from Talmadge Creek, but not from the Kalamazoo River sample locations. Please provide an explanation for the presence of molybdenum in multiple cores collected from Talmadge Creek.
- 16. Section 3.3.2.2, Page 15: The high concentrations of DRO and ORO in upstream sediment samples collected from the Kalamazoo River suggest that TPH cannot be used to quantify the amount of oil present (resulting from the Line 6B spill) in the Kalamazoo River due to potential interferences of naturally occurring organic matter and/or historical contamination from sources other than the Line 6B spill.
- 17. Attachment B:
 - a. Please provide box plots for Total Organic Carbon (TOC) and bulk density.
 - b. Please provide a clearer labeling of the summarized groups on Figures 7 through 12 as the USCS codes are not commonly used acronyms.
 - c. Please provide an explanation or "key" for the box plots. There are several variants, distinguished by the methods used to delimit whiskers and classify the outside values plotted as points.
- 18. Attachment D, Figures 1 through 58: Please add clarifying labels to indicate whether graphs are plotting results for Talmadge Creek sample area (cf. section 3.3.1) or one of the Kalamazoo River or Battle Creek sample areas (cf. section 3.3.2).
- 19. Tables:
 - a. Tables 1 through 4:
 - i. Please identify if the reporting limits for reported non-detects are adjusted for percent moisture.
 - ii. Please identify reporting limits for results reported as non-detect.
 - b. Table 5:
 - i. "Sample volume" column has mass density units rather than volume units. Please revise.
 - ii. Please clarify whether moisture, particle size, and TOC units are percent by weight or percent by volume.

- c. Table 6:
 - i. Please explain why there is a maximum value shown when all the results were non-detect (i.e., below reporting limits).
 - ii. Please add group medians to Table 6.
 - iii. Please clarify the column labeled "Average." Explain whether this is an arithmetic mean, geometric mean, weighted average, or some other type of average statistic.
- d. Tables 6 and 7: Both tables list a maximum value for GRO concentration in sampled bed sediment, but also list a 100% rate of non-detection of GRO. Please revise and clarify.
- 20. General:
 - a. Please identify if split sediment samples analyzed by the Michigan Department of Environmental Quality (MDEQ) were considered in the evaluations presented in the report.
 - b. Some samples appear to have been analyzed for Fraction Organic Content (FOC) while others were analyzed for TOC. Please clearly identify which samples were analyzed for each analysis and the rationale for the method used. Also please explain any correlation and/or comparability, if any, between TOC and FOC in the samples analyzed.
 - c. Please provide a method reference for the TOC analysis utilized.

Please submit five copies of the revised report, as modified, to U.S. EPA no later than 17:00 hours Eastern, January 5, 2012. The document shall also be concurrently submitted electronically in Microsoft Word format for the text and in Microsoft Excel for spreadsheet/graphs/tables.

If you have any questions regarding this conditional approval, please contact me immediately at (231) 301-0559.

Sincerely,

ul. D.F

Ralph Dollhopf Federal On-Scene Coordinator and Incident Commander U.S. EPA, Region 5

cc: L. Kirby-Miles, U.S. EPA, ORC M. Durno, U.S. EPA, Dep. IC, Section Chief S. Vega, U.S. EPA Records Center, U.S. EPA, Reg. V M. Ducharme, MDEQ M. Alexander, MDEQ M. Delong, MDEQ