

June 21, 2005

MEMORANDUM

To: Technical Review Committee

From: Don Yee and Meg Sedlak

Re: RMP Laboratories with QA/QC Issues

As a result of our standard QA/QC data review process, Don Yee has identified several QA/QC issues with the following analytes and has requested that the respective laboratories implement corrective measures. We have summarized the issues by analyte and laboratory below.

AXYS Analytical

- PAHs – Based on a review of 2003 water data set, significant blank contamination was observed in the dissolved and particulate fractions. The particulate fraction had the highest and most pronounced contamination. Further discussions with the laboratory and discussions among staff suggested that the cause of this blank contamination may be the result of the extraction method used for the glass filters. Toluene (an aromatic ring with a methyl group) is used to extract PAHs from a ground glass filter. The toluene is heated for several hours in a soxhlet distillation apparatus. It is thought that the toluene polymerizes to form PAHs during this process.

Corrective action: The laboratory has investigated several ideas to mitigate this problem; however, none look particularly promising. At this time, we have authorized AXYS to begin the extraction of the filters for the 2004 analytes (e.g., PBDEs, PCBs, PAHs, etc.); however, it is likely that we will again have significant blank contamination with PAHs. (The filter is extracted into one sample that is divided up for five analyses (i.e., PBDEs, PCBs, PAHs, Pesticides, and backup). Because no promising methodology is presently available to address the PAH contamination issue, it was decided not to hold up the analyses of the other chemicals.) We are presently evaluating other alternatives for 2005. One idea is to collect whole water samples for PAH. The samples would not be filtered through a glass filter in the field and therefore, the issue of forming PAHs during the extraction process would be avoided. Collecting whole water samples will be somewhat cumbersome and will increase our costs because a separate liquid-liquid extraction method will be needed (approximately \$12,500).

- PBDEs In reviewing the 2003 PBDE water data set, review of the QA/QC data suggested that recovery of surrogate congener for 209 was abnormally low and resulted in this congener not being reportable. In addition, based on the laboratory notes it appears that there may be some issues with laboratory contamination. Several other important congeners were not detected in a few samples (e.g., congeners 47, 99, and 100), making it difficult to calculate PBDE sums.

Corrective action: We have asked the laboratory to review and comment on this data set. Depending on the response from the laboratory, we may ask the laboratory to reanalyze the backup extract for PBDEs.

California Department of Fish and Game

- PAHs - Recovery of the standard reference materials for PAHs was poor. In addition, there were a significant number of non-detects for PAHs.

Corrective action:

Revise analytical procedures for tissue PAHs until acceptable results for SRMs can be obtained?

- PBDEs - Several important and biologically relevant congeners such as congeners 153 and 154 were not detected in bivalves. An intercomparison study of egg homogenate between AXYS and CDFG suggests that CDFG may be under reporting PBDE data by approximately 50 percent.

Corrective action:

CDFG and AXYS will be participating in a NIST intercomparison exercise. Upon completion of this exercise, the RMP will re-evaluate CDFG data and provide suggestions.

Brooks Rand Laboratories

- Selenium in Water – As mentioned at the previous TRC meeting, dissolved concentrations of selenium occasionally exceed total concentrations. Brooks Rand is investigating using Dynamic Reaction Cell ICP-MS method instead of hydride generation atomic absorption. Brooks Rand should be providing additional data on the methods the week of June 20th. We will need to make a decision shortly regarding which method we will use.
- Selenium in Sediment – Selenium concentrations in sediments in a pair of field replicate samples showed 10x differences in concentration. Some of this difference may be that seen in EBMUD organics, i.e. RMP field homogenized sediments may not be totally uniform. However differences EBMUD found in analyzing multiple sediment aliquots typically differed by 2-3x. A second

contributing factor may be incomplete recovery of selenium seen in the total fraction water analysis of Se. Brooks Rand has located remaining material from the replicate sites and is reanalyzing. On the basis of the results, we may investigate modifications to the field homogenization procedures, or use of larger samples for extraction by laboratories.

UCSC

- Methyl mercury in sediment. It has recently been suggested that methyl mercury may be lost as a result of the homogenization process that is conducted on board prior to freezing the sample. The RMP is considering for the 2005 sampling event a comparative study of replicate grab sediment samples to composite homogenized sediment samples for the same sites to see if there is significant variation among the samples.