UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

February 20, 2012

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Re: Sampling Plans Prepared by the Scientific Support Coordination Group (SSCG) for Aquatic Toxicity Testing and Oil Fingerprinting
Enbridge Line 6B MP608 Release, Marshall, MI

Dear Ron and Faith:

I have reviewed the above-referenced Sampling Plans and accompanying memos that were prepared in response to Charges 1, 2, and 3 submitted to the SSCG:

Charge 1

a) Provide an evaluation of viable analytical approaches, including benefits and draw backs for each, to quantify the amount of submerged oil in the Kalamazoo River sediments attributable to the Enbridge Oil pipeline Release.

Charge 2

a) Identify and evaluate viable procedures for assessing the toxicity of remaining submerged oil.
b) Provide a recommendation for the best procedure to accomplish this goal.

Charge 3

a) Provide an evaluation of viable procedures, including benefits and draw backs for each, to assess whether remaining submerged oil will biodegrade over time.
b) Provide a recommendation for the best approach to accomplish this goal.

I hereby accept the group's recommendations on these issues. Our Environmental Unit and Enbridge have already initiated its implementation. Field work will begin on Monday February 20, 2012 to collect these sediment samples.

I am very pleased with the ability of your SSCG subgroups to coordinate their efforts and produce a unified approach that will address the near-term data needs for this project. Please extend my regards to all members of the subgroups for their valued participation.

In my review of the Plans I see that there is discussion of potential "next steps" beyond the February/March timeframe, and I will be thinking about these concepts and will discuss them soon with both of you. In addition my preference is that we continue to consider that chronic toxicity testing may yet be useful to our evaluation of potential ecological impacts and will not necessarily fall to MDEQ for implementation.

I am glad to hear that the SSCG is energized and actively advancing our collaborative effort.

Once again I must extend my since appreciation for the level of professionalism and diligence displayed by the SSCG members. I know that our overall project will be more successful because of their efforts.

Sincerely,

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

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February 16, 2012

Mr. Ralph Dollhopf
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Subject: Toxicity Testing of Kalamazoo River Sediments
Enbridge Line 6B MP 608, Marshall, MI Pipeline Release

Dear Mr. Dollhopf,

With this memorandum, the Ecological Risk and Toxicity Subgroup of the Scientific Support Coordination Group (SSCG) presents its response to the FOSC's Charge No. 2:

a) Identify and evaluate viable procedures for assessing the toxicity of remaining submerged oil.

b) Provide a recommendation for the best procedure to accomplish this goal.

Recommendations herein are in detail for immediate action (February 2012) and include:

- Conduct standard procedures for acute sediment toxicity tests for freshwater invertebrates (10-day survival and growth for *Chironomus* and *Hyalella*) for a range of oil contaminated sediment collected from the Kalamazoo River at about 25-30 locations.

- Conduct standard procedures for acute sediment toxicity tests for freshwater invertebrates (10-day survival and growth for *Chironomus* and *Hyalella*) for reference sediment spiked with various concentrations of weathered source oil samples recovered during the response (EPA, 2000).

Based on subgroup members' experience in addressing issues related to oil spills and contaminated sediment related ecological toxicity, we strongly recommend the adoption of this technical approach to initiate an assessment of the toxicity level of residual oil remaining in the Kalamazoo River.

On behalf of the SSCG Ecological Risk and Toxicity Subgroup,
/s/
Faith Fitzpatrick, Ph.D.
Research Hydrologist, U.S. Geological Survey

Recommendations to the FOSC for Submerged Oil Toxicity Testing Enbridge Line 6B MP 608 Marshall, MI Pipeline Release

Background and FOSC Charge

Spring 2012 cleanup strategies for the Kalamazoo River in response to the Enbridge Line 6B Pipeline Release are expected to be based primarily on a synthesis of acute toxicity test results, sediment chemistry, and benthic community assessment (EPA sediment triad) in combination with information on project chemical fate and a Net Environmental Benefit Analysis (NEBA). Sediment toxicity testing is needed to determine the ecological risk associated with remaining submerged oil and oil-containing sediment in the inundated areas of the Kalamazoo River and its backwaters and will be used in spill response decisions. The SSCG Ecological Risk and Toxicity Subgroup examined and discussed the relevance of toxicity testing within the realm of EPA strategies for submerged oil cleanup starting in Spring 2012.

Specifically, these recommendations are in response to the two parts of the FOSC's Charge No. 2:

a) Identify and evaluate viable procedures for assessing the toxicity of remaining submerged oil.

b) Provide a recommendation for the best procedure to accomplish this goal.

In the discussion process, the Subgroup recognized that toxicity testing could take place over longer time periods that may be of interest as oil recovery techniques transition from active toolbox techniques to passive techniques and as EPA transitions authority to the State of Michigan. Recommendations herein are in detail for immediate action (February 2012). Considerations for additional follow-up studies that may take place during summer 2012 and post 2012 are presented as preliminary ideas that need further discussion and development. Initial results from the immediate testing will be used to help guide the scope and content of additional toxicity test designs, as appropriate. The recommendations are outlined in terms of viable procedures, advantages and disadvantages of the procedures, and recommended approach for immediate action.

Viable Procedures

Multiple potential experimental procedures were identified as possible approaches in determining the toxicity of oil/sediment mixtures in the Kalamazoo River. They are listed in order of priority. Procedures 1 and 2 are independent of each other. The Subgroup strongly recommends performing procedure 1 with procedure 2 simultaneously. Procedure 3 would follow procedures 1 and 2. Procedure 4 is a combination of procedures 1 and 3.

1. Conduct standard procedures for acute sediment toxicity tests for freshwater invertebrates (10-day survival and growth for *Chironomus* and *Hyalella*) for a range of oil contaminated sediment collected from the Kalamazoo River.

2. Conduct standard procedures for acute sediment toxicity tests for freshwater invertebrates (10-day survival and growth for *Chironomus* and *Hyalella*) for reference sediment spiked with various concentrations of weathered source oil samples recovered during the response (EPA, 2000). Potential weathered oil sources include those stored at Griffith, Indiana and recently relocated to the Marshall area and (or) weathered oil recently found in the overbank at MP 13.4 on 2/15/12.

3. Conduct standard procedures for chronic sediment toxicity tests for freshwater invertebrates (28-day survival and growth for *Hyalella* and *Chironomus)* for a range of oil contaminated sediment collected from the Kalamazoo River. May be recommended following results of acute tests and will be conducted in the future as part of MDEQ's authority.

4. Conduct parallel acute (10-day) and chronic (28-day) sediment toxicity tests on *Chironomus* and *Hyalella* for a range of oil contaminated sediment collected from the Kalamazoo River.

Advantages and Disadvantages of the Procedures

1. Conduct standard procedures for acute sediment toxicity tests (10-day survival and growth for *Chironomus* and *Hyalella*) for Kalamazoo River sediment from the field.
    - Advantages
        - Survival endpoint is consistent with imminent and substantial endangerment to the environment, and facilitates direct comparison with potential lethal effects of response actions to ecological receptors.
        - Fast results, requiring 10 days of laboratory exposure, plus data evaluation and report preparation.
        - Same analysis was done on Talmadge Creek samples in the fall 2011.
        - Initial tests can be used to design additional toxicity sampling, such chronic toxicity tests.
        - Typically used as an EPA assessment tool for contaminated sediment at spill and Superfund sites and MDEQ's preferred methodology.
        - Conducting tests on two species strengthen results, in case something happens with one species the other serves as a backup.
        - *Hyalella* is generally a shredder and *Chironomus* is more of a deposit feeder. Both species are equally sensitive to contaminants.

        - Can generate valuable information within the timeframe specified by the FOSC (end of April, 2012)
    - Disadvantages
        - May not show full toxicity (e.g., sublethal and chronic effects) effects of oil.
        - May not be fitting for all affected species (such as fish) or life stages (such as juveniles).
        - Hard to get representative field samples of the surficial sediment in which most benthic invertebrates live that have a gradient of oil exposure.
        - Requires complimentary sediment chemistry analyses. Causality needs to be established through contaminant analyses.

2. Conduct standard procedures for acute sediment toxicity tests for freshwater invertebrates (10-day survival and growth for *Chironomus* and *Hyalella*) for reference sediment spiked with various concentrations of weathered oil recovered by Enbridge during cleanup operations.
    - Advantages
        - Fast results, requiring 10 days of laboratory exposure, plus data evaluation and report preparation.
        - Typically used as an EPA assessment tool for contaminated sediment at spill and Superfund sites
        - Shows effects from weathered oil along a gradient. Oil reaching river sediments was likely weathered enough to lack the majority of lighter diluent and lighter-molecular weight components in the source oil.
        - Allows for assessment of oil toxicity.
        - Can identify toxicity thresholds for survival and growth that may be related to sediment chemistry measurements, and may provide a step toward identifying cleanup criteria.
        - Can generate valuable information within the timeframe specified by the FOSC (end of April, 2012)
    - Disadvantages
        - Reference sediment may be physically or chemically different than Kalamazoo River sediment, resulting in erroneous interpretation of results
        - Residual submerged oil in river is likely more weathered than that which was recovered earlier.
        - Requires complimentary sediment chemistry analyses. Causality needs to be established through contaminant analyses.

3. Conduct standard procedures for chronic sediment toxicity tests (28-day survival and growth for *Hyalella* and *Chironomus)*
    - Advantages
        - Typically used as a State assessment tool for determining impairments for designated uses

        - Results may show more subtle or delayed toxic effects of oil/sediment mixture.
    - Disadvantages
        - State will run chronic tests, no need for EPA to do before acute tests results can be analyzed.
        - Harder to interpret results, false positives are common
        - May not be fitting for all affected species (such as fish) or life stages (such as juveniles).
        - Hard to get representative field samples of the surficial sediment in which most benthic invertebrates live that have a gradient of oil exposure.
        - Chronic test duration (28 days) and data interpretation extend timeframe beyond that specified by the FOSC (end of April, 2012).
        - Causality needs to be established through contaminant analyses.

4. Conduct parallel acute (10-day) and chronic (28-day) sediment toxicity tests on *Chironomus* and *Hyalella* for a range of oil contaminated sediment collected from the Kalamazoo River.
    - Advantages
        - Tests the relative sensitivity of the two test species and the applicability of the short and long-term tests over a relatively short time period
        - Based on this series of tests, a "preferred" test species (*Chironomus* or *Hyalella*) and test duration (10 days or 28 days) could be determined.
    - Disadvantages
        - No benefit for response decision making by the end of April, 2012.
        - This evaluation would require 28 days to complete (not including data evaluation and report preparation) and thus may not generate information within the timeframe specified by the FOSC (end of April, 2012)

Recommended Procedure 1 for February 2012 Study

1. Laboratory Analysis -- Perform acute sediment toxicity tests (10-day survival and growth for *Chironomus and Hyalella*) for sediment samples collected from approximately 25-30 locations in the Kalamazoo River. Use standard EPA procedures for measuring toxicity of sediment-related contaminants with freshwater invertebrates (EPA, 2000). Consider focusing primarily on survival endpoints for decision making, but use growth measurements as secondary lines of evidence.

2. Sampling Locations -- Sample sediment from approximately 25-30 locations along the Kalamazoo River that include a range of oil as well as background samples from the Kalamazoo River upstream of Talmadge Creek confluence and Battle Creek.

Considerations for site selection within the oil affected reach of the Kalamazoo River (about 20 locations):

- Sample a range of oil contamination based on the 2011 late summer reassessment poling results
  - None
  - Light
  - Moderate
  - Heavy
- Sample primary geomorphic settings in the active channel/submerged oil setting
  - Backwater/channel margins/side channels associated with the active channel
  - Impoundments/Morrow delta
- Possible secondary settings of lesser priority
  - Oxbows/standing water in the floodplain (connected to the river during flood events only)
  - Spring channels and pools in groundwater discharge areas (these may represent movement of overbank oil from floodplain soils towards the river)
- Track oil recovery history for selected sites
  - Oiled, no recovery to date (near 35th Street bridge)
  - Oiled, recovery, site remains free of oil
  - Oiled, recovery, oil returned
- Prioritize samples from passive sediment collection areas, remaining STRIKE/OSCAR sites, biodegradation studies, and 2012 focus areas
- Locations should be distributed along the length of the affected river, with recognition that submerged oil behavior may be different downstream of Ceresco dam compared to upstream, based on observations of spilled oil behavior during initial release and flooding.

Considerations for site selection for background sediment samples (about 2-3 locations from 3 settings for a total of 6-9 samples):

- Select subset from 2011 background core locations in depositional areas from three reaches in similar hydrogeomorphic settings as the oil-affected reach of the Kalamazoo River:
  - The Kalamazoo River upstream of its confluence with Talmadge Creek
  - Battle Creek River upstream of its confluence with the Kalamazoo River
  - The Kalamazoo River in Marshall impoundment.

3. Sediment Sample Collection—Collect sediment in the field with a clamshell style Eckman or Ponar dredge. Sample the top 3-4 inches in depositional area. The top 3-4 inches is the most representative for benthic life.

- Collect 2 gallons of wet sediment.
- Fill out field log, similar to those done for cores.
- Process samples for labs. Toxicity lab will prepare split for chemistry after large debris is removed and sample goes through minor amount of homogenization.
- Split sample for archive (approximately 2 liters). Keep archive sample cold or frozen and sealed for future potential biodegradation, toxicity, or chemical analyses.
- Collect a separate sample for acid volatile sulfides (needs to be submitted with minimal disturbance and transported to lab frozen)
- Perform UV sheen test evaluation and visual oil determination similar to that done for fall 2011 core logging
- Consider performing fluorescence test on sample (methods under development by MDEQ and needs further investigation and follow-up)
- Make casual observations of living aquatic invertebrates encountered in the sample processing (e.g., worms, insect larvae, mussels).
- Note whether the sediment has a strong hydrogen sulfide smell (often described as ”rotten eggs”).

4. Sediment Chemical Analysis—Laboratory analysis of sediment chemistry should be consistent with SSCG Chemistry Subgroup recommendations (Analytical Quality Assurance Plan). Tests that will be used to help distinguish sources for toxicity and should be done on all samples:

- Polycyclic Aromatic Hydrocarbons and sulfur heterocyclic compounds, including alkyl homologues by gas chromatography with low resolution mass spectrometry using selected ion monitoring,
- Total extractable hydrocarbons (TEH) representing the total aromatic and aliphatic hydrocarbon content of sample extracts after silica gel cleanup and analysis by GC/FID
- Petroleum chemical biomarkers by GC/MS-SIM, including hopane (help to determine weathering)
- Trace elements by ICP/MS
- Total organic carbon
- Particle size analysis (sand, silt, clay breaks)

Laboratory analysis to consider for a subset of samples (approximately 5-10 samples):

- Acid volatile sulfides (AVS) and simultaneously extracted metals (SEM). Not typically done for oil spill-related toxicity sampling. Tracers of beryllium, molybdenum, nickel, and vanadium are associated with the crude oil and nickel concentrations in crude oil samples were above sediment quality guidelines for probable effects concentrations (MacDonald et al. 2000; Enbridge, 2011). Nickel concentrations were above probable effects concentrations in some sediment samples from

Marshall impoundment and may represent historical inputs of other industrial metals that have accumulated in fine sediment depositional areas in impoundments in the affected area of the Kalamazoo River. AVS may help explain trace elements related toxicity (needs separate sample and frozen for shipment).

Post- February 2012 Additional Approaches and Ideas

Based on acute toxicity results, SSCG subgroup will continue to discuss additional toxicity tests that might be appropriate. Some potential alternatives:

1. Design and(or) develop toxicity tests for more specific freshwater species important in the Kalamazoo River:

    - 48-hr *Daphnia* tests – easy to perform and sensitive acute methods but water column test – not sure on usefulness for submerged oil.
    - Mussels – early life stages
    - Crustaceans
    - Fish-- early life stage of smallmouth bass

2. In-situ toxicity assays (e.g., Burton et al. 2005 Envir. Pollut. 134: 133-144.) – These resemble the lab assays with *Hyalella* but are conducted in screened containers placed on the sediment surface in the river, to better represent the conditions in the benthic environment.

3. Perform laboratory study to test agitation effects on sediment toxicity:
    a. Spike field sediment in lab with weathered oil sample along gradient of concentration
    b. Agitate and recover liberated oil (measuring % recovery)

4. Perform field study of agitation effects on toxicity of resuspended sediments

5. Collect and run toxicity tests on samples collected from streambed sediment traps (open tubes on the bed in settling areas downstream of active agitation areas)

6. Re-examine the side-by-side acute and chronic toxicity test results and determine if more acute or chronic testing are needed, and if so, with which species.

References

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