

Laboratory QA Summary

CW4CB Task 3, Phases I and II

May 7, 2014

Submitted to:

Mr. Geoff Brosseau
Bay Area Stormwater Management Agencies Association (BASMAA)
P.O. Box 2385
Menlo Park, CA 94026
info@BASMAA.org

Submitted by:



4749 Bennett Drive, Suite L
Livermore, CA 94551
925-373-7142

1. Introduction

The Bay Area Stormwater Agencies Association (BASMAA) contracted with Applied Marine Sciences, Inc. (AMS) to support implementation of CW4CB Task 3 and Task 5. As part of its contract with BASMAA, AMS is providing project quality assurance for all Task 3 and Task 5 activities. Don Yee at SFEI is the Project QA Officer (QAO), and has completed data review of Task 3 (Phase 1 & 2) and Task 5 (Phase 1) analytes. Below are narrative summaries of reviews of QA/QC samples analyzed with reported field samples for the project. QA/QC samples were evaluated using the procedures and measurement quality objectives (MQOs) described in the project QAPP (BASMAA 2013).

2. Task 3 Phase 1

Task 3 Phase 1 field monitoring was conducted in September and October of 2012. Hg samples were analyzed by two laboratories – (1) Soil Control Lab (SCL) for samples collected by KLI within San Mateo and Santa Clara Counties, and (2) ALS Global / Columbia Analytical Services (ALS) for samples collected by AMS within Alameda and Contra Costa Counties. In the case of PCBs, all samples collected for the Project were analyzed by ALS.

QA/QC results generally met project MQOs, with some minor deviations. Some mercury contamination was found in blanks from one lab (SCL), and some congeners in PCB blanks (ALS), likely affecting results for some of the lowest concentration samples reported by the labs, which were censored (not reported) in those samples as a result. PCBs in sediment samples showed moderate to large variation in replicate samples for some of the less abundant congeners; one congener (PCB 20/28) showed very large variation (>100% RPD) and was therefore censored. Details on the individual data submittals by various labs are provided below. All data should be considered preliminary until release of final data submittal by BASMAA.

2.1. PCBs - ALS

PCBs in sediment samples were analyzed by ALS. Samples were collected between September 24 and October 4, 2012, and were analyzed between October 24 and November 25, 2012. The 40 PCB congeners reported by the Regional Monitoring Program for Water Quality in the San Francisco Estuary¹ were reported for 81 field samples (including replicates). Blank and LCS (recovery) samples were also reported.

2.1.1. Sensitivity

PCB 008 was not detected (ND) in about 1/3 of the samples, but aside from that, most of the analytes were ND in only 2 to 6 of the 81 samples analyzed for PCBs.

2.1.2. Blanks

About half the analytes (20) were found in the blank at least 2x the MDL in one or more batches (QAPP page 7-5). For 13 of those, blanks were possibly > 10% of the field sample value in 2 or 3 samples - cases with ND results for diluted samples. It is unknown whether the blank signal would constitute a significant portion of the sample in these cases due to their dilution, so those results were censored with

¹ The RMP 40 list of PCBs has been the historic suite of PCB congeners analyzed by BASMAA agencies.

VRIP flag (Data rejected - Analyte detected in field or lab generated blank, flagged by QAO) as a worst-case assumption. Other samples were flagged with VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning but not censored since the blank contamination was <10% of those field sample concentrations.

2.1.3. Recovery

Recovery results were only reported for LCSs, with good recovery (within the 70-130% recovery (30% error) QAPP Table 26-2 target for PCBs. Only 3 out of the 29 reported analytes in the LCS were in the RMP 40 target analytes; although a range of PCB homologs were included in the LCS, some interferences are congener specific and would not necessarily be identified in the LCS.

2.1.4. Precision

Precision was calculated only for sample pairs where an analyte was detected in both samples. Precision results were averaged across batches for analytes, and those with average RPDs > the 25% MQO (QAPP Table 26-2) were flagged as having marginal precision but not censored, and those with RPDs grossly above the MQO (average RPD >50%) were censored (see table below). Only one analyte, PCB 20/28, fell into this latter category.

| PARAMETER | avgRPD |
|---------------------------|----------------|
| PCB 020/28 | 100.76% |
| PCB 086/87/97/109/119/125 | 32.80% |
| PCB 105 | 44.86% |
| PCB 110/115 | 31.82% |
| PCB 118 | 26.64% |
| PCB 132 | 33.76% |
| PCB 141 | 45.14% |
| PCB 147/149 | 26.00% |
| PCB 156/157 | 27.53% |
| PCB 158 | 33.14% |

2.2. Hg - ALS

Mercury in sediment samples collected by AMS/ADH was analyzed by ALS. Samples were collected between September 25 and October 4, 2012, and were analyzed between October 23 and 25, 2012. Total mercury in the <2mm sediment fraction was reported for 47 field samples (including replicates and an intercomparison sample). Blank and LCS (recovery) samples were also reported.

2.2.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for total mercury.

2.2.2. Blanks

No mercury blank contamination was found.

2.2.3. Recovery

Recovery results were only reported for LCSs, with good recovery (within the QAPP Table 26-4 target 75-125% recovery (25% error) for mercury (actual average 4.6%). The LCS used was ERA D076-540, a metal spiked soil from ERA (external supplier).

2.2.4. Precision

Precision on lab replicates was good, averaging 6.6% RPD, less than the 25% MQO (QAPP Table 26-4), so no results were flagged for marginal or poor precision. Field replicates were more variable, with individual pairs up to 37% RPD, suggesting field sample heterogeneity, although the average of all replicate pairs was still <25% RPD.

2.2.5. Intercomparison sample

Results included one sample analyzed for intercomparison to a second lab. The PUL8 result of 0.073 mg/kg dw here, was 51% lower (67% RPD) than the average of SCL results for the same site of 0.22, 0.11 and 0.11 mg/kg dw (for a split sample, a split blind field duplicate, and its lab duplicate, respectively). The intra-lab variation between the split sample and its blind dupe suggest some variation due to sample heterogeneity

2.3. Hg - SCL

Mercury in sediment samples collected by KLI was analyzed by SCL. Samples were collected between September 24 and October 2, 2012, and were analyzed on October 12, 2012. Total mercury in a <2mm sediment fraction was reported for 36 field samples (including replicates). Blanks, MS/Ds, LCM, and CRM (recovery) samples were also reported.

2.3.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for total mercury.

2.3.2. Blanks

Mercury blank contamination was found slightly over 2x the MDL (QAPP page 7-5), but at concentrations averaging only 0.0057 mg/kg. Four of the lowest concentration samples (all from Pulgas) were < 10x that and flagged VRIP (censored), but the remainder were flagged without censoring (VIP flag).

2.3.3. Recovery

Recovery samples had good recovery (within the 75-125% recovery (25% error) target (QAPP Table 26-4) for mercury. Recovery errors on the LCM & CRM averaged 6.7%, and on the MS/Ds 4.4%, well within the target, so no recovery qualifiers were needed.

2.3.4. Precision

Precision on lab replicates was good, averaging 2.2% RPD, less than the 25% MQO (QAPP Table 26-4), so no results were flagged for marginal or poor precision. Field replicates were more variable, with 45% RPD suggesting field sample heterogeneity, but results were not flagged for potentially variable field replicates.

2.3.5. Intercomparison sample

Results included one sample analyzed for intercomparison to a second lab. The ETT122a result of 0.27 mg/kg dw here, 41% lower (51% RPD) than the average of ALS results for the same site of 0.4 and 0.51

mg/kg dw. Intra-lab variation on sample replicates was 24%, suggesting some sample heterogeneity, which may account for a portion of the total variation.

2.4. PAHs - ALS

PAHs in sediment samples collected by AMS were analyzed by ALS. Samples were collected between September 27 and October 4, 2012, and were analyzed on October 20-23, 2012. PAHs in a <2mm sediment fraction were reported for 4 field samples. A blank, MS/Ds, and two LCS samples were also reported.

2.4.1. Sensitivity

Sensitivity was sufficient to have no NDs for most PAHs, but Biphenyl, 2,6-Dimethylnaphthalene, Dibenzothiophene, and Fluorene had 1 ND each, and Acenaphthene had 2.

2.4.2. Blanks

PAHs were not found in the blank at concentrations over the detection limit.

2.4.3. Recovery

Matrix spike recovery was never outside the target MQO of 50-150%, but average recovery errors above 35% were seen for Dibenz(a,h)anthracene, 2,6-Dimethylnaphthalene, Indeno(1,2,3-c,d)pyrene, and 2-Methylnaphthalene.

2.4.4. Precision

Precision on lab replicates was good for most analytes, with RPDs less than the 25% MQO (QAPP Table 26-2), except for Anthracene, Biphenyl, 2,6-Dimethylnaphthalene, and Fluorene, with RPDs ranging 26-37%.

2.5. PAHs - ATL

PAHs in sediment samples collected by KLI were analyzed by ATL. Samples were collected between September 24 and October 2, 2012, and were analyzed on October 30 and November 8, 2012. PAHs in a <2mm sediment fraction were reported for 4 field samples. A blank, MS/Ds, and an LCS sample were also reported.

2.5.1. Sensitivity

Sensitivity was sufficient to have no NDs for most PAHs, but Acenaphthylene, Anthracene, Fluorene, 2-Methylnaphthalene, and Naphthalene each had one ND result.

2.5.2. Blanks

Fluorene was found in the blank at a concentration over the detection limit, and greater than or equal to one-third the concentration in field samples, so all Fluorene results were censored.

2.5.3. Recovery

Matrix spike recoveries were never outside the target MQO of 50-150%, and all had <25% error for analytes spiked in a quantitative range (at least 3x MDL).

2.5.4. Precision

Precision on matrix spike replicates was good for the 3 analytes spiked, with RPDs less than 10%, well within the 25% MQO (QAPP Table 26-2).

2.6. Pesticides - ALS

Pesticides in sediment samples collected by AMS/ADH were analyzed by ALS. Samples were collected between September 27 and October 4, 2012, and were analyzed on November 6, 2012. 31 pesticides, mostly legacy organochlorine compounds, in a <2mm sediment fraction were reported for 4 field samples. A blank, MS/Ds, and two LCS samples were also reported.

2.6.1. Sensitivity

Sensitivity was sufficient to have detections of only about half the pesticides, and all but 6 analytes had one or more NDs, despite MDLs mostly <1 ug/kg, meeting the project QAPP targets (most 1 ug/kg or more).

2.6.2. Blanks

Pesticides were not found in the blank at concentrations over the detection limit.

2.6.3. Recovery

Matrix spike recovery was outside the target MQO of 50-150% only for Endrin Aldehyde (47%), with Endosulfan I, Endrin, and Hexachlorobenzene approaching those limits with deviations above 35%.

2.6.4. Precision

Precision on was good for most analytes, with RPDs less than the 25% MQO (QAPP Table 26-2), except for Oxychlordan, Isodrin, Hexachlorobenzene, and Endrin Ketone, with RPDs ranging 28-63%.

2.7. Pesticides - ATL

Pesticides in sediment samples collected by KLI were analyzed by ATL. Samples were collected between September 24 and October 2, 2012, and were analyzed on October 10 to 15, 2012. 22 pesticides (mainly legacy organochlorines) in a <2mm sediment fraction were reported for 4 field samples and 1 intercomparison sample. A blank, MS/Ds, and an LCS sample were also reported. The analytes reported omitted a number requested in the project QAPP, namely all of the o,p' DDT derivatives, as well as hexachlorobenzene, mirex, cis & trans nonachlor, and oxychlordan.

2.7.1. Sensitivity

Samples were 100% NDs except for the chlordanes and DDTs. Most of the MDLs were <1ug/kg dw so met the project requirements

2.7.2. Blanks

None of the target analytes were detected in blanks.

2.7.3. Recovery

Recovery was evaluated from matrix spikes and spike dupes, spiked for 6 of the analytes. Recoveries on all the spiked analytes were <70%, always biased low, but within the project target of 50-150%.

2.7.4. Precision

Precision was measured via matrix spike duplicates, with RPDs within the MQO target of <25% (QAPP Table 26-2) except for p,p' DDT (RPD 32%) which was flagged VIL (not meeting precision target) but not censored

2.7.5. Intercomparison samples

Results included one sample analyzed by ALS. RPDs between lab results for analytes reported by both ranged from 17 to 90%, with the 90% RPD occurring on an analyte <3xMDL for one of the labs. There were a half dozen or so analytes reported by ALS without results from ATL in the intercomparison sample.

2.8. PBDEs - ALS

PBDEs in sediment samples collected by AMS/ADH and KLI were analyzed by ALS. Samples were collected between September 24 and October 4, 2012, and were analyzed on November 26 and 27, 2012. 17 PBDE congeners, in a <2mm sediment fraction were reported for 8 field samples. A blank and two LCS samples were also reported.

2.8.1. Sensitivity

Detection limits ranged 14 to 174 ug/kg dw (highest for PBDEs 206 and 209), about 100x above the project target MRLs of 0.1 to 1 ug/kg. PBDEs were NDs in all samples except for 47, 99, and 209. These are typically among the most abundant PBDEs.

2.8.2. Blanks

No PBDEs were found in the blank at concentrations over the detection limit.

2.8.3. Recovery

LCSs were used to evaluate accuracy. Average recoveries had <25% error for all congeners, within the MQO target, so no records needed to be qualified.

2.8.4. Precision

Replicates of the LCS were used to evaluate precision. The average RPD was <7% for all congeners in the LCS, below the target MQO of 25%. No additional qualifiers were added.

2.9. Dioxins - ALS

Dioxins in sediment samples collected by AMS/ADH and KLI were analyzed by ALS. Samples were collected between September 24 and October 4, 2012, and were analyzed between October 18 and November 2, 2012. 17 dioxin and furan congeners with 2,3,7,8-TCDD activity, in a <2mm sediment fraction were reported for 8 field samples. Two blanks and 3 LCS samples were also reported.

2.9.1. Sensitivity

Detection limits ranged 0.07 to 22.5 ng/kg dw (highest for OCDD), slightly above the project target MRLs of 1 to 10 ng/kg for some of the analytes. 1,2,3,7,8,9-HxCDF was ND in all samples, and 2,3,7,8-TCDD, and 1,2,3,7,8-PeCDF were ND in one sample each.

2.9.2. Blanks

Two lab blanks were reported with 41% (7 out of 17) of the individual analytes having some blank contamination. Most blank contaminations was <10% of field sample concentrations, but one 1,2,3,4,7,8,9- HpCDF result was censored for a higher contribution of the blank.

2.9.3. Recovery

LCSs were used to evaluate accuracy. Average recoveries had <25% error for all analytes, within the MQO target, so no records needed to be qualified.

2.9.4. Precision

Replicates of the LCS were used to evaluate precision. The average RPD was below the target MQO of 25% for all congeners in the LCS so no additional qualifiers were added.

2.10. TOC - ALS

Total organic carbon (TOC) in sediment samples collected by AMS/ADH were analyzed by ALS. Samples were collected between September 27 and October 4, 2012, and were analyzed on October 22 to 23, 2012. TOC in a <2mm sediment fraction were reported for 42 field samples, with 3 lab replicates and one field replicate. Lab blanks, MS/Ds, and LCS samples were also reported.

2.10.1. Sensitivity

The detection limits were above the project QAPP target of 0.01%, but all TOC results were above the detection limits (TOC MDL 0.05%) with no NDs reported.

2.10.2. Blanks

TOC was not found in the blanks at concentrations over the detection limit.

2.10.3. Recovery

MSs were spiked to around 20% TOC, near the high end of field sample concentrations. Recovery errors for the MSs averaged 4%, well within the target MQO of 80-120%. LCSs spiked to only 5x the MDL (0.28% TOC, below any samples) had OK recovery as well, averaging 19% error, so no additional qualifiers were needed.

2.10.4. Precision

Lab replicates were used to evaluate precision for TOC. The average RPD, around 3.5%, was well within the target MQO of 25% for TOC. A field sample replicate was analyzed, but not used to assess precision, and had a similarly small RSD of 2.5% (analyzed 3x total, paired with a sample with a lab replicate).

2.11. TOC - ATL

Total organic carbon (TOC) in sediment samples collected by KLI were analyzed by ATL. Samples were collected between September 24 and October 2, 2012, and were analyzed on October 11, 2012. TOC in a <2mm sediment fraction was reported for 31 field samples, along with lab replicates, field blind replicates, lab blanks, and CRMs.

2.11.1. Sensitivity

All TOC results were above the detection limits (TOC MDL 0.01, meeting the project QAPP target) with no NDs reported.

2.11.2. Blanks

Some TOC was measured in blanks, but at concentrations (0.02% dw) less than 10% of the lowest field sample (0.28% dw TOC) so no results were censored.

2.11.3. Recovery

Two certified reference materials (CRMs) were run for TOC and were used to evaluate accuracy. Recovery for the CRMs was 97 to 98%, within the target MQO of 80-120%.

2.11.4. Precision

The average RPD for lab replicates was within the target MQO of 25% for TOC (individual RPDs of 12.6 and 1.2%). Field sample replicates were not used to flag precision, also met the target with an average RPD of 17.8%.

2.12. Total Solids - ALS

Total solids in sediment samples collected by AMS/ADH and KLI were analyzed by ALS. Samples were collected between September 24 and October 4, 2012, and were analyzed between October 8 and November 1, 2012. Total solids in a <2mm sediment fraction were reported for 179 samples, including 15 lab replicates and 9 field replicates. No other sample types were reported.

2.12.1. Sensitivity

Total solids were reported to within 0.1%, with no NDs reported.

2.12.2. Precision

Lab replicates were used to evaluate precision for total solids, with RPDs <1%, well within the <25% RPD for other sediment conventional analytes.

2.13. Total Solids - ATL

Total solids were reported in sediment samples analyzed by ATL for intercomparison for chemical analytes. Samples were collected between September 24 and October 2, 2012, and were analyzed on October 8 to 12, 2012. Total solids in a <2mm sediment fraction was reported for 5 field samples, plus 1 lab replicate.

2.13.1. Sensitivity

Total solids were reported to the nearest 1%, with no NDs reported.

2.13.2. Precision

The RPD for lab replicates was <1%, within the target MQO of 25% for other sediment conventional analytes.

2.13.3. Intercomparison samples

The difference in average total solids between the two labs was good, always <3% RPD.

2.14. Grainsize - ALS

Grainsize in sediment samples collected by AMS/ADH were analyzed by ALS. Samples were collected between September 27 and October 4, 2012, and were analyzed between October 23 and 26, 2012. Grainsize in 9 fractions (from <75 to <0.005 mm) using the ASTM scale were reported in sediment samples for 45 samples, including 3 lab replicates and 4 field replicates. Some of the ASTM sizes are slightly offset from the ranges requested in the project QAPP. No other sample types were reported.

2.14.1. Sensitivity

Grainsize fractions were reported to within the nearest 0.1% or better, with no NDs reported.

2.14.2. Precision

Lab replicates were used to evaluate precision for grainsize. The average RPD was below the target MQO of 25% for all size ranges except Medium Gravel (55% RPD), flagged but not censored (CW4CB has no listed censoring threshold). Field sample replicates showed similar variability, with some RPDs higher and others lower than for lab replicates,

2.15. Grainsize - SCL

Grainsize in sediment samples collected by KLI were analyzed by SCL. Samples were collected between September 24 and October 2, 2012, and were analyzed October 19, 2012. Grainsize in 17 fractions (from <64 to <0.00098 mm) using the Wentworth/Plumb scale were reported in sediment samples for 31 field samples, plus 2 lab replicates and 3 field replicates. No other sample types were reported.

2.15.1. Sensitivity

Grain sizes were reported to the nearest 0.01% or better, with frequent (sometimes 100%) NDs reported for some of the coarser (pebble) fractions.

2.15.2. Precision

Lab replicates were used to evaluate precision, except the three pebble fractions which were mostly NDs. Average RPD was above the target MQO (25%) for the majority of analyte/fraction combinations (eight of 14 fractions with detects), and were flagged with a qualifier "VIL" for precision outside the MQO target. Field sample replicates were analyzed, but not used to assess precision, and had similarly large RPDs ranging from 0.02 to 145% (the latter for a pebble fraction, likely to be heterogeneous), with 47% of analyte/fraction combinations having average RPDs>25%. The frequent exceedance of the 25% RPD MQO may in part be due to the numerous fractions; with each fraction representing a smaller portion of the total size range, equivalent shifts in absolute percentages of total mass become amplified when expressed as RPD (a percentage relative to the average of a given fraction).

3. Task 3 Phase 2

Task 3 Phase 2 field monitoring was conducted May 14 & 15, 2013. ALS Global / Columbia Analytical Services (ALS) analyzed samples for Hg and sediment quality (TOC, bulk density, grainsize), dioxins/furans, PAHs, PBDEs, PCBs, and pesticides. Soil Control Lab (SCL) analyzed samples for Hg and sediment quality (grainsize and TOC).

QC results generally met project MQOs, with some minor deviations, particularly for organic contaminants. For example, some contamination was found in PCB blanks, likely affecting results for some of the lowest concentration samples reported by the labs, which were censored (not reported) in those samples as a result. PCBs in sediment samples showed moderate to large variation in replicate samples for many congeners, with some showing large variation (>50% RPD) and therefore those were flagged as estimated. Details on the individual data submittals by various labs are provided below. All data should be considered preliminary until release of final data submittal by BASMAA.

3.1. Hg – ALS

Mercury in a small number of sediment samples collected for Task 3 Phase 2 was analyzed by ALS. Samples were collected May 14, 2013 and were analyzed May 24, 2013. Total mercury in a <2mm sediment fraction was reported for 4 samples (including one replicate). Blank, LCM (recovery), and MS/MSD samples were also reported.

3.1.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for total mercury.

3.1.2. Blanks

Mercury was not detected in blanks above the MDL.

3.1.3. Recovery

Recovery for matrix spikes averaged 3.1% error (within the QAPP Table 26-3 target 75-125% recovery (25% error) for mercury. The average error for laboratory control materials of 1.6% was also good.

3.1.4. Precision

Precision on the lab replicates was acceptable, 8.4% RPD, within the 25% MQO (QAPP Table 26-3).

3.2. TOC, bulk density, total solids – ALS

Total organic carbon (TOC) and bulk density in a small number of sieved (<2mm) sediment samples collected for Task 3 Phase 2 were analyzed by ALS. A larger number of samples (30, including replicates) was reported for total solids. Samples were collected May 14 & 15, 2013, and were analyzed May 20 to June 26, 2013. Blank, LCM (recovery), and MS/MSD samples were also reported.

3.2.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for TOC, bulk density, or total solids.

3.2.2. Blanks

TOC was not detected in blanks above the MDL. Bulk density and total solids are not concentrations measurable in blanks.

3.2.3. Recovery

Recovery for TOC matrix spikes averaged 1.8% error (within the QAPP Table 26-1 target 75-125% recovery (25% error) for TOC.

3.2.4. Precision

Precision on TOC lab replicates was acceptable, with 2.5% RPD, within the 25% MQO (QAPP Table 26-1). Total solids was also highly repeatable, with 0.28% RPD.

3.3. Grainsize – ALS

Grainsize fractions for two sediment samples collected for Task 3 Phase 2 were analyzed by ALS. Typically blanks and recovery standards are not reported for grainsize determination, but replicate analyses sometimes are. Samples from another project reported by the same lab were provided as QC for measurement precision.

The lab originally reported grainsize as a percentage of an estimated total dry mass based on an initial sample total (wet) mass and moisture loss in a 10-15g subsample. However, on occasion that estimated mass was less than even just the calculated coarse material (>63µm), sieved from a 50-100g subsample. Although estimated total dry mass from the smaller subsample is still useful as an indicator of potential subsampling variability, the summed masses of fractions from the latter (larger) subsample is likely to be more precise and representative of the full sample due to its larger size. Results have therefore been rescaled and expressed as a percentage of the summed masses of separated fractions instead.

3.3.1. Sensitivity

Only the medium gravel (4.75 to 75mm) grain size fraction was not reported (due to its absence) in some samples.

3.3.2. Blanks

Typically blanks are not reported for grainsize determination

3.3.3. Recovery

Typically recovery standards are not reported for grainsize determination

3.3.4. Precision

Precision was evaluated using replicate analyses of samples from another project analyzed by the lab around the same time. RPDs on replicates were within the target MQO (<25% RPD, QAPP Table 26-1) for all sizes except for coarse sand (2 to 4.75mm), with an RPD of 66% due to its small contribution (average <1% of total mass).

3.4. Dioxins & Furans (PCDD/Fs) – ALS

Dioxins and furans in 2 sediment samples were analyzed by ALS. Samples were collected May 14 & 15, 2013 and were analyzed between June 25 and July 3, 2013. The 17 2,3,7,8- substituted congeners were reported. A blank, and LCS (recovery samples in duplicate) were also reported.

3.4.1. Sensitivity

A number of the more toxic isomers (including TCDD/F, and some of the PeCDD/Fs) were not detected (ND) in 1 of the 2 samples. Non-detects for these less abundant isomers often occur.

3.4.2. Blanks

Most of the analytes (13) were found in the blank at least 2x the MDL. For 4 congeners, (1,2,3,7,8,9-HxCDF, 1,2,3,4,7,8,9- HpCDF, 1,2,3,4,7,8- HxCDD, and 1,2,3,7,8-PeCDD) blanks were possibly > 10%

of the field sample value in one or both samples. Those results were censored with VRIP flag (Data rejected - Analyte detected in field or lab generated blank, flagged by QAO) as a worst-case assumption. Other samples were flagged with VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning but not censored the blank contamination was <10% of those field samples.

3.4.3. Recovery

Recovery was evaluated using LCSs as no reference materials were available. Good recovery was found for LCSs (within the 70-130% recovery (30% error) QAPP Table 26-3 target for dioxins).

3.4.4. Precision

Precision on LCS replicates was good, with RPDs 16% or less, within the 25% MQO (QAPP Table 26-3) for all analytes. LCSs generally are a simple matrix with fewer interferences, so recoveries represent a best case scenario.

3.5. PAHs – ALS

Seventeen PAHs in 2 sediment samples were analyzed by ALS. Samples were collected May 14 & 15, 2013 and were analyzed June 4, 2013. A blank, and LCSs (recovery samples in duplicate) were also reported.

3.5.1. Sensitivity

There were no non-detects for any of the 17 reported PAH compounds.

3.5.2. Blanks

No PAHs were found above detection limits in blank samples.

3.5.3. Recovery

Average recovery errors for LCSs were below 17% of the target MQO of 25% for all analytes (maximum error was 17%)

3.5.4. Precision

Precision on LCS replicates was good, with RPDs 16% or less, within the 25% MQO (QAPP Table 26-3) for all analytes. LCSs generally are a simple matrix with fewer interferences, so recoveries represent a best case scenario.

3.6. PBDEs –ALS

PBDEs in 2 sediment samples were analyzed by ALS. Samples were collected May 14 & 15, 2013 and were analyzed June 4, 2013. A blank and LCSs (recovery samples in duplicate) were also reported.

3.6.1. Sensitivity

There were no non-detects for any of the 17 reported PAH compounds.

3.6.2. Blanks

No PAHs were found above detection limits in blank samples.

3.6.3. Recovery

Average recovery errors for LCSs were below 17% the target MQO of 25% for all analytes (maximum error was 17%)

3.6.4. Precision

Precision on LCS replicates was good, with RPDs 16% or less, within the 25% MQO (QAPP Table 26-3) for all analytes. LCSs generally are a simple matrix with fewer interferences, so recoveries represent a best case scenario.

3.7. PCBs –ALS

PCBs in sediment samples were analyzed by ALS. Samples were collected May 14 & 15, 2013 and were analyzed between June 20 and July 12, 2013. The 40 PCB congeners reported by the Regional Monitoring Program for Water Quality in the San Francisco Estuary² were reported for 29 field samples (including 3 replicates). Blank, LCS (recovery), and MS/MSD samples were also reported.

3.7.1. Sensitivity

PCB 141 was not detected (ND) in about 11% of the samples, but aside from that, most congeners were ND in <5% of samples analyzed for PCBs.

3.7.2. Blanks

About half the analytes (22) were found in the blank at least 2x the MDL in one or more batches (QAPP page 7-5). For 3 congeners, blanks were possibly > 10% of the field sample value in 3% of samples. Those congener results were censored with VRIP flag (Data rejected - Analyte detected in field or lab generated blank, flagged by QAO) as a worst-case assumption. Other samples were flagged with VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning but not censored since the blank contamination was <10% of those field sample concentrations.

3.7.3. Recovery

Recovery was evaluated using LCSs because MS/MSDs were spiked at levels not sufficiently higher than the native samples to quantify recovery. Good recovery was found for LCSs (within the 70-130% recovery (30% error) QAPP Table 26-2 target for PCBs) except for PCB 15 just marginally (<1%) above that limit. Only 3 out of the 29 reported analytes in the LCS were in the RMP 40 target analytes; although a range of PCB homologs were included in the LCS, some interferences are congener and matrix specific, and thus LCSs represent of a somewhat best case scenario for recovery.

3.7.4. Precision

Precision was calculated only for sample pairs where an analyte was detected in both samples. Precision results were averaged across batches for analytes, and those with average RPDs > the 25% MQO (QAPP Table 26-2) were flagged as having marginal precision but not censored, and those with RPDs grossly above the MQO (average RPD >50%) were flagged as estimated (see table below). The many analytes with large RPDs suggests PCBs were quite heterogeneous despite efforts to homogenize.

| Congener | Average RPD |
|-----------------|-------------|
| PCB 021/33 | 32.75% |
| PCB 066 | 75.51% |
| PCB 090/101/113 | 27.09% |
| PCB 095 | 25.14% |

² The RMP 40 list of PCBs has been the historic suite of PCB congeners analyzed by BASMAA agencies.

| | |
|-----------------|--------|
| PCB 128/166 | 25.80% |
| PCB 129/138/163 | 64.86% |
| PCB 132 | 44.79% |
| PCB 135/151 | 67.26% |
| PCB 141 | 68.46% |
| PCB 147/149 | 66.08% |
| PCB 153/168 | 49.19% |
| PCB 156/157 | 33.63% |
| PCB 158 | 45.31% |
| PCB 170 | 54.61% |
| PCB 174 | 57.56% |
| PCB 177 | 57.97% |
| PCB 180/193 | 56.06% |
| PCB 183 | 57.32% |
| PCB 187 | 79.81% |
| PCB 194 | 37.80% |
| PCB 195 | 48.77% |
| PCB 201 | 44.61% |
| PCB 203 | 40.91% |

3.8. Pesticides –ALS

Pesticides in 2 sediment samples were analyzed by ALS. Samples were collected May 14 & 15, 2013 and were analyzed May 31, 2013. A blank, MS/MSD, and LCSs (in duplicate) were also reported for 31 pesticides.

3.8.1. Sensitivity

A majority of the pesticides (29 of 31) had some non-detects (NDs), with 21 of them showing extensive NDs (>50% NDs).

3.8.2. Blanks

No pesticides were found above detection limits in the blank sample.

3.8.3. Recovery

Matrix spike recoveries of cis-Chlordane, trans-Chlordane, Endrin Aldehyde, cis-Nonachlor, trans-Nonachlor, and Oxychlordane had an average % Error above the target MQO of 25% and were flagged but not censored. Cis-Chlordane, cis-Nonachlor, trans-Nonachlor, and Oxychlordane had average %Errors >50% and were also flagged as estimated values (VJ) due to recovery uncertainties.

3.8.4. Precision

Matrix spike replicates were used to evaluate precision. Cis-Chlordane, trans-Chlordane, and trans-Nonachlor had average RPDs above the target MQO of 25% and were flagged but not censored.

3.9. Grainsize – SCL

Grainsize in sediment samples collected for Task 3 Phase 2 was analyzed by SCL. Samples were collected May 14 & 15, 2013 and were analyzed June 10, 2013. 17 sediment grainsize fractions were reported for 24 samples (including 2 field replicates).

3.9.1. Sensitivity

Sensitivity was sufficient for most fractions with non-detects reported for some of the pebble fractions and medium clay (0.00098 to <0.00195 mm).

3.9.2. Blanks

Typically blanks are not measured for grainsize determination.

3.9.3. Recovery

Recovery samples are often not run for grainsize, and are not specified by the project QAPP.

3.9.4. Precision

Precision on field replicates was generally within 25% RPD, except for fine and medium clay, medium and coarse silt, and small and medium pebble, flagged (VIL) for RPDs between 25% and 50%.

3.10. Hg – SCL

Mercury in sediment samples collected for Task 3 Phase 2 was analyzed by SCL. Samples were collected May 14 & 15, 2013 and were analyzed May 20 to June 26, 2013. Total mercury in a <2mm sediment fraction was reported for 4 samples (including a replicate). Blank, LCS (recovery), and MS/MSD samples were also reported.

3.10.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for total mercury.

3.10.2. Blanks

Mercury was detected in blanks but at concentrations <2x the MDL.

3.10.3. Recovery

Recovery for matrix spikes averaged 10.8% error (within the QAPP Table 26-4 target 75-125% recovery (25% error) for mercury. The average error for laboratory control materials of 1.9% was also good.

3.10.4. Precision

Precision on lab replicates was acceptable, averaging 18.9% RPD, within the 25% MQO (QAPP Table 26-4). Field replicates averaged 27.9% RPD, suggesting field sample heterogeneity.

3.11. TOC – SCL

TOC in sediment samples collected for Task 3 Phase 2 was analyzed by SCL. Samples were collected May 14 & 15, 2013 and were analyzed May 30, 2013. TOC in a <2mm sediment fraction was reported for 26 samples (including 2 lab and 2 field replicates). Blank and CRM samples were also reported.

3.11.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for TOC.

3.11.2. Blanks

TOC was detected in blanks but at concentrations just under <2x the MDL.

3.11.3. Recovery

Recovery for CRMs averaged 4.9% error (within the QAPP Table 26-1 target 80-120% recovery (20% error) for TOC).

3.11.4. Precision

Precision on lab replicates was acceptable, averaging 10.3% RPD, within the 25% MQO (QAPP Table 26-1). Field replicates averaged 7.0% RPD, also meeting the MQO.

4. Task 3 Phase 2 – Ettie Street

4.1. Hg – ALS

Hg in sediment samples from Ettie St. for Task 3 Phase 2 collected May 21, 2013 were analyzed by ALS June 11, 2013. Hg in a <2mm sediment fraction was reported for 10 samples (including 1 lab and 1 blind field replicates). Blank, LCS, and MS/MSD samples were also reported.

4.1.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for Hg.

4.1.2. Blanks

Mercury was not detected in blanks above the MDL.

4.1.3. Recovery

Recoveries were within the MQO target 25% error for MS/MSDs (17%) and the LCS (4%).

4.1.4. Precision

RPD was 23% on the field replicate and 17% on the lab replicate, within the MQO target of 25%. The MS/MSD RPD was also good, around 1%.

4.2. Total Organic Carbon and Total Solids – ALS

Sediment samples from Ettie St. for Task 3 Phase 2 collected May 21, 2013 were analyzed for total organic carbon (TOC) and total solids by ALS June 12-13, 2013. Results for a <2mm sediment fraction were reported for 10 samples (including 1 lab and 1 blind field replicates). Blank, LCS, and MS/MSD samples were also reported for TOC.

4.2.1. Sensitivity

No non-detects were reported for TOC or total solids.

4.2.2. Blanks

TOC was not detected in blanks above the MDL.

4.2.3. Recovery

Recoveries are not routinely measured for total solids, and errors were within the MQO target 25% error for TOC in MS/MSDs (2%) and the LCS (5%).

4.2.4. Precision

RPD was <1% field and lab replicates for total solids. RPD on TOC was 0% for the lab replicate, 10% for the blind field replicate, and averaged 2% on the MS/MSD, all within the MQO target of 25%.

4.3. Grainsize – ALS

Sediment samples from Ettie St. for Task 3 Phase 2 collected May 21, 2013 were analyzed for grainsize by ALS June 1, 2013. 10 samples (including 1 lab and 1 blind field replicates) were reported for 9 size fractions.

4.3.1. Sensitivity

All size fractions were reported in all samples.

4.3.2. Blanks

Blank samples are not typically measured for grainsize determination.

4.3.3. Recovery

Recoveries are not routinely measured for grainsize.

4.3.4. Precision

The lab replicate was used to evaluate precision of analytes. Average RPD was below the target MQO (25%) for all grain size/fraction combinations. The blind field replicate was examined but not used to flag precision. Average RPD for the blind field replicate was below the target MQO of 25% for all grain size/fraction combinations, except for Gravel 4.75 to <75 mm which had a RPD of 80%, not unexpected due to the challenges in uniformly subsampling sparse and large objects in sediment

4.4. Organics (PAHs, PBDEs, PCBs, Pesticides) – ALS

Sediment samples from Ettie St. for Task 3 Phase 2 collected May 21, 2013 were analyzed for organic contaminants by ALS June 20 to July 11, 2013. 10 samples, including 1 lab and 1 field blind replicate were analyzed for PAHs, PCBs, and PBDEs and a suite of pesticides were analyzed in 1 sample. A blank, LCS, and MS/MSD samples were also reported for all analytes.

4.4.1. Sensitivity

A majority of the PBDEs and pesticides were not detected in the one sample analyzed for those analytes. PAHs and PCBs were mostly detected, with only one non-detect for PCB 141.

4.4.2. Blanks

A number of the PCBs were found in the blank >2x the MDL, but all field concentrations were at least 10x higher, so results were flagged for blank contamination, but none censored.

4.4.3. Recovery

Recoveries on the LCSs were within the QAPP target 30% error for reference materials for most analytes, except for chlordanes and nonachlors, which were flagged but not censored, and MSs were within 50% of their target, except (o,p')DDT, similarly flagged but not censored.

4.4.4. Precision

Replicate RPDs on field or matrix spike replicates were outside of the QAPP target 25%, for 3 PBDEs (47, 100, 138), 4 PCBs (56, 60, 66, 105), and (p,p')DDT, and as a result, those results were flagged but not censored.

5. References

BASMAA, 2013. *Quality Assurance Project Plan: Clean Watersheds for a Clean Bay – Implementing the San Francisco Bay’s PCBs and Mercury TMDLs with a Focus on Urban Runoff, EPA San Francisco Bay Water Quality Improvement Fund Grant # CFDA 66.202, Revision 1*. Prepared by Applied Marine Sciences, Inc. August 15, 2013.