

Peer Review of Sediment TIE Guidance Document

Overall Questions

- 1) Yes; generally well presented, described and discussed.
- 2) Yes; some of the approaches are not particularly specific in terms of what classes of contaminants they might affect, but the authors describe the need for multiple lines of evidence and ways to narrow the focus in follow-up work.
- 3) Generally yes.
- 4) Sections 1-5. Generally seem reasonable to me. This is a guidance document, and also presumes some level of knowledge of TIE and laboratory procedures on the part of the reader.
- 5) Section 6. Laid out and presented well.
- 6) Section 7. Same.
- 7) Section 8. Same.
- 8) Section 9. Same.
- 9) Section 10. Same.

Overall, the authors have a wealth of experience in this area, and they have done a remarkable and commendable job of putting it on paper. That being said, I do have some specific comments, both technical and editorial. These follow:

- 1) Page iii, 3rd para, 2nd line: suggest comma between “sediments” and “including”.
- 2) Page 13, Section 6.2.1, 1st para, 5th line: delete hyphen between “of” and “toxicants”.
- 3) Page 20. Under General Considerations, would suggest considering measuring sulfide as well, since it is frequently present, and can be quite toxic. Similar reasoning as for measuring ammonia.
- 4) Page 22, last para, 4th and 5th lines: suggest inserting “and” before “not intended”, and replacing “only” with “but”.
- 5) Page 27, Section 7.2.1. A couple of comments on this section. First, since many sediment samples do not produce 100 percent mortality, I would lead with the statement that such samples be tested at full-strength in Phase I. This actually doesn't get mentioned until the bottom of pg. 29. Would then follow-up with the justification and procedures for dilution. Also, there is much reference to not “overwhelming” the manipulations with toxicity, but no clear guidance. In the effluent TIE, typically the treatments will be effective up to 4X the LC50. To provide assistance to the reader, the authors should give more quantitative estimates of the effectiveness of the treatments; i.e., 1.5X? 2X? The implication is that most of the treatments are not highly effective at removing excess levels of toxicity, but the reader needs a better idea as to the width of the window he/she can expect to be working with.
- 6) Page 31, foot note to Figure 7-2. insert “criteria document” between “quality” and “is”.
- 7) Page 32, 1st para under Interpretation: I believe this is the first mention of zero-valent magnesium, and should be accompanied by the location in the document (Section) where more information can be found, as well as where it fits in a TIE

- context (Phase 2). Otherwise the reader is going “what?”, since it is not listed in the Phase 1 flow chart. At least I was....
- 8) Page 33, 1st para, line 3. Need space between “charge” and “(Rozić...)”
 - 9) Page 35, 1st para, last line: insert “also” between “hydroxides” and “play”
 - 10) Page 44, 1st para, is there a measure of “under vacume”? What are we actually trying to achieve?
 - 11) Page 44, 2nd para, the 1st sentence is not a sentence; suggest combining 1st and 2nd sentences, deleting “For that reason,”.
 - 12) Page 45, 1st para. I think this is the first time I have ever seen the word “egregiously” in a scientific document. Awesome! Again this section begs a more definitive characterization of the extent of toxicity that these treatments can accommodate without “breakthrough”.
 - 13) Page 47, 1st para under Method: there are 2 “although” s in the first sentence; suggest deleting one of them.
 - 14) Page 47, 2nd para under Method, 1st sentence, change “determine” to “determining”
 - 15) Same para, line 5, add space between “hours” and “to”.
 - 16) Page 48, 2nd para. Again, please provide more definitive guidance as to appropriate dilution of sediment... would 50% dilutions work, and then testing the lowest concentration that produces 100% mortality, for example? Given that sediment can be limiting and expensive to collect, it is important to try and get this right the first time, and not have to back into it over a period of several tests...
 - 17) Page 49, top. Here the authors use sand as a dilution blank; is this application consistent with the discussion of appropriate sediment to use for dilution that takes place on page 28?
 - 18) Page 53, top. Suggest saying adding 5 mL of excess sample to compensate for Rather than introducing a term such as “slop”. On the other hand, if you really like it, use it!
 - 19) Page 55, top. We did find one case where it mattered whether the manipulation was done before or after salting up... Again, careful records are important (why did this treatment work before and not now, kind of thing). Also, we don’t do a lot of LC50 calculations with our treatments. Obviously dilutions are necessary to calculate LC50s, but % mortality, time to mortality, and so on are also reasonable indicators of effects. In this case, we might put more emphasis on replicates, rather than dilutions... Just a thought, not sure that it matters....
 - 20) Page 55, section 8.2.9: delete the period between “the” and “marine” (1st line), and insert “this” between “While” and “possibility” (4th line).
 - 21) Page 57 Overview: we use a side-arm flask to capture sublatables—the flask is aerated, the air flows out through the top (port in stopper), and the wet foam flows out through the side arm where it can be collected. No fuss, no muss.
 - 22) Page 57 Interpretation: hydrogen sulfide is also readily oxidizable, and volatility (and toxicity) are pH dependent? Doesn’t blow off so well at higher pHs. Aeration with pure oxygen knocks it out pretty quickly—minutes.
 - 23) Page 58, 1st para, line 6: suggest adding a hyphen between “particle” and “active”.
 - 24) Page 58, 1st para, line 9: suggest adding commas around “therefore”

- 25) Page 58, 3rd para. An alternative to less polar solvents is to use SPE columns with lower binding affinity; e.g., C8, for example. Bailey et al (1996) compared elution patterns and efficiencies for different SPE columns for three pesticides (ET&C 15:837-845).
- 26) Page 58, last para. A thought: we generally hold the column until the results of the phase 1 tests are in. If organics appear to be implicated, or if multiple treatments are effective (e.g., EDTA and SPE), we would then elute the column with solvent (no gradient) to confirm/deny the presence of toxic organics. Usually we would test the eluate at 1, 2 and 4X to ensure that we are not adding artifactual toxicity by concentrating an organic that is present near but not over the toxic threshold. Columns can be stored for extended periods; if an organic is implicated, we generally load a bunch of columns so we have something to work with in case toxicity diminishes over time.
- 27) Page 60, footnote to Table 8-2: *M. lateralis* should be in italics.
- 28) Page 61, top para: I did not find a reference that corresponded to Anderson 2006.
- 29) Page 61, bottom para, 3rd line from the bottom: insert “in” between “high” and “IWs”.
- 30) Page 62, you can’t say something was used in two ways and then have three bullets... Suggest adding the 1st sentence of the 3rd bullet to the end of the 2nd bullet, and then have the remainder of the 3rd bullet be its own stand-alone paragraph.
- 31) Page 62, last para: The sentence that begins on line 7 looks like a conglomeration of two separate thoughts. Also suggest inserting “at 25C in freshwater” following “For example” in line 9.
- 32) Page 63, top. Table 8-3 is supposed to show different toxicity of ammonia at different pHs. However, the table appears to show sensitivity of different species to different toxicants. The title of the table should be revised to include “to Different Toxicants”
- 33) Page 63, middle para: Delete period between “and” and “Broderius”, line 4.
- 34) Page 65, in the equation in the middle of the page, it looks like the right third or so is not on the same line as the rest of the equation.
- 35) Page 66, para 2, line 2, insert “CO₂” after “0.04%”
- 36) Page 69, 1st para. Would the closed cup work with the mysid and amphipod if they were tested in a larger volume (reduced loading)? Or is the demand such that the volume would have to be increased dramatically...
- 37) Page 69, Method, 3rd para: This may be me, but I can’t figure out how the 360-mL volume equates to the three 60-mL aliquots... Are there extra treatments/repes that I am missing?
- 38) Page 69-70; does use of a laboratory film create any issues for sorbing organics?
- 39) Page 75. para 3, lines 6-7: suggest deleting phrase “within that static exposure”.
- 40) Page 78, section 9.2.2, 1st line: insert space between “magnesium” and “(“, and “mg” should be “Mg”.
- 41) Page 79, 3rd para, lines 2 and 3: suggest inserting comma after “24 hours” and deleting “This test requires longer than 24 hours to take effect”.
- 42) Page 81, section 9.2.4, line 5: the Mg₀ should have the 0 superscripted.

- 43) Page 85, section 9.3.1, 2nd para, line 7, insert parenthesis following “.. from now on”
- 44) Page 89, 1st para, last line: Heinis et al requires a date.
- 45) Page 99 middle: three bullets for two factors. Suggest making the 3rd bullet a stand-alone paragraph.
- 46) Page 100, 2nd para, 3rd line: the context of the phrase “science application guidance” is not clear.
- 47) Page 102, 2nd bullet: delete parentheses around Cornelissen et al
- 48) Page 103: the last bullet does not refer to a specific manipulation, and should be a separate concluding sentence.
- 49) Page 107, 5th line up from bottom: suggest inserting hyphen between spot and checked.
- 50) Page 108, 1st para, line 4: delete “#1095”
- 51) Page 111, 2nd para, lines 3 and 4: Another example would be diazinon and ammonia (Shamelessly citing Bailey et al 2001 ET&C 20:2877-2882).
- 52) Page 111, 6 lines up from bottom: suggest inserting hyphen between “well” and “capped”.
- 53) Page 112, 2nd para. Insert period at end of paragraph.

Note to the authors: Awesome job. Especially good insights in the interpretation (Phases 2 and 3) sections. This part is often neglected in the cookbook cookie cutter one size fits all approach....