August 8, 2012

Mr. Ralph Dollhopf
Federal OSC and Incident Commander
U.S. EPA, Region 5
Emergency Response Branch
801 Garfield Avenue, #229
Traverse City, MI 49686

Re: Experiments for Evaluating the Efficacy and Potential Ecological Effects of In-Situ Sediment Agitation (Summer 2012)
Effects of Sediment Agitation During Oil Recovery in Kalamazoo River Sediments
Enbridge Line 6B MP 608, Marshall, MI Pipeline Release

Dear Mr. Dollhopf,

Attached are my recommendations for methodologies to further evaluate the potential ecological effects of agitation during the response to the Enbridge Line 6B Oil Spill, based on the individual scientific opinions that I have received. Recommendations herein are for physical experiments as part of the previously recommended Tier II evaluation to assess the potential effects of agitation, as described in a March 21, 2012 submittal to you. The attached documents represent additional response to the Federal On-Scene Coordinator's (FOSC) Charge No. 4:

> *Identify viable procedures to assess the potential for adverse ecological effects resulting from further oil recovery using sediment agitation ("toolbox") techniques.*

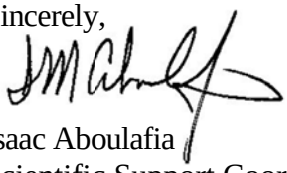
The attached two documents represent my synthesis (as a Scientific Support Coordinator) of the applicable opinions and recommendations received from individuals involved with the Effects of Agitation Subgroup (a subgroup of the Ecological Risk and Toxicity Subgroup) of the Scientific Support Coordination Group (SSCG). The following two documents are attached:

> ***Evaluating the Efficacy and Potential Ecological Effects of In-Situ Sediment Agitation (Summer 2012), August 7, 2012***
>
> ***Toxicological Assessment of the Effects of Residual Weathered Oil and Increased Suspended Solids Resulting from Sediment Agitation, August 7, 2012***

Each individual scientist's opinion was provided to me based on his or her prior experiences in addressing issues related to oil spill recovery and potential effects of recovery. Opinions expressed by individuals from the SSCG and its subgroup are included in the attached documents, or are otherwise documented in supporting documents maintained in the response files.

I recommend adoption of this technical approach to further develop the understanding of potential effects of sediment agitation for the purposes of oil recovery from the Kalamazoo River.

Sincerely,



Isaac Aboulafia
Scientific Support Coordinator to the FOSC for Enbridge Line 6B Oil Spill
Sr. Vice President, MEC[X], LP, a Weston Solutions Region 5 S.T.A.R.T. Subcontractor

SSC RECOMMENDATION TO THE FOSC

EVALUATING THE EFFICACY AND POTENTIAL ECOLOGICAL EFFECTS OF IN-SITU SEDIMENT AGITATION (SUMMER 2012)

ENBRIDGE LINE 6B MP 608 MARSHALL, MI PIPELINE RELEASE
AUGUST 8, 2012

BACKGROUND

Kalamazoo River sediment has been extensively agitated as part of the "agitation toolbox" techniques used to recover submerged oil after the July 2010 Enbridge Line 6B release. The toolbox techniques were approved by the U.S. EPA for use on the Enbridge Line 6B oil recovery operations and represent a set of recovery techniques intended to minimize potential impacts to the environment while liberating and recovering submerged oil.

The toolbox techniques have already been used in many depositional areas along the 40 miles of affected river, but particularly in the impounded areas and most extensively in the delta of Morrow Lake in 2011. However, direct quantitative determination of the efficacy and the potential adverse ecological effects of using agitation techniques have not been performed. The potential adverse effects include, but are not necessarily limited to: increased toxicity to aquatic organisms from residual oil suspended in the water column and transported downstream; increased turbidity and downstream burial and smothering of sand, silt, and clay associated with the agitation process; and increased erodibility of residual oil and sediment on the streambed following agitation.

On March 21, 2012, individual experts participating in the Scientific Support Coordination Group (SSCG) recommended implementation of the following summarized Tier I and II evaluations to assess the potential ecological effects of sediment agitation techniques:

- Tier I: review of existing project data (chemistry, toxicity, etc.) and other published literature regarding the potential effects of sediment agitation;
- Tier II: perform bench-scale and/or field applications of agitation techniques while simultaneously collecting water quality measurements and collecting samples for analyses.

As part of the Tier II activities, the field evaluation described herein is intended to provide the following information to evaluate the potential consequences of the sediment agitation toolbox techniques that may be used in future oil recovery operations during the Enbridge Line 6B response to recover submerged oil in the Kalamazoo River.

This document represents my synthesis of the opinions and recommendations that I received from individual scientists and engineers of the SSCG.

EVALUATION GOALS

The overall goals of the evaluation are to describe the chemical and physical traits of submerged oil and sediment in three riverine components (sheen, suspension and bottom) before, during, and after agitation for representative geomorphic settings where submerged oil resides in the Kalamazoo River. Chemical and potential toxicity differences among the components shall be identified. The time course of settling of the resuspended particulate matter and the oil associated with it will help understand how it may be transported downstream, and will inform the hydrodynamic modeling of sediment/oil dynamics. Such information can only be obtained with empirical observations because the physical characteristics of the submerged oil in the particulate (or globule) fraction are not known.

Specific goals of the evaluation are:

A. To determine the relative proportions of submerged oil that appear on the water surface as sheen versus the amount re-suspended in the water column (dissolved or on particulate matter) as well as the amount of residual oil left in the bottom sediment after agitation.
B. To estimate the worst case toxicity (i.e., in stagnant water not subject to dilution via flow) of resuspended particulate matter as inferred based on chemical characterization.
C. To provide an estimate of the physical components of resuspended material in the absence of current and wind to better characterize the traits and weathering of submerged oil and inform the hydrodynamic modeling.

BENEFITS AND USES OF THE EVALUATION RESULTS

Results from these evaluations will aid future decision-making by enabling the comparison of the environmental benefits and risks associated with potential agitation toolbox use, as developed in the Net Environmental Benefits Analysis (NEBA). These results will be particularly useful for:

- Evaluating recovery options in Morrow Lake delta, Morrow Lake fan and in Morrow Lake, where residual submerged oil persists across broad areas.
- Providing sediment particle size, concentration and settling velocities to better represent river sediment conditions in the hydrodynamic model.
- Aiding in understanding the migration of submerged oil in Morrow Lake delta and fan following the intensive agitation work in 2011.
- Better characterizing the oiled sediment characteristics with sediment/turbidity data for future scenarios of the hydrodynamic model related to oil migration in the delta and lake, especially for a 3-D version.

EVALUATION ENCLOSURES

These tests shall be conducted in in-situ enclosures, or mesocosms, of cylindrical or oval shape with an equivalent circular diameter of approximately 4-5 feet. Enclosing the system is the only way to quantify the fractions of oil appearing as sheen compared to resuspended material in the water column. While an enclosure precludes the effects of current and waves in maintaining material in suspension and thus provides conditions more conducive to sedimentation, it offers an environment of quiescent conditions that is comparable across various evaluation runs and

unaffected by the variable flow and wind conditions that would be encountered during the evaluation.

Flexible plastic sheets or rigid cattle tanks with the bottoms cut out are possibilities for the enclosure design. The enclosure shall be small and light enough to carry and handle in a boat, ideally stackable and adjustable to different water depths, but large enough to allow easy access with agitation and sampling equipment, to minimize the wall effects, and to cover enough sediment for sampling.

LOCATIONS OF THE EVALUATION

The tests shall be conducted at 6 U.S. EPA-approved locations (for illustration purposes, identified as locations A through F herein) in water depths of 1-3.5 feet and in places where the presence of submerged oil is indicated by production of moderate to heavy sheen in response to poling. The Morrow Lake Delta, where water is generally less than 3 feet deep and submerged oil is likely to be present, is a particularly critical place to perform the evaluation described herein; other possibilities also exist including the upriver end of the Ceresco impoundment. These are the locations likely to require future oil recovery operations, possibly by agitation. Each of the 6 locations (A through F) shall have an evaluation cell (agitated) enclosure.

Ideally, a diversity of locations shall be tested that span the variable sediment characteristics (grain texture and organic matter content), geomorphic settings and recovery history that exist within the areas targeted for agitation and NEBA rankings in 2012. The evaluation described herein should precede future agitation or other toolbox activity in the vicinity. Locations that overlap with other assessments and activities shall be prioritized.

The full suite of laboratory analyses will be conducted initially on 3 (locations A, B, C) of the 6 (locations A through F) experiment locations, with the other samples (locations D, E, F) preserved appropriately. The lab results will be evaluated to consider whether high variability demands analysis of samples from the other 3 experiments (D, E, F) to reach conclusions.

TEST PROCEDURE

The approach is to conduct agitation within enclosures using a method similar to the "stinger" water jet that is part of the existing agitation toolbox, followed immediately by sampling of the water column, with additional samplings at various times afterward up to 4 hours (see also the sampling and measurement schedule in Attachment 1).

The following step-wise procedure should be employed to execute the evaluation at each location.

Experiment Day 1 at Each Location

1. After verifying that water temperatures are conducive to submerged oil agitation techniques as determined by the FOSC (i.e., >60°F), use poling to locate areas of the appropriate water depth (< 3.5 feet) that yield moderate to heavy sheen and thus contain submerged oil. Mark exact poled locations with floats or other appropriate devices and record GPS coordinates. Record water, sediment and air temperatures prior to and following the evaluation.

2. The evaluations would best be conducted under calm conditions and stable river levels, with good underwater visibility.
3. Moor the boat securely in place using at least two anchors, facing upstream alongside the target site.
4. Form the enclosure by placing a premade enclosure into the sediments close to, but not on top of, the poled location. Orient the long axis with the current if the enclosure is oval to reduce pressure from the flowing water. Drill, or otherwise cut round openings on opposite sides of the enclosures and temporarily seal the holes using bungs or threaded PVC fittings. These openings could be opened later to permit some water flow-through during the stabilization period, if it was determined desirable by the U.S. EPA or its representatives during execution of the evaluation (e.g., to equilibrate water levels inside and outside of the enclosure).

   Push the enclosure down through soft sediment to the underlying consolidated layer to form the enclosure. Be sure to leave at least 6 inches of the enclosure as "freeboard" (i.e., extending the above the top of water surface).

   a. Use at least two fence posts and polypropylene rope to hold the enclosure in place.
   b. Place a flag alongside the enclosure to mark the location to minimize potential navigation hazard. Other measures as appropriate shall be taken to minimize navigation hazards and other potential Health and Safety related concerns.
   c. If using cattle tanks, stack two enclosures (top one inverted) and affix them with C clamps or similar devices if the water depth warrants a taller enclosure.
   d. If significant resuspension of the sediments occurs during installation, leave them alone for at least an hour before commencing with the pre-agitation sampling.
5. Perform **poling** at four points outside of but close to (i.e., within 0.5 m) of the enclosure and record sheen intensity as per the U.S. EPA-approved SOPs. Record the depth to the top of the sediment surface and bottom of sediment surface/top of firm sediments using the U.S. EPA-approved poling procedures. Record water, sediment and air temperatures.
6. Collect **depth-integrated and water-surface (sheen) samples** exactly as described below *before* ("pre-agitation") as well as immediately after agitation to show background conditions. Record water, sediment and air temperatures. Conduct **pre-agitation turbidity** and **Laser In-Situ Scattering and Transmissometry** (**LISST)** for concentration and particle size distribution profiles. Note GPS coordinates of each enclosure. Photograph water surface inside each enclosure. This shall be done for all locations A through F.
7. Collect 3 **"Day-1 pre-agitation" cores** from within the evaluation as per the SSCG sediment coring work plan (in preparation), sampling on one side of the enclosure to leave the other side for later core sampling. Coring should not be done close to the enclosure walls where the sediment was disturbed. Record water depth, penetration

depth, depth to refusal (1-, 2-hand push) recovery ratio, corer type. This shall be done for all locations A through F.

8. Using a water pump for agitation, place the pump intake outside of the enclosure. Agitate the upper 6-12 inches of sediments using techniques similar to 2011 recovery efforts for at least 2 minutes, until further liberation of sheen stops or until the top of the water level inside the mesocosm is 4 inches below the top of the enclosure. Avoid agitation of the sediments along the edges of the enclosure, which may destabilize it. This shall be done for all locations A through F.
    a. Be careful to avoid contamination from gasoline or oil used for the pump or boat motor.
    b. As agitation is performed, the enclosure may need to be pushed downward to maintain a seal with the sediment surface.
    c. Record the equipment used for agitation and the associated operational parameters. This shall include, but not be limited to: pump manufacturer; model number; inlet size; outlet size; types and length of pipes/hoses used; actual flow rates observed; pump curves; operational parameters (height difference between the pump inlet and final discharge); distance from the outlet to the top of the sediment layer. This shall be done for all locations A through F.
9. Immediately after agitation ceases ("time zero" on Attachment 1):
    a. Collect an **integrated sample of the water column** using a vertical tube water column sampler (or 3 Van Dorn samplers placed in each third of the vertical column and used simultaneously), avoiding sheen and transfer contents to a new and properly decontaminated carboy for sub-sampling. Do not approach the sediment surface with the sampler. This shall be done for all locations A through F. Repeat sampling quickly if necessary to obtain at least 10 L of water.
        i. If the water is <1 foot deep, collect water from the middle of the water column using a horizontally mounted Van Dorn sampler.
    b. Record the time, location and sampling depth. Photograph water surface inside enclosure.
    c. Immediately after agitation ceases measure **turbidity** every 6 to 12 inches (minimum 4 different representative depths if deeper than 2 feet) using a properly calibrated field meter (in accordance with the approved Sampling and Analysis Plan). This could be done at the same time as the water sampling. Maintain the probe in the center of the enclosure, away from the edges. This shall be done for all locations A through F.
    d. Measure a vertical profile of **dissolved oxygen** at the same depths as turbidity is measured. This shall be done for all locations A through F.
    e. Measure vertical profile of particle sizes and concentration using the **LISST instrument**. This shall be done for all locations A through F.

10. Collect all of the **sheen** using the sheen skimmer kit using SSCG's sheen collection SOP. Continue to collect any significant sheen that continues to appear later during the experiment, up until the end of the experiment when sediments are again disturbed by coring. This shall be done for all locations A through F.

11. Thoroughly but gently mix the water-column sample in the properly decontaminated churn splitter, and collect subsamples for the following laboratory analyses from at least 3 (A, B, C; which shall include at least one sample from the Morrow Lake Delta and one from the Ceresco impoundment) of the 6 sample locations:

    a. **Total suspended solids** (0.5 L).

    b. **Chemical fingerprinting** (at least three 1-L samples; or other minimum amount dictated by the laboratory performing the chemical analyses). Particulate matter shall be collected on a filter (pre-cleaned 0.7-µm glass fiber or Teflon). This particulate matter and the filtrate (filtered water) sample shall be analyzed for sediment-associated and dissolved oil-derived compounds. Total organic carbon (TOC) should also be analyzed. Analytical methods and data quality objectives are listed in SSC's analytical chemistry QAP.

    c. Perform **epifluorescence UV microscopy** of oil-sediment particles to determine size and concentration of oiled particles in the resuspended sediment.

    d. The remainder shall of water from locations A through C shall be transferred to a properly decontaminated container for possible additional laboratory analyses.

    The water column samples from locations D, E and F shall be mixed and prepared for analysis similar to samples from locations A, B and C. However, freeze and archive the remaining water samples from locations D, E and F, where hydrocarbon chemistry is not currently being performed, at -20°C until otherwise directed by the U.S. EPA. The need for analysis of these samples will be determined after reviewing results from the first 3 locations analyzed.

12. At t=15 minutes, collect another depth-integrated water sample as well as another round of turbidity, LISST, and oxygen measurements at the same depths as before. Record the time and depth for each reading. This will provide a measure of the rate at which the resuspended particulate matter settles out of the water column.

13. Repeat above (turbidity, LISST and oxygen measurements) at t=30 minutes, and t=1 hour, 2 hours, and 4 hours.

    a. If significant turbidity (i.e., ≥125% of the original measurement) is apparent at t=4 hours, repeat turbidity, LISST and oxygen measurements at t=6 hours.

14. After the t=4 hour sampling (or t=6 hour, if collected), collect 3 **"Day-1 post-agitation" cores** from within the evaluation enclosure as per SOPs. Avoid the part of the enclosure footprint that was sampled for the pre-agitation cores. These cores will also serve as "**Day-2 pre-agitation**" cores.

15. At the completion of the monitoring period and measurements on Day 1, place buoys and other safety devices in the vicinity of the mesocosm test chamber and allow the chamber to remain undisturbed overnight. This would require coordination with Safety and approval by the Michigan Department of Environmental Quality (MDEQ).

Experiment Day 2 at Each Location

1. Record water, sediment and air temperatures prior to and following the evaluation.
2. Moor the boat securely in place using at least two anchors, facing upstream alongside the target site.
3. Carefully decant water from inside the mesocosm back into the open water until the water level inside the mesocosm is the same as pre-agitation levels on Day-1. Methods used to remove the water should prevent, to the maximum extent practical, disturbance of the sediment.
4. Collect **depth-integrated and water-surface (sheen) samples** exactly as described below *before* ("pre-agitation") as well as immediately after agitation to show background conditions. Record water, sediment and air temperatures. Conduct **pre-agitation turbidity** and **LISST** for concentration and particle size distribution profiles. Note GPS coordinates of each enclosure. Photograph water surface inside each enclosure. This shall be done for all locations A through F.
5. Using a water pump for agitation, place the pump intake outside of the enclosure. Agitate the upper 6-12 inches of sediments using techniques similar to 2011 recovery efforts for at least 2 minutes, until further liberation of sheen stops or until the top of the water level inside the mesocosm is 4 inches below the top of the enclosure. Avoid agitation of the sediments along the edges of the enclosure, which may destabilize it. This shall be done for all locations A through F.
    a. Be careful to avoid contamination from gasoline or oil used for the pump or boat motor.
    b. As agitation is performed, the enclosure may need to be pushed downward to maintain a seal with the sediment surface.
    c. Record the equipment used for agitation and the associated operational parameters. This shall include, but not be limited to: pump manufacturer; model number; inlet size; outlet size; types and length of pipes/hoses used; actual flow rates observed; pump curves; operational parameters (height difference between the pump inlet and final discharge); distance from the outlet to the top of the sediment layer. This shall be done for all locations A through F.
6. Immediately after agitation ceases ("time zero" on Attachment 1):
    a. Collect an **integrated sample of the water column** using a vertical tube water column sampler (or 3 Van Dorn samplers placed in each third of the vertical column and used simultaneously), avoiding sheen and transfer contents to a new and properly decontaminated carboy for sub-sampling. Do not approach the sediment surface with

the sampler. This shall be done for all locations A through F. Repeat sampling quickly if necessary to obtain at least 10 L of water.

i. If the water is <1 foot deep, collect water from the middle of the water column using a horizontally mounted Van Dorn sampler.

b. Record the time, location and sampling depth. Photograph water surface inside enclosure.

c. Immediately after agitation ceases measure **turbidity** every 6 to 12 inches (minimum 4 different representative depths if deeper than 2 feet) using a properly calibrated field meter (in accordance with the approved Sampling and Analysis Plan). This could be done at the same time as the water sampling. Maintain the probe in the center of the enclosure, away from the edges. This shall be done for all locations A through F.

d. Measure a vertical profile of **dissolved oxygen** at the same depths as turbidity is measured. This shall be done for all locations A through F.

e. Measure vertical profile of particle sizes and concentration using the **LISST instrument**. This shall be done for all locations A through F.

7. Collect all of the **sheen** using the sheen skimmer kit using SSCG's sheen collection SOP. Continue to collect any significant sheen that continues to appear later during the experiment, up until the end of the experiment when sediments are again disturbed by coring. This shall be done for all locations A through F.

8. Thoroughly but gently mix the water-column sample in the properly decontaminated churn splitter, and collect subsamples for the following laboratory analyses from at least 3 (A, B, C; which shall include at least one sample from the Morrow Lake Delta and one from the Ceresco impoundment) of the 6 sample locations:

   a. **Total suspended solids** (0.5 L).

   b. **Chemical fingerprinting** (at least three 1-L samples; or other minimum amount dictated by the laboratory performing the chemical analyses). Particulate matter shall be collected on a filter (pre-cleaned 0.7-µm glass fiber or Teflon). This particulate matter and the filtrate (filtered water) sample shall be analyzed for sediment-associated and dissolved oil-derived compounds. Total organic carbon (TOC) should also be analyzed. Analytical methods and data quality objectives are listed in SSC's analytical chemistry QAP.

   c. Perform **epifluorescence UV microscopy** of oil-sediment particles to determine size and concentration of oiled particles in the resuspended sediment.

   d. The remainder shall of water from locations A through C shall be transferred to a properly decontaminated container for possible additional laboratory analyses.

   The water column samples from locations D, E and F shall be mixed and prepared for analysis similar to samples from locations A, B and C. However, freeze and archive the remaining water samples from locations D, E and F, where hydrocarbon chemistry is not

currently being performed, at -20°C until otherwise directed by the U.S. EPA. The need for analysis of these samples will be determined after reviewing results from the first 3 locations analyzed.

9. At t=15 minutes, collect another depth-integrated water sample as well as another round of turbidity, LISST, and oxygen measurements at the same depths as before. Record the time and depth for each reading. This will provide a measure of the rate at which the resuspended particulate matter settles out of the water column.
10. Repeat above (turbidity, LISST and oxygen measurements) at t=30 minutes, and t=1 hour, 2 hours, and 4 hours.
    a. If significant turbidity (i.e., ≥125% of the original measurement) is apparent at t=4 hours, repeat turbidity, LISST and oxygen measurements at t=6 hours.
11. After the t=4 hour sampling (or t=6 hour, if collected), collect 3 **"Day-2 post-agitation" cores** from within the evaluation enclosure as per SOPs. Avoid the part of the enclosure footprint were sediment cores were collected on Day-1. These cores will also serve as "**Day-3 pre-agitation**" cores.
12. At the completion of the monitoring period and measurements on Day 2, place buoys and other safety devices in the vicinity of the mesocosm test chamber and allow the chamber to remain undisturbed overnight. This would require coordination with Safety and approval by the MDEQ.

Experiment Day 3 at Each Location

1. Record water, sediment and air temperatures prior to and following the evaluation.
2. Moor the boat securely in place using at least two anchors, facing upstream alongside the target site.
3. Carefully decant water from inside the mesocosm back into the open water until the water level inside the mesocosm is the same as pre-agitation levels on Day-1. Methods used to remove the water should prevent, to the maximum extent practical, disturbance of the sediment.
4. Collect **depth-integrated and water-surface (sheen) samples** exactly as described below *before* ("pre-agitation") as well as immediately after agitation to show background conditions. Record water, sediment and air temperatures. Conduct **pre-agitation turbidity** and **LISST** for concentration and particle size distribution profiles. Note GPS coordinates of each enclosure. Photograph water surface inside each enclosure. This shall be done for all locations A through F.
5. Using a water pump for agitation, place the pump intake outside of the enclosure. Agitate the upper 6-12 inches of sediments using techniques similar to 2011 recovery efforts for at least 2 minutes, until further liberation of sheen stops or until the top of the water level inside the mesocosm is 4 inches below the top of the enclosure. Avoid agitation of the sediments along the edges of the enclosure, which may destabilize it. This shall be done for all locations A through F.

a. Be careful to avoid contamination from gasoline or oil used for the pump or boat motor.

b. As agitation is performed, the enclosure may need to be pushed downward to maintain a seal with the sediment surface.

c. Record the equipment used for agitation and the associated operational parameters. This shall include, but not be limited to: pump manufacturer; model number; inlet size; outlet size; types and length of pipes/hoses used; actual flow rates observed; pump curves; operational parameters (height difference between the pump inlet and final discharge); distance from the outlet to the top of the sediment layer. This shall be done for all locations A through F.

6. Immediately after agitation ceases ("time zero" on Attachment 1):

   a. Collect an **integrated sample of the water column** using a vertical tube water column sampler (or 3 Van Dorn samplers placed in each third of the vertical column and used simultaneously), avoiding sheen and transfer contents to a new and properly decontaminated carboy for sub-sampling. Do not approach the sediment surface with the sampler. This shall be done for all locations A through F. Repeat sampling quickly if necessary to obtain at least 10 L of water.

      i. If the water is <1 foot deep, collect water from the middle of the water column using a horizontally mounted Van Dorn sampler.

   b. Record the time, location and sampling depth. Photograph water surface inside enclosure.

   c. Immediately after agitation ceases measure **turbidity** every 6 to 12 inches (minimum 4 different representative depths if deeper than 2 feet) using a properly calibrated field meter (in accordance with the approved Sampling and Analysis Plan). This could be done at the same time as the water sampling. Maintain the probe in the center of the enclosure, away from the edges. This shall be done for all locations A through F.

   d. Measure a vertical profile of **dissolved oxygen** at the same depths as turbidity is measured. This shall be done for all locations A through F.

   e. Measure vertical profile of particle sizes and concentration using the **LISST instrument**. This shall be done for all locations A through F.

7. Collect all of the **sheen** using the sheen skimmer kit using SSCG's sheen collection SOP. Continue to collect any significant sheen that continues to appear later during the experiment, up until the end of the experiment when sediments are again disturbed by coring. This shall be done for all locations A through F.

8. Thoroughly but gently mix the water-column sample in the properly decontaminated churn splitter, and collect subsamples for the following laboratory analyses from at least 3 (A, B, C; which shall include at least one sample from the Morrow Lake Delta and one from the Ceresco impoundment) of the 6 sample locations:

a. **Total suspended solids** (0.5 L).

b. **Chemical fingerprinting** (at least three 1-L samples; or other minimum amount dictated by the laboratory performing the chemical analyses). Particulate matter shall be collected on a filter (pre-cleaned 0.7-µm glass fiber or Teflon). This particulate matter and the filtrate (filtered water) sample shall be analyzed for sediment-associated and dissolved oil-derived compounds. Total organic carbon (TOC) should also be analyzed. Analytical methods and data quality objectives are listed in SSC's analytical chemistry QAP.

c. Perform **epifluorescence UV microscopy** of oil-sediment particles to determine size and concentration of oiled particles in the resuspended sediment.

d. The remainder shall of water from locations A through C shall be transferred to a properly decontaminated container for possible additional laboratory analyses.

The water column samples from locations D, E and F shall be mixed and prepared for analysis similar to samples from locations A, B and C. However, freeze and archive the remaining water samples from locations D, E and F, where hydrocarbon chemistry is not currently being performed, at -20°C until otherwise directed by the U.S. EPA. The need for analysis of these samples will be determined after reviewing results from the first 3 locations analyzed.

9. At t=15 minutes, collect another depth-integrated water sample as well as another round of turbidity, LISST, and oxygen measurements at the same depths as before. Record the time and depth for each reading. This will provide a measure of the rate at which the resuspended particulate matter settles out of the water column.
10. Repeat above (turbidity, LISST and oxygen measurements) at t=30 minutes, and t=1 hour, 2 hours, and 4 hours.
    a. If significant turbidity (i.e., ≥125% of the original measurement) is apparent at t=4 hours, repeat turbidity, LISST and oxygen measurements at t=6 hours.
11. After the t=4 hour sampling (or t=6 hour, if collected), collect 3 **"Day-3 post-agitation" cores** from within the evaluation enclosure as per SOPs. Avoid the part of the enclosure footprint that was sampled for the Day 1 and Day 2 cores.
12. At the completion of the monitoring period and measurements on Day 3, remove the mesocosm enclosure.

SEDIMENT CORE HANDLING, STORAGE AND ANALYSIS REFERENCE CORE SAMPLING WORKPLAN

1. Use U.S. EPA-approved procedures for core sampling and logging.
2. Measure **sediment thickness** in each of the pre- and post-agitation sediment cores. For one pre- and post- agitation core, log according to SSC's sediment coring work plan (in preparation). Measure the oil thickness in pre- and post-agitation cores using standard visible and UV lamination of oil. Take **photographs** of vertical sediment distribution in each core.

3. Compare and visually evaluate pre- and post- sediment cores to determine the depth of sediment affected by the agitation process. Record the observed maximum depth (D) of sediment affected by the agitation.
4. Combine sediment from the top of the sediment surface to the maximum depth (D) determined in step 3 above from the 3 pre-agitation cores at each location to produce a single pre-agitation composite sediment sample for each test cell.
5. Combine sediment from the top of the sediment surface to the maximum depth (D) determined in step 3 above from the 3 post-agitation cores from each test cell to produce a single post-agitation composite sediment sample for each test cell.
6. For at least 3 (locations A, B, C; which shall include at least one sample from the Morrow Lake Delta and one from the Ceresco impoundment) of the 6 sample locations, analyze one pre- and one post- agitation sediment sample for **hydrocarbon chemistry** (SSC's analytical chemistry QAP).
7. For at least 3 (locations A, B, C; which shall include at least one sample from the Morrow Lake Delta and one from the Ceresco impoundment) of the 6 sample locations, analyze one pre- and one post- agitation sediment sample for **wet and dry bulk density, and TOC**. **Epifluorescence UV microscopy** of oil-sediment particles shall be used to determine sizing and concentration of oiled particles in sediments. It is critical that oil globules are collected from these cores according to the SSCG's sediment core work plan.
8. Freeze and archive the remaining core intervals at -20°C.
9. Freeze and archive the remaining cores from locations where hydrocarbon chemistry is not currently being performed (locations D, E, F) at -20°C until otherwise directed by the U.S. EPA. The need for analysis of these samples will be determined after reviewing results from the first 3 locations analyzed.

TIMING OF THE EVALUATIONS

Not including the poling to locate suitable test locations, which is being conducted regularly, each evaluation should take approximately 4-5 hours to set up and execute. Timing of sampling is approximate; exact time intervals are not important except that frequent sampling is necessary in the first hour after agitation (record all sampling times). The test shall be performed at each of the 6 locations over as short of a time period as possible (ideally in one week) to facilitate intercomparison of the results. A summary of the estimated time to perform each task described is presented in Attachment 1.

MATERIALS

- Stable boat with at least two anchors
- Small floats with lines and weights to mark locations
- Agitation equipment: 10 GPM water pump similar to stinger device, generator, lance, etc.
- Large open-ended enclosures: Possibly plastic tubs with the bottoms cut out, with holes at the top to affix a fence post on the outside (make sets of two so they can be stacked if needed in deeper water). Options include flexible sheets of plastic or sheet metal formed into enclosures. Ideally these would be 4-5 feet in diameter and light enough to handle in a boat. Need two setups per evaluation run (paired treatment and control).

- Fence posts (4+ feet, studded T-post design)(3 per enclosure) and polypropylene rope to hold enclosure in place, driven into the bed at least 6 inches.
- C-clamps (4 or 5 inch), 4 per evaluation, to stack enclosures if needed.
- Sledge hammer or post driver.
- Flags to mark enclosures.
- Sheen sampling kit (one per evaluation), properly prepared as per Gregg Douglas' protocol (WIP). Be prepared to clamp the sheen net to an extension pole to extend its reach into the enclosure.
- Integrating water sampler (vertical tube design) and three horizontal Van Dorn samplers (for shallow water), properly cleaned.
- Poling equipment as per SOPs (including high resolution GPS to record locations).
- Core collection equipment as per SOPs. Use a push-style or piston corer.
- Turbidity meter with submersible probe and cable >5 feet long
- LISST 100x
- Clean churn splitter (polypropylene or Teflon) to receive and mix water samples for subsampling.
- Bottles for sheen samples (1L, amber, certified VOC clean)
- Bottles for TSS, chemical fingerprinting subsamples, UV microscopy (40mL, VOC certified clean)
- Bottles for LISST samples (if done at shoreline rather than in situ)
- Carboy for remaining water samples (10L+)
- Coolers for samples
- Field notebooks, pencils, label tape, pens, watch, bathymetric map, etc.

ATTACHMENTS

Attachment 1 - Sampling Tasks and Schedule per Mesocosm Location

ATTACHMENT 1: SAMPLING TASKS AND SCHEDULE PER MESOCOSM LOCATION

EVALUATING THE EFFICACY AND POTENTIAL ECOLOGICAL EFFECTS OF IN-SITU SEDIMENT AGITATION (SUMMER 2012)

	Time after Agitation (hours)							
	-1.0	0.0	0.25	0.5	1.0	2.0	4.0	4.5
Field Task / Measurement								
Poling and associated measurements outside of enclosure	☒							
Collect depth-integrated water sample	☒	☒					☒	
Collect sheen sample	☒	☒						
Collect sediment cores (3) within cylinder and composite sediment	☒							☒
Measure turbidity within cylinder (6 inch intervals)	☒	☒	☒	☒	☒	☒	☒	
Measure DO within cylinder (6 inch intervals)	☒	☒	☒	☒	☒	☒	☒	
LISST particle size analysis	☒	☒	☒	☒	☒	☒	☒	
Laboratory Analysis Task								
TSS on depth-integrated water sample	☒	☒					☒	
Oil fingerprinting on suspended solids and filtrate (filtered water) from depth-integrated water sample	☒	☒						
Oil fingerprinting on sediment core samples	☒							☒
Wet + dry bulk density, TOC on sediment core samples	☒							☒
UV fluorescence microscopy on suspended solids and sediment core samples	☒	☒						
UV fluorescence microscopy on sediment core samples	☒							☒
Oil fingerprinting on sheen sample	☒	☒						

RECOMMENDATION TO THE FOSC

TOXICOLOGICAL ASSESSMENT OF THE EFFECTS OF RESIDUAL WEATHERED OIL AND INCREASED SUSPENDED SOLIDS RESULTING FROM SEDIMENT AGITATION

ENBRIDGE LINE 6B MP 608 MARSHALL, MI PIPELINE RELEASE
AUGUST 8, 2012

1.0 BACKGROUND

Several questions are being considered regarding the potential ecological effects on aquatic resources of weathered residual Enbridge Line 6B oil in sediments, and concerning the effects associated with increased suspended solids (SS; as measured by turbidity) during sediment agitation. This information is needed to better assess the potential ecological risks of future agitation tool-box techniques that may be used by Enbridge in the Kalamazoo River. This scope of work provides a series of recommendations for a set of toxicological experiments that would facilitate collection of data to directly address these data gaps.

The main objectives of this work include:

1) Determine the response of a representative aquatic test species to "worst-case" concentrations of SS/weathered residual oil in the water column (Phase 1);
2) Determine if weathered Enbridge Line 6B residual oil in sediments poses toxicological risks to benthic test species, and establish a weathered Enbridge Line 6B oil threshold in sediments (Phase 2A);
3) Determine if increased sediment SS (absent any Enbridge Line 6B weathered oil) pose toxicological risks to a representative fish species in the water column, and establish a SS threshold (Phase 2B);
4) Determine if a range of weathered oil/SS combinations pose toxicological risks to a representative fish species in the water column (Phase 3);

2.0 EXPERIMENTAL DESIGN

2.1 Selection of Test Sediments

Critical to addressing the above objectives is the selection of test sediments. Two options were considered: Boardman River sediments and Kalamazoo River sediments (both upstream and downstream of Talmadge Creek.

2.1.1 Option 1: Test Sediment Collected from the Boardman River

Advantages:

1) The Boardman River is a relatively undisturbed river that has a primarily forested watershed in the Pere Marquette State Forest.
2) The Boardman River has traditionally been used as the laboratory control sediment in sediment toxicity assays previously performed on Kalamazoo River sediments.
3) Previous analyses[1] showed polynuclear aromatic hydrocarbons (PAH) concentrations in Boardman River sediment below detection limits, and metal concentrations ranging between below detection limits to concentrations much lower than those in field reference sites of the Kalamazoo River watershed.
4) The total organic carbon content (TOC) in sediments from the Boardman River system is lower than the TOC found in field reference sites of the Kalamazoo River watershed and, therefore, the confounding factors associated with TOC may be reduced.
5) The particle size distribution is similar to the distribution measured in Kalamazoo River samples[2].

Disadvantages:

1) This river system is relatively pristine, suggesting the possibility that sediments of the Boardman River may not be representative of the impacted area of the Kalamazoo River prior to the Line 6B oil release, thereby potentially affecting the bioavailability of weathered oil constituents in ways that are different from the Kalamazoo River sediments prior to the Line 6B oil release.

2.1.2 Option 2: Test Sediment Collected from the Kalamazoo River

2.1.2.1 Option 2A: Kalamazoo River Sediment (Upstream)

Unconsolidated sediment samples from the Kalamazoo River upstream of the Talmadge Creek confluence. Sediments would be collected in close proximity of the location where samples SEKR0000L020 and SEKR0000L021 were collected, and avoiding the channel and left side of the river (where SEKR0000C019, SEKR0000C023 and SEKR0000R022 samples were collected) as these areas may have sources of contamination not consistent with those within the impacted section of the river.

Advantages:

1) Sediment characteristics may be most representative of the area of the Kalamazoo River affected by and prior to the Line 6B oil spill.

Disadvantages:

1) Samples collected from this area (particularly those on the left bank and channel) had PAH and total extractable hydrocarbons (TEH) concentrations equal or greater than the average concentration measured in heavily oiled areas downstream of the Talmadge Creek confluence[2]. This suggests that these sites may have additional sources of contamination not typical of the Line 6B-impacted section of the river.

2) The recent analysis of the initial Kalamazoo River sediment toxicity test results [2] highlighted the selection of upstream Kalamazoo River reference sites within the Kalamazoo River system as a major source of uncertainty in understanding the toxicological effects of oiled sediments in the two target species.

2.1.2.2 Option 2B Kalamazoo River Sediment (Line 6B Oiled Areas)

Sediment samples from the Kalamazoo River within the impacted area that have been *consistently* classified as having no oil/sheen during all poling periods in 2011 and 2012.

Advantages:

1) Two of the three sediments samples categorized as having no oil in 2011 and used for toxicity testing had lower PAH and THE concentrations than all other samples collected within the impacted zone[2]. These results suggest that these sites (which are within the impacted zone of the Kalamazoo River) could be considered to be representative of the background contamination in the River before the oil spill.

Disadvantages:

1) Site selection may require a more rigorous assessment based on the most current poling data and perhaps collection of samples for chemical analyses to ensure that these sediments are not toxic or highly contaminated with residual oil or other organic/inorganic contaminants.

2.1.2.3 Recommendation

Because of the uncertainty associated with the characteristics and non-oil contamination of the Kalamazoo River sediments, use of Boardman River sediments (Option 1) for each of the tests described below is recommended.

2.2 Phase 1 Testing

The test described below is intended to exclusively address the first objective of the SOW. The information generated through this Phase 1 test will provide a conservative estimate of the toxicological effects resulting from simultaneous exposures to "worst-case" weathered oil and SS concentrations in the water column (measured as turbidity).

For the purpose of this Phase 1 Test, "worst-case" weathered oil is being defined as ***3 times*** the highest weathered oil concentration (based on the 2012 chemical analyses of sediment samples); and "worst-case SS concentrations is being defined as 780 NTU, which is ***3 times*** the 95th percentile of turbidity levels (260 NTU) reported during sediment agitation. These "worst-case" conditions are intended to be conservative, given the uncertainty associated with field SS and weathered oil concentrations.

2.2.1 Test Species

Juvenile (2-3 months old) fathead minnows *Pimephales promelas* will be used as the target species in the proposed studies related to the assessment of weathered oil and turbidity effects. This species is common in Michigan, and is representative of the species that could possibly be impacted by sediment agitation in the Kalamazoo River.

2.2.2 Source Oil

The source oil will consist of weathered Line 6B oil for these experiments. The weathered oil may be obtained from the MP-13.4 excavation or other potential sources of weathered oil in the river system. Alternatively, weathered oil may be created by weathering Line 6B oil that was released.

2.2.3 Recommended Toxicity Testing Experiments

a) Test Species: 2-3 month old fathead minnows.

b) Test Substance: Boardman River sediment dosed with an amount of weathered oil to result in a concentration of ***3 times*** the highest weathered oil concentration based on 2012 chemical analyses of sediment samples (see above).

c) Exposure Duration: 24 hours; 10 hours of sustained elevated SS (agitation performed as necessary to sustain elevated SS), followed by 14 hours of exposure post agitation.

d) Experimental Setting: a static test system (5 gallon glass carboy) containing a sustained SS concentration equivalent to 780 NTU, which is ***3 times*** the 95th percentile of turbidity levels (260 NTU) reported during sediment agitation.

e) The exposures described in this document will be conducted on four replicates of the test sediment.

f) In addition, two replicates of control sediment exposure will be conducted on a Boardman River sediment sample absent of Line 6B weathered oil.

g) Endpoints: Percent survival and observations of sublethal impacts (loss of equilibrium, lethargic behavior, unresponsive to mild tactile stimulation).

h) Decision Criterion: If, under these "worst-case" conditions, the mixture is non-toxic to the test species, and there are no observable sublethal effects, then no additional water column testing would be recommended.

2.2.4 Experimental Details

This test will be performed by exposing juvenile fathead minnows to the Boardman River test sediment spiked with weathered oil. The sediment/oil exposure conditions will be comparable to ***3 times*** the highest known oil concentrations in field sediments (as quantified based on recent 2012 chemistry and field collected data), and 780 NTU, which is ***3 times*** the 95th percentile of turbidity levels (260 NTU) reported during sediment agitation.

The test organisms will be exposed for a total of 24 hours: 10 hours of sustained elevated SS during agitation, followed by 14 hours post agitation settling. These exposure conditions are intended to simulate worst-case SS/weathered oil levels observed during sediment agitation. Any noticeable changes in behavior observed during the exposure period will be recorded, and effects on both survival and behavior noted at the end of the exposure period. Turbidity measurements (as a surrogate for SS measurements) will be made throughout the exposure period to monitor the SS levels. During the course of the agitation, SS samples will be taken to characterize the weathered oil exposure concentrations (suite of PAHs and TEHs). Biomarker analyses will be required only if excavated oiled sediments are used as the source oil.

2.3 Phase 2 Testing

In order to understand the potential synergistic/antagonistic effects resulting from combined exposures to weathered oil and elevated SS, it is important to characterize the contribution of each potential source of effects to the overall biological responses. Consequently, the tests below focus on testing the effects of weathered oil alone (Phase 2A), as well as the effects of elevated SS alone (measured as turbidity) (Phase 2B).

2.3.1 Phase 2A

The tests described below are intended to exclusively address the second objective of the SOW. The information generated through these tests will provide a conservative estimate of the toxicological effects resulting from exposures to weathered oil, and may provide information that will allow the derivation of thresholds of concern for residual oil.

2.3.1.1 Test Species

One or both of the species used in previous toxicity tests[3], the freshwater insect *Chironomus dilutus* and/or the benthic amphipod *Hyalella azteca*, will be used to assess the toxicity of residual weathered oil. Exposures will be performed with weathered oil-dosed reference sediments from the Boardman River, a relatively uncontaminated site, thereby controlling for anthropogenic sources of contamination and sediment characteristics, which may have confounded the results of earlier toxicity tests (i.e., sand content, and TOC concentration)[2].

2.3.1.2 Source Oil

The source oil will consist of weathered Line 6B oil for these experiments. The weathered oil may be obtained from the MP-13.4 excavation or other potential sources of weathered oil in the river system. Alternatively, weathered oil may be created by weathering Line 6B oil that was released.

2.3.1.3 Recommended Toxicity Testing Experiments

One or both of the benthic species used in earlier toxicity testing[3] will be used as the test species. The tests below follow the previous recommendations of the Scientific Support Coordinator (SSC) to U.S. EPA. For the purposes of this investigation, a "worst-case" weathered oiling condition in the sediments is defined as ***3 times*** the highest oil concentration, based on 2012 sediment samples from the River.

a) Test Species: freshwater insect *Chironomus dilutus* and/or the benthic amphipod *Hyalella azteca*

b) Exposure Duration: 10 day exposures[3]

c) Experimental Setting: same as those used in earlier investigations[3]; clean and spiked reference sediments from the Boardman River (5 oiled sediment concentrations; 4 replicates each, plus controls).

d) Endpoints: Percent survival, growth and biomass[3].

Benthic invertebrates will be exposed to oiled and unoiled reference sediments using the same methodology used in earlier investigations[3]. Reference sediments collected from the Boardman River will be spiked with a range of amounts of weathered oil to generate dose-response exposure treatments. Sediment-spiked weathered oil treatments will range from unoiled

reference sediments (control treatment) to a "worst-case" weathered oiling condition, defined as ***3 times*** the highest oil concentrations in field sediments, to include a total of 5 oiled sediment treatments (oil concentration ranges to be determined).

Sediment samples (unoiled and oiled sediments) will be collected and analyzed (suite of PAHs and TEHs) to characterize the exposure concentrations. Biomarker analyses will be required only if excavated oiled sediments are used as the source oil.

2.3.2 Phase 2B

The tests described below are intended to exclusively address the third objective of the SOW. The information generated through these tests will provide conservative estimates of the effects associated with pulse exposures to elevated SS levels (measured as turbidity) expected during sediment agitation. This information will allow the derivation of effects thresholds to be avoided during submerged oil recovery with agitation tool-box operations.

2.3.2.1 Test Species

Juvenile (2-3 months old) fathead minnows *Pimephales promelas* will be used as the primary target species in the proposed studies related to the assessment of turbidity effects. This species is common in Michigan, and is representative of the species that could possibly be impacted by sediment agitation in the Kalamazoo River.

2.3.2.2 Recommended Toxicity Testing Experiments

The test below (which is identical to the Phase 1 test, except that oil will not be added to any of the sediments) follows the recommendations of the SSC to the U.S. EPA. For the purposes of this investigation, a "worst-case" SS concentration (measured as turbidity) is defined as 780 NTU, which is ***3 times*** the 95th percentile of turbidity levels (260 NTU) reported during sediment agitation.

a) Test Species: 2-3 month old fathead minnows.

b) Test Substance: elevated SS in the absence of oil.

c) Exposure Duration: 24 hours: 10 hours of sustained elevated SS (agitation performed as necessary to sustain elevated SS), followed by 14 hours of sediment settling.

d) Experimental Setting: a static test system (5 gallon glass carboy) containing a sustained elevated SS concentration equivalent to 780 NTU, which is ***3 times*** the 95th percentile of turbidity levels (260 NTU) reported during sediment agitation; replicates-4.

e) Endpoints: Percent survival and observations of sublethal impacts (loss of equilibrium, lethargic behavior, unresponsive to mild tactile stimulation).

This test will be performed by exposing juvenile fish to unoiled test sediment at SS (measured as turbidity) concentrations ranging from background to the "worst case" sustained concentration defined as 780 NTU, which is ***3 times*** the 95th percentile of turbidity levels (260 NTU) reported during sediment agitation. There will be a total of 5 SS treatments (concentration ranges to be determined). For each of the four replicates, juvenile fathead minnows will be exposed for a total of 24 hours: 10 hours of sustained elevated SS (3 times the highest SS concentration observed, and measured as turbidity) during agitation, followed by 14 hours post agitation. Any noticeable changes of behavior observed during the exposure period will be recorded, and effects on

survival noted at the end of the exposure period. Turbidity and SS measurements will be taken throughout the exposure period to monitor the variability of the exposure conditions among the four replicates.

2.4 Phase 3

The decision on whether or not to perform this test will depend on the results of Phase 2 tests. Therefore, the experimental settings and details described below are tentative and subject to change, pending results of previous tests. The test described below is intended to exclusively address the fourth objective of the SOW.

2.4.1 Test Species

Juvenile (2-3 months old) fathead minnows *Pimephales promelas* will be used as the primary target species in the proposed studies related to the assessment of weathered oil and turbidity effects. This species is common in Michigan, and is representative of the species that could possibly be impacted by sediment agitation in the Kalamazoo River.

2.4.2 Recommended Toxicity Testing Experiments

a) Test Species: 2-3 month old fathead minnows.

b) Test Substance: Boardman River sediment dosed with a concentration of weathered oil at concentrations to be determined based on tests from Phase 2.

c) Exposure Duration: 48 hours total, where each 24 hour period consists of: 10 hours of sustained elevated SS (agitation performed as necessary to sustain SS), followed by 14 hours of sediment settling.

d) Experimental Setting: a static test system (5 gallon glass carboy) containing SS concentrations to be determined based on tests from Phase 2; replicates-4.

e) Endpoints: Percent survival and observations of sublethal impacts (loss of equilibrium, lethargic behavior, unresponsive to mild tactile stimulation).

This test will be performed by exposing juvenile fish to reference sediments spiked with weathered oil. Exposure conditions include a background (unoiled reference sediments) and oiled sediments, and it will include a total of 3 oiled sediment treatments (oil concentration ranges to be determined). These sediments will be used to generate three suspended solid treatments (concentration ranges to be determined) (Table 1). Within each oiled sediment treatment, juvenile fish will be exposed to elevated turbidity levels for 10 hours every day of the two day exposure period. These conditions will represent a recurrent exposure to upstream agitation events.

Turbidity and SS measurements will be taken throughout the exposure period to monitor the variability of the exposure conditions among the four replicates and among treatments. Sediment samples (oiled sediments and reference sediments) will be collected to characterize the exposure concentrations (suite of PAHs and TEHs). Biomarker analyses will be required only if excavated oiled sediments are used as the source oil.

Table 1
Weathered Oil and Turbidity Treatment Combinations for Fish Tests Only

Oiled Sediments	SS (as measured by Turbidity)		
	Treatment 1	**Treatment 2**	**Treatment 3**
Unoiled reference	X	X	X
Treatment 1	X	X	X
Treatment 2	X	X	X
Treatment 3	X	X	X

Note: Each treatment combination will have 4 replicates.

REFERENCES

[1] Analysis of Boardman River Reference Sediment, Report by RTI laboratories, Inc, submitted to the Great Lakes Environmental Center, February 16, 2011.

[2] Bejarano AC, 2012. Analysis on the Association between Aquatic Toxicity Results and Sediment Characteristics from Samples Collected within the Kalamazoo River Enbridge Line 6B MP 608 Marshall, MI Pipeline Release June 27, 2012. Appendix A in Net Environmental Benefit Analysis (NEBA) Risk Ranking. Kalamazoo River System Enbridge Line 6B MP 608 Marshall, MI Pipeline Release.

[3] Great Lakes Environmental Center, Inc. (GLEC) 2012. Preliminary 10-day Whole Sediment Toxicity Test Report *Hyalella azteca* and *Chironomus dilutus* Kalamazoo River Sediment Sampling, February 2012 Line 6B Spill, Marshall, Michigan. March 21, 2012.