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**Rich Adams**
Vice President, U.S. Operations


November 13, 2012

**VIA EMAIL & OVERNIGHT DELIVERY**

Richard Karl
Superfund Division Director
US EPA Region 5
77 W. Jackson Blvd.
Mail Code: S-6J
Chicago, IL 60604-3507

**VIA EMAIL & HAND DELIVERY**

Ralph Dollhopf
Federal On-Scene Coordinator and Incident Commander
U.S. Environmental Protection Agency
801 Garfield Avenue, #229
Traverse City, MI 49686

**RE: In the Matter of Enbridge Energy Partners, L.P., *et al.***
**Docket No. SWA 1321-5-10-001**

Dear Mr. Karl and Mr. Dollhopf:

Enbridge Energy, Limited Partnership ("Enbridge") sets forth in the attached report views on the recent study by Kenneth Lee et al. entitled "UV-Epifluorescence Microscopy Analysis of Sediments Recovered from the Kalamazoo River." Enbridge first received a copy of the Lee study from EPA on November 1, 2012. Given the limited amount of time to comment made available, a complete analysis of the study has not been possible. Nonetheless, as is set forth in the attached report, Enbridge believes that the Lee study establishes the likelihood that the quantity of oil remaining in the affected areas of the River is limited to trace amounts not visible even with the use of sophisticated techniques such as UV screening. Further, the trace amounts of oil remaining appear to be undergoing conversion to oil-mineral aggregates (OMA) as part of a process of natural attenuation.

We look forward to the opportunity to further discuss our views with you.

Sincerely,

ENBRIDGE ENERGY, LIMITED PARTNERSHIP
By Enbridge Pipelines (Lakehead) L.L.C.
Its General Partner

Richard L. Adams
Vice President, U.S. Field Operations

cc: Robert Kaplan, U.S. EPA, Region 5 (via email only)
Leslie Kirby-Miles, U.S. EPA, Region 5 (via email only)
Michelle DeLong, MDEQ (via email only)
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THIRD PARTY REVIEW OF COOGER EPIFLUORESCENCE MICROSCOPY STUDY

# FOR THE KALAMAZOO RIVER OIL SPILL

At the request of Enbridge Energy, Limited Partnership (Enbridge), a third party review was conducted of the Centre for Offshore Oil, Gas and Energy Research (COOGER) UV-Epifluorescence Microscopy Analysis of Sediments Recovered from the Kalamazoo River (**UV Microscopy Report**) dated October 24, 2012. The report provides a detailed description of the epifluorescence and conventional microscopic analysis of crude oil in sediments and the formation of oil-mineral-aggregates (OMA). Previous studies of OMA formation as a remediation process have demonstrated that both mineral fines and organic particles can stabilize oil droplets in the water column. The formation of OMA reduces the adhesion of oil to river sediments and increases the oil-water interface where microbial activity primarily occurs. Epifluorescence is useful in verifying the formation of OMA within sediments. The study also attempted to correlate epifluorescence analysis with gas chromatographic analysis and found that fluorescence quenching and low oil concentrations prevented reliable quantification by epifluorescence.

The general observations noted below were compiled from comments provided by separate third-party reviewers.

**Study Objectives**

- One of the objectives of the study was to determine if UV-epifluorescence microscopy is a useful tool for determining which sediment interval to sample for subsequent laboratory analysis in sediment cores. The **UV Microscopy Report** concludes:

  "it appears that in the initial stages of the cleanup operations, UV-fluorescence was able to highlight the presence of bulk oil within the split sediment cores. However, as cleanup operations proceeded by the use of strategies such as sediment agitation, oil within the sediments was reduced to low concentrations by a combination of the recovery of the mobilized oil, and dilution and dispersion of oil within the sediments through the natural process of OMA formation. This, coupled with quenching of dispersed oil droplets, resulted in our subsequent inability to detect traces of the residual oil by image analysis of whole sediment cores under UV illumination."

  Therefore, UV-epifluorescence microscopy is not selected for future use to assist with the selection of sample intervals for chemical analysis.

- At the onset of the study, it was hypothesized that the agitation procedure used in the spill cleanup operations in the Kalamazoo River following the Line 6B spill resulted in a significant amount of OMA formation. The **UV Microscopy Report** documents that when the crude oil from Line 6B is mixed with sediment from the Kalamazoo River that the oil will form OMA (Section 4.2). OMA formation is between colloidal particles and oil, increasing the amount of emulsified mass of oil and increasing its dispersion and eventual biodegradation. The study showed that the OMA are stable based on observations of the spiked sample after two days and concludes that "oil within the sediments was reduced to low concentrations by a combination of the recovery of the mobilized oil, and dilution and dispersion of oil within the sediments through the natural process of OMA formation". "Thus, OMA formation is an integral part of natural attenuation process..."

**Omitted Information**

- The epifluorescence study did not quantify total organic carbon in sediments to evaluate its potential influence on OMA formation and on UV-fluorescence.

**Identified Inaccuracies (Selective)**

- On page 3 the report states that "it was noted that oil was no longer observed in the analysis of split cores under UV illumination".
    - Residual amounts of oil were observed under UV illumination in a small percentage of cores collected for quantification of oil.
- Section 2 eludes that UV fluorescence was intended to quantify oil in sediment in several places such as on pages 2 and 3.
    - The general intentions of the study were to determine if UV-epifluorescence microscopy is a useful tool for determining which sediment interval to sample for subsequent laboratory analysis in sediment cores and to document that Line 6B oil forms OMA in the presence of Kalamazoo River sediment. While quantification was not the intent of the study, the UV Microscopy Report nonetheless shows that the volume of oil was very small.
- On page 18 the report states that "Alpha Labs was capable of distinguishing line 6B oil from other potential oil sources detected in the samples."
    - Alpha Labs is not capable of distinguishing between Line 6B oil and other sources, this requires more detailed forensic analysis.
- On page 18 the report states that "Figure 18 illustrates the graphical distribution of total oil (a different data set than the TPH values used in Figure 17), line 6B oil, contributions of the sum of saturates and aromatics, and the sum of resins and asphaltenes in the samples collected in the vicinity of the spill."
    - Figure 18 does not adequately differentiate Line 6b oil from all other sources of oil in the river. These data were provided to Dr. Lee prior to the completion of the forensic chemist analysis.

**General Conclusions**

- In general, the study reaffirms that any remaining oil in the sediments is at trace concentrations, below detection limits of typical investigation techniques such as UV screening.
- The study has confirmed that residual oil has formed OMA, which is an integral part of natural attenuation process.
- Epifluorescence has limited application to the analysis of oil in sediments as a tool to qualitatively assess the presence and distribution of oil. As the authors acknowledge, quantitative analysis with epifluorescence requires extensive evaluation of a large quantity of slide fields in order to have a statistically valid representation of the small sediment sample placed on a slide. The method involves analysis of a few hundredths to tenths of a gram of sediment at most, making extrapolation to field

concentrations practically impossible. Sample selection is likely to skew the analysis either in favor of sediments with oil or towards clean sediments.

- Although epifluorescence is a powerful tool for the evaluation of fluorescent compounds in various matrixes, its utility for quantifying oils in sediments is limited. This tool is best suited for evaluating the associations between sediment particles and oil and assessing the functional groups in the fluorescence molecules and how they influence interactions with soil minerals.

- Due to the limited number of samples that scored positive for oil under UV-epifluorescence microscopy and the relatively low level of oil fluorescence observed as oil droplets within OMA, no correlation or limited correlation could be made between OMA and chemical analyses. The inability to observe the oil in the sediment cores is attributed to the cleanup operations that have been performed along with the weathering of the small amounts of remaining oil.

- The UV Microscopy Report confirms the presence of contaminant petroleum hydrocarbons from multiple sources (non Line 6B) within the sediments.

Contributors:

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