Annual Report for the California Bay-Delta Authority Project

Programmatic Quality Assurance and Quality Control for California Bay-Delta Authority (CBDA) Mercury Research and Monitoring Projects

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by

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INTRODUCTION

Created as a part of the CALFED Science Program, the California Bay-Delta Authority (CBDA) Mercury Studies Program will deal with questions specifically related to mercury in the Bay-Delta system. The sub-tasks carried out under the program address a wide array of topics, with the common goals of broadening the base of knowledge while controlling and eventually decreasing biotic exposure to methylmercury. In order to ensure that the data generated during the life of the project are of known and documented quality, the CBDA Mercury Project Quality Assurance (QA) Oversight Program was implemented in March 2005. The personnel implementing the QA Oversight Program (referred to as the QA Team) work closely with the Department of Fish and Game (DFG), CBDA, principal investigators (PIs), and laboratories involved to accomplish the following goals.

- Build comparability with other monitoring and research efforts in California. Ecosystem managers, regulatory groups, and other end user groups benefit from comparable data. There are many monitoring and research efforts to help characterize mercury in California including the CBDA mercury studies, the Surface Water Ambient Monitoring Program (SWAMP), Total Maximum Daily Load (TMDL) programs, the Nonpoint Source Programs, and individual projects conducted by Regional Water Quality Control Boards. It is a goal that the CBDA mercury program tenants and QA Project Plan will be used for other mercury collection efforts. This allows agencies to leverage larger data sets for use in areas. Just a few examples of uses are: Section 303(b) listing/delisting, Section 305(d) reports, and TMDLs.
- Ensure that the results generated by the various sub-tasks are comparable. In other words, environmental samples collected and submitted to the contract laboratories should yield comparable results regardless of who collects or analyzes the samples.
- Ensure that the data is of known and documented quality. This refers to what is commonly referred to as the “transparency” of the generated data. All of the information that contributes to the generation of the final results should be readily available and well documented. In addition, the process used to calculate the results should be clear and reproducible by a third party.
- Ensure that the data will be generated in a manner that is comparable with other mercury programs.

Realization of these goals will enhance the quality of the individual sub-tasks, the CBDA Mercury Project as a whole, and even future mercury projects. Data of this quality can be confidently referenced to supplement the results of other mercury work and can be built upon in the design of future studies. Comparability widens the use of data to other programs and organizations to benefit the State of California as a whole.

The goals of the QA Oversight Program will be accomplished by the execution of the following activities.

1. Collaboration with other programs and agencies to help build comparability between monitoring and research efforts. For example, the CBDA QA Team actively collaborates on QA issues with SWAMP (Beverly H. van Buuren is also the SWAMP QA Officer), TMDL, NPS, US EPA R9 Office of Water, SWRCB Office of Information Technology, SWRCB Department of Financial Assistance, USGS mercury work outside the CBDA program, San Francisco Estuary Institute, Southern California Coastal Water Research Project, Agricultural Waiver Programs, storm water monitoring and the discharger community.
2. A series of intercomparison studies conducted over the life of the CBDA Mercury Studies Program. The intercomparison studies will cover all matrix and analyte combinations represented by program samples.
3. The collection and analysis of replicate samples totaling five percent of the total number of samples collected and covering all matrix/analyte combinations. These split samples are analyzed by the contract laboratory and an independent Referee Laboratory, and the results from both labs will be evaluated by the CBDA QA Team.
4. Evaluation of the contract laboratories by a series of on-site audits conducted by the QA Team
5. Method detection limit (MDL) studies conducted by the contract laboratories and reviewed by the QA Team
6. Third-party validation of a selection of contract laboratory data sets to assess QA/QC practices and data reporting
7. Scientific and QA review of all methods used to ensure state-of-the-art procedures and good laboratory practices (GLP)

The intercomparison studies and split sample analyses give an indication of the comparability of the results generated by the various sub-tasks. The remaining tasks ensure transparency of the results. Taken as a whole, the QA Oversight Program provides a framework that allows one entity to evaluate the data generated by the many different participants in the project. This provides an early opportunity to identify and efficiently resolve any issues regarding data quality. Also, since many of the current members of the QA Team have extensive experience with mercury analysis, the QA Oversight Program has been able to increase the flow of valuable information between the participants. While the confidentiality of each laboratory's work is always held in high regard, it remains the case that many of the participants are performing related work. Valuable tips, taken both from the QA Team's personal experience, and those that are passed on by the

participants, can be shared with everyone to help solve common problems and improve the overall quality of the work.

PROJECT TIMETABLE AND MILESTONES

As a whole, the QA Oversight Program is behind schedule due to lengthy contracting delays. For example, the QA Project Plan (QAPP) was originally to be finalized by May 2004. However, the contract between CBDA and Department of Fish and Game (DFG) was delayed, as was the contract between DFG and Van Buuren Consulting (VBC). The latter contract was finally executed in March 2005, though it covers only one-half of the mercury projects. To accommodate the remaining mercury projects, CBDA approved a contract amendment in November of 2004. At the time of writing, the CBDA contract amendment is in processing at DFG. It is anticipated that project timelines will be extended to accommodate the contracting delays.

Details of the progress to date and future tasks are highlighted below and are separated by specific project activity. Please note that the reports cited, and standard operating procedures (SOPs) referenced, are (or will be) posted on the VBC website (www.vanbuurenconsulting.com).

INTERCOMPARISON STUDIES

A total mercury in freshwater sample and a methyl mercury in sediment sample were sent to the contract laboratories on June 7, 2005. These samples were distributed as part of the first CBDA Intercomparison Study designed and executed by the CBDA QA Team. Reference values for the samples were established as part of the preparation for the study, due to the paucity of certified reference materials for both methyl mercury in sediment and low-level total mercury in water. The results were received, compiled, and evaluated by calculating a z-score for each participant. Calculation of the z-score, and the criterion used to evaluate the laboratory performance are as follows.

$$z - score = \frac{\mu_{lab} - x_{ref}}{\sigma_{ref}}$$

Where:

μ_{lab} = the mean of the three values reported by the participating laboratory

x_{ref} = the reference value established for the intercomparison study

σ_{ref} = the target standard deviation for the study

To establish a target standard deviation of 10%, the value of σ_{ref} is defined to be,

$$\sigma_{ref} = 0.05 x_{ref}$$

With respect to the calculated z-scores, the laboratory results are evaluated as follows.

|z-score| ≤ 2 indicates that the laboratory results were within 10% of the study reference value

2 < |z-score| ≤ 5 indicates that laboratory results were between 10% and 25% of the reference value

|z-score| > 5 indicates that laboratory results were greater than 25% removed from the study reference value

A summary of the results of CBDA Mercury Studies QA Program Intercomparison Study-1 is provided below. The full report will be available on the VBC website (www.vanbuurenconsulting.com).

CBDA Laboratory Intercomparison Exercise 1: Total Mercury in Freshwater

QC Parameter	Laboratory Intercomparison Study Summary: Total Mercury in Freshwater				
	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E
Freshwater Reference Material Mean Value	**7.08 ng/L** 0.2% RSD n = 3	**7.59 ng/L** 0.2% RSD n = 3	**7.37 ng/L** 10.3% RSD n = 6	**6.75 ng/L** 1.3% RSD n = 3	**Pending**
Mean Method Blank	**0.17 ng/L** n = 3	**0.19 ng/L** n = 3	**Not Reported**	**<MDL**	**Pending**
Estimated MDL	**0.00 ng/L**	**0.03 ng/L**	**n/a**	**n/a**	**Pending**
z-score	**-1.417**	**-0.079**	**-0.656**	**-2.275**	**Pending**
Preparation Method	**BrCl Oxidation**	**BrCl Oxidation**	**BrCl Oxidation**	**BrCl Oxidation**	**UV Oxidation**
Analytical Method	**$SnCl_2$ Reduction CVAFS**	**$SnCl_2$ Reduction CVAFS**	**$SnCl_2$ Reduction CVAFS**	**$SnCl_2$ Reduction CVAFS**	**$NaBH_4$ Reduction CVAFS**

Note: Referee Laboratory is designated as Laboratory B

Results received thus far indicate good comparison between the laboratories for total mercury in water. The total mercury concentration of the study reference sample is on the order of total mercury concentrations seen in water samples submitted for the five percent split study, indicating good comparability on real samples. The results from Laboratory E are still pending, and will be appended in future reports.

CBDA Laboratory Intercomparison Exercise 1: Methylmercury in Sediment

QC Parameter	Laboratory Intercomparison Study Summary: Methylmercury in Sediment				
	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E
Mean Sediment Reference Material	**2.82 ng/g** 2.6% RSD n=3	**3.31 ng/g** 5.5% RSD n=3	**6.29 ng/g** 11.3% RSD n=5	**3.41 ng/g** 9.7% RSD n=3	**3.76 ng/g** 1.2% RSD n=3
Mean Method Blank	**0.008 ng/g** n=3	**0.003 ng/g** n=3	**Not Reported**	**<MDL**	**0.011 ng/g** n=3
Estimated MDL	**0.000 ng/g**	**0.002 ng/g**	**n/a**	**<MDL**	**0.011 ng/g**
z-score	**-3.585**	**-0.736**	**n/a-Outlier**	**-0.155**	**1.860**
Preparation Method	**$MeCl_2$ Extraction**	**$MeCl_2$ Extraction**	**Distillation**	**$MeCl_2$ Extraction**	**$MeCl_2$ Extraction**
Analytical Method	**Aqueous Phase Ethylation CVAFS**	**Aqueous Phase Ethylation CVAFS**	**Aqueous Phase Ethylation CVAFS**	**Aqueous Phase Ethylation CVAFS**	**Aqueous Phase Ethylation CVAFS**

Note: Referee Laboratory is designated as Laboratory B
Results from Laboratory C have been excluded as outliers

Of the four contract laboratories, two submitted results for methylmercury in sediment that could be rated "good", one submitted results that rated "fair", and the remaining lab submitted results that were approximately double the established reference concentration and were excluded as outliers. The comparability of methylmercury in sediment results, as evidenced by the first round of intercomparison samples, is not consistent across the laboratories participating in the program. In order to identify and correct any possible problems leading to the discrepancies noted, discussions are continuing with laboratories that received other than a "good" rating.

The first intercomparison exercise was completed on schedule, with the exception that results are still pending from Laboratory E. This delay is beyond the control of the laboratory, and will not be reported here so that confidentiality will be maintained.

The second CBDA Intercomparison Study is currently being planned, with shipment of the samples currently scheduled for the second half of November 2005. This study will cover all matrix/analyte combinations (total and methylmercury in water, sediment, and tissues). Contract labs are required to participate within the scope of their work on the project, but are welcome to participate in the full study.

Samples for the second intercomparison exercise were scheduled to be shipped in September. However, they are currently scheduled for shipment in the second half of November 2005.

FIVE PERCENT SPLIT SAMPLE ANALYSIS

All PIs are required to collect a number of duplicate field samples (split samples). One of these samples is submitted to the contract laboratory, while the other is submitted to a designated referee laboratory (Brooks Rand, LLC). The number of split samples collected

is to total five percent of the total quantity collected for each sub-task, and should represent the requested sample type and target analyte(s).

At this point in the project, the total number and type of split samples that have been analyzed and reported are as follows.

- 23 water samples for total mercury
- 41 water samples for methylmercury
- 5 sediment samples for total mercury
- 8 sediment samples for methylmercury

To date, no tissue samples have been submitted for split sample analysis.

Results for the split field samples were submitted by the contract and referee laboratories and compiled by the CBDA QA Team. Concentrations from the parent and duplicate sample were compared side-by-side, and a relative percent difference (RPD) for each pair was calculated using the following formula.

$$\text{RPD} = \left(\frac{|\text{contract} - \text{referee}|}{\text{mean}} \right) \times 100$$

Where:

contract = the result submitted by the contract laboratory
referee = the result submitted by the Referee Laboratory
mean = the calculated mean of the two submitted results

Pairs of results requiring further scrutiny and possible corrective action were identified using the measurement quality objective (MQO) for field replicates detailed in the CALFED QAPP (Puckett et al, 2002). Split sample pairs with an RPD greater than the acceptance criterion where the difference between the results is greater than a specified reporting limit (RL) were highlighted and investigated. The RL specified in the CALFED QAPP is by definition 10 times the method detection limit (MDL) for each matrix/analyte combination. For the purposes of evaluating the split sample results, however, an RL defined to be five times the MDL is used to evaluate the data. This tighter limit better reflects the type and quality of data needed to meet the data quality objectives (DQO) of the project.

The RPDs and reporting limits specific to each matrix/analyte combination are detailed in the following table.

Matrix/Analyte Combination	MQO (RPD)	RL (5xMDL)
Total Mercury in Water	25%	1.0 ng/L
Methylmercury in Water	25%	0.100 ng/L
Total Mercury in Sediment	35%	2.5 ng/g
Methylmercury in Sediment	35%	0.025 ng/g
Total Mercury in Tissue	25%	2.5 ng/g
Methylmercury in Tissue	25%	5.0 ng/g

The specific number and type of samples submitted by each PI is detailed below.

Principle Investigator	Total Mercury In Water	Methyl Mercury In Water	Total Mercury In Sediment	Methyl Mercury In Sediment
Chris Foe Central Valley Regional Water Quality Control Board	11	14	0	0
Mark Stephenson Department of Fish and Game	2	17	0	3
Gary Gill Texas A&M University (Galveston)	4	4	2	2
Mark Marvin-DiPasquale USGS	6	6	3	3
Total	**23**	**41**	**5**	**8**

A full accounting of the results and required corrective actions is detailed in The California Bay-Delta Authority (CBDA) Mercury Studies QA Program Analysis of Split Samples-Report 1 (will be available on the VBC website (www.vanbuurenconsulting.com)).

ON-SITE ASSESSMENTS (AUDITS)

All of the laboratories participating in the CBDA Mercury Studies program have been scheduled to participate in an on-site audit by the QA Team. Laboratory audits are performed in accordance with Van Buuren Consulting SOP Hg432v1 "On-Site Assessment Procedure for the California Bay-Delta Mercury Studies Quality Assurance Program". The current status of laboratory audits is summarized below.

Laboratory	Date of On-site Audit	Final Audit Report Issued
Moss Landing Marine Lab (MLML)	February 9, 2005	March 25, 2005
Brooks Rand LLC (designated referee laboratory)	May 23, 2005	June 27, 2005
Texas A&M University (TAMUG)	June 29, 2005	Pending
Battelle Marine Sciences Laboratory (Sequim)	September 13, 2005	October 12, 2005
University of Connecticut (UConn)	Pending	Pending

Prior to the on-site visit, a desk-top audit was conducted by a member of the QA Team. The desk-top audit consists of a review of the laboratory documentation relevant to the project, such as standard operating procedures (SOPs), laboratory datasets, and method detection limit (MDL) studies.

On-site audits are conducted by one or more members of the QA Team. An audit checklist covering several aspects of laboratory operations is completed during the desk-top and on-site phases of the audit. Face-to-face meetings held with key laboratory personnel allow the QA Team to address any questions that arise during the auditing process. The meetings also give the laboratories an opportunity to submit questions to the QA Team regarding the project's quality requirements.

Following the on-site audit, a formal audit report is generated and submitted to the laboratory for comment. The report details findings, observations, and recommendations noted during the audit, and may include suggested corrective actions. The laboratory is then required to make a formal response to the audit report, detailing proposed corrective actions to the findings and observations. Following the initial visit, and at the discretion of the project QA Officer, future assessments may be limited to desk-top audits.

The first round of audits will be completed early in 2006 following a visit to the University of Connecticut laboratory. While the first audits were scheduled to have been completed at this point, the schedule has been extended in the case of the University of Connecticut to give them time to set up their laboratory. A schedule of future audits will be released in July 2006.

METHOD DETECTION LIMIT (MDL) STUDIES

All laboratories participating in the CBDA Mercury Studies Program are required to submit MDL studies reflecting the scope of their contract. An SOP detailing specific instructions detailing the design and reporting of MDL studies was distributed to the laboratories and PIs during the CBDA Mercury Studies Kickoff Meeting in May 2005 (VBC SOP#433v0:*Draft* Method Detection Limit Study Procedure for the California Bay-Delta Authority Mercury Studies Quality Assurance Program).

MDL studies must be performed for each matrix/analyte combination on an annual basis. In March, laboratories were permitted to submit MDL studies performed prior to the start of the program as long as these studies conformed to the requirements in 40 CFR Section 136 Appendix B: Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11.

A listing of the MDL studies submitted to the QA Team at this point is detailed in the following table.

Laboratory	Analyte	Matrix	MDL	Study Date
Moss Landing Marine Laboratory (MLML)	Total Hg	Water	0.16 ng/L	1/8/2004
	Methyl Hg	Water	0.009 ng/L	10/28/2004
	Total Hg	Sediment	5.64 ng/g	8/31/2004
	Methyl Hg	Sediment	0.012 ng/g	8/18/2004
Battelle Marine Sciences Laboratory	Total Hg	Water	0.17 ng/L	1/21/2005
	Methyl Hg (A)	Water	0.011 ng/L	2/1/2005
	Methyl Hg (B)	Water	0.019 ng/L	1/20/2005
Texas A&M University	Methyl Hg	Water	0.006 ng/L	6/21/2005
	Methyl Hg	Sediment	0.033 ng/g	7/1/2005

These can be contrasted with the currently proposed project-mandated MDLs specified for the analysis of low-level environmental samples.

Matrix/Analyte Combination	MDL
Total Mercury in Water	0.2 ng/L
Methylmercury in Water	0.020 ng/L
Total Mercury in Sediment	0.5 ng/g
Methylmercury in Sediment	0.02 ng/g
Total Mercury in Tissue	0.5 ng/g
Methylmercury in Tissue	1.0 ng/g

MDL studies from the University of Connecticut are pending since the laboratory was recently set up and is still in the process of starting up and validating its procedures.

To date, verification of laboratory MDL studies is incomplete. In order to fulfill this portion of the QA Oversight Program, the QA Team is in the process of individually contacting the laboratories to collect supporting information. While details are considered confidential, the QA Team will be working with the laboratories to address the following deficiencies:

- Supporting information that has not yet been provided with MDL studies that have already been received, such as raw data.
- New MDL studies to replace ones that have expired or did not conform to 40 CFR Section 136 Appendix B.
- MDL studies for matrix/analyte combinations that have not been received.

DATA SET REVIEW AND VALIDATION

Each year, contract laboratories are required to submit two datasets from each matrix/analyte combination for review by the CBDA Mercury Studies QA Team. Data sets are evaluated for documentation issues, consistency of calculations, QC sample use, scientific coherence, and data usability.

The QA Team is still in the process of collecting and verifying data sets from the contract laboratories. Initial review has revealed potential issues with regard to the transparency of the data - more specifically the ability of third parties to evaluate and the data and recreate the final results based on the information provided. In some instances, critical information is missing, in others the method of data reduction is difficult to recreate.

Data review is critical to the program, both with regard to validating data for the current project, and for ensuring that the data is able to be considered for use in future projects. It is in this area that the QA Team can offer a great deal of assistance. The QA Team has background both in mercury analysis and in the day-to-day workings of an analytical laboratory. Based on this experience, it is the opinion of the QA Team that transparency can be addressed without increasing the amount of paperwork performed by the laboratories. Doing so streamlines work and frees up valuable time for other activities. The QA Team will continue to work with the laboratories on a case-by case-basis.

WRITTEN METHODS EVALUATION

A preliminary review of each laboratory's SOPs has been conducted as part of each desk-top audit conducted by the QA Team. This review covered administrative details, such as how often they are reviewed and revised, and document tracking procedures. It also assessed conformance to the QC requirements of the CBDA Mercury Studies Program, which were communicated to participating laboratories in the Kick-off information packet. Results of this portion of the review can be found in the final audit report of each contract laboratory.

Full validation and acceptance of all laboratory SOPs is still in progress. The majority of the SOPs reviewed so far contain sufficient detail with regard to involved chemistry, but several have been found to lack details of QC samples, results calculation, and corrective actions. While the goal was to have validation completed prior to allowing the laboratories to analyze project samples, this would have brought significant portions of the mercury project to a halt. Using the intercomparison studies and the split analysis study as an evaluation tool, analyses have been allowed to continue. However, the QA Team is working with the laboratories to ensure that the necessary details are added to revisions of the laboratory procedures.

CONCLUSIONS

Based on the results of both the first intercomparison study and the comparison of the results from the split field samples, the majority of the data being generated for the program compare well across the board. This assertion is based on the small amount of data collected so far, and as the QA oversight program progresses a clearer picture of comparability of the project data will emerge. This is an important finding, not only with regard to the current project, but for the quality of other projects as well, allowing end users of the data to confidently reference information from the program to apply to planning and interpretation of future studies.

Initial review of the data sets and SOPs by the various laboratories has revealed one area where improvement would greatly increase the usability of the data, both for this current project and for future projects. This area involves what is usually referred to as the transparency of the data - the ability to gather various pieces of information used to generate the final results and recreate and verify these results. Transparency is important in evaluating the data for the current project, and will be critical if the information generated by this project is used to support future work.

The QA Team will continue to work with the laboratories on this issue. The main focus will be to create systems for documenting the work so that the information being used to generate the data is clear and easy to retrieve. The goal is to improve this area of data quality without creating a large amount of paperwork for either the principle investigators or the laboratories. So far, two specific areas have been identified that would greatly improve data transparency.

- The amendment of SOPs to address the calculation of the final data, including details of how the results are corrected for blanks.
- The use of spreadsheets to calculate the data, where all of the information (such as sample volumes, sample masses, and dilution factors) is clearly detailed so that the calculation of results can be followed step-by-step.

Based on the personal experience of the QA Team, these objectives should be achievable without a large amount of extra work, and will result in a great improvement in the quality of the project.

REPORTS, PUBLICATIONS, AND PRESENTATIONS

Reports and publications are available at www.vanbuurenconsuling.com.

DOCUMENTS and SOPs ISSUED MARCH - OCTOBER 2005

The CBDA Mercury Studies QA Program Kick-off Meeting Information Packet, The CBDA QA Team at VBC, May 2005.

SOP Topic	CBDA SOP Title	Version #	Revision Date
Lab Audits	On-Site Systems assessment Procedure for the California Bay-Delta Authority Mercury Studies Quality Assurance Program	Hg432v1	2/4/2005
MDL Studies	Method Detection Limit Study Procedure for the California Bay-Delta Authority Mercury Studies Quality Assurance Program	Hg433v0 ***Draft***	5/4/2005
Data V&V	Data Verification and validation Procedure for the California Bay-Delta Authority Mercury Studies Quality Assurance Program	Hg451v0 ***Draft***	5/5/2005
Method Evaluation	Method Evaluation Procedure for the California Bay-Delta Authority Mercury Studies Quality Assurance Program	Hg435v1 ***Draft***	5/13/2005
Split Samples	Split Sample Processing Procedure for the California Bay-Delta Authority Mercury Studies Quality Assurance Program	Hg452v1	2/28/2005

PRESENTATIONS/PUBLICATIONS ISSUED MARCH - OCTOBER 2005

Van Buuren, B.H., Hagan, W., and Deanovic, L., 2005, "Quality Assurance and Quality Control Tools for Monitoring Projects – The CBDA Mercury QA Program as an Exercise," Part of a full-day workshop entitled, "Monitoring Design – An Introduction," at The 2005 California Nonpoint Source Pollution Conference, Sacramento, California, November 2005.

Van Buuren, B.H., 2005, "The Quality Assurance Oversight Program for Mercury Studies," The San Francisco Estuary Institute Newsletter, February 2005.

Van Buuren, B.H., and Crane, D.B., 2005, "Elements of the QA Oversight Program for Mercury Speciation," The Annual Mercury Workshop hosted by SFEI, San Francisco, California.

REPORTS ISSUED: MARCH - OCTOBER 2005

The California Bay-Delta Authority (CBDA) Mercury Studies QA Program Intercomparison Study 1-June 2005:Total Mercury in Freshwater, Methylmercury in Sediment, The CBDA QA Team at VBC, September 2005.

The California Bay-Delta Authority (CBDA) Mercury Studies QA Program Analysis of Split Samples-Report 1, The CBDA QA Team at VBC, October 2005.