

Laboratory QA Summary

CW4CB Task 4

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Submitted to:

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1. Introduction

The Bay Area Stormwater Agencies Association (BASMAA) contracted with Applied Marine Sciences, Inc. (AMS) to support implementation of CW4CB Task 4. As part of its contract with BASMAA, AMS is providing project quality assurance for all Task 4 activities. SFEI has completed final data review of Task 4 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).

Task 4 field monitoring encompasses five distinct projects: (1) street sweeping effectiveness evaluations conducted at four locations for ten events each (soil samples); (2) a follow-up to the sweeping effectiveness study analyzing archived street dirt at different particle size fractions (soil samples); (3) a Pulgas Street Flushing Study (soil and water samples); (4) a Leo Ave. Storm Drain Line Cleanout (soil and water samples); and (5) an Ettie Street Pump Station Cleanout (soil samples).

All monitoring was conducted between November 2013 and September 2014. Analytical laboratories employed for each study, along with analyses conducted by each, are described in the sections that follow. In some cases, data for specific analytes from multiple sampling efforts were batched and reviewed together to increase efficiency of data management and quality assurance process. The text below reflects these groupings where applicable.

2. Street Sweeping Effectiveness Evaluation – Events 1 thru 10

2.1. Sediment Hg - ALS

Mercury in sediment samples collected by AMS were analyzed by ALS. Samples were collected between November 25, 2013 and June 18, 2014, and were analyzed between December 6, 2013 and June 25, 2014. Total mercury in the <2mm sediment fraction was reported for 80 sediment field samples, 8 blind field replicates, and lab replicates. Blanks (method and field), matrix spike/matrix spike replicates (MS/MSDs), and laboratory control samples (LCSs) were also analyzed.

2.1.1. Sensitivity

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

2.1.2. Blanks

Mercury was not found in either the method or field blanks at concentrations over the detection limit.

2.1.3. Recovery

Recoveries for total mercury matrix spike results with an average error of 37.66% were outside the 25% target (QAPP Table 26-4 target 75-125% recovery (25% error)) so results were flagged, but not censored. LCS samples were also examined and the average error for total mercury of 4.07% was well below the 25% target.

2.1.4. Precision

Precision on lab replicates for mercury with an average RPD of 24.98% fell just below the 25% MQO (QAPP Table 26-4), so no results were flagged for marginal or poor precision. Field replicates were examined but not used for the evaluation, with the mercury average RPD for lab and field samples

combined being more variable at 30%. Likewise, LCS and matrix spike replicates were examined, but not used for the evaluation, with an average RPD of 30.39% for the mercury matrix spikes (LCS average RPD was <25% target (5.89%)).

2.2. Sediment PCBs - ALS

PCBs in sediment samples collected by AMS were analyzed by ALS. Samples were collected between November 25, 2013 and June 18, 2014 and were analyzed between December 18, 2013 and July 11, 2014. The 40 PCB congeners reported by the Regional Monitoring Program for Water Quality in the San Francisco Estuary¹ were reported in the <2mm sediment fraction for 103 field samples (including blind field and lab replicates). Method blanks, matrix spike/matrix spike replicate (MS/MSD), field blank, and laboratory control samples (LCS) were also reported.

2.2.1. Sensitivity

PCB 008 was not detected (ND) in about 5% (5 out of 103) of the samples, other not detected analytes were ND in only 1 to 3 of the 103 samples analyzed for PCBs.

2.2.2. Blanks

All of the analytes were found in the blank in at least one or more of the batches. All were flagged with the non-censoring qualifier VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning, except for 6% of PCB 044/47/65, 4% of PCB 194, 2% of PCB 187, 2% of PCB 180/193, 2% of PCB 203, 1% of PCB 128/166, and 1% of PCB 156/157 results, which were flagged with the censoring flag VRIP (Data rejected - Analyte detected in field or lab generated blank, flagged by QAO), because the results were <10x the blank concentration. However, a few samples of each of these analytes were <3x the blank concentrations, so may not be real field quantities.

Blank contamination was found for all congeners and “totals” measured in the field blank, except for PCB 008, PCB 056, PCB 060, PCB 128/166, PCB 156/157, PCB 201, and Total Di-PCB, but at levels that were less than 0.4% of the average concentration measured in the field samples.

2.2.3. Recovery

Matrix spike samples were used to evaluate the accuracy of only PCB 156/157; because the majority of matrix spikes were not useable having not been spiked at concentrations high enough to avoid analytical variability in the native sample concentrations from dominating the recovery calculations. The recovery for PCB 156/157 was good (within the 50-150% recovery (50% error) QAPP Table 26-2 target for PCBs. Accuracy for PCB 105, 118 and 156/15 measured in the LCS samples was also within the 50-150% recovery range. Only 3 out of the 29 reported analytes in the matrix spike and LCS samples 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.

2.2.4. 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

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

above the MQO (average RPD >50%) were flagged with the qualifier of VJ and a compliance code of Est to indicate the results are estimated values (see table below). Only one analyte, Total Nona-PCB, fell into this latter category.

PARAMETER	avgRPD
Total Nona-PCB	68.94%
PCB 187	49.29%
PCB 201	33.04%
Total Octa-PCB	30.94%
PCB 203	29.85%
PCB 128/166	28.61%
PCB 066	28.18%
PCB 195	27.63%

2.3. Sediment Bulk Density, Density, TOC, and Total Solids - ALS

Bulk density, density, total organic carbon (TOC), and Total Solids in sediment samples collected by AMS were analyzed by ALS. Samples were collected between November 25, 2013 and June 18, 2014, and were analyzed on November 27, 2013 to July 11, 2014. Bulk density and density were reported for 4 sediment and 76 sediment samples, respectively (ALS seems to use Bulk Density and Density interchangeably citing the same method and reporting concentrations in the same units – the analyte names contained within the final Task 4 data deliverable remain as reported by ALS). Density results were also reported for eight blind field replicates. TOC in a <2mm sediment fraction were reported for 80 sediment samples, 8 blind field replicate, lab replicates, MS/MSDs, method blanks, and LCS samples. Total Solids in a <2mm sediment fraction were analyzed in 84 sediment samples, 8 blind field replicates, lab replicates, and 1 field blank. Several Bulk Density and Density results exceeded the 7 day holding time specified in the QAPP and were flagged with the qualifier VH (Holding time violation occurred, flagged by QAO).

2.3.1. Sensitivity

The detection limits were above the project QAPP target of 0.01% for TOC, but all TOC results were above the detection limits (TOC MDL 0.02%). No non-detects were reported for bulk density, density, or Total Solids.

2.3.2. Blanks

Bulk density, density, TOC, and Total Solids were not found in the blanks at concentrations over the detection limit (all NDs). Field blanks were examined, but not used in the evaluation, with Total Solids reported as being 99.9% in the dried clean sample, which is a reasonable result.

2.3.3. Recovery

Matrix spike samples were used to evaluate the accuracy of Total Organic Carbon. Recoveries were good, with an average error of 4.1% that was well within the target MQO of 80-120% (QAPP Table 26-1). LCS recovery samples were examined and the average 3.43% error was well within the target for TOC.

2.3.4. Precision

Lab replicates were used to evaluate precision for TOC. The average TOC RPD of 3.82% was less than the target MQO of 25%. Field replicates, LCS, and matrix spike replicates were also examined but not used for the evaluation, with the average RPDs, likewise, less than the 25% MQO target.

2.4. Sediment Grainsize - ALS

Grainsize in sediment samples collected by AMS were analyzed by ALS. Samples were collected between November 25, 2013 and June 18, 2014 and were analyzed between December 10, 2013 and June 26, 2014. Results for 12 samples exceeded the 28 day holding time specified in the QAPP and were flagged with the qualifier VH (Holding time violation occurred, flagged by QAO). Grainsize in 9 fractions (from <75 to <0.005 mm) using the ASTM scale were reported in sediment samples for 99 samples, including 11 lab replicates and 8 field replicates. Some of the ASTM sizes are slightly offset from the ranges requested in the project QAPP. No other sample types were reported. Note: the various fractions for a sample do not always sum to 100%.

2.4.1. Sensitivity

No method detection or reporting limits were provided, but all analyte/fraction combinations had results greater than 0, except for Gravel (4.75 to <75 mm fraction), which had 11 of 100 results (11%) equal to 0.

2.4.2. Precision

Lab replicates were used to evaluate precision for grainsize. Coarse grained fractions were heterogeneous. The average RPD was below the target MQO of 25% for all size ranges except Gravel (4.75 to <75 mm fraction), Sand (Coarse 2.0 to <4.75 mm fraction), and Clay (<0.005 mm fraction) which had RPDs above the target MQO of 25% (81.83%, 36.21%, and 26.69%, respectively), these were flagged but not censored (CW4CB has no listed censoring threshold). The average RPD for Gravel (4.75 to <75 mm fraction) being >50% the results were flagged with the qualifier of VJ and a compliance code of Est to indicate the results are estimated values. Field sample replicates showed similar variability, with some RPDs higher and others lower than for the lab replicates.

3. Street Sweeping Effectiveness Evaluation – Re-analyses

For the above discussion of the Street Sweeping Effectiveness Evaluation project, samples were collected between January 27, 2014 and June 18, 2014. A subset of samples archived associated with project implementation were re-analyzed on September 11, 2014 on two particle size fractions not analyzed previously. The results from these re-analyses are discussed below.

3.1. Sediment Hg (Reanalyzed Samples) - ALS

Mercury in a small number of sediment samples collected by AMS was reanalyzed by ALS. Total mercury was reported in “sediment, <75um” and “sediment, >75um” fractions for 10 sediment samples (5 Pre- and Post-sweeping pairs). One lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), 1 method blank, and 1 laboratory control sample (LCS) were run for only the “sediment, >75um” matrix, but were used to evaluate both fractions as all samples were analyzed in the same batch.

3.1.1. Sensitivity

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

3.1.2. Blanks

Mercury was not found in the method at concentrations over the detection limit.

3.1.3. Recovery

Recoveries for total mercury matrix spike results with an average error of 3.07% were well below the target MQO of 25% (QAPP Table 26-4 target 75-125% recovery (25% error)). LCS samples were examined, but not used for the evaluation; with the average error of 2.51% also well below the 25% target.

3.1.4. Precision

Precision on lab replicates for mercury with an average RPD of 25.31% was just above the 25% MQO (QAPP Table 26-4), so results were flagged with the non-censoring qualifier “VIL” for marginal precision. Matrix spike replicates were examined, but not used for the evaluation, and the average RPD of 4.6% was well below the target MQO.

3.2. Sediment PCBs (Reanalyzed Samples) - ALS

PCBs in a small number of sediment samples collected by AMS was reanalyzed by ALS. Results were reported for 48 PCB congeners (including co-elutions and “sums”) in “sediment, <75um” and “sediment, >75um” fractions for 10 sediment samples (5 Pre- and Post-sweeping pairs). One lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), 1 method blank, and 1 laboratory control sample (LCS) were run for only the “sediment, >75um” matrix, but were used to evaluate both fractions as all samples were analyzed in the same batch.

3.2.1. Sensitivity

Method detection limits were sufficient with non-detects (NDs) reported for only Total Monochlorobiphenyls and Total Nonachlorobiphenyls, both 100% NDs.

3.2.2. Blanks

About 63% (30 out of 48) of the analytes were found in the blank in at least one or more of the batches. All were flagged with the non-censoring qualifier VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning, except for 100% of Total Monochlorobiphenyls results which were flagged with the censoring flag VRIP (Data rejected - Analyte detected in field or lab generated blank, flagged by QAO), because the results were <10x the blank concentration.

3.2.3. Recovery

Laboratory control samples (LCSs) were used to evaluate accuracy because matrix spikes were not useable having not been spiked at concentrations high enough to avoid analytical variability in the native sample concentrations from dominating the recovery calculations. The average error for PCB 105, PCB 118, and PCB 156/157 was good, 9.88%, 3.59% and 11.23% respectively, well below the target MQO of 25% (within the 50-150% recovery (50% error) QAPP Table 26-2 target for PCBs). Only 3 out of the 29 reported analytes in the matrix spike and LCS samples 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.

3.2.4. Precision

Lab replicates were quite variable for most analytes. Average RPDs ranged from 1% to 86.67% with 42 out of 46 (91%) of the analytes having RPDs greater than the target MQO of 25%, so were flagged with the non-censoring qualifier “VIL”. About 90% (38 out of 42) of the analytes had average RPDs >50% and were flagged with the QACode of VJ and a compliance code of Est to indicate estimated values. Matrix spike replicates were examined, but not used in the evaluation, and were also highly variable. PCB 105, PCB 118, and PCB 156/157 had average RPDs well above the target MQO of 25% (142%, 57%, and 39%, respectively).

4. Leo Ave Outflow

4.1. Sediment Hg - SCL

Mercury in one sediment sample collected by KLI was analyzed by SCL. Sample was collected on September 4, 2013, and analyzed on September 20, 2013. Total mercury was reported for 1 sediment field sample, 1 field replicate, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), two laboratory control materials (LCMs), and one method blank. Mercury samples were analyzed ~16 days after collection exceeding the 14 day holding time specified in the QAPP (there is no indication the samples were stored frozen at -20 degrees centigrade). Mercury results were flagged with the qualifier VH (Holding time violation occurred, flagged by QAO).

4.1.1. Sensitivity

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

4.1.2. Blanks

Mercury was not found in the method blank at concentrations over the detection limit.

4.1.3. Recovery

Matrix spike recoveries were used to assess accuracy. Recoveries were good, with the average error of 9.86% less than the target MQO of 25% (within the QAPP Table 26-4 target 75-125% recovery (25% error)). LCMs were examined, but not used in the evaluation of mercury, with the average error of 5.16% being well below the target MQO.

4.1.4. Precision

Precision on laboratory replicates was good, averaging 5.7%, well below the 25% target MQO (QAPP Table 26-4). LCMs, matrix spike, and field replicates RPDs were also examined and were likewise well below the target MQO (6.12%, 1.42%, and 5.81%, respectively).

4.2. Sediment Trace Metals - ALS

Trace metals in sediment samples collected by KLI were analyzed by ALS. Samples were collected on September 4, 2013, and were analyzed between September 9 and September 11, 2013. Trace element results were reported for 1 field sample, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), 1 method blank, and 1 LCS sample.

4.2.1. Sensitivity

Sensitivity was sufficient with no non-detects reported for any of the trace metals.

4.2.2. Blanks

Lead, Molybdenum, Nickel, Vanadium, and Zinc were measured in the lab blank at levels 2-14 times higher than their MDL, but this is still well less than the average field sample and lab replicate concentration (~3% of average field sample and lab replicate concentration). Lead, Molybdenum, Nickel, Vanadium, and Zinc were flagged with the qualifier VIP, but not censored.

4.2.3. Recovery

Matrix spike sample recoveries were used to evaluate the accuracy of Antimony, Arsenic, Beryllium, Cobalt, Molybdenum, Selenium, Silver, and Thallium; other trace metals were evaluated using the recoveries from the laboratory control samples (LCSs). Recoveries were generally good, with an average error below the target MQO of 25% for all analytes, except Antimony (error of 82.67%). Antimony with an average error >50% was flagged as an estimated value with a compliance code of Est, and the non-censoring qualifiers of VIU (Percent Recovery exceeds laboratory control limit, flagged by QAO) and VJ (Estimated value - flagged by QAO).

4.2.4. Precision

Precision on lab replicate samples was used to evaluate all trace metals. Average RPDs were less than the target MQO of 25% for all parameters (all less than 12%), except for Antimony (RPD = 33.73%). Antimony results were flagged with the non-censoring qualifier VIL (RPD exceeds control limit, flagged by QAO).

4.3. Sediment PAHs, Pesticides, Phthalates, Semi- and Volatile Organic Compounds–ALS

PAHs in sediment samples collected by KLI were analyzed by ALS. Samples were collected on September 4, 2013 and analyzed on September 12 and October 7-8, 2013. Results were reported for 1 sediment field sample, matrix spike/matrix spike replicates (MS/MSDs), method blanks, and laboratory control samples (LCSs) for 70 analytes (PAHs, Pesticides, Phthalates, Semi- and Volatile Organic Compounds).

4.3.1. Sensitivity

A majority of the analytes, 59% (49 out of the 70 reported in the single sample) were non-detects.

4.3.2. Blanks

The analyte “TPH as Gasoline C6-C10” was found in the blank of at least one or more of the batches (field result for TPH as Gasoline C6-C10 was not much (<3x) above the blank level), and was flagged with the censoring qualifier VRIP (Data rejected - Analyte detected in field or lab generated blank, flagged by QAO).

4.3.3. Recovery

Matrix spike recoveries were used to evaluate Benzene, Ethylbenzene, Toluene, and TPH as Gasoline C6-C10; other parameters were evaluated using the recoveries of the laboratory control samples (LCSs). Recoveries were marginal, with an average error above the target MQO of 25% for all analytes, except for Benzene, Bis(2-ethylhexyl)phthalate, Dibenz(a,h)anthracene, Indeno(1,2,3-c,d)pyrene, Ethylbenzene, Toluene, and Total Xylenes. All analytes with an error >25% were flagged but not censored; analytes with an error >50% were flagged with the qualifier of VJ and a compliance code of Est to indicate the results are estimated values.

4.3.4. Precision

Matrix spike/matrix spike replicate samples were used to evaluate the precision of Benzene, Ethylbenzene, Toluene, and TPH as Gasoline C6-C10 results; other parameters were evaluated using the laboratory control samples (LCSs). Ethylbenzene and Toluene had average RPDs above the target MQO of 25% (QAPP Table 26-2), and were flagged with the qualifier VIL (RPD exceeds control limit, flagged by QAO), but not censored.

4.4. Sediment Bulk Density and TOC – SCL

Bulk Density and total organic carbon (TOC) in sediment samples collected by KLI were analyzed by SCL. Sample was collected on September 4, 2013, and was analyzed on September 9 and 19, 2013. Bulk Density results were reported for 1 sediment field sample, and one field replicate. TOC results were reported in 1 sediment sample, 1 field replicate, 1 lab replicate, 1 certified reference material (CRM), and 1 method blank.

4.4.1. Sensitivity

Sensitivity was sufficient with no non-detects reported for Bulk Density or TOC.

4.4.2. Blanks

No lab blanks were analyzed/required per the QAPP for Bulk Density. TOC was measured in the one method blank at a level nine times the MDL (0.09% compared to 0.01% dw), but this well below the average result of the field sample and replicates (3.86% dw). TOC results were flagged with the non-censoring qualifier VIP (Analyte detected in field or lab generated blank, flagged by QAO).

4.4.3. Recovery

No spiked samples were analyzed/required per the QAPP for Bulk Density. The CRM, LECO Soil 502-308 (1006), was used to evaluate the accuracy of TOC. The CRM recovery was good, with an average error of 8.06%, which was less than the target 25% MQO.

4.4.4. Precision

Laboratory replicates were used to evaluate precision of TOC with the average RPD of 2.91% being below the 25% target MQO. Field replicate RPDS were examined, but not used to evaluate the precision of Bulk Density and TOC, and with average RPDs of 1.04% and 5.57%, respectively, were similarly well below the target MQO.

4.5. Sediment GrainSize – SCL

Grainsize in sediment samples collected by KLI were analyzed by SCL. One sample collected on September 4, 2013 was analyzed on September 18, 2013. Grainsize in 17 fractions (from <64 to <0.00098 mm) were reported for 1 sediment field sample, and one field replicate. No other sample types were reported.

4.5.1. Sensitivity

Method detection limits were sufficient with all analyte/fraction combinations having results, except for Pebble (Medium 8 to <16 mm fraction), Pebble (Large 16 to <32 mm fraction), and Pebble (V. Large 32 to <64 mm fraction), which were non-detects.

4.5.2. Precision

Precision could not be evaluated as no laboratory replicates were analyzed; informed by PM that they are tracking to ensure the minimum percentage of replicates are analyzed by the end of the project. Field replicate RPDs were examined and besides Pebble (Small 4 to <8 mm fraction), all were all below the 25% target MQO (Pebble/Small 4 to <8 mm RPD = 48.25%). This is somewhat variable, but it is often hard to get the larger fractions homogeneous.

4.6. Water Hg – SCL

Mercury in a water sample collected by KLI was analyzed by SCL. Sample was collected on September 4, 2013, and was analyzed on September 9, 2013. Total mercury was reported for 1 field sample, 1 matrix spike/matrix spike replicate (MS/MSD), 1 method blank, 1 laboratory control material (LCM), and other client samples (for QA purposes).

4.6.1. Sensitivity

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

4.6.2. Blanks

Mercury was not found in the single method blank.

4.6.3. Recovery

Matrix spike sample recoveries were used to evaluate the accuracy of mercury. Recoveries were good (within the QAPP Table 26-8 target 75-125% recovery (25% error)) for mercury (actual average 4.55%). LCM recoveries were examined and, with an error of 1.3%, was also well below the 25% target MQO.

4.6.4. Precision

Matrix spike replicates were used to evaluate the precision of the mercury result. Mercury had an average RPD of 1.36% well below the target MQO of 25% (QAPP Table 26-8).

4.7. Water SSC and TOC – SCL

Suspended Sediment Concentration (SSC) and Total Organic Carbon (TOC) in a water sample collected by KLI were analyzed by SCL. Sample was collected on September 4, 2013 and was analyzed on September 18, 2013. Results were reported for SSC in 1 water sample and a method blank. TOC results were reported in 1 water sample, 1 lab replicates, 1 matrix spike/matrix spike replicate (MS/MSD), 1 method blank, and 1 LCS sample. The LCS used was LECO Soil 502-309 (1002), carbon and nitrogen in soil from the LECO Corporation (external supplier). The SSC result was analyzed more than 7 days after the date of sample collection (holding time requirement in the QAPP is 7 days) so was flagged with the QACode of VH (Holding time violation occurred, flagged by QAO).

4.7.1. Sensitivity

The method detection limits were sufficient with no non-detects reported for SSC or TOC.

4.7.2. Blanks

Suspended Sediment Concentration (SSC) and total organic carbon (TOC) were not found in the blanks at concentrations over the detection limit (all NDs).

4.7.3. Recovery

Matrix spike recoveries were used to evaluate the accuracy of TOC. Recoveries were good with an average error of 2.34%, well below the target MQO of 25%. LCM recoveries were examined, but not used in the evaluation, and were also well below the 25% target MQO (average error for TOC of 7%). Accuracy could not be evaluated for SSC as no spiked samples were analyzed/required by the QAPP.

4.7.4. Precision

Lab replicate was used to evaluate the precision of TOC. The average RPD for TOC was 0.27%, well below the 25% target MQO. The average RPD for the matrix spike replicates were examined and were likewise well below the target MQO (RPD 3.4%).

Other samples collected on September 4, 2013 for the Leo Outflow study were analyzed and QA reviewed together with the samples collected for the Pulgas Flushing Study (see Section 5 for QA summaries).

5. Pulgas Flushing Study and Leo Outflow

5.1. Sediment Hg - ALS

Mercury in sediment samples collected by KLI was analyzed by ALS. Samples were collected between September 4 (Leo Outflow sample) and 20, 2013, and were analyzed between September 9 and October 10, 2013. Total mercury was reported for 9 sediment field samples, 1 field replicate, 1 lab replicate, 1 matrix spike, 3 method blanks, 1 field blank, and 5 LCS samples. Four mercury samples exceeded the 14 day holding time listed in the QAPP so were flagged as VH (Holding time violation occurred, flagged by QAO).

5.1.1. Sensitivity

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

5.1.2. Blanks

Mercury was not found in either the method or field blanks at concentrations over the detection limit.

5.1.3. 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 1.82%).

5.1.4. Precision

Precision on lab replicates was good, averaging 4.11% RPD, less than the 25% MQO (QAPP Table 26-4), so no results were flagged for marginal or poor precision. LCS and matrix spike replicates were examined, but not used for the evaluation of mercury, with average RPDs less than the 25% target MQO.

5.1.5. Intercomparison sample

Results included one sample analyzed for intercomparison to a second lab. The average LeoOut-S-01 result of 1.46 mg/kg dw here (for field sample and lab replicate), was 93% greater (64% RPD) than the average of SCL results (see Section 4.1 for QA summary) for the same site of 0.73, 0.79, and 0.7462 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.

5.2. Sediment PCBs – ALS

PCBs in sediment samples collected by KLI were analyzed by ALS. Samples were collected between September 4 (Leo Outflow sample) and September 20, 2013, and were analyzed between October 1 and October 24, 2013. Results were reported for 9 sediment field samples, 2 field replicates, 2 lab replicates (1 for field blank), 2 matrix spike/matrix spike replicate (MS/MSD; 1 for field blank), 2 method blanks, 1 field blank, and 2 laboratory fortified samples (LCSs) for 38 PCB congeners (including coelutors), and 10 PCB sums.

5.2.1. Sensitivity

Method detection limits were sufficient with non-detects reported for only the “sums” Total Mono-PCB and Total Nona-PCB.

5.2.2. Blanks

About 32% (12 out of 38) of the analytes were found in the blank in at least one or more of the batches. All were flagged with the non-censoring qualifier VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning.

Blank contamination was found for 40% (15 out of 38) of the congeners measured in the field blank, but all at level that was less than 0.05% of the average concentration measured in the field samples.

5.2.3. Recovery

LCS samples were used to evaluate accuracy because the matrix spike samples were not spiked at concentrations high enough to avoid analytical variability in the native sample concentrations from dominating the recovery calculations. Recoveries were good, within the 50-150% recovery (50% error) QAPP Table 26-2 target for PCBs. Only 3 of the reported analytes in the LCS samples 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.

5.2.4. Precision

Precision was calculated only for sample pairs where an analyte was detected in both the field and lab replicate 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 with the qualifier of VJ and a compliance code of Est to indicate the results are estimated values (see table below). Only four analytes, PCB 056, PCB 060, PCB 066, and PCB 061/70/74/76 fell into this latter category.

PARAMETER	avgRPD
PCB 061/70/74/76	187.56%
PCB 066	146.74%
PCB 060	107.06%
PCB 056	73.01%
PCB 187	28.57%

Matrix spike and LCS replicates were examined, but not used in the evaluation. Average RPDs for the analytes measured in the matrix spike and LCS replicates (PCB 105, PCB 118, and PCB 156/157) were all less than the 25% target MQO. RPDs for the lab replicates and field replicates (combined) ranged from 13.44% to 105.47%, the majority greater than the target MQO of 25%.

5.3. Sediment Density, pH, TOC, and Total Solids – ALS

Density, pH, total organic carbon (TOC), and Total Solids in sediment samples collected by KLI were analyzed by ALS. Samples were collected between September 4 (Leo Outflow sample) and 20, 2013, and were analyzed between September 12 and October 14, 2013. Density results were reported for 8 sediment field samples, and one field replicate. Results for pH were reported for 1 sediment sample and one laboratory control sample (LCS). TOC results were reported in 8 sediment samples, 1 field replicate, 1 lab replicate, 1 MS/MSD, 2 method blanks, and 2 LCS samples. Total Solids were measured in 9 sediment samples, 2 field replicates, 2 lab replicates, and 1 field blank.

5.3.1. Sensitivity

The detection limits were above the project QAPP target of 0.01% for TOC, but all TOC results were detects above the detection limits (TOC MDL 0.02%). No non-detects were reported for density or Total Solids.

5.3.2. Blanks

Density, TOC, and Total Solids were not found in the blanks at concentrations over the detection limit (all NDs). Field blanks were examined, but not used in the evaluation, with Total Solids reported as being 100% in the dried clean sample, a reasonable result.

5.3.3. Recovery

Matrix spike recoveries were used to evaluate the accuracy of Total Organic Carbon. Recoveries were generally good, with the average error of 20.5%, below the 25% target MQO. LCS recoveries were also examined, and with an average error of 2% which was well below the MQO target.

5.3.4. Precision

Lab replicates were used to evaluate precision for TOC and Total Solids. The average RPDs for TOC and Total Solids of 5.44% and 2.27% respectively, were less than the target MQO of 25%. Field, LCS, and matrix spike replicates were also examined but not used for the evaluation, with the average RPDs, likewise, less than the 25% MQO target.

5.4. Sediment GrainSize – ALS

Grainsize in sediment samples collected by KLI were analyzed by ALS. Samples were collected between September 13 and September 20, 2013, and were analyzed between October 1 and 8, 2013. Grainsize in 9 fractions (from <75 to <0.005 mm) using the ASTM scale were reported in sediment samples for 9 sediment field samples, one lab replicate, and one field replicate. Some of the ASTM sizes are slightly offset from the ranges requested in the project QAPP. No other sample types were reported. Note: the various fractions for a sample do not always sum to 100%.

5.4.1. Sensitivity

No method detection or reporting limits were provided, but all analyte/fraction combinations had results greater than 0, except for gravel (4.75 to <75 mm fraction) which had 10 of 11 results (91%) reported as zero.

5.4.2. Precision

Lab replicates were used to evaluate precision, except the gravel fraction which was mostly zeros. Grainsize was somewhat variable/heterogeneous with Silt (0.005 to <0.075 mm fraction), Sand (Fine 0.075 to <0.106 mm fraction), and Sand (Medium 0.850 to <2.0 mm fraction) having average RPDs above the target MQO of 25% (27.41%, 35.18%, and 25.41%, respectively), and were flagged with the qualifier “VIL” for precision outside of the MQO target. Field replicate RPDs were examined and were all below the 25% target MQO.

5.5. Water Hg – ALS

Mercury in water samples collected by KLI was analyzed by ALS. Samples were collected between September 13 and 20, 2013, and were analyzed between October 11 and 15, 2013. Total mercury was reported for 13 field sample, 1 field replicate, 2 matrix spike/matrix spike replicate (MS/MSD), and 6 method blanks.

5.5.1. Sensitivity

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

5.5.2. Blanks

Mercury was found in one of the lab batches at a level just above the MDL (average result of 0.07 ng/L versus average MDL of 0.06 ng/L); results in that batch were flagged with the non-censoring qualifier of VIP (Analyte detected in field or lab generated blank, flagged by QAO).

5.5.3. Recovery

Matrix spike sample recoveries were used to evaluate the accuracy of mercury. Recoveries were generally good (within the QAPP Table 26-8 target 75-125% recovery (25% error)) for mercury (actual average 13.52%).

5.5.4. Precision

Matrix spike replicates were used to evaluate precision of mercury. The average RPD of 6.16% was less than the target MQO of 25% (QAPP Table 26-8). Field replicates were examined, but not used for the evaluation of mercury, and the average RPD of 14.38% was below the 25% target MQO.

5.6. Water PCBs – ALS

PCBs in water samples collected by KLI were analyzed by ALS. Samples were collected between September 4 (Leo Outflow sample) and September 20, 2013, and were analyzed between September 30 and October 24, 2013. Results were reported for 38 PCBs (with some co-eluting) in 11 field samples, method blanks, equipment blanks, matrix spike/matrix spike replicate (MS/MSD), and laboratory control samples (LCSs). Many of the analytes in the LCS samples were not target analytes in the field samples, and the MS samples included only 3 of the target analytes.

5.6.1. Sensitivity

Method detection limits were sufficient with no non-detects reported for the field samples.

5.6.2. Blanks

Three analytes (PCB 044/47/65, PCB 153/168, and PCB 180/193), all of them co-eluting groups, were detected in the blanks. All were flagged with the non-censoring qualifier VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning. Concentrations were always 10x lower or better compared to field samples.

5.6.3. Recovery

Matrix spike results were not useful for evaluating recovery, as analytes spiked into the matrix spike samples were low (typically <10%) of the native concentrations. LCS recoveries were good, with average errors <25% for all PCBs, except PCB 037 at just over 26%. Most of the LCS analytes were not targets in native field samples, but there were no patterns suggesting recovery issues with any homolog groups.

5.6.4. Precision

Precision was calculated only for sample pairs where an analyte was detected in both the field and lab replicate samples. Precision results were averaged across batches for analytes, and those with average RPDs > the 25% MQO (QAPP Table 26-9) were flagged as having marginal precision but not censored, and those with RPDs grossly above the MQO (average RPD >50%) were flagged with the qualifier of VJ and a compliance code of Est to indicate the results are estimated values (see table below). Only one analyte, PCB 095 fell into this latter category.

PARAMETER	avgRPD
PCB 095	67.61%
PCB 052	45.19%
PCB 141	28.85%
PCB 183	27.73%
PCB 132	26.39%
PCB 174	25.92%
PCB 008	25.75%

5.7. Water Copper, TOC, and Total Solids – ALS

Copper, total organic carbon (TOC), and Total Solids in water samples collected by KLI were analyzed by ALS. Samples were collected between September 13 and 20, 2013, and were analyzed between September 20 and October 21, 2013. Results were reported for copper in 4 water samples, 2 laboratory control samples (LCSs), and 2 method blanks. TOC results were reported in 4 water samples, 1 field replicate, 11 lab replicates, 1 MS, 3 method blanks, and 3 LCS samples. Total Solids were measured in 4 water samples, 1 field replicate, 2 lab replicates, 2 method blanks, and 2 LCS samples.

TOC results exceeded the 28 day holding time specified in the QAPP and were flagged with the qualifier “VH”. Several Total Solids results exceeded by several hours the 7 day holding time so were also flagged as “VH”.

5.7.1. Sensitivity

The method detection limits were sufficient with no non-detects reported for any of the parameters.

5.7.2. Blanks

Copper, total organic carbon (TOC), and Total Solids were not found in the blanks at concentrations over the detection limit (all NDs).

5.7.3. Recovery

Matrix spike sample recoveries were used to evaluate the accuracy of Total Organic Carbon; Copper and Total Solids were evaluated for accuracy using the recoveries from the laboratory control samples (LCSs). Recoveries were generally good, with an average error below the target MQO of 25% (all below 14%).

5.7.4. Precision

Lab replicate samples were used to evaluate precision of TOC and Total Solids, and LCS replicates were used to evaluate the precision of Copper. Average RPDs were less than the target MQO of 25% for all parameters (all less than 7%). Field replicates were examined, but not used for the evaluation of TOC and Total Solids with the average RPDs all less than the 25% target MQO.

5.8. Water Grainsize – SCL

Grainsize in water samples collected by KLI were analyzed by SCL. Water samples collected between September 13 and 20, 2013 were analyzed on September 26, 2013. Grainsize in 10 fractions, or combinations of sizes (from <2 to <0.00098 mm) were reported for 5 field samples, and one field replicate in both water concentration (mg/L) and % (of particle mass) units. No other sample types were reported.

Overall, the dataset is acceptable if used primarily for the silt/sand split. For some of the smaller sub-fractions, the quantitation is highly variable (up to over 100% RPD), possibly due to issues with subsampling suspended particles.

5.8.1. Sensitivity

Method detection limits were sufficient with no non-detects.

5.8.2. Precision

No lab replicates were run, only a field replicate of one sample. For the largest size fractions (Sand plus Silt: everything <2mm, and Silt: everything <0.0625mm) replicates were fairly good, with RPDs <5%. However, for Sand (~8% of total mass, RPD ~35%) and several other smaller fractions, both the concentration and percent grainsize reported were highly variable, with RPDs up to ~130%. Those results were flagged VIL for variable precision, but not censored.

6. Leo Ave Cleanout

6.1. Sediment Hg (Leo Avenue Cleanout) – SCL

Mercury in a small number of sediment samples added on to Task 4 Phase 1 was analyzed by SCL. Samples were collected on June 17, 2014 and analyzed on July 1, 2014. Total mercury was reported for 2 sediment field samples, 1 blind field replicate, 1 lab replicate, 1 certified reference material, 1 matrix

spike/matrix spike replicate (MS/MSD), and 1 method blank. Mercury samples exceeded the 14 day holding time listed in the QAPP, by 8 hours, but were not flagged.

6.1.1. Sensitivity

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

6.1.2. Blanks

Mercury was not detected in blanks above the method detection limits.

6.1.3. Recovery

The certified reference material, FLUKA SQC001-50G (021436); metals spiked in soil, obtained from Sigma-Aldrich (as external supplier) was used to evaluate the accuracy of the mercury results. Recoveries were good with an average error of 7.42% (within the QAPP Table 26-4 target 75-125% recovery (25% error)), well below the target MQO of 25%. Matrix spike recoveries were examined and the average error was below the 25% target for mercury (19.63%).

6.1.4. Precision

Precision on the lab replicates was acceptable, 0.32%, within the 25% MQO (QAPP Table 26-4). Field replicates were examined but not used for the evaluation, with the average RPD of 12%, being less than the 25% target MQO. Precision on the matrix spike replicates was examined, and with an average RPD of 8.72% was well below the target MQO.

6.2. Sediment PCBs and Total Solids – ALS

PCBs in sediment samples collected by KLI were analyzed by ALS. Samples were collected on June 17, 2014, and were analyzed on July 14, August 25, and August 26, 2014. Results were reported for 2 sediment samples for two fractions (“sediment, <63 um” and “sediment, 63 to 2000 um”), 1 blind field replicate (both fractions), 1 lab replicate (“sediment, <63 um”, only), 1 matrix spike/matrix spike replicate (MS/MSD; “sediment, <63 um”, only), 1 method blank, and 1 laboratory control sample (LCS) for 46 PCB congeners (including co-elutions and “sums”). Total Solids were analyzed for 2 sediment samples (both fractions), two blind field replicates, and one lab replicate (sediment, <63 um, only).

6.2.1. Sensitivity

Method detection limits were sufficient with non-detects (NDs) reported for only PCB 008 and Total Dichlorobiphenyls in both fractions, both 33.33% NDs in sediment, 63 to 2000 um and 25% NDs in sediment, <63 um; none had extensive NDs (>50% NDs).

6.2.2. Blanks

About 63% (29 out of 46) of the analytes were found in the blank in at least one or more of the batches. All were flagged with the non-censoring qualifier VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning.

6.2.3. Recovery

LCS samples were used to evaluate accuracy because the matrix spike samples were not spiked at concentrations high enough to avoid analytical variability in the native sample concentrations from dominating the recovery calculations. Recoveries were good, within the 50-150% recovery (50% error) QAPP Table 26-2 target for PCBs. Only 3 of the reported analytes (PCB 105, PCB 118, and PCB

156/157) in the LCS samples 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.

6.2.4. Precision

Precision was calculated only for sample pairs where an analyte was detected in both the field and lab replicate 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 with the qualifier of VJ and a compliance code of Est to indicate the results are estimated values (see table below). Five analytes, PCB 018/30, PCB 020/28, PCB 021/33, PCB 031, and Total Trichlorobiphenyls fell into this latter category.

PARAMETER	avgRPD
PCB 021/33	71.10%
Total Trichlorobiphenyls	58.65%
PCB 018/30	58.37%
PCB 020/28	58.02%
PCB 031	55.95%

Field replicates were examined for both fractions (“sediment, <63 um” and “sediment, 63 to 2000 um”), but not used in the evaluation. As would be expected, field replicates were more variable, than the lab replicates. PCB 018/30, PCB 020/28, PCB 021/33, PCB 031, PCB 044/47/65, PCB 049/69, PCB 052, PCB 056, PCB 060, PCB 061/70/74/76, PCB 066, Total Tetrachlorobiphenyls, and Total Trichlorobiphenyls had average RPDs above 25% for the sediment, <63 um fraction; ranging from 27.25% to 50.40%. For the fraction sediment, 63 to 2000 um, analytes above the target MQO of 25% were PCB 008, PCB 018/30, PCB 020/28, PCB 031, PCB 105, PCB 170, PCB 180/193, Total Dichlorobiphenyls, and Total Trichlorobiphenyls; ranging from 25.06% to 200%.

Matrix spike replicates were examined, but not used in the evaluation. Average RPDs for the analytes measured in the matrix spike replicates (PCB 105, PCB 118, and PCB 156/157) were all below the 25% target MQO (7.07%, 20.86%, and 13.36%, respectively).

6.3. Sediment Bulk Density and TOC – SCL

Bulk Density and Total Organic Carbon (TOC) in a small number of sediment samples added on to Task 4 Phase 1 were analyzed by SCL. Samples were collected on June 17, 2014 and were analyzed on June 18 and July 2, 2014. Bulk Density was reported for 2 sediment samples, and 1 blind field replicate. Total Organic carbon (TOC) results were reported for 2 sediment field samples, 1 blind field replicate, 1 lab replicate, 1 certified reference material, and 1 method blank.

6.3.1. Sensitivity

Sensitivity was sufficient with no non-detects reported for Bulk Density or TOC.

6.3.2. Blanks

TOC was not detected in blanks above the method detection limits.

6.3.3. Recovery

No spiked samples were analyzed/required per the QAPP for Bulk Density. The certified reference material, LECO Soil 502-308 (1006), carbon and nitrogen in soil from the LECO Corporation (external supplier) was used to evaluate the accuracy of the TOC results. Recoveries were good with an average error of 13.03% well below the target MQO of 25%.

6.3.4. Precision

Lab replicate samples were used to evaluate the precision of TOC. Precision on the lab replicates was acceptable, 10.08%, within the 25% MQO. Field replicates were examined for Bulk Density and TOC, but not used for the evaluation, with the respective average RPDs of 1.63% and 6.17%, both less than the target MQO.

6.4. Sediment GrainSize (Leo Avenue Cleanout) – SCL

Grainsize in sediment samples collected by KLI were analyzed by SCL. Samples collected on June 17, 2014 were analyzed on July 3, 2014. Grainsize in 17 fractions (from <64 to <0.00098 mm) were reported for 2 sediment field samples, 1 blind field replicate, 1 lab replicate, and 1 method blank.

6.4.1. Sensitivity

Method detection limits were sufficient with all analyte/fraction combinations having results, except for Pebble (Medium 8 to <16 mm fraction), Pebble (Large 16 to <32 mm fraction), and Pebble (V. Large 32 to <64 mm fraction) with 50%, 100% and 100% of results non-detects (NDs), respectively.

6.4.2. Blanks

No lab blanks are required per the QAPP, but one lab blank was analyzed with no blank contamination found.

6.4.3. Precision

Precision was evaluated using laboratory replicates. The analyte/fraction combinations Sand (V. Coarse 1.0 to <2.0 mm fraction) and Pebble (Small 4 to <8 mm fraction) had RPDs above the target MQO of 25% (43.15% and 133.56%, respectively), and were flagged with the non-censoring qualifier VIL (RPD exceeds control limit, flagged by QAO). The average RPD for Pebble (Small 4 to <8 mm fraction) being >50% results were flagged with the qualifier VJ and a compliance code of Est to indicate the results are estimated values. Field replicate RPDs were examined, but not used in the evaluation, and average RPDS were below the 25% target MQO, except for Sand (V. Coarse 1.0 to <2.0 mm fraction (29.16%)) and Pebble (Small 4 to <8 mm fraction (103.76%)). Similar to other grainsize analyses, the larger fractions are pretty heterogeneous.

7. Ettie Street Cleanout

7.1. Sediment Hg – ALS

Mercury in a small number of sediment samples were collected by AMS and analyzed by ALS. Samples were collected on September 16, 2014 and analyzed on October 15, 2014 and February 6, 2015. Total mercury was reported for three fractions; “sediment, <75 um”, “sediment, >75 um”, and “sediment, <2 mm”. Results were reported for 2 sediment samples, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), method blanks, and laboratory control samples (LCSs) for the “sediment, <75 um” fraction.

For the fraction “sediment, >75 um”, results were reported for 2 sediment samples, method blanks, and laboratory control samples (LCSs). Results were reported for 4 sediment samples, 1 blind field replicate, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), method blanks, and laboratory control samples (LCS) in the “sediment, <2 mm” fraction.

All samples were analyzed outside the QAPP hold time for sediment mercury samples, as they were not analyzed “within 14 days of collection or thawing”. K1410101 samples were not frozen till after analysis; they were analyzed 29 days after collection. K1410101 samples were later thawed and sieved to obtain the K1500147 samples, which were subsequently analyzed 143 days after initial collection. Results were flagged with the QA code of “VH”.

7.1.1. Sensitivity

Sensitivity was sufficient so no non-detects were reported for total mercury in any of the three fractions.

7.1.2. Blanks

Mercury was not detected in blanks above the method detection limits.

7.1.3. Recovery

Laboratory control sample (LCS) recoveries were used to evaluate accuracy for the “sediment, <75um” and “sediment, >75um” fractions. Recoveries were good with average errors well below the target MQO of 25% (within the QAPP Table 26-4 target 75-125% recovery (25% error)). Matrix spike samples for the “sediment, <75um” fraction were not usable as the recoveries were dominated by the analytical noise of the parent samples. No matrix spike samples were analyzed for the “sediment, >75um” fraction.

Matrix spike recoveries were used to evaluate accuracy of the “sediment, <2 mm” fraction. The average error for mercury was 5.18%, well below the target MQO of 25%. Laboratory control samples were examined, but not used in the evaluation, and with an average error of 2.51% were likewise well below the MQO target.

7.1.4. Precision

Precision on the lab replicates was acceptable for the “sediment, <75um” and “sediment, <2 mm” fractions, 0.58% and 0% respectively, within the 25% MQO (QAPP Table 26-4). Matrix spike replicates (for “sediment, <75um” fraction), and field replicate and laboratory control sample replicates (for “sediment, <2 mm” fraction) were examined but not used for the evaluation, with average RPDs all being below the MQO target. No lab replicates, field replicates, matrix spike replicates, or laboratory control sample replicates were analyzed for the “sediment, >75 um” fraction so results were flagged with the non-censoring qualifier of “VBS” to indicate insufficient QC procedures.

7.2. Sediment PCBs and Total Solids – ALS

PCBs and Total Solids in sediment samples collected by AMS were analyzed by ALS. Samples were collected on September 16, 2014, and analyzed between October 7, 2014 and February 27, 2015. PCBs and Total Solid were reported for three fractions; “sediment, <75 um”, “sediment, >75 um”, and “sediment, <2 mm”. Results were reported for 49 PCB congeners (including co-elutions and “sums”) in 2 sediment samples, method blanks, and laboratory control samples (LCSs) in the “sediment, <75 um” fraction. Total Solids (ww basis) were reported for this fraction in 2 sediment samples.

For the “sediment, >75 um” fraction, results were reported for 49 PCB congeners (including co-elutions and “sums”) in 2 sediment samples, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), method blanks, and laboratory control samples (LCSs). Total Solids (ww basis) were reported for 2 sediment samples.

Results were reported for 46 PCB congeners (including co-elutions and totals) in 4 sediment samples, 1 blind field replicate, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), method blanks, and laboratory control samples (LCSs) in the “sediment, <2 mm” fraction. Total Solids (ww and dw basis) were reported for this fraction in 4 sediment samples, 1 blind field replicate, and 1 lab replicate.

7.2.1. Sensitivity

Method detection limits were sufficient with non-detects (NDs) reported for only PCB 061 in the “sediment, <75 um” fraction, and PCB 061 and PCB 093/100 in the “sediment, >75 um” fraction. Method detection limits were sufficient for the “sediment, <2 mm” fraction with no NDs reported.

7.2.2. Blanks

For the “sediment, <75 um” and “sediment, >75 um” fractions, about 22% (11 out of 49) of the analytes, and about 63% (29 out of the 46) of the analytes for the “sediment, <2 mm” fraction were found in the blank in at least one or more of the batches. All were flagged with the non-censoring qualifier VIP (analyte detected in field or lab generated blank, flagged by QAO) as a warning.

7.2.3. Recovery

LCS samples were used to evaluate accuracy for all three sediment fractions as the matrix spike samples were not spiked at concentrations high enough to avoid analytical variability in the native sample concentrations from dominating the recovery calculations. Recoveries were good, within the 50-150% recovery (50% error) QAPP Table 26-2 target for PCBs. Only 3 of the reported analytes (PCB 105, PCB 118, and PCB 156/157) in the LCS samples 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.

7.2.4. Precision

Precision was calculated only for sample pairs where an analyte was detected in both the field and lab replicate samples, and then averaged across batches for the “sediment, >75 um”, and “sediment, <2 mm” fractions. No lab replicates, field replicates, matrix spike replicates, or laboratory control sample replicates were analyzed for the “sediment, <75 um” fraction. Results were consequently flagged with the non-censoring qualifier of “VBS” because of insufficient QC procedures.

Lab replicates for the “sediment, >75 um” fraction were quite variable for most analytes. Average RPDs ranged from 9.17% to 125.86% with 38 out of 47 (81%) of the analytes having RPDs greater than the target MQO of 25%, so were flagged with the non-censoring qualifier “VIL”. About 79% (30 out of 38) of the analytes had average RPDs >50% and were flagged with the QACode of VJ and a compliance code of Est to indicate estimated values. Matrix spike replicates were examined, but not used in the evaluation of this fraction. PCB 105, PCB 118, and PCB 156/157 all had average RPDs below the target MQO of 25% (16.01%, 6.84%, and 4.47%, respectively).

Lab replicates for the “sediment, <2 mm” fraction were likewise very heterogeneous with average RPDs ranging from 0.92% to 197.24%. About 93% (43 out of 46) of the analytes had RPDs greater than the target MQO, so were flagged with the non-censoring qualifier “VIL”. Thirty-nine out of 43 (91%) of those PCBs had average RPDs >50% and were assigned the QACode of VJ and a compliance code of Est to indicate estimated values. Field replicates were examined, but not used in the evaluation, of the “sediment, <2 mm” fraction. Average RPDs ranged from 3.59% to 177.24%, and 45 out of 46 (98%) of the analytes had RPDs greater than the 25% target MQO; 38 out of those 45 (84%) analytes had average RPDs >50%. Likewise, matrix spike replicates were examined, and PCB 105, PCB 118, and PCB 156/157 had average RPDs of 12.23%, 26.46%, and 22.02%, respectively.

7.3. Sediment Bulk Density and TOC – ALS

Bulk Density and Total Organic Carbon (TOC) in a small number of sediment samples collected by AMS were analyzed by ALS. Samples were collected on September 16, 2014 and were analyzed on October 14 and 31, 2014. Bulk Density was reported for 4 sediment samples, and 1 blind field replicate. Total Organic Carbon (TOC) in the <2mm sediment fraction was reported for 4 sediment field samples, 1 blind field replicate, 1 lab replicate, 1 matrix spike/matrix spike replicate (MS/MSD), 1 method blank, and 1 laboratory control sample (LCS). All bulk density samples exceeded the 7 day holding time specified in the QAPP so results were flagged with the code “VH” to indicate a holding time violation.

7.3.1. Sensitivity

The detection limits were above the project QAPP target of 0.01% for TOC, but all TOC results were non-detects above the detection limits (TOC MDL 0.02%). No non-detects were reported for Bulk Density.

7.3.2. Blanks

TOC was not detected in blanks above the method detection limits.

7.3.3. Recovery

No spiked samples were analyzed/required per the QAPP for Bulk Density. Matrix spike samples were used to evaluate the accuracy for Total Organic Carbon. Recoveries were good with the average error of 4.72% well below the target MQO of 25%. The LCS sample was examined, but not used for the evaluation, with the average error (6.91%) also below the 25% target.

7.3.4. Precision

Lab replicate samples were used to evaluate the precision of TOC. Precision on the lab replicates was acceptable, 12.54%, within the 25% MQO. Field replicates were examined, but not used for the evaluation of Bulk Density and TOC results, with average RPDs of 18.06% and 27.86%, respectively. Matrix spike replicates were examined for TOC, with the average RPD of 9.91% being well below the 25% target.

7.4. Sediment GrainSize – ALS

Grainsize in a small number of sediment samples collected by AMS were analyzed by ALS. Samples were collected on September 16, 2014 and were analyzed on October 10, 2014. Grainsize in 9 fractions (from <75 to <0.005 mm) using the ASTM scale were reported for 4 sediment field samples, 1 blind field replicate, 1 lab replicate, and 1 method blank. Some of the ASTM sizes are slightly offset from the ranges

requested in the project QAPP. Note: the various fractions for a sample did not always sum to 100%, therefore, results were rescaled so that analyte/fraction combinations for a sample summed to 100.

7.4.1. Sensitivity

No method detection or reporting limits were provided, and all analyte/fraction combinations had reportable results.

7.4.2. Precision

Lab replicates were used to evaluate precision. Grainsize was somewhat variable/heterogeneous with Silt (0.005 to <0.075 mm fraction), Gravel (4.75 to <75 mm fraction), and Clay (<0.005 mm fraction) having RPDs above the target MQO of 25% (27.78%, 32.24%, and 37.36%, respectively), and were flagged with the qualifier “VIL” for precision outside of the MQO target. Field replicate RPDs were examined, but not used in the evaluation, with average RPDS above the 25% target MQO, except for Sand (V. Fine 0.0625 to <0.125 mm fraction (RPD 9.55%)) and Sand (V. Coarse 1.0 to <2.0 mm fraction (RPD 3.14%)).

8. 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*. Prepared by Applied Marine Sciences, Inc. August 15, 2013.