

Dr. Robert Spies, Applied Marine Sciences, Livermore, CA

SPIES MAJOR COMMENTS:	RESPONSE TO COMMENTS:
1. Applicability of the approach a. The sampling scheme appears to capture and be representative of the range of water quality in runoff samples. The sampling was extensive enough that it is not likely to have missed many important sources of runoff from naval facilities to San Diego Bay. The inclusion of first-flush samples was important as these are well established now as the most toxic component of runoff, especially in a climate with long periods without precipitation. There are some questions raised under minor comments (below) as to why some of the larger discharges at some sites were bypassed in the selection process for drains that did not recruit from very large areas.	No comment necessary.
b. The chemical analyses appeared to be carried out according to best practices of environmental chemistry. However, there are several aspects that deserve comment.	No comment necessary.
First, there were no field blanks taken and analyzed, which are an important quality assurance precaution, especially when collecting water samples for determining concentrations of dissolved trace substances in an industrial settings.	Field blanks were taken for metals but text identifying that fact was left out of Table 10. Text was added to Table 10 to indicate that field blanks were included as part of the QA/QC.
Second, since trace organic substances are a concern for the NAVY (e.g., PCBs and PAH) why were larger water samples not taken to avoid the large proportion of non-detects?	The laboratory and methods used provide the best detection limits for aqueous organic compounds available anywhere in the country. The volume of storm water sample needed for all analyses was a difficult logistical requirement. Increasing the sample volume by more than a factor of two would have been very difficult to meet logistically and would have been cost prohibitive.

SPIES MAJOR COMMENTS (cont.):	RESPONSE TO COMMENTS:
Third, only total tin (Sn) was measured, which was rather surprising considering the very high toxicity of the alkylated tins and the fact that the US Navy is the largest user of these anti-fouling compounds. Alkylated tin toxicity has a chronic component, as these compounds are known endocrine disruptors. For example they cause the development of male sex organs in female gastropod mollusks.	The Navy does not use alkylated tins for anti-fouling coatings.
Fourth, while it is impractical to measure every possible chemical, the attribution of toxicity in some TIE analyses to nonylphenol suggests that this surfactant may be an important contaminant originating from some of the Navy bases. Nonylphenol is both lethal in some conditions and an endocrine disrupting compound in some organisms (e.g., fish).	Nonylphenol was identified as a partial cause of toxicity in one sample in the last year of the project. It was identified as a probable cause of toxicity in the TIE based on previous observations of toxic levels by the toxicity laboratory. However, saltwater aquatic life criteria just became available (EPA, February 2006). The acute criterion is 7.0 ug/L. The estimated concentration of nonylphenol in the TIE first-flush sample was 0.18 ug/L. These latest EPA criteria will be included in the report text.

SPIES MAJOR COMMENTS (cont.):	RESPONSE TO COMMENTS:
c. The toxicity tests carried out for this study on effluent and on bay waters were 96 hours long and were only capable of measuring short-term, and, for two of the three tests, acute toxicity. Longer-term effects that are expressed after 96 hours are not captured by these tests. The mussel embryo deformity test is not on the RWQCB list of assays, but was carried out on many samples. This test measures acute effects (occurring in 96 hours) but could be considered to measure sublethal effects since some of the deformed larvae are alive at the end of the test. It is probably predictive of longer-term toxicity for at least mussels. The requirements that the test organisms survive the whole undiluted effluent at high rates for storm water discharges provide some assurance against longer-term effects (expressed after 96 hours) and against sublethal effects. The largest criticism that I have of this report is that it appears to undermine this strategy by suggesting that dilution of the whole effluent after it enters San Diego Bay so that it is no longer acutely toxic is sufficient by implication to protect marine life in San Diego Bay. There is not sufficient data in this report about the fate and effects of discharged contaminants originating from Navy facilities to show that there is no harm to Bay life. Having Bay waters pass acute toxicity tests is not a sufficient basis for establishing a lack of harm. For example, contaminants that are not acutely toxic in storm runoff can accumulate in sediments to levels that affect benthic organisms.	For clarification purposes to the comment, the mussel test uses a 48-h, not 96-h exposure. We agree that the mussel embryo development test can be considered a sublethal effect that is predictive of longer-term toxicity. The test was chosen because mussels are endemic to San Diego Bay and it is one of the most sensitive toxicity tests to metals, the reason it was used to set EPA's aquatic life copper criterion for marine waters. As such, the test is one of the most sensitive toxicity endpoints available to evaluate either acute or chronic toxicity in marine waters. We understand the criticism and realize we may have not focused the report findings sufficiently on the major goal of evaluating the efficacy of using WET testing in "assessing and protecting against impacts upon water quality and designated uses caused by the aggregate toxic effects of the discharge of pollutants" (EPA, 1991). The study showed that the toxicity threshold used at the end-of-pipe was not predictive of a toxicological impact in receiving waters. This is because WET testing usually takes into account the exposure concentration after an effluent mixes with the receiving environment. It is also because toxicity testing is subject to method variability and the current 90% threshold has no power to detect a true toxic result. By using one of the most sensitive toxicity tests available, acute or chronic, for measuring receiving water toxicity; by evaluating contaminants against chronic aquatic life criteria; and by quantifying the duration and extent of storm plumes, the report can conclude that receiving water quality was protected against impacts in 99% of all cases, regardless of what the end-of-pipe WET test indicated. The study cannot conclude that there is no potential for impacts to sediments, though meeting the current permit requirement also does not guarantee this. These types of impacts are best evaluated under current TMDL and Baywide monitoring programs.

SPIES MAJOR COMMENTS (cont.):	RESPONSE TO COMMENTS:
d. Toxicity identification evaluations (TIEs) are useful in helping to identify causative agents for toxicity in standard bioassays. However, these tests are only guides and they do carry their own set of problems that must be kept in mind. For example, stage 2 testing is usually carried out 5 days after collection of the original bioassay water sample. The samples obviously cannot be acidified to preserve their chemical properties at the time of collection as one would for chemical analysis. So, the water samples can be altered chemically in this 5-day period. Heavy metals can bind to the sides of the container, eliminating this source of toxicity. Other processes such as volatilization, biodegradation of organic compounds, or possibly photo-oxidation can occur depending on sample storage conditions. In addition, TIEs are a reductionist approach and cannot account for the interactive effects of contaminants.	We agree with and understand the limitations of TIEs. However, there are no other standardized methods available for identifying potential causative agents. The Tier III copper and zinc tests performed on some samples were able to evaluate their interactions though this portion of the TIE was a special effort.
2. General considerations for protecting marine life in San Diego Bay. Chronic effects on bay organisms. It appears that some engineering solutions will be required to meet the current standards of the RWQCB. I do not think that the current discharges can be established as safe without much more detailed study of long-term effects. If the Navy wishes to go beyond the legal requirements for obtaining a permit and acquire a deeper understanding of the possible contributions its operations make to San Diego Bay then longer term testing would be in order and more studies of the relationships between the impairment of marine life and the particular suite of contaminants that are discharged from its San Diego facilities. Such studies might include the possible combined effects of copper, zinc, alkylated tins, polychlorinated biphenyls (PCBs) and polynuclear aromatic hydrocarbons (PAHs) at ambient Bay concentrations on growth, reproduction and fitness over the life cycle of key native organisms. Particularly useful would be participation in a Bay-wide study of contaminant effects and mass balance budgets of key contaminants. This of course should involve as well other sponsors that contribute to contamination of San Diego Bay.	The Navy agrees that if the current toxicity standard stands that the only alternative is an engineering solution that is estimated to cost over \$300M. The focus of this study was to evaluate the potential for toxicity occurring in receiving waters as a result of storm water discharges. The best way to evaluate long-term impacts to sediments is within current TMDL programs that are used to evaluate the magnitude and extent of impaired sediments using a weight-of-evidence approach and identify sources of the impairment. These programs are currently underway at several locations in San Diego Bay including at Navy facilities. Additional programs that can provide a better evaluation of long-term impacts to sediments include the Bay Protection and Toxic Cleanup Program, the Bight'98 program organized by the Southern California Coastal Water Research Program, and the Port of San Diego/Regional Water Quality Control Board Baywide Monitoring program.

SPIES MAJOR COMMENTS (cont.):	RESPONSE TO COMMENTS:
a. The role of the surface microlayer in ambient toxicity. In urban bays the very top layer of the water often has a microlayer that is about 75-100 micrometers thick. This layer is very important as it contains concentrations of most contaminants that may be several orders of magnitude higher than in the underlying water. In addition, it is subject to intense sunlight which photo-oxidizes some compounds (e.g., PAH) to much more toxic forms. Marine animals that spend anytime in this layer as adults, or in the larval or egg stages (some fish), will be subject to much greater toxicity than they otherwise might experience in water from beneath the surface. The microlayers persist under surprisingly energetic conditions and only break up in rather rough seas. I have little doubt that the microlayer plays a role in toxicity of contaminants in San Diego Bay in ways that were not anticipated in the design of the present study. It is quite likely that contaminants entering the Bay as storm water expose surface-dwelling organisms at higher concentrations than they experienced in toxicity tests carried out in this study with ambient sub-surface water. I would recommend that any future studies take the potential sequestration of storm water contaminants in the microlayer and their toxicity into account.	The study of the microlayer is a highly specialized area of research that has shown the potential for elevated concentrations of certain contaminants at levels above those found in the underlying water. The Navy study also did not specifically evaluate the potential for PAH photoactivation. Though the study did not evaluate this unique portion of the receiving water habitat, it did capture the exposure and toxic responses of the bulk surface water. An end-of-pipe measurement of toxicity is not likely to better predict toxicity that might occur in the microlayer. Techniques and studies to evaluate toxicity to microlayer organisms are in still in their infancy and would be highly impractical to implement at this time.
b. Was SDB4 an unusual circumstance? San Diego has a climate in which there are long dry periods in many years, so a 180-d period without rain before this particular event is probably not that unusual.	As stated in the report, the 182 day dry period was the longest dry period ever recorded (156 yr). Though dry periods can be relatively long in San Diego, 85% of dry periods were less than 127 days (National Weather Service, personal communication).

SPIES MINOR COMMENTS:	RESPONSE TO COMMENTS:
1. p. 17. Why weren't some of the largest drains of NAB (15, 17, 41) sampled? The drains that were measured appear to be rather minor ones.	As stated in the text, the choice of drains was based on several criteria including the presence or absence of industrial activities, logistical constraints and safe access during all hours. The reference to "minor" would only relate to size and not potential for impact. The data show that some of the smaller sites had similar contaminant levels and impacts as larger drainages.
2. p. 23. Where are results of the contaminant mass loading calculations mentioned here?	Mass loading data were not included in the report as they were not required to evaluate the efficacy of the toxicity requirement.
3. pp. 26-27. The RV ECOS-MESC system for real time chemical analyses and flow-through bioassays is innovative. Was there a comparison made between the outcome of split samples run in this system and those run under the usual conditions?	No comparisons were made during this effort. However, previous bay surveys have validated these techniques (Katz, 1998; Blake et al., 2004).
4. p. 33. Water samples could have been adjusted in volume or filtered part way through the study to provide greater detection limits for some analytes.	MDLs from our contract lab were as low or lower than any available from any lab in the country. Filtering samples for organics analysis is not recommended because of potential losses onto the filter during filtration
5. p. 39. Were mass flows reported in the Appendix?	No.
6. p. 43. The TIE exercise here and elsewhere was done for a small storm event and is probably not representative of a larger storm event.	The key component for performing TIEs is that the samples show a toxic response, not the size of the event.
7. p. 48. Hull coatings are apparently leaching copper into the Bay. What about alkylated tins?	The Navy does not use alkylated tins for hull coatings.
8. p. 51. Recheck latest literature on PAH chronic toxicity levels. In some cases concentrations of PAH in the low parts per billion have been chronically toxic to developing fish (e.g., Heintz et al., 2000; Carls et al., 1999).	We have rechecked the literature that was reviewed as a part of the study and found an additional comprehensive review article by Scannell et al., 2005 that expands our database and cites the two papers identified in the comment. None of the receiving water samples contained PAHs above a chronic toxic effect level, including effects with photoactivation under UV light. The text in the report was modified to account for the additional database information.

SPIES MINOR COMMENTS (cont.):	RESPONSE TO COMMENTS:
9. p. 32. Because of naphthalene contamination in Battelle's laboratory blanks, the elevated naphthalenes in Fig. 21 may be artifacts.	Procedural blanks were run with each batch of samples. Methylated naphthalene values in these blanks were typically between the MDL and the reporting limit though one blank analysis showed an elevated MDL of 16 ng/L. The methylated naphthalene data shown in Figure 21 were well above values found in the associated blanks and not a result of high blanks.
10. p. 55. Chronic WQOs are exceeded for DDT in these samples. Why were chronic WQOs not dealt with in more detail when considering the effects of the effluents in San Diego Bay?	The appropriate way to evaluate short-lived episodic discharge such as storm water is to compare levels against acute WQS. Results in receiving water samples, when available were compared to chronic WQS.
11. p. 78. Nonylphenol is implicated in the toxicity of NAB runoff but is not analyzed chemically in other samples on a routine basis.	MBAS, not nonylphenol was identified as a partial cause of toxicity in these samples.
12. p. 79-81. Copper and zinc exceeded chronic WQOs in Bay water after SDB4 and apparently were in the dissolved phase. This is at odds with your overall conclusion that these compounds are quickly diluted or chemically complexed to harmless levels in the Bay.	The overall conclusions of the study are based on the observation that 99% of bay water samples showed no toxicity and with some exceptions for copper and two for zinc, no elevation above chronic water quality standards of any contaminant. The study showed two instances of toxic effects. Discharges at these levels are not acceptable and should be targeted for additional BMPs. The proposed toxicity alternatives would identify these samples as a permit exceedance.
13. pp. 88-89. Another case of the apparent toxic effects of surfactants.	No comment necessary.
14. p. 97. 6484 ng/L is enough PAH to be of concern for chronic toxicity to some fish.	The value identified in the comment was for storm water discharges which should be compared to acute rather than chronic WQS. No receiving water sample exceeded a chronic toxic threshold identified in the literature.
15. p. 105. Combining top smelt and larval fish bioassay results is not well justified and I would question this.	We believe the comment should read: "combining topsmelt and mysid data is not well justified". The tests were combined mainly for the purposes of evaluating the percentage of tests failing or passing the toxicity thresholds. Because both tests can be used to meet the requirement, their results are in essence, interchangeable and can reasonably be combined for this purpose.

SPIES CONCLUSIONS:

This is a very extensive study and was competently carried out by the Navy and its contractors. It sets a new standard for storm water runoff studies. As in all studies there are some aspects that should be done differently if the study is to be repeated. Some important contaminants were not analyzed (tin chemical species, i.e., alkylated tins, and surfactants). Fifty-eight percent of the first flush samples failed the 90% survival criteria for whole effluent and copper and zinc are strongly implicated as serious problems in the runoff with other compounds possibly also contributing. While acute toxicity generally quickly dissipates with the mixing of the effluent in the Bay, this is not always the case. Chronic effects of the effluent in the Bay are not considered or addressed in any meaningful way. This reviewer does therefore not accept the conclusion that "The Bay is able to rapidly assimilate storm water discharges and effectively attenuate potential impacts, thus meeting the Clean Water Act narrative of 'no toxics in toxic amounts' (33 U.S.C. 125)." Attenuation of acute toxicity does not assure lack of chronic toxicity.

RESPONSE TO COMMENTS:

We appreciate that the reviewer acknowledged the magnitude of the effort conducted. We agree that every study could be improved or modified, particularly after the fact, when all the results become available. However, we believe that the main criticism that "chronic effects...are not considered...in any meaningful way" disregards the bulk of the findings that show there is no chronic exposure present in bay waters.

The study showed in every instance that the magnitude of storm water plumes were very short lived, lasting typically less than 24 hours. The plume measurements also showed that their magnitude was negligible away from the immediate shoreline source. The special bioassay study showed that peak exposures are on the order of minutes before falling off to background levels. The chemical data suggest that there were only limited instances when concentrations of contaminants exceeded an aquatic life chronic toxicity threshold. These measures that show a lack of potential for chronic toxicity are supported by the fact that 99% of all receiving water toxicity tests, using one of the most sensitive endpoints available, acute or chronic, showed no toxicity. The goal was not show that there could never be chronic toxicity, but rather, to identify the appropriate test that can be used to determine when that occurs.

COMMENTS ON ALTERNATIVE TOXICITY REQUIREMENT

SPIES ALTERNATIVES COMMENTS:	RESPONSE TO COMMENTS:
This draft set of more lenient criteria than those in force under Permits CA 0109163, CA 0109169 and CA0109185 are proposed based on the assumption that dilution of storm water in San Diego Bay with a corresponding reduction of acute toxicity in 96-h assays is protective of marine life in San Diego Bay. The changes in the criteria would allow testing to be carried out on diluted Bay water close to the effluent discharges and the testing to be of 48 h duration rather than 96 h. I am unable to support this change in the criteria as protection of marine life in San Diego Bay means protection from chronic effects of discharged contaminants as well. In the report which I reviewed there were no studies of long-term effects of discharged contaminants. However, this report did include data on discharge of nonylphenols and tin, and since nonylphenol and some chemical forms of tin are known endocrine disruptors in marine organisms, these specific sources and others with the potential for long-term toxicity to marine life in San Diego have not been investigated. In the storm water toxicity studies carried out by the Navy under these permits 58% of the first flush samples failed the 90% survival criteria for whole effluent. This reviewer does therefore not accept the conclusion that "The Bay is able to rapidly assimilate storm water discharges and effectively attenuate potential impacts, thus meeting the Clean Water Act narrative of 'no toxics in toxic amounts' (33 U.S.C. 125)." Attenuation of acute toxicity does not assure lack of chronic toxicity. The high threshold of effects imposed for storm water discharge in California are to compensate for less vigorous mixing in the inshore marine environment that allow point dischargers in offshore environments to use mixing zones and effluent dilution in their toxicity testing criteria. These high criteria for inshore discharges in water bodies with restricted circulation such as San Diego Bay also compensate to some degree for the lack of data on long-term chronic effects that can occur from low concentrations of water-borne contaminants. Many of these contaminants accumulate in sediments and organisms to much higher concentrations than in water through partitioning and bioaccumulation. I therefore cannot support the more lenient criteria as there are not sufficient data to demonstrate lack of harm to marine life from their implementation.	The main thesis of Dr. Spies' disagreement with the proposed alternatives is that the study failed to measure or account for chronic toxicity caused by low levels of chemicals that may or may not have been measured in the study. A secondary thesis is that chemicals derived from storm discharges may accumulate in sediments and/or organisms and eventually lead to impairment. The study results show that the current end-of-pipe toxicity requirement is not predictive of acute or chronic toxic effects in receiving waters. Of all the storm water samples identified as "toxic", only two receiving water samples showed a toxic result. The study dataset covers the full range of conditions likely to occur, including a condition that clearly represents a chronic exposure condition to bay organisms (SDB5 collected outside 4 outfalls after 6" of rainfall over a two-week period). We know of no other EPA approved toxicity endpoint, chronic or acute, that would provide a more sensitive measure of effects, particularly to the main CoCs of copper and zinc. The magnitude and extent of storm water plumes indicates and the special floating bioassay study show that chronic exposures are not likely. The current permit requirement for end-of-pipe toxicity has little ability to predict acute or chronic toxicity or other impacts to sediments. We believe that an evaluation of impairment to benthic organisms is best done within sediment TMDLs that are already underway at two Navy bases. These studies evaluate the magnitude and extent of impairment using multiple lines of evidence and can be used to identify likely sources of the contaminants causing the impairment.

References cited that were not included in the draft report:

- Chadwick, D.b., A. Zirino, I. Rivera-Duarte, C. N. Katz, and A. C. Blake (2004). *Modeling the mass balance and fate of copper in San Diego Bay*. Limnol. Oceanogr., 49(2), 2004, 355–366.
- Scannell, P.W, D.D. Duffy, R. Perkins, and T. O'Hara (2005). *Acute and chronic toxicity of hydrocarbons in marine and fresh water with an emphasis on Alaska species, A review of the literature*. Review performed for the Alaska Dept. Environmental Conservation, 610 University Avenue, Fairbanks, AK 99709/
- EPA (2006). *FACT SHEET Aquatic Life Ambient Water Quality Criteria - Nonylphenol – Final*, United States Environmental Protection Agency Office of Water 4304T EPA-822-F-05-003, February 2006.

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DEPARTMENT OF THE NAVY
COMMANDER NAVY REGION SOUTHWEST
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IN REPLY REFER TO:
5090
Ser N40JRR.cs/073
August 20, 2012

Ms. Jeanine Townsend
Clerk of the Board
State Water Resources Control Board
1001 I Street
Sacramento, CA 95814

Dear Ms. Townsend:

Subject: COMMENT LETTER - POLICY FOR TOXICITY ASSESSMENT AND
CONTROL

On behalf of the Department of Defense (DoD) Regional Environmental Coordinator (REC) in California, we appreciate the opportunity to provide comments on the Water Board's Draft Policy for Toxicity Assessment and Control. We previously submitted comments on 18 November 2010, and January 21, 2011 (Enclosures). We are offering the below comments on the latest draft.

We believe the Test of Significant Toxicity (TST) provides certainty in the evaluation of Whole Effluent Toxicity (WET) testing that was not provided previously by the Technical Support Document for Water Quality Based Toxics Control, 1991(TSD) and Percent Minimum Significant Difference (PMSD) documentation and commend the work to develop its use and implementation. We believe that when the TST is applied appropriately, at a concentration equal to the Instream Waste Concentration (IWC), the test results will provide good prediction of toxicological effects in receiving waters.

We have considerable data to support that the use of toxicity testing when applied to 100% storm water is not predictive of toxicological effects in receiving waters. This leads us to conclude that the portion of the Policy definition of IWC that states: "A discharge of 100 percent effluent will be considered the IWC whenever mixing zones or dilution credits are not authorized by the applicable Water Board" will potentially be misapplied and result in an unintended consequence of an overly conservative outcome that is costly to dischargers with no benefit to the environment and the State.

We suggest that the Policy provide clear guidelines to Regional Boards for the application of mixing zones to storm water discharges. Specifically, mixing zones or dilution credits

Subject: COMMENT LETTER - POLICY FOR TOXICITY ASSESSMENT AND CONTROL

should generally be applied to storm water discharges unless a Regional Board finds specific factual reasons to support using 100% storm water. To further clarify, we also suggest the Policy include specific conditions when mixing zones or dilution credits standard should NOT be applied. Some examples of these conditions include:

- a) When a storm water discharge makes up the majority of the flow or volume of the receiving water body;
- b) When the initial zone of dilution is large enough to preclude clear passage of threatened and/or endangered species through the water body; and
- c) When there is no natural mechanism for flushing of the water body.

We have consistently identified the need for California's water programs to consider the significant contribution of deposition from aerial and mobile sources in storm water toxicity. Substantial research continues to show that sources such as automobile brake pads, and their contribution of metals, are key sources of toxicity. These sources are beyond the immediate control of facility operators whether that operator be a public agency, private businesses, or municipality.

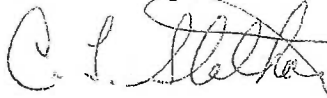
We believe that any toxicity policy must recognize that the reduction of toxicity, especially in urbanized, areas must come from holistic changes such as the DTSC brake pad program created by SB 346 (Kehoe.) Absent this approach, the multiple testing requirements of the Toxicity Policy will merely affirm what we already know: first flush urban storm water runoff is toxic. Instead, we believe the policy should focus on promoting transformational changes and using toxicity testing that actually seeks to measure a true impact to receiving waters.

In summary, we believe that allowing Regional Boards authority to use an IWC=100% effluent, in most situations, is an incorrect application of WET testing procedures. This approach lacks scientific basis and is not a good method of predicting receiving water impacts. Regional Boards have inconsistently applied toxicity testing creating uncertainty for regulated

Subject: COMMENT LETTER - POLICY FOR TOXICITY ASSESSMENT AND
CONTROL

parties. In addition, we believe the Toxicity Policy should promote transformational changes in how storm water toxicity is addressed. If you have questions or concerns regarding this letter please contact Brian Gordon at (619) 532-2273 or Chris Haynes at 619 532-2285.

Sincerely

A handwritten signature in dark ink, appearing to read "C. L. Stathos", written over a horizontal line.

C. L. STATHOS

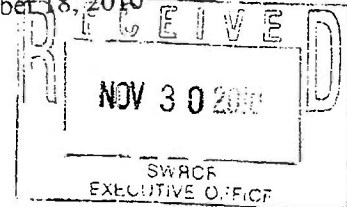
By direction

Enclosures: 1. DoD REC Comment Ltr dated Nov 18, 2010
2. Navy Region Southwest Comment Ltr dated Jan 21, 2011



DEPARTMENT OF DEFENSE
REGIONAL ENVIRONMENTAL COORDINATOR, REGION 9
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5090
Ser N40JRR.cs/0034
November 18, 2010



Ms. Jeanine Townsend
Clerk to the Board
State Water Resources Control Board (SWRCB)
1001 I Street
Sacramento, CA 95814

Subject: **COMMENTS ON THE DRAFT POLICY FOR TOXICITY ASSESSMENT AND CONTROL**

On behalf of the Department of Defense (DoD) Regional Environmental Coordinator (REC) in California, we appreciate this opportunity to provide the comments below on the Water Board's Draft Policy for Toxicity Assessment and Control.

In the Water Board's Staff Report on "Policy for Toxicity Assessment and Control" Project Background Section identifies the triggering event for this draft policy as the renewal of two NPDES permits for two publicly owned treatment plants (page 5 and 6). While chronic toxicity may be an established problem with discharges from publicly owned treatment plants, the staff report never indicates there is a demonstrated problem with chronic toxicity from storm water discharges. The proposed four chronic toxicity tests per year for storm water discharge (Part III, Section B.3, page 14) should not be required until it is established that chronic toxicity from storm water run-off has the reasonable potential to cause or contribute to an excursion above the chronic toxicity objective.

Should the state elect to move forward with the chronic toxicity monitoring requirements for storm water discharges, the definition for "instream waste concentration" (page 2, Definition H.) should be revised to clarify storm water discharges can only be assessed after considering mixing in the receiving water, and read as follows:

"Instream waste concentration (IWC) is the concentration of a toxicant or effluent in the receiving water after mixing (the inverse of the dilution factor). For discharges other than stormwater, a discharge of 100% effluent will be considered the IWC whenever mixing zones or dilution credits are not authorized by the applicable Water Board."

The draft policy allows Regional Water Boards to determine "reasonable potential" by applying toxicity testing to whole effluent storm water runoff instead of considering the actual exposure to aquatic life. Storm water discharges are generally short term, intermittent discharges that typically do not cause toxicity in receiving waters after mixing. Applying toxicity testing and objectives directly to storm water discharges is overly conservative and will result in reasonable potential determinations that do not reflect actual affects to aquatic life. The acute and chronic

toxicity reasonable potential analyses and effluent limitations for storm water discharges and other intermittent, short term discharges should be performed on the effluent after considering the mixing that occurs in the receiving water. This will provide for the consistent statewide application of toxicity limitations and objectives and prevent the application of overly conservative standards that are not based on real impacts to beneficial uses.

In addition, we would request that the policy on compliance schedules be modified to recognize the differences between acute toxicity and chronic toxicity. As currently written, a discharger with existing toxicity monitoring requirements is ineligible to receive a compliance schedule. This would mean that a discharger with existing acute toxicity monitoring requirements would not be eligible for a compliance schedule for the proposed chronic toxicity requirements. The SWRCB's "Policy for Compliance Schedules in NPDES Permits" allows for a compliance schedule for a new permit limitation "more stringent than the limitation previously imposed." We believe requiring compliance with chronic toxicity tests constitutes a more stringent limitation than a previously imposed acute toxicity limitation. As such, we propose insertion of the word "chronic" into the last sentence of Part III, Section B.4 (page 14) so it would read:

"Phase I and Phase II MS4 dischargers and individual industrial storm water dischargers with existing chronic toxicity monitoring requirements are not eligible to receive a compliance schedule."

Please direct any questions or concerns you may have regarding this letter to Mr. Michael Huber at (619) 532-2303.

Sincerely,



C.L. STATHOS

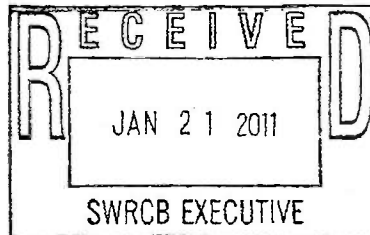
By Direction



DEPARTMENT OF THE NAVY
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IN REPLY REFER TO:
5090
Ser JWB.bg/0016
January 21, 2011

Ms. Jeanine Townsend
Clerk of the Board
State Water Resources Control Board
1001 I Street
Sacramento, CA 95814



Subject: Comment Letter – Policy for Toxicity Assessment and Control

On behalf of the Commander, Navy Region Southwest (CNRSW), we appreciate the opportunity to provide comments below on the Water Board's Draft Policy for Toxicity Assessment and Control. We previously submitted comments on the draft policy in August 2010. The comments in this letter address our concerns with economic considerations associated with the policy, and the unguided discretion provided to Regional Boards in utilizing 100% effluent as the Instream Waste Concentration (IWC).

Economic Considerations

We believe that the State Board should not adopt this policy until it considers the full costs of implementation/compliance similar to how the Air Resources Board quantifies the costs of its proposed air quality regulations. The Staff Report provides very little consideration of the costs associated with compliance where chronic toxicity limitations are included in permits. This is particularly true if the policy provides discretion to the Regional Boards to apply chronic toxicity limitations where the IWC=100 effluent. We question whether or not compliance in this situation for storm water discharges is even consistently feasible with costly treatment and whether diversion to sanitary sewage systems is the only method to achieve compliance, if this option is even available. These potential costs were not considered in the Staff Report. We have in the past provided a number of written letters to the State and Regional Board concerning this issue. We also point to the fact that should Regional Boards apply chronic toxicity limitations utilizing an IWC=100% effluent for storm water discharges from general urban populated areas it may have tremendous costs both fiscally and socially.

We further believe that implementation of this standard may have serious impacts on the ability of several of our major installations to conduct their missions as it may prevent the ability to do ongoing maintenance activities that are a standard part of home-porting.

Instream Waste Concentration

The draft policy inappropriately provides the Regional Boards unguided discretion in utilizing whole effluent toxicity (WET) testing on discharge samples in which the instream waste concentration (IWC) is equal to 100% effluent. We believe that this is a misapplication of WET testing procedures in predicting receiving water community impacts and are concerned that this requirement would be applied in a non-uniform or inappropriate way. The allowance for Board discretion is found in the second version of the Policy which defines the Instream Waste Concentration as:

"Instream waste concentration (IWC) is the concentration of a toxicant or effluent in the receiving water after mixing (the inverse of the dilution factor). A discharge of 100% effluent will be considered the IWC whenever mixing zones or dilution credits are not authorized by the applicable Water Board."

This definition was altered from the first version of the Draft State Policy by inclusion of the second sentence, thereby allowing Water Boards authority to make the IWC=100% effluent. According to the Staff Report on the Draft Policy, the underlying rationale to use an IWC=100% effluent was for "...water for which mixing zones would not be allowed (e.g., ephemeral and low flow streams, impaired water bodies)". However, this or any other rationale has not been included in the policy and the historical usage of toxicity testing in the State has shown that Regional Boards will inconsistently utilized the IWC=100% effluent toxicity testing on all manner of receiving water conditions so that there is no standardized approach for toxicity testing. As a member of the regulated community it would be difficult or impossible to know in advance what standard would be applied. The Staff Report on the Draft Policy identifies numerous examples of current and historical requirements to evaluate toxicity at an IWC=100%, regardless of receiving water conditions, and there is no discernible, predictable regulatory pattern. This data also includes discharges from Navy facilities that are also subject to WET testing of an IWC=100% effluent, even though receiving water conditions warrant mixing zones or dilution credits.

We believe that WET testing is appropriate for evaluating potential impacts in receiving water when the stated methods, conditions, and evaluations for WET testing are conducted in accordance with how the EPA's Toxicity Support (TSD) Document, Percent Minimum Significant Difference (PMSD) Document, and Test of Significant Toxicity (TST) Document. These documents identify methods, data, and study results designed to show that WET testing is appropriate for predicting receiving water community impacts. The testing in all of these EPA studies evaluates the Instream Waste Concentration (IWC) against a control sample, where the *"IWC is the concentration of a toxicant or effluent in the receiving water after mixing. The IWC is the inverse of the dilution factor. It is sometimes referred to as the receiving water concentration (RWC)."* We agree that WET testing results can be used for the purpose of hypothesis testing that will successfully predict receiving water impacts when the test sample is the IWC. However, we do not agree that a 100% effluent sample collected at the end-of-pipe represents the IWC.

The Navy's position on this point is based on the EPA's stated goals, hypothesis testing, and its own extensive research and datasets used to develop WET test methods and guidance. To our knowledge the EPA has never published data or an evaluation of the use of 100% effluent samples in predicting receiving water impacts. In particular, the EPA's TSD specifically points out the efficacy of its large database and WET tests conducted on samples that were correctly diluted to their ambient condition and the appropriateness of considering dilution:

"Together, these studies comprise a large data base specifically collected to determine the validity of toxicity tests to predict receiving water community impact. In order to address the correlation of effluent and ambient toxicity tests to receiving water impacts, EPA evaluated the results of the studies discussed above [29]. The results, when linked together, clearly show that if toxicity is present after considering dilution, impact will also be present."

The Navy conducted its own extensive research, described in a 2006 report and provided to the San Diego Regional Board, which conclusively showed that WET testing of 100% storm water effluent was not predictive of effects in an estuarine environment. The Navy's study showed that 34% of 64 acute toxicity tests conducted on 100% effluent samples failed (using t-testing for significance) even though acute toxicity was never found in 129 receiving water samples collected adjacent to outfall pipes. The Navy's data also showed that 90% of 40 chronic toxicity tests conducted on 100% storm water effluent samples failed even though chronic toxicity testing was found only twice in 60 (3%) receiving water samples collected adjacent to outfall pipes. The major difference in results between WET tests conducted on 100% effluent and WET tests conducted on receiving waters clearly shows a lack of test predictability. This result was the basis for the Navy recommending that samples be measured in the ambient or adjusted for true exposure conditions in the receiving environment (i.e., samples that represent the IWC) when performing WET testing instead of using 100% effluent.

The reason end-of-pipe 100% effluent tests are not predictive of effects in the receiving environment is that they do not account for the true exposure conditions that organisms in the receiving environment are subject to during storm events. While the permit-required WET tests in the study were conducted on 100% storm water over a 96-hr period, organisms in the receiving environment were subject to 100% effluent on the order of minutes, if ever, and typically at effluent concentrations less than 5% for periods of less than 12 hours. Additionally, there is a well-known capacity of estuarine waters to mitigate the toxic effects of pollutants through natural complexation (biotic ligand model) that is not taken into account in WET testing procedures on 100% effluent.

In summary, we believe that allowing Regional Boards authority to use an IWC=100% effluent is an incorrect application of WET testing procedures and lacks scientific basis as a good method of predicting receiving water impacts. In addition, Regional Boards have inconsistently applied toxicity testing to IWC=100% effluent, creating uncertainty for regulated parties. Finally, chronic WET testing on 100% effluent is an inappropriate and unproductive testing method because storm water discharges will almost always fail even though there may be no impact in the receiving water. We therefore urge you not to grant the Regional Boards authority

to allow IWC=100% effluent, or at minimum provide clear direction when an IWC=100% can be utilized (e.g., ephemeral and low flow streams, impaired water bodies).

If you have questions or concern regarding this letter I can be contacted at (619) 532-2273.

Sincerely

A handwritten signature in cursive script, appearing to read "B. S. Gordon". The signature is written in dark ink and is positioned above the printed name.

B. S. GORDON

By direction

METALS QA/QC (CONT.)

MSL Code	Rep	Instrument Sponsor I.D.	ICP-MS Ag	ICP-MS Al	ICP-MS As	ICP-MS Cd	ICP-MS Cr	ICP-MS Cu	ICP-MS Fe	CVAH Hg	ICP-MS Mn	ICP-MS Ni	ICP-MS Pb	ICP-MS Se	ICP-MS Sn	ICP-MS Zn
METHOD BLANK																
Method Blank		Hg-03/13/03	NA	NA	NA	NA	NA	NA	NA	0.00014 U	NA	NA	NA	NA	NA	NA
Method Blank		Hg-03/14/03	NA	NA	NA	NA	NA	NA	NA	0.00014 U	NA	NA	NA	NA	NA	NA
Method Blank		Hg-03/18/03	NA	NA	NA	NA	NA	NA	NA	0.00014 U	NA	NA	NA	NA	NA	NA
1979-blk TRM r1		ICP-MS	0.0038 U	0.0008 U	0.0007 U	0.0008 U	0.245 J	0.0029 U	NA	NA	0.003 U	0.0114 U	0.0044 U	0.0991 U	0.0185 J	0.0493 U
1979-blk TRM r2		ICP-MS	0.0038 U	0.0038 U	0.00929 J	0.0008 U	0.321 J	0.0029 U	NA	NA	0.003 U	0.0114 U	0.0044 U	0.0991 U	0.00810 J	0.0493 U
1979-blk dissolved		ICP-MS	0.00463 J	0.00463 J	0.00039 J	0.0039 J	0.024 U	0.0029 U	0.993 U	NA	0.003 U	0.0259 J	0.0044 U	0.0991 U	0.0103 J	0.0493 U
Blank Trm r1		ICP-MS (Al, Fe)	NA	0.823 U	NA	NA	NA	NA	0.993 U	NA	NA	NA	NA	NA	NA	NA
METHOD DETECTION LIMIT			0.0038	0.823	0.0087	0.0008	0.024	0.0029	0.993	0.00014	0.003	0.0114	0.0044	0.0991	0.0024	0.0493
Project Target			0.50	50.0	0.50	0.05	1.00	0.05	10.0	0.01	0.50	0.05	0.05	0.20	0.50	0.50
STANDARD REFERENCE MATERIAL																
1979-1640 TRM		ICP-MS	7.49	58.2	28.3	23.1	41.5	87.8	NA	NA	132	29.2	27.7	21.9	1.54	54.9
1640 Direct		ICP-MS	7.63	52.7	30.8	25.3	40.4	89.9	NA	NA	127	29.2	27.6	28.2	1.56	84.4
1640 TRM		ICP-MS (Al, Fe)	NA	50.6	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
1640		certified value	7.6	52.0	26.7	22.8	38.6	85.2	34.3	NC	122	27.4	27.9	22.0	NA	NA
1640		range	±0.25	±1.5	±0.73	±0.96	±1.6	±1.2	±1.6	NC	±1.1	±0.8	±0.14	±0.51	NC	±1.1
		% difference	2%	12%	6%	1%	8%	3%	3%	NA	9%	7%	1%	0%	NA	3%
		% difference	0%	3%	15%	11%	5%	6%	NA	NA	5%	7%	1%	28%	NA	21%
		% difference	NA	3%	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
SLRS-3 (Fe)		ICP-MS	NA	NA	NA	NA	NA	NA	119	NA	NA	NA	NA	NA	NA	NA
SLRS-3 (Fe)		ICP-MS (Al, Fe)	NA	NA	NA	NA	NA	NA	92.2	NA	NA	NA	NA	NA	NA	NA
		certified value	NA	NA	NA	NA	NA	NA	100	NA	NA	NA	NA	NA	NA	NA
		range	NA	NA	NA	NA	NA	NA	±2	NA	NA	NA	NA	NA	NA	NA
		% difference	NA	NA	NA	NA	NA	NA	19%	NA	NA	NA	NA	NA	NA	NA
		% difference	NA	NA	NA	NA	NA	NA	8%	NA	NA	NA	NA	NA	NA	NA
16410031203		Hg-03/13/03	NA	NA	NA	NA	NA	NA	NA	1565	NA	NA	NA	NA	NA	NA
16410031303		Hg-03/14/03	NA	NA	NA	NA	NA	NA	NA	1466	NA	NA	NA	NA	NA	NA
16410031703		Hg-03/18/03	NA	NA	NA	NA	NA	NA	NA	1573	NA	NA	NA	NA	NA	NA
1641d		certified value	NC	NC	NC	NC	NC	NC	NC	1590	NC	NC	NC	NC	NC	NC
1641d		range	NC	NC	NC	NC	NC	NC	NC	±4.00	NC	NC	NC	NC	NC	NC
		% difference	NA	NA	NA	NA	NA	NA	NA	2%	NA	NA	NA	NA	NA	NA
		% difference	NA	NA	NA	NA	NA	NA	NA	8%	NA	NA	NA	NA	NA	NA
		% difference	NA	NA	NA	NA	NA	NA	NA	1%	NA	NA	NA	NA	NA	NA
ICV/CCV RESULTS																
ICV		ICP-MS or Hg 1	102%	102%	103%	100%	103%	102%	104%	93%	103%	103%	101%	104%	101%	102%
CCV		ICP-MS or Hg 1	103%	113%	107%	102%	109%	105%	110%	100%	110%	106%	100%	104%	102%	107%
CCV		ICP-MS or Hg 1	104%	113%	105%	102%	108%	106%	115%	98%	109%	106%	98%	104%	102%	105%
CCV		ICP-MS or Hg 1	103%	113%	105%	100%	108%	106%	113%	101%	109%	105%	98%	104%	101%	105%
CCV		ICP-MS or Hg 1	101%	114%	104%	100%	108%	103%	111%	94%	108%	104%	98%	101%	100%	105%
ICV		ICP-MS (Al, Fe) or Hg 2	NA	97%	NA	NA	NA	NA	92%	94%	NA	NA	NA	NA	NA	NA
CCV		ICP-MS (Al, Fe) or Hg 2	NA	101%	NA	NA	NA	NA	96%	92%	NA	NA	NA	NA	NA	NA
CCV		ICP-MS (Al, Fe) or Hg 2	NA	NA	NA	NA	NA	NA	NA	94%	NA	NA	NA	NA	NA	NA
CCV		ICP-MS (Al, Fe) or Hg 2	NA	NA	NA	NA	NA	NA	NA	97%	NA	NA	NA	NA	NA	NA
ICV		Hg 3	NA	NA	NA	NA	NA	NA	NA	100%	NA	NA	NA	NA	NA	NA
CCV		Hg 3	NA	NA	NA	NA	NA	NA	NA	101%	NA	NA	NA	NA	NA	NA
CCV		Hg 3	NA	NA	NA	NA	NA	NA	NA	103%	NA	NA	NA	NA	NA	NA
CCV		Hg 3	NA	NA	NA	NA	NA	NA	NA	96%	NA	NA	NA	NA	NA	NA

[illegible]

MSL	Code	Rep	Instrument Sponsor I.D.	ICP-MS	Aq	ICP-MS	Al	As	ICP-MS	Cd	Cr	ICP-MS	Cu	Fe	CVAF	ICP-MS	Mn	Ni	ICP-MS	Pb	Sa	ICP-MS	Sn	Zn	
REPLICATE RESULTS																									
1979-23			NAV-PR6-SDB2-FF	0.0266		30.4	NA	1.41		1.23	3.58	177		161	0.0133		81.5	17.2		0.879		0.289	J	288	
1979-23			2 NAV-PR6-SDB2-FF			NA	NA	NA		NA	NA	NA	NA	NA	0.0132		NA	NA		NA		NA			
			% difference	NA	NA					NA	NA	NA	NA	NA	1%		NA	NA		NA		NA			
1979-24			NAVPR3-SDB2-COMP	0.00809		15.1	NA	1.18		0.303	1.12	14.2		17.6	0.00219	J	5.94	1.88		0.533		0.247	0.0603	J	80.8
1979-24			2 NAVPR3-SDB2-COMP	0.00670		14.8	NA	1.10		0.295	1.14	13.6		16.2	NA		5.88	1.91		0.502		0.0691	U	79.7	
			% difference	18%		2%	2%	7%		3%	2%	4%		8%	NA		1%	2%		6%		13%		1%	

D-72

PAHs

CLIENT SAMPLE ID	SUB- OF11B-SDB2- FF		SUB- OF24-SDB2- FF		SUB- OF26-SDB2- FF	
Battelle Sample ID	U7094		U7093		U7095	
Battelle Batch ID	03-0203		03-0203		03-0203	
Data File	A1890.D		A1889.D		A1891.D	
Extraction Date	03/04/03		03/04/03		03/04/03	
Acquired Date	03/20/03		03/20/03		03/20/03	
Matrix	Water		Water		Water	
Sample Size (L)	2.66		2.66		2.66	
Dilution Factor	1.667		1.667		1.667	
PIV (mL)	0.3		0.3		0.3	
Min Reporting Limit	0.94		0.94		0.94	
Amount Units	ng/L		ng/L		ng/L	
Naphthalene	5.02	B	3.57	B	5.73	B
C1-Naphthalenes	2.78	B	2.87	B	4.62	
C2-Naphthalenes	5.20		6.25		5.17	
C3-Naphthalenes	5.55		4.86		4.95	
C4-Naphthalenes	11.32		11.31		10.95	
2-Methylnaphthalene	2.73	B	2.60	B	4.13	
1-Methylnaphthalene	1.72	B	1.67	B	2.11	B
2,6-Dimethylnaphthalene	1.27		1.50		1.38	
2,3,5-Trimethylnaphthalene	0.95		0.99		0.37	U
Biphenyl	2.43		1.61		2.60	
Acenaphthylene	1.37		0.56	J	1.40	
Acenaphthene	0.96		0.59	J	4.93	
Fluorene	1.91		1.25	B	7.16	
C1-Fluorenes	3.62		3.82		3.78	
C2-Fluorenes	7.61	B	18.92		35.65	
C3-Fluorenes	29.93	B	45.48		48.59	
Phenanthrene	18.47		20.80		59.33	
Anthracene	1.46	B	1.72	B	3.08	
C1-Phenanthrenes/Anthracenes	15.40	B	17.01	B	17.91	B
C2-Phenanthrenes/Anthracenes	34.66	B	24.22	B	33.99	B
C3-Phenanthrenes/Anthracenes	27.77	B	11.19	B	21.92	B
C4-Phenanthrenes/Anthracenes	13.41	B	2.74	B	7.38	B
1-Methylphenanthrene	4.32	B	4.54	B	4.56	B
Dibenzothiophene	7.27		4.62		9.30	
C1-Dibenzothiophenes	13.07		8.54	B	14.98	
C2-Dibenzothiophenes	42.40	B	23.78	B	28.80	B
C3-Dibenzothiophenes	42.08	B	16.79	B	28.21	B
Fluoranthene	28.49		16.92		42.51	
Pyrene	31.56	B	15.45	B	28.96	B
C1-Fluoranthenes/Pyrenes	18.74	B	9.61	B	14.74	B
C2-Fluoranthenes/Pyrenes	22.76	B	7.37	B	16.75	B
C3-Fluoranthenes/Pyrenes	25.77	B	7.12	B	13.95	B
Benzo(a)anthracene	5.68		1.76		2.20	
Chrysene	29.63		9.65		18.35	
C1-Chrysenes	25.16		6.55	B	10.57	B
C2-Chrysenes	29.19	B	10.97	B	11.54	B
C3-Chrysenes	28.50	B	0.28	U	0.28	U
C4-Chrysenes	0.28	U	0.28	U	0.28	U
Benzo(b)fluoranthene	11.63		4.70		9.75	
Benzo(k)fluoranthene	9.02		3.81		5.41	
Benzo(e)pyrene	12.23		4.78		7.21	
Benzo(a)pyrene	6.62		2.81		3.15	
Perylene	4.47		0.91	J	0.73	J
Indeno(1,2,3-cd)pyrene	7.20		2.87		4.26	
Dibenz(a,h)anthracene	1.89		0.45	J	0.74	J
Benzo(g,h,i)perylene	17.99		7.40		10.12	
Total Priority Pollutant PAHs	178.90		94.32		207.07	
Surrogate Recoveries (%)						
Naphthalene-d8	61		58		69	
Phenanthrene-d10	70		67		77	
Chrysene-d12	69		80		89	

PAHs QA/QC

PROJECT: SPAWAR TO0011, Contaminant Analysis of Stormwater and San Diego Bay
Seawater
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: The water samples were collected February 25, 2003. They were received in Duxbury on February 28, 2003 in good condition in six coolers. The cooler temperature on arrival ranged from 0.2 °C to 1.3 °C. Samples were stored at 4 °C until processing.

QA/QC DATA QUALITY OBJECTIVES:

	Reference Method	Surrogate Recovery	LCS/MS Recovery	Sample Replicate Relative Precision	Procedural Blank
PAH	General NS&T	30-130% Recovery	LCS: 40-120% Recovery for at least 80% of analytes MS: 50-150% Recovery for at least 70% of analytes; analyte conc. in MS must be >5x background	≤30% RSD analyte conc. in MS must be <5x background	<3X MDL

METHOD: Water samples were extracted for PAH following general NS&T methods. Full water samples were spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with Recovery Internal Standard (RIS) and split for analysis. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS) with the MS operating in the selected ion monitoring (SIM) mode, following general NS&T methods. Sample data were quantified by the method of internal standards, using the RIS compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch.

Samples were extracted within the 7-day holding time for waters. Extracts were analyzed within the 40-day holding time for extracts

<u>Batch</u>	<u>Extraction Date</u>	<u>Analysis Date</u>
03-0203	3/4/2003	3/19/2003 – 3/20/2003

BLANKS: A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0203 – Several target analytes were detected at concentrations greater than 3X the MDL.

Comments – All samples are appropriately flagged. The contamination in the blank does not appear to have the same PAH homologue pattern as the samples indicating that the contamination is likely isolated to the blank and that the samples are not impacted by the blank contamination. This is supported by the fact that no alkyl homologues were detected in the LCS (blank spike) sample – the LCS is prepared in the same manner as the blank, with the addition of a spike of the target analytes of interest (in this case, the parent PAH).

LABORATORY **Note:** The 2003 MDL for substituted naphthalenes were updated.
A laboratory control sample (LCS) was prepared with each analytical batch. The percent

**CONTROL
SAMPLE:**

recoveries of target PAH were calculated to measure data quality in terms of accuracy.

03-0203 – All target analytes were recovered within the laboratory control limits specified by the client.

Comments – None.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS)/matrix spike duplicate (MSD) pair was prepared with each analytical batch. The percent recoveries of target PAH were calculated to measure data quality in terms of accuracy; the relative percent difference between the pair was calculated to measure data quality in terms of precision.

03-0203 – All target analytes were recovered within the laboratory control limits specified by the client. The relative percent differences between the MS and MSD recoveries were within the laboratory control limits for all target PAH.

Comments – None.

SURROGATES:

Three surrogate compounds were added prior to extraction, including naphthalene-d8, phenanthrene-d10, and chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0203 – All surrogate percent recoveries were within the laboratory control limits specified by the client.

Comments – None.

PAHs QA/QC (CONT.)

CLIENT SAMPLE ID	LABORATORY CONTROL SAMPLE			MATRIX SPIKE-NAV-OF9-SDB2-FF			MATRIX SPIKE DUPLICATE-NAV-OF9-SDB2-FF				PROCEDURAL BLANK	
Battelle Sample ID	BB593LCS			U7083MS			U7083MSD				BB592PB	
Battelle Batch ID	03-0203			03-0203			03-0203				03-0203	
Data File	A1873.D			A1875A.D			A1876.D				A1872.D	
Extraction Date	03/04/03			3/4/2003			03/04/03				03/04/03	
Acquired Date	03/19/03			3/19/2003			37699				03/19/03	
Matrix	Water			Water			Water				Water	
Sample Size (L)	2			1.18			1.18				2	
Dilution Factor	1.667			1.67			1.67				1.667	
PIV (mL)	0.30			0.30			0.30				0.3	
Min Reporting Limit	1.25			2.13			2.13				1.25	
Amount Units	ng	Rec%	Q	ng/L	Rec%		ng/L	Rec%	Q	RPD	Q	ng/L
Naphthalene	352.38		70	543.19		63	554.69		64	2.14		2.33
C1-Naphthalenes	0.66	U	NA	1.13	U	NA	1.13	U	NA	NA		1.23
C2-Naphthalenes	0.66	U	NA	1.13	U	NA	1.13	U	NA	NA		0.66
C3-Naphthalenes	0.66	U	NA	1.13	U	NA	1.13	U	NA	NA		0.66
C4-Naphthalenes	0.66	U	NA	1.13	U	NA	1.13	U	NA	NA		0.66
1-Methylnaphthalene	360.50		72	577.66		67	577.08		67	0.10		1.09
2-Methylnaphthalene	339.07		68	553.60		64	554.86		64	0.23		0.82
2,6-Dimethylnaphthalene	343.64		69	579.78		68	595.69		70	2.72		0.47
2,3,5-Trimethylnaphthalene	371.42		74	645.28		76	686.38		80	6.19		0.50
Biphenyl	337.28		67	583.63		68	594.79		69	1.91		0.28
Acenaphthylene	379.80		76	644.53		75	674.66		79	4.59		0.41
Acenaphthene	361.28		72	612.93		72	636.85		74	3.84		0.54
Fluorene	398.80		80	685.50		80	731.35		85	6.51		0.57
C1-Fluorenes	0.49	U	NA	0.83	U	NA	0.83	U	NA	NA		1.13
C2-Fluorenes	0.49	U	NA	0.83	U	NA	0.83	U	NA	NA		4.77
C3-Fluorenes	0.49	U	NA	0.83	U	NA	0.83	U	NA	NA		13.84
Phenanthrene	419.23		84	774.11		82	869.69		94	12.77		5.78
Anthracene	430.88		86	683.27		80	747.23		87	9.00		0.84
C1-Phenanthrenes/Anthra	0.38	U	NA	0.64	U	NA	0.64	U	NA	NA		15.14
C2-Phenanthrenes/Anthra	0.38	U	NA	0.64	U	NA	0.64	U	NA	NA		58.14
C3-Phenanthrenes/Anthra	0.38	U	NA	0.64	U	NA	0.64	U	NA	NA		81.62
C4-Phenanthrenes/Anthra	0.38	U	NA	0.64	U	NA	0.64	U	NA	NA		108.36
1-Methylphenanthrene	425.88		85	721.31		84	796.73		92	10.07		2.70
Dibenzothiophene	5.39		NA	21.83		NA	23.72		NA	NA		0.82
C1-Dibenzothiophenes	0.46	U	NA	0.79	U	NA	0.79	U	NA	NA		3.14
C2-Dibenzothiophenes	0.46	U	NA	0.79	U	NA	0.79	U	NA	NA		22.71
C3-Dibenzothiophenes	0.46	U	NA	0.79	U	NA	0.79	U	NA	NA		47.04
Fluoranthene	464.01		93	861.68		89	1001.92		106	16.88		5.12
Pyrene	477.18		95	879.40		92	1033.03		110	17.89		17.73
C1-Fluoranthenes/Pyrenes	0.44	U	NA	0.75	U	NA	0.75	U	NA	NA		31.70
C2-Fluoranthenes/Pyrenes	0.44	U	NA	0.75	U	NA	0.75	U	NA	NA		66.39
C3-Fluoranthenes/Pyrenes	0.44	U	NA	0.75	U	NA	0.75	U	NA	NA		68.03
Benzo(a)anthracene	472.04		94	760.93		88	892.16		103	16.14		0.69
Chrysene	469.94		94	820.97		88	995.84		108	20.99		0.37
C1-Chrysenes	0.37	U	NA	0.62	U	NA	0.62	U	NA	NA		6.13
C2-Chrysenes	0.37	U	NA	0.62	U	NA	0.62	U	NA	NA		25.02
C3-Chrysenes	0.37	U	NA	0.62	U	NA	0.62	U	NA	NA		30.09
C4-Chrysenes	0.37	U	NA	0.62	U	NA	0.62	U	NA	NA		0.37
Benzo(b)fluoranthene	432.60		86	776.18		86	964.01		108	22.83		0.43
Benzo(k)fluoranthene	439.70		88	747.55		83	919.30		103	21.64		0.43
Benzo(e)pyrene	388.05		78	685.24		77	836.81		95	21.05		1.48
Benzo(a)pyrene	418.30		84	725.55		81	893.33		101	21.63		0.65
Perylene	384.14		77	659.17		77	789.49		92	18.15		0.70
Indeno(1,2,3-cd)pyrene	390.28		78	728.87		81	810.93		91	11.20		0.43
Dibenz(a,h)anthracene	421.03		84	740.55		86	814.99		95	9.65		1.04
Benzo(g,h,i)perylene	347.96		70	658.67		71	751.71		82	14.29		0.69
Total Priority Pollutant PAHs												33.49
Surrogate Recoveries (%)												
Naphthalene-d8	74			63			66					74
Phenanthrene-d10	81			77			82					77
Chrysene-d12	93			83			95					91

PCBs

CLIENT SAMPLE ID:	SUB- OF11B-SDB2 FF		SUB- OF24-SDB2- FF		SUB- OF26-SDB2 FF	
Battelle Sample ID:	U7094		U7093		U7095	
Battelle Batch ID:	03-0203		03-0203		03-0203	
Data File:	sc0382,54,1		sc0382,53,1		sc0382,55,1	
Extraction Date:	3/04/03		3/04/03		3/04/03	
Aquired Date:	3/19/03		3/19/03		3/19/03	
Matrix:	Water		Water		Water	
Sample Volume (L):	2.660		2.660		2.660	
Dilution Factor:	1.667		1.667		1.667	
Pre Injection Volume (µL):	300		300		300	
Minimum Reporting Limit (ng/L):	0.188		0.188		0.188	
Units:	ng/L		ng/L		ng/L	
CI2 08	1.805 U		1.805 U		1.805 U	
CI3 18	0.156 U		0.156 U		0.156 U	
CI3 28	0.195 U		0.279 NC		0.195 U	
CI4 44	0.164 U		0.475 NC		0.731 NC	
CI4 49	4.229 NC		0.168 U		0.168 U	
CI4 52	0.162 U		0.162 U		0.162 U	
CI4 66	0.168 U		0.168 U		0.168 U	
CI4 77	1.320 NC		1.841 NC		0.239 U	
CI5 87	0.086 NC		0.127 U		0.127 U	
CI5 101	0.129 U		1.338 NC		0.129 U	
CI5 105	0.065 U		0.065 U		0.065 U	
CI5 114	0.111 U		0.111 U		0.111 U	
CI5 118	0.098 U		0.098 U		0.098 U	
CI5 123	0.111 U		0.111 U		0.111 U	
CI5 126	0.139 U		0.139 U		0.139 U	
CI6 128	0.732 NC		0.281 NC		0.304 NC	
CI6 138	0.413 NC		0.149 U		0.149 U	
CI6 153	0.120 U		0.698		0.120 U	
CI6 156	0.135 U		0.135 U		0.135 U	
CI6 157	0.135 U		0.135 U		0.135 U	
CI6 167	0.815		0.135 U		0.135 U	
CI6 169	0.108 U		0.108 U		0.108 U	
CI7 170	0.114 U		0.114 U		0.114 U	
CI7 180	0.893		0.221 NC		0.108 U	
CI7 183	0.105 U		0.105 U		0.105 U	
CI7 184	0.104 U		0.104 U		0.104 U	
CI7 187	0.097 U		0.197		0.097 U	
CI7 189	0.106 U		0.106 U		0.106 U	
CI8 195	0.122 U		0.122 U		0.122 U	
CI9 206	0.137 U		0.363		0.137 U	
CI10 209	1.463		0.793		0.346	
Total PCB	9.136		6.488		1.381	
Surrogate Recoveries:						
CI3(34)	89		77		85	
CI5(112)	66		69		75	

PCBs QA/QC

PROJECT: SPAWAR TO0011, Contaminant Analysis of Stormwater and San Diego Bay Seawater
PARAMETER: PCB Congener
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: The water samples were collected on February 25, 2003. They were received in Duxbury on February 28, 2003 in good condition in six coolers. The cooler temperature on arrival ranged from 0.2 °C to 1.3 °C. Samples were stored at 4 °C until processing.

QA/QC DATA QUALITY OBJECTIVES:

	Reference Method	Surrogate Recovery	LCS/MS Recovery	Sample Replicate Relative Precision	Procedural Blank
PAH	General NS&T	30-130% Recovery	LCS: 40-120% Recovery for at least 80% of analytes MS: 50-150% Recovery for at least 70% of analytes; analyte conc. in MS must be >5x background	≤30% RSD analyte conc. in MS must be <5x background	<3X MDL

METHOD: Water samples were extracted for PCB Congener following general NS&T methods. Full water samples were spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with Recovery Internal Standard (RIS) and split for analysis. Extracts were analyzed using gas chromatography/electron capture detection (GC/ECD), following general NS&T methods. Sample data were quantified by the method of internal standards, using the RIS compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch.

Samples were extracted with in the 7-day holding time for waters. Extracts were analyzed within the 40-day holding time for extracts

<u>Batch</u>	<u>Extraction Date</u>	<u>Analysis Date</u>
03-0203	3/4/2003	3/17/2003 – 3/22/2003

BLANKS: A procedural blank (PB) was prepared with each analytical batch. Blanks were analyzed to ensure the sample extraction and analysis methods were free of contamination.

03-0203 – No analytes identified at greater than 3X the MDL.

Comments – None.

LABORATORY CONTROL SAMPLE: A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB Congeners were calculated to measure data quality in terms of accuracy.

03-0203 – All target analytes were recovered within the laboratory control limits specified by the client.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

Comments – None.

A matrix spike (MS)/matrix spike duplicate (MSD) pair was prepared with each analytical batch. The percent recoveries of target PCB Congeners were calculated to measure data quality in terms of accuracy; the relative percent difference between the pair was calculated to measure data quality in terms of precision.

03-0203 – All target analytes were recovered within the laboratory control limits specified by the client. The relative percent differences between the MS and MSD recoveries were within the laboratory control limits for all target PCB Congeners.

SURROGATES:

Comments – None.

Two surrogate compounds were added prior to extraction, including PCB34 and PCB112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

03-0203 – All surrogate percent recoveries were within the laboratory control limits specified by the client.

Samples:

Comments – None.

The condition of the confirmation column was in question after the analysis. It was decided to report all “hits” from the primary column regardless if confirmed or not confirmed. The analytes are appropriately flagged if reported, but not confirmed.

PCBs QA/QC (CONT.)

CLIENT SAMPLE ID:	LABORATORY CONTROL SAMPLE		MATRIX SPIKE- NAV-OF9-SDB2-FF		MATRIX SPIKE DUPLICATE-NAV-OF9-SDB2-FF		
Battelle Sample ID:	BB593LCS		U7083MS		U7083MSD		
Battelle Batch ID:	03-0203		03-0203		03-0203		
Data File:	sc0382,38,1		sc0382,40,1		sc0382,41,1		
Extraction Date:	3/04/03		3/04/03		3/04/03		
Aquired Date:	3/17/03		3/18/03		3/18/03		
Matrix:	Water		Water		Water		
Sample Volume (L):	2.000		1.175		1.175		
Dilution Factor:	1.667		1.667		1.667		
Pre Injection Volume (µL):	300		300		300		
Minimum Reporting Limit (ng/L):	0.250		0.426		0.426		
Units:	ng	% Recovery	ng/L	% Recovery	ng/L	% Recovery	% RPD
Ci2 08	20.509	68	17.345	68	20.449	80	16
Ci3 18	21.498	72	19.854	66	20.959	70	6
Ci3 28	35.064	117	21.372	84	25.258	99	17
Ci4 44	25.535	85	21.262	83	23.531	92	10
Ci4 49	25.348	84	24.907	97	25.302	99	2
Ci4 52	24.280	81	20.705	81	21.345	84	3
Ci4 66	27.632	92	22.603	89	25.267	99	11
Ci4 77	24.023	80	20.028	73	22.256	82	11
Ci5 87	24.470	82	20.388	77	21.738	82	7
Ci5 101	25.400	85	21.352	76	23.998	86	13
Ci5 105	26.157	87	20.806	80	21.916	84	5
Ci5 114	NA	NA	NS	NA	NS	NA	NA
Ci5 118	23.286	78	19.969	75	21.834	82	9
Ci5 123	NA	NA	NS	NA	NS	NA	NA
Ci5 126	28.227	94	19.566	77	20.869	82	6
Ci6 128	26.487	88	23.105	85	21.485	79	8
Ci6 138	25.310	84	22.841	80	24.760	88	9
Ci6 153	22.656	76	22.155	79	23.904	86	8
Ci6 156	NA	NA	NS	NA	NS	NA	NA
Ci6 157	NA	NA	NS	NA	NS	NA	NA
Ci6 167	NA	NA	NS	NA	NS	NA	NA
Ci6 169	28.949	96	23.429	91	25.035	98	7
Ci7 170	25.778	86	22.468	85	24.052	91	7
Ci7 180	25.907	86	23.798	85	26.011	94	10
Ci7 183	25.158	84	21.384	84	22.322	87	4
Ci7 184	23.828	79	19.885	78	20.606	80	4
Ci7 187	23.085	77	19.872	75	20.867	79	5
Ci7 189	NA	NA	NS	NA	NS	NA	NA
Ci8 195	25.317	84	20.354	80	21.844	86	7
Ci9 206	23.978	80	20.081	79	21.885	86	9
Ci10 209	23.396	78	18.226	66	19.594	71	8
Total PCB	631.280	NA	527.755	NA	567.085	NA	NA
Surrogate Recoveries:							
Ci3(34)	84		90		99		
Ci5(112)	78		74		77		

SDB3- 2/2/2004

METALS

MSL	Rep	Sponsor	As (µg/L)	Se (µg/L)	Ag (µg/L)	Cd (µg/L)	Sn (µg/L)	Pb (µg/L)	Hg (µg/L)
Code		I.D.	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF
SAMPLE RESULTS									
2157*1		SUB-OF11B-SDB3-COMP (T)	1.09	0.561	0.0403	0.237	0.50 U	12.2	0.04315
2157*4		SUB-OF11B-SDB3-COMP (D)	0.721	0.650	0.009 U	0.0880	0.50 U	0.400	0.01546
2157*3		SUB-OF23CE-SDB3-COMP (T)	2.08	0.260 t	0.0633	2.60	0.874	20.1	0.01657
2157*6		SUB-OF23CE-SDB3-COMP (D)	1.52	0.237 t	0.009 U	0.855	0.50 U	0.742	0.02654
2157*2		SUB-OF26-SDB3-COMP (T)	4.62	0.629	0.0722	0.995	0.537	7.82	0.01740
2157*5		SUB-OF26-SDB3-COMP (D)	4.31	0.20 U	0.0256	0.451	0.50 U	0.521	0.00740
2157*8		Field Blank-Filtered	0.129 t	0.20 U	0.009 U	0.023 U	0.50 U	0.0345	0.01046

MSL	Rep	Sponsor	Al (µg/L)	Fe (µg/L)	Cr (µg/L)	Mn (µg/L)	Ni (µg/L)	Cu (µg/L)	Zn (µg/L)
Code		I.D.	ICP-OES	ICP-OES	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-OES
SAMPLE RESULTS									
2157*1		SUB-OF11B-SDB3-COMP (T)	2190	3210	6.16	78.4	6.76	24.9	123
2157*4		SUB-OF11B-SDB3-COMP (D)	9.05	31.6	0.890	11.1	3.18	15.2	37.4
2157*3		SUB-OF23CE-SDB3-COMP (T)	1550	1980	6.71	89.7	7.68	37.3	792
2157*6		SUB-OF23CE-SDB3-COMP (D)	18.2	33.5	0.948	35.9	3.14	18.0	505
2157*2		SUB-OF26-SDB3-COMP (T)	529	2300	4.79	48.7	9.31	216	442
2157*5		SUB-OF26-SDB3-COMP (D)	17.5	30.9	1.80	23.8	5.76	142	263
2157*8		Field Blank-Filtered	0.638 b	10.0 U	0.0712	0.50 U	0.01 U	0.018 U	0.140 U

SAMPLE ID	DISSOLVED COPPER (ppb)	TOTAL COPPER (ppb)	DISSOLVED ZINC (ppb)	TOTAL ZINC (ppb)
SUB-SDB3-BAY11B-PRE	1.1	1.6	3.8	4.3
SUB-SDB3-BAY11B-DUR	1.6	1.9	7.6	7.0
SUB-SDB3-BAY11B-AFT	0.66	1.1	2.5	2.9
SUB-SDB3-BAY23CE-PRE	1.1	1.6	4.5	4.5
SUB-SDB3-BAY23CE-DUR	0.78	1.2	2.8	3.4
SUB-SDB3-BAY23CE-AFT	0.60	0.80	1.9	2.07
SUB-SDB3-BAY26-PRE	1.6	2.4	4.0	4.6
SUB-SDB3-BAY26-DUR	1.4	1.8	6.3	6.2
SUB-SDB3-BAY26-AFT	0.59	0.88	1.6	1.93
SUB-SDB3-BAY26A-PRE	0.79	1.1	2.2	2.7
SUB-SDB3-BAY26A-DUR	0.34	0.55	1.2	1.19
SUB-SDB3-BAY26A-AFT	0.42	0.62	1.3	1.24

METALS QA/QC

QA/QC SUMMARY

PROGRAM: SPAWAR STORMWATER, Task 15
PARAMETER: Metals
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Stormwater

QA/QC DATA QUALITY OBJECTIVES

	Reference Method	Range of Recovery	SRM Accuracy	Relative Precision	Target Detection Limit (µg/L)
Aluminum	ICP/MS	50-150%	±20%	±50%	50.0
Iron	ICP/MS	50-150%	±20%	±50%	10.0
Manganese	ICP/MS	50-150%	±20%	±30%	0.5
Chromium	ICP/MS	50-150%	±20%	±30%	1.0
Nickel	ICP/MS	50-150%	±20%	±30%	0.05
Copper	ICP/MS	50-150%	±20%	±30%	0.05
Zinc	ICP/MS	50-150%	±20%	±30%	0.5
Arsenic	FIAS	50-150%	±20%	±30%	0.5
Selenium	FIAS	50-150%	±20%	±30%	0.2
Silver	GFAA	50-150%	±20%	±30%	0.5
Cadmium	ICP/MS	50-150%	±20%	±30%	0.05
Tin	ICP/MS	50-150%	±20%	±30%	0.5
Lead	ICP/MS	50-150%	±20%	±30%	0.05
Mercury	CVAF	50-150%	±25%	±30%	0.01

METHOD

Seven (7) samples were analyzed for ten (10) metals; chromium (Cr), manganese (Mn), nickel (Ni), copper, (Cu), arsenic (As), selenium (Se), silver (Ag), cadmium (Cd), tin (Sn) and lead (Pb) by inductively coupled plasma mass spectroscopy (ICP/MS) following EPA Method 1631m, three (3) metals: aluminum (Al), iron (Fe), and zinc (Zn) by inductively coupled plasma optic emission spectroscopy (ICP/OES) following EPA Method 200.7, and mercury (Hg) by cold vapor atomic fluorescence (CVAF) following EPA Method 1631e.

Samples were preserved with nitric acid prior to arrival at MSL. The samples were analyzed for all metals except Hg by ICP/MS. Results for Al, Fe, and Zn were outside the range of the ICP/MS and were then analyzed by ICP/OES. Samples analyzed for Hg by CVAF were pre-treated with bromine chloride and stannous chloride to oxidize and convert all Hg compounds to volatile Hg, which is subsequently trapped onto a gold-coated sand trap.

HOLDING TIMES

Eight (8) samples were received on 2/5/2004 and were logged into Battelle's sample tracking system. Following a phone call from Joel Guerrero, sample 7 was designated for archive instead of analysis. Seven samples were digested and analyzed within the six-month holding time for metals and 90 days for Hg. The following list summarizes all analysis dates:

Task	Date Performed
Hg	2/12/04
ICP-MS	2/17/04
ICP-OES	3/9/04
ICP-OES (reanalysis of sample 2159*4)	3/12/04

DETECTION LIMITS

The target detection limit was met for all metals. The method detection limit was met for all metals. An MDL is determined by multiplying the standard deviation of the results of a minimum of 7 replicate low level spikes by the Student's t value at the 99th percentile.

METHOD BLANKS

One method blank was analyzed with this batch of samples. Results were less than 3 times the MDL for all metals, except Al. Sample results that are less than 3 x the blank have been "b" flagged.

BLANK SPIKES

One sample of reagent water was spiked at one level with metals. Recoveries were within the QC limits of 50-150% for all metals.

MATRIX SPIKES

One sample was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

REPLICATES

A duplicate was not requested for this task.

SRM

Two matrix-appropriate standard reference materials (SRM) were analyzed for each method; 1641d, river water, and 1640, natural water, obtained from the National Institute of Science and Technology.

SRM 1640 has 22 certified metals. Recovery for all metals reported were within the control limit of $\pm 20\%$ of the certified value. Tin and Hg are not certified in 1640. SRM 1641d is certified for Hg. Recovery for Hg was within the control limit of $\pm 25\%$ of the certified value.

REFERENCES

EPA. 1991. Methods for the Determination of Metals in Environmental Samples. EPA-600/4-91-010. Environmental Services Division, Monitoring Management Branch.

METALS QA/QC (CONT.)

MSL	Rep	Sponsor	Al (µg/L)	Fe (µg/L)	Cr (µg/L)	Mn (µg/L)	Ni (µg/L)	Cu (µg/L)	Zn (µg/L)	As (µg/L)	Se (µg/L)	Ag (µg/L)	Cd (µg/L)	Sn (µg/L)	Pb (µg/L)	Hg (µg/L)
Code		I.D.	ICP-OES	ICP-OES	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-OES	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF
PROCEDURAL BLANK																
METHOD DETECTION LIMIT			0.785	10.0	0.107	0.50	0.01	0.018	0.160	0.051	0.20	0.009	0.023	0.50	0.0110	0.00014
Project Target Detection Limit			0.20	NA	0.047	NA	0.01	0.018	0.140	0.051	NA	0.009	0.023	NA	0.011	0.00014
STANDARD REFERENCE MATERIAL			50.0	10.0	1.00	0.50	0.05	0.05	0.50	0.50	0.20	0.50	0.05	0.50	0.05	0.01
1640			61.3	36.9	38.7	124	27.9	86.3	56.8	25.7	21.3	7.59	22.9	1.47	27.3	NA
1640		certified value	52.0	34.3	38.6	122	27.4	85.2	53.2	26.7	22.0	7.62	22.8	NC	27.9	NC
1640		range	±1.5	±1.6	±1.6	±1.1	±0.8	±1.2	±1.1	±0.73	±0.51	±0.25	±0.96	NC	±0.14	NC
1640		% difference	18%	8%	0%	2%	2%	1%	7%	4%	3%	0%	0%	NA	2%	NA
1641d			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	1613
1641d		certified value	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	1557
1641d		range	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	±4.00
1641d		% difference	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	4%
ICV/CCV RESULTS																
ICV			101%	100%	101%	103%	101%	101%	103%	102%	102%	102%	102%	102%	102%	93%
CCV			99%	98%	98%	101%	98%	99%	102%	99%	100%	101%	101%	101%	99%	101%
CCV			99%	98%	99%	101%	97%	99%	101%	98%	100%	100%	100%	101%	100%	94%
CCV			100%	98%	102%	104%	100%	102%	103%	103%	102%	102%	101%	103%	101%	NA
CCV			NA	NA	98%	95%	95%	97%	NA	99%	99%	102%	102%	106%	106%	NA
BLANK SPIKE RESULTS																
Amount Spiked			2500	2500	50.0	50.0	50.0	50.0	250	50.0	50.0	50.0	50.0	50.0	50.0	0.00496
Blank			0.785	10.0	0.107	0.50	0.010	0.018	0.160	0.051	0.20	0.009	0.023	0.50	0.011	0.000368
Blank + Spike			2538	2477	48.5	47.4	46.5	50.7	255	48.1	47.7	50.3	50.9	52.5	52.6	0.00543
Amount Recovered			2537	2477	48.4	47.4	46.5	50.7	255	48.1	47.7	50.3	50.9	52.5	52.6	0.00506
Percent Recovery			101%	99%	97%	95%	93%	101%	102%	96%	95%	101%	102%	105%	105%	102%
MATRIX SPIKE RESULTS																
Amount Spiked			NS	NS	50.0	50.0	50.0	50.0	NS	10.0	10.0	10.0	10.0	10.0	50.0	NS
SUB-OF11B-SDB3-COMP (T)			N/A	N/A	6.16	78.4	6.76	24.9	N/A	1.09	0.561	0.0403	0.237	0.50	12.2	N/A
SUB-OF11B-SDB3-COMP (T)+ Spike			NS	NS	59.4	127	59.5	76.1	NS	12.3	11.8	10.6	11.3	8.00	62.4	NS
Amount Recovered			N/A	N/A	53.2	48.6	52.7	51.2	N/A	11.2	11.2	10.6	11.1	8.00	50.2	N/A
Percent Recovery			N/A	N/A	106%	97%	105%	102%	N/A	112%	112%	106%	111%	80%	100%	N/A
Amount Spiked			2336.0	2336	NS	NS	NS	NS	234	NS	NS	NS	NS	NS	NS	0.0103
SUB-OF26-SDB3-COMP (T)			529	2300	N/A	N/A	N/A	N/A	442	N/A	N/A	N/A	N/A	N/A	N/A	0.01740
SUB-OF26-SDB3-COMP (T) + Spike			2970	4794	NS	NS	NS	NS	703	NS	NS	NS	NS	NS	NS	0.0274
Amount Recovered			2441	2494	N/A	N/A	N/A	N/A	261	N/A	N/A	N/A	N/A	N/A	N/A	0.0100
Percent Recovery			104%	107%	N/A	N/A	N/A	N/A	112%	N/A	N/A	N/A	N/A	N/A	N/A	97%

U = not detected at or above detection limit; NC = not certified; NA = not analyzed or available; N/A = not applicable; b = Sample results are less than 5 x the blank; w = spike recovery is out of control due to inappropriate spiking level; t = 0.1 LLS recovery was outside default limits of 50-150%; result reported is an estimate.

PAHS

CLIENT ID	SUB-OF11B-SDB3-FF	SUB-OF11B-SDB3-COMP	SUB-BAY11B-SDB3-PRE	SUB-BAY11B-SDB3-DUR	SUB-BAY11B-SDB3-AFT	SUB-OF23CE-SDB3-FF	SUB-OF23CE-SDB3-COMP	SUB-BAY23CE-SDB3-PRE
Bottle ID	S0887-P	S0890-P	S0887-P	S0889-P	S0883-P	S0883-P	S0882-P	S0877-P
Sample Type	SA	SA	SA	SA	SA	SA	SA	SA
Collection Date	02/03/04	02/03/04	02/03/04	02/03/04	02/04/04	02/03/04	02/03/04	02/02/04
Extraction Date	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04
Analysis Date	02/21/04	02/22/04	02/21/04	02/21/04	02/21/04	02/22/04	02/22/04	02/21/04
Analytical Instrument	MS	MS	MS	MS	MS	MS	MS	MS
% Moisture	NA	NA	NA	NA	NA	NA	NA	NA
% Lipid	NA	NA	NA	NA	NA	NA	NA	NA
Matrix	FRESHWATER	FRESHWATER	SEAWATER	SEAWATER	SEAWATER	FRESHWATER	FRESHWATER	SEAWATER
Sample Size	2.62	2.64	2.64	2.62	2.62	2.64	1.32	2.65
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
Naphthalene	6.28 B	10.72	6.39 B	128.92	5.15 B	10.83	16.08	34.76
C1-Naphthalenes	3.39 B	6.6	3.5 B	140.93	3.28 B	5.87	9.08	26.19
C2-Naphthalenes	3.76 B	6.56	2.75	59.53	2.6 J	6.45	9.45	12.37
C3-Naphthalenes	12.41	17.46	1.29 J	23.5	0.85 J	38.59	47.96	4.36
C4-Naphthalenes	6.3	10.82	1.27 J	9.23	0.51 J	8.38	17.55	2.9 J
2-Methylnaphthalene	3.87 B	7.67	3.41 B	155.82	3.43 B	6.33	9.9	28.03
1-Methylnaphthalene	1.74 J	3.39	2.35 J	81.94	2.01 J	3.36	5.13	16.51
Biphenyl	2.34 J	4.09	0.72 J	8.87	0.78 J	5.31	7.94	2.13 J
2,6-dimethylnaphthalene	1.24 J	2.06 J	0.95 J	30.79	0.83 J	2.4 J	3.61 J	5.61
Acenaphthylene	1.21 J	1.83 J	1.59 J	17.54	0.57 J	2.4 J	2.44 J	4.87
Acenaphthene	0.62 J	1.12 J	2.09 J	3.12 J	1.78 J	1.04 J	1.29 J	1.75 J
2,3,5-trimethylnaphthalene	0.88 J	1.22 J	0.38 J	3.89	0.17 J	1.61 J	2.11 J	1.14 J
Dibenzofuran	1.94 J	3.33	1.23 J	2.03 J	1.02 J	5	5.6 J	0.98 J
Fluorene	2.09 J	2.82 J	1.36 J	5.93	1.09 J	3.29	3.64 J	1.93 J
C1-Fluorenes	3.18	3.55	0.44 J	3.05 J	0.35 J	3.87	4.65 J	1.01 J
C2-Fluorenes	9	20.72	0.74 J	4.9	0.52 U	18.23	27.9	1.79 J
C3-Fluorenes	23.58	29.04	0.52 U	4.55	0.48 J	60.59	65.07	0.51 U
Anthracene	2.79 J	3.11 J	0.65 J	2.32 J	0.48 J	4.77	4.1 J	0.91 J
Phenanthrene	18.33	25	1.76 J	9.92	2.16 J	41.96	41.21	3.22
C1-Phenanthrenes/Anthracenes	13.79	18.47	1.16 J	7.84	0.78 J	33.69	36.06	2.47 J
C2-Phenanthrenes/Anthracenes	22.91	22.91	0.87 J	6.88	0.62 J	37.64	45.4	2.07 J
C3-Phenanthrenes/Anthracenes	18.58	22.66	0.98 J	3.2	0.82 U	28.5	31.93	1.55 J
C4-Phenanthrenes/Anthracenes	9.7	14.9	0.82 U	0.8 J	0.82 U	18.7	18.14	0.81 U
1-Methylphenanthrene	4.27	5.56	0.37 J	1.53 J	0.17 J	9.74	10.54	0.54 J
Dibenzophenone	2.11 J	7.33	0.22 J	0.56 J	0.21 J	5.13	10.7	0.22 J
C1-Dibenzothiophenes	8.82	17.4	0.56 J	0.83 J	0.34 J	15.46	27.9	0.51 J
C2-Dibenzothiophenes	23.98	43.08	0.59 J	1.53 J	0.41 J	39.54	59.87	0.7 J
C3-Dibenzothiophenes	26.73	41.94	0.38 U	1.05 J	0.38 U	40.45	54.5	0.38 U
C4-Dibenzothiophenes	19.14	27.41	0.38 U	0.38 U	0.38 U	31.6	32.6	0.38 U
Fluoranthene	31.6	33.09	4.97	9.03	2.89 J	70.33	61.54	4.4
Pyrene	34.4	36.9	2.58 J	10.89	1.75 J	58.98	51.2	3.5
C1-Fluoranthenes/Pyrenes	12.04	15.75	0.94 J	2.76 J	0.7 J	20.03	19.91	1.23 J
C2-Fluoranthenes/Pyrenes	16.43	24.07	0.68 U	1.42 J	0.68 U	28.84	32.14	0.68 U
C3-Fluoranthenes/Pyrenes	17.04	28.76	0.68 U	0.68 U	0.68 U	30.94	31.38	0.68 U
Benzo(a)anthracene	3.27	6.37	0.43 J	0.65 J	0.24 J	7.94	8.49	0.33 J
Chrysene	21.96	25.1	0.84 J	1.32 J	0.48 J	43.25	38.21	0.69 J
C1-Chysenes	12.38	22.06	0.27 J	0.36 J	0.45 U	22.32	21.03	0.24 J
C2-Chysenes	14.04	28.34	0.45 U	0.45 U	0.45 U	24.31	23.03	0.44 U
C3-Chysenes	11.84	28.93	0.45 U	0.45 U	0.45 U	22.3	23.09	0.44 U
C4-Chysenes	0.45 U	15.53	0.45 U	0.45 U	0.45 U	11.37	11.37	0.44 U
Benzo(b)fluoranthene	10.31	13.42	0.76 J	0.65 J	0.29 J	23.11	22.95	0.55 J
Benzo(k)fluoranthene	9.14	11.28	0.72 J	0.69 J	0.31 J	18.07	18.56	0.51 J
Benzo(e)pyrene	10.06	15.37	0.47 J	0.51 J	0.31 J	20.67	20.32	0.37 J
Benzo(a)pyrene	4.56	8.54	0.55 J	0.55 J	0.32 J	9.15	11.49	0.35 J
Perylene	2.01 J	4.96	0.14 J	0.16 J	1.47 U	2.69 J	3.31 J	1.46 U
Indeno(1,2,3-cd)pyrene	4.67	8.9	0.35 J	0.31 J	0.11 J	11.48	13.29	0.25 J
Dibenz(a,h)anthracene	1.35 J	1.9 J	0.3 J	0.2 J	0.64 U	1.98 J	2.47 J	0.11 J
Benzo(g,h,i)perylene	7.51	16.78	0.29 J	0.58 J	0.11 J	16.48	16.93	0.27 J

PAHs (CONT.)

CLIENT ID	SUB- OF11B-SDB3-FF	SUB- OF11B-SDB3- COMP	SUB- BAY11B-SDB3- PRE	SUB- BAY11B-SDB3- DUR	SUB- BAY11B-SDB3- AFT	SUB- OF23CE-SDB3-FF	SUB- OF23CE-SDB3- COMP	SUB- BAY23CE-SDB3- PRE
Surrogate Recoveries (%)								
Naphthalene-d8	54	61	51	49	55	59	54	48
Phenanthrene-d10	78	85	73	79	74	86	79	70
Chrysene-d12	81	86	84	89	83	87	83	80

PAHs (CONT.)

CLIENT ID	SUB-BAY23CE-SDB3-DUR	SUB-BAY23CE-SDB3-AFT	SUB-OF28-SDB3-FF	SUB-OF28-SDB3-COMP	SUB-BAY26-SDB3-PRE	SUB-BAY26-SDB3-DUR	SUB-BAY26-SDB3-AFT	SUB-BAY26A-SDB3-PRE	SUB-BAY26A-SDB3-DUR	SUB-BAY26A-SDB3-AFT
Bottle ID	S0885-P	S0885-P	S0888-P	S0881-P	S0876-P	S0880-P	S0884-P	S0876-P	S0882-P	S0886-P
Sample Type	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA
Collection Date	02/03/04	02/04/04	02/03/04	02/03/04	02/02/04	02/03/04	02/04/04	02/02/04	02/03/04	02/04/04
Extraction Date	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04	02/06/04
Analysis Date	02/21/04	02/21/04	02/22/04	02/22/04	02/21/04	02/21/04	02/21/04	02/21/04	02/21/04	02/21/04
Analytical Instrument	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS
% Moisture	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
% Lipid	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Matrix	SEAWATER	SEAWATER	FRESHWATER	FRESHWATER	SEAWATER	SEAWATER	SEAWATER	SEAWATER	SEAWATER	SEAWATER
Sample Size	2.64	2.64	2.64	2.64	2.63	2.64	2.64	2.64	2.64	2.64
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
Naphthalene	3.66 B	7.52 B	11.9	9.37 B	14.18	3.33 B	9.76 B	2.03 J	7.96 B	6.53 B
C1-Naphthalenes	1.7 J	4.39 B	5.68	5.9	12.58	1.83 J	6.13	0.94 J	4.95 B	3.78 B
C2-Naphthalenes	2.13 J	2.63 J	5.86	6.87	8.72	1.95 J	2.97 J	1.08 J	2.53 J	1.89 J
C3-Naphthalenes	0.89 J	0.81 J	14.52	42.09	3.52	1.11 J	0.87 J	0.67 J	0.99 J	0.72 J
C4-Naphthalenes	1.01 J	0.89 J	6.49	69.49	2.44 J	1.29 J	0.85 J	0.5 J	0.98 J	0.78 J
2-Methylnaphthalene	1.62 J	4.57 J	6.11	5.57	12.76	1.7 J	6.55	0.96 J	5.12	4.01 B
1-Methylnaphthalene	1.09 J	2.72 J	3.42	3.66	8.49	1.22 J	3.72	0.62 J	3.1 J	2.28 J
Biphenyl	0.51 J	0.6 J	3.43	3.44	1.38 J	0.52 J	0.76 J	0.43 J	0.57 J	0.84 J
2,6-dimethylnaphthalene	0.58 J	0.78 J	1.95 J	1.87 J	3.74	0.59 J	1.06 J	0.27 J	1.02 J	0.59 J
Acenaphthylene	0.77 J	0.63 J	3.11 J	2.9 J	3.34	1.01 J	0.84 J	0.38 J	1.04 J	0.59 J
Acenaphthene	0.87 J	1.15 J	1.8 J	2.06 J	1.35 J	1.1 J	0.99 J	0.35 J	0.2 J	0.53 J
2,3,5-trimethylnaphthalene	0.19 J	0.18 J	1.02 J	0.99 J	0.81 J	0.32 J	0.21 J	0.15 J	0.21 J	0.16 J
Benzo(a)fluoranthene	0.53 J	0.59 J	5.6	3.94	0.85 J	0.8 J	0.68 J	0.48 J	0.28 J	0.4 J
Fluorene	0.57 J	0.89 J	3.28	3.66	1.38 J	0.83 J	0.89 J	0.33 J	0.46 J	0.53 J
C1-Fluorenes	0.32 J	0.31 J	2.52 J	4.46	0.81 J	0.39 J	0.52 J	0.34 J	0.23 J	0.52 J
C2-Fluorenes	0.72 J	0.52 J	19.36	39.6	1.61 J	1.04 J	0.52 J	0.52 J	0.52 J	0.52 J
C3-Fluorenes	0.52 J	0.52 J	63.92	69.71	0.52 J	0.52 J	0.52 J	0.52 J	0.52 J	0.52 J
Anthracene	0.54 J	0.38 J	4.62	1.73 J	0.88 J	0.59 J	0.36 J	0.25 J	0.14 J	0.19 J
Phenanthrene	0.74 J	1.71 J	50.61	31.03	1.95 J	1.07 J	2.11 J	0.47 J	0.92 J	1.15 J
C1-Phenanthrenes/Anthracenes	0.87 J	0.77 J	23.82	21.28	2.01 J	1.08 J	0.82 J	0.68 J	0.75 J	0.64 J
C2-Phenanthrenes/Anthracenes	0.78 J	0.7 J	29.52	25.69	1.7 J	1.11 J	0.82 J	0.8 J	0.7 J	0.68 J
C3-Phenanthrenes/Anthracenes	0.82 J	0.82 J	37.69	23.51	1.43 J	1.11 J	0.82 J	1.02 J	0.82 J	0.82 J
C4-Phenanthrenes/Anthracenes	0.82 J	0.82 J	32.56	13.78	0.82 J	0.82 J	0.82 J	0.82 J	0.82 J	0.82 J
1-Methylphenanthrene	0.2 J	0.16 J	6.27	6.29	0.47 J	0.24 J	0.27 J	0.27 J	0.22 J	0.21 J
Dibenzothiophene	0.12 J	0.17 J	4.15	10.34	0.19 J	0.2 J	0.19 J	0.07 J	0.08 J	0.09 J
C1-Dibenzothiophenes	0.25 J	0.36 J	9.78	24.24	0.47 J	0.55 J	0.34 J	0.48 J	0.28 J	0.12 J
C2-Dibenzothiophenes	0.48 J	0.51 J	29.55	50.5	0.76 J	0.79 J	0.48 J	0.55 J	0.44 J	0.38 J
C3-Dibenzothiophenes	0.36 J	0.36 J	34.25	46.69	0.38 J	0.63 J	0.38 J	0.38 J	0.38 J	0.38 J
C4-Dibenzothiophenes	0.38 J	0.38 J	28.76	37.25	0.38 J	0.38 J	0.38 J	0.38 J	0.38 J	0.38 J
Fluoranthene	2.76 J	2.43 J	71.87	24.57	4.48	4.72	2.61 J	2.4 J	0.68 J	1.15 J
Pyrene	1.5 J	1.25 J	53.39	20.45	3.3	2.64 J	1.42 J	0.96 J	0.59 J	0.64 J
C1-Fluoranthenes/Pyrenes	0.78 J	0.82 J	22.34	12.94	1.09 J	0.9 J	0.65 J	0.66 J	0.32 J	0.36 J
C2-Fluoranthenes/Pyrenes	0.68 J	0.68 J	41.41	14.82	0.68 J	0.68 J	0.68 J	0.68 J	0.68 J	0.68 J
C3-Fluoranthenes/Pyrenes	0.68 J	0.68 J	32.69	15.5	0.68 J	0.68 J	0.68 J	0.68 J	0.68 J	0.68 J
Benzo(a)anthracene	0.15 J	0.23 J	7.12	2.7 J	0.88 J	0.68 J	0.68 J	0.68 J	0.68 J	0.68 J
Chrysene	0.46 J	0.4 J	39.53	10.06	0.84 J	0.21 J	0.15 J	0.18 J	0.07 J	0.07 J
C1-Chrysenes	0.45 J	0.45 J	16.53	6.9	0.65 J	0.97 J	0.51 J	0.36 J	0.23 J	0.31 J
C2-Chrysenes	0.45 J	0.45 J	21.55	8.26	0.23 J	0.22 J	0.45 J	0.45 J	0.45 J	0.45 J
C3-Chrysenes	0.45 J	0.45 J	22.7	7.7	0.45 J	0.45 J	0.45 J	0.45 J	0.45 J	0.45 J
C4-Chrysenes	0.45 J	0.45 J	8.39	0.45 U	0.45 J	0.45 J	0.45 J	0.45 J	0.45 J	0.45 J
Benzo(b)fluoranthene	0.26 J	0.19 J	17.6	7.07	0.45 J	0.34 J	0.2 J	0.3 J	0.88 J	0.88 J
Benzo(k)fluoranthene	0.39 J	0.28 J	16.34	5.82 J	0.35 J	0.3 J	0.27 J	0.21 J	0.99 J	0.99 J
Benzo(e)pyrene	0.18 J	0.17 J	15.95	6.7	0.38 J	0.31 J	0.2 J	0.22 J	0.39 J	0.39 J
Benzo(a)pyrene	0.23 J	0.2 J	7.92	4.21	0.37 J	0.22 J	0.17 J	0.26 J	0.76 J	0.76 J
Perylene	1.46 J	1.46 J	2.18 J	0.79 J	1.47 U	1.46 U	1.46 U	1.46 U	1.46 U	1.46 U
Indeno(1,2,3-cd)pyrene	0.1 J	0.08 J	8.15	4.54	0.24 J	0.13 J	0.06 J	0.12 J	0.75 U	0.05 J
Dibenz(a,h)anthracene	0.63 J	0.63 J	1.62 J	0.82 J	0.19 J	0.11 J	0.63 J	0.08 J	0.63 J	0.63 J
Benzo(g,h,i)perylene	0.13 J	0.06 J	15.94	5.63	0.33 J	0.19 J	0.09 J	0.12 J	0.75 U	0.05 J

PAHs (CONT.)

CLIENT ID	SUB- BAY23CE-SDB3- DUR	SUB- BAY23CE-SDB3- AFT	SUB- OF 76-SDB3-FF	SUB- OF 76-SDB3- COMP	SUB- BAY76-SDB3-PRE	SUB- BAY76-SDB3- DUR	SUB- BAY76-SDB3-AFT	SUB- BAY26A-SDB3- PRE	SUB- BAY26A-SDB3- DUR	SUB- BAY26A-SDB3- AFT
Surrogate Recoveries (%)										
Naphthalene-d8	61	68	53	68	50	51	51	57	56	64
Phenanthrene-d10	81	88	76	88	73	80	76	77	75	79
Chrysene-d12	83	89	78	88	83	89	84	85	85	86

PAHs QA/QC

PROJECT: Task Order TO0015 –Contaminant Analysis of Stormwater and San Diego Bay Seawater
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected over three days 2/2/04 – 2/4/04. Samples were shipped in three containers to Battelle Duxbury via Federal Express. The samples were received on 2/5/04. The cooler temperatures were recorded at 2.1°C, 2.6°C, and 3.4°C upon arrival. No custody issues were noted. Samples were stored at 4° C until sample preparation could begin. Samples were extracted as one analytical batch, 04-0039.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PAH	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.50 – 1.93

METHOD: Water samples were extracted for PAH following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
04-0039	2/6/04	2/20/04 – 2/22/04

SURROGATES: Three surrogate compounds were added prior to extraction, including Naphthalene-d8, Phenanthrene-d10, and Chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0039 - Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (40 - 120% recovery).

Comments - None.

(BD935PB), however all compounds were below the laboratory control limit (< 5 times MDL) and below the reporting limit (RL). These data points were qualified with a "J" in the procedural blank. Any authentic field sample concentrations that were less than five times the concentration detected in the blank were qualified with a "B". No further corrective action is necessary.

**LABORATORY
CONTROL
SAMPLE:**

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PAH were calculated to measure data quality in terms of accuracy.

04-0039 - All target analytes were recovered within the laboratory control limits.

Comments - None.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair was prepared with each analytical batch. The percent recoveries of target PAH and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

04-0039 - All target analytes were recovered within the laboratory control limits. All RPDs were within the laboratory control limits.

Comments - None.

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy.

04-0039 - No exceedences noted.

Comments - None.

SURROGATES:

Three surrogate compounds were added prior to extraction, including Naphthalene-d8, Phenanthrene-d10, and Chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0039 - Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (40 - 120% recovery).

Comments - None.

PAHs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE		MATRIX SPIKE- OF23CE-SDB3-COMP		MATRIX SPIKE DUPLICATE- OF23CE-SDB3-COMP			PROCEDURAL BLANK	PROCEDURAL BLANK- DUXBURY BAY SEAWATER BACKGROUND	
Battelle ID	BD936LCS-P		S0892MS-P		S0892MSD-P			BD935PB-P	BD936PB-P	
Sample Type	LCS		MS		MSD			PB	PB	
Collection Date	02/06/04		2/3/2004		2/3/2004			02/06/04	2/6/2004	
Extraction Date	02/06/04		2/6/2004		2/6/2004			02/06/04	2/6/2004	
Analysis Date	02/20/04		2/22/2004		2/22/2004			02/20/04	2/20/2004	
Analytical Instrument	MS		MS		MS			MS	MS	
% Moisture	NA		NA		NA			NA	NA	
% Lipid	NA		NA		NA			NA	NA	
Matrix	LIQUID		LIQUID		LIQUID			LIQUID	LIQUID	
Sample Size	2.00		0.65		0.65			2.00	2	
Size Unit-Basis	L LIQUID		L LIQUID		L LIQUID			L LIQUID	L LIQUID	
Units	NG/L LIQUID	% Rec	NG/L LIQUID	% Rec	NG/L LIQUID	% Rec	RPD (%)	NG/L LIQUID	NG/L LIQUID	
Naphthalene	864.16	69	1811.73	47	1735.32	45	4.3	2.11	J	8.27
C1-Naphthalenes	0.66	U	2.04	U	2.04	U		1.1	J	3.18
C2-Naphthalenes	0.66	U	2.04	U	2.04	U		1.18	J	2.23
C3-Naphthalenes	0.66	U	2.04	U	2.04	U		0.54	J	1.3
C4-Naphthalenes	0.66	U	2.04	U	2.04	U		0.66	U	0.66
2-Methylnaphthalene	988.62	79	2195.25	57	2115.43	55	3.6	0.85	J	3.17
1-Methylnaphthalene	861.6	69	1941.62	50	1891.28	49	2.0	0.56	J	2.19
Biphenyl	929.05	74	2239.03	58	2272.63	59	1.7	0.45	J	1.28
2,6-dimethylnaphthalene	927.6	74	2260.22	59	2276.76	59	0.0	0.36	J	0.92
Acenaphthylene	1038.32	83	2685.66	70	2749.98	71	1.4	0.7	U	0.61
Acenaphthene	945.09	76	2409.61	63	2473.72	64	1.6	0.75	U	1.25
2,3,5-trimethylnaphthalene	985.41	79	2743.51	71	2849.58	74	4.1	0.17	J	0.44
Dibenzofuran	0.3	U	13.18		0.93	U		0.3	U	1.39
Fluorene	945.1	76	2630.92	68	2733.85	71	4.3	0.16	J	1.37
C1-Fluorenes	0.68	U	2.09	U	2.09	U		0.68	U	0.43
C2-Fluorenes	0.68	U	2.09	U	2.09	U		0.68	U	0.68
C3-Fluorenes	0.68	U	2.09	U	2.09	U		0.68	U	0.68
Anthracene	1131.51	90	3117.3	81	3211.72	83	2.4	0.51	U	0.25
Phenanthrene	1006.84	81	2923.26	75	3080.18	79	5.2	0.37	J	3.44
C1-Phenanthrenes/Anthracenes	1.08	U	3.32	U	3.32	U		0.52	J	1.19
C2-Phenanthrenes/Anthracenes	1.08	U	3.32	U	3.32	U		0.48	J	1.02
C3-Phenanthrenes/Anthracenes	1.08	U	3.32	U	3.32	U		1.08	U	1.38
C4-Phenanthrenes/Anthracenes	1.08	U	3.32	U	3.32	U		1.08	U	1.08
1-Methylphenanthrene	1168.53	93	3368.48	87	3524.52	91	4.5	0.18	J	0.4
Dibenzothiophene	12.33		52.04		54.37			0.08	J	0.25
C1-Dibenzothiophenes	0.5	U	1.55	U	1.55	U		0.5	U	0.36
C2-Dibenzothiophenes	0.5	U	1.55	U	1.55	U		0.54	J	0.6
C3-Dibenzothiophenes	0.5	U	1.55	U	1.55	U		0.5	U	0.5
C4-Dibenzothiophenes	0.5	U	1.55	U	1.55	U		0.5	U	0.5
Fluoranthene	1177.07	94	3370.11	86	3523.89	90	4.5	0.37	J	2.37
Pyrene	1183.96	95	3337.92	85	3510.25	90	5.7	0.39	J	1.34
C1-Fluoranthenes/Pyrenes	0.9	U	2.76	U	2.76	U		0.32	J	0.62
C2-Fluoranthenes/Pyrenes	0.9	U	2.76	U	2.76	U		0.9	U	0.52
C3-Fluoranthenes/Pyrenes	0.9	U	2.76	U	2.76	U		0.9	U	0.9
Benzo(a)anthracene	1240.16	99	3478.77	90	3635.88	94	4.3	0.16	J	0.46
Chrysene	1127.61	90	3141.02	81	3288.97	84	3.6	0.39	J	1.07
C1-Chrysenes	0.59	U	1.81	U	1.81	U		0.2	J	0.27
C2-Chrysenes	0.59	U	1.81	U	1.81	U		0.59	U	0.59
C3-Chrysenes	0.59	U	1.81	U	1.81	U		0.59	U	0.59
C4-Chrysenes	0.59	U	1.81	U	1.81	U		0.59	U	0.59
Benzo(b)fluoranthene	1192.45	95	3395.92	88	3518.19	91	3.4	0.15	J	0.63
Benzo(j,k)fluoranthene	1320.96	106	3792.52	98	3910.57	101	3.0	0.2	J	0.67
Benzo(e)pyrene	1187.42	96	3394.05	89	3538.22	93	4.4	0.15	J	0.53
Benzo(a)pyrene	1213.38	97	3462.33	90	3604.14	93	3.3	0.14	J	0.59
Perylene	1187.17	95	3514.76	91	3651.85	95	4.3	1.93	U	0.17
Indeno(1,2,3-cd)pyrene	1136.94	91	3400.22	88	3531.49	91	3.4	0.25	J	0.61
Dibenz(a,h)anthracene	1169.01	93	3508.51	91	3633.68	94	3.2	0.56	J	0.55
Benzo(g,h,i)perylene	850.35	68	2454.91	63	2554.38	66	4.7	0.23	J	0.49

CLIENT ID	LABORATORY CONTROL SAMPLE		MATRIX SPIKE- OF23CE-SDB3-COMP		MATRIX SPIKE DUPLICATE- OF23CE-SDB3-COMP			PROCEDURAL BLANK	PROCEDURAL BLANK- DUXBURY BAY SEAWATER BACKGROUND	
Surrogate Recoveries (%)										
Naphthalene-d8	76		53		52			71		67
Phenanthrene-d10	87		80		85			80		80
Chrysene-d12	94		83		86			84		88

PCBs

CLIENT ID	SUB- OF11B-SDB3-COMP	SUB- OF23CE-SDB3- COMP	SUB- OF26-SDB3-COMP
Battelle ID	S0890-P	S0892-P	S0891-P
Sample Type	SA	SA	SA
Collection Date	02/03/04	02/03/04	02/03/04
Extraction Date	02/06/04	02/06/04	02/06/04
Analysis Date	02/17/04	02/17/04	02/17/04
Analytical Instrument	MS	MS	MS
% Moisture	NA	NA	NA
% Lipid	NA	NA	NA
Matrix	FRESHWATER	FRESHWATER	FRESHWATER
Sample Size	2.64	1.32	2.64
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
C12(8)	0.06 U	0.13 U	0.06 U
C13(18)	0.06 U	0.12 U	0.06 U
C13(28)	0.06 U	0.12 U	0.06 U
C14(44)	0.11 U	0.23 U	0.11 U
C14(49)	0.11 U	0.23 U	0.11 U
C14(52)	0.11 U	0.23 U	0.11 U
C14(66)	0.11 U	0.23 U	0.11 U
C14(77)	0.14 U	0.28 U	0.14 U
C15(87)	0.11 U	0.23 U	0.11 U
C15(101)	U	0.23 U	0.11 U
C15(105)	0.11 U	0.21 U	0.11 U
C15(114)	0.23 U	0.46 U	0.23 U
C15(118)	0.07 U	0.14 U	0.07 U
C15(123)	0.08 U	0.16 U	0.08 U
C15(126)	0.12 U	0.24 U	0.12 U
C16(128)	0.15 U	0.31 U	0.15 U
C16(138)	0.15 U	0.31 U	0.15 U
C16(153)	0.15 U	0.31 U	0.15 U
C16(156)	0.08 U	0.15 U	0.08 U
C16(157)	0.14 U	0.29 U	0.14 U
C16(167)	0.27 U	0.54 U	0.27 U
C16(169)	0.11 U	0.22 U	0.11 U
C17(170)	0.19 U	0.37 U	0.19 U
C17(180)	0.11 U	0.21 U	0.11 U
C17(183)	0.11 U	0.23 U	0.11 U
C17(184)	0.11 U	0.23 U	0.11 U
C17(187)	0.11 U	0.23 U	0.11 U
C17(189)	0.08 U	0.16 U	0.08 U
C18(195)	0.21 U	0.42 U	0.21 U
C19(206)	0.34 U	0.67 U	0.34 U
C110(209)	0.4 U	0.81 U	0.4 U
Surrogate Recoveries (%)			
C12(14)	86	76	89
C13(34)	83	76	87
C15(104)	83	77	86
C15(112)	86	82	86

PCBs QA/QC

PROJECT: Task Order TO0015 –Contaminant Analysis of Stormwater and San Diego Bay Seawater

PARAMETER: PCB

LABORATORY: Battelle, Duxbury, MA

MATRIX: Water

SAMPLE CUSTODY: Water samples were collected over three days 2/2/04 – 2/4/04. Samples were shipped in three containers to Battelle Duxbury via Federal Express. The samples were received on 2/5/04. Upon arrival, the cooler temperatures were recorded at 2.1°C, 2.6°C, and 3.4°C. No custody issues were noted. Samples were stored in the upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 04-0039.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PCB	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.08 – 0.53

METHOD: Water samples were extracted for PCB following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS). The method is based on key components of the PCB congener analysis approach described in EPA Method 1668A. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
04-0039	2/6/04	2/17/04

BLANK:

Two blank (PB) samples were prepared with the analytical batch. One procedural blank BD935PB was analyzed to ensure the sample extraction and analysis methods were free of contamination. The other blank, BD938PB, was analyzed to give a background value for "clean" seawater. All analytical data has been qualified according to the concentrations detected in BD935PB.

04-0039 - No exceedences noted.

Comments - No target analytes were detected in the procedural blank.

**LABORATORY
CONTROL
SAMPLE:**

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB were calculated to measure data quality in terms of accuracy.

04-0039 - One exceedence noted.

Comments - All target analytes were recovered within the laboratory control limits specified by the client (40-120%), except for PCB 180. PCB 180 was over-recovered at 130%. The chromatograms and calculations were reviewed. The analyst notes an interfering compound, possibly a phthalate, co-eluting with this peak. A spectral comparison versus a standard has been included in the data a package. The exceedence was qualified with an "N". No further corrective action was taken.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair was prepared with each analytical batch. The percent recoveries of target PCB and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

04-0039 - Two percent recovery exceedences noted.
No RPD exceedences noted.

Comments - All RPDs were within the laboratory control limits (<30% RPD). All target analytes were recovered within the laboratory control limits specified by the client (40-120%), except for PCB 170. PCB 170 was over-recovered in both the MS and MSD samples at 131% and 126%, respectively. Calculations and chromatograms were reviewed. No discrepancies were found. Accuracy for this compound was demonstrated in both the LCS and SRM samples. The exceedences

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy.

04-0039 - No exceedence noted.

Comments - None.

SURROGATES:

Four surrogate compounds were added prior to extraction, including PCB 14, PCB 34, PCB 104, and PCB 112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0039 - Percent recoveries for all surrogate compounds were within the laboratory control limits (40 - 120% recovery).

Comments - None.

PCBs QA/QC (CONT.)

[illegible]

PESTICIDES

CLIENT ID	SUB- OF11B-SDB3- COMP	SUB- OF26-SDB3- COMP	SUB- OF23CE-SDB3- COMP
Battelle ID	S0890-P	S0891-P	S0892-P
Sample Type	SA	SA	SA
Collection Date	02/03/04	02/03/04	02/03/04
Extraction Date	02/06/04	02/06/04	02/06/04
Analysis Date	02/27/04	02/27/04	02/27/04
Analytical Instrument	ECD	ECD	ECD
% Moisture	NA	NA	NA
% Lipid	NA	NA	NA
Matrix	FRESHWATER	FRESHWATER	FRESHWATER
Sample Size	2.64	2.64	1.32
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
2,4'-DDD	0.82 U	0.62 U	1.23 U
2,4'-DDE	0.52 U	0.52 U	1.04 U
2,4'-DDT	0.37 U	0.37 U	0.73 U
4,4'-DDD	0.72 U	0.72 U	1.44 U
4,4'-DDE	0.52 U	0.52 U	1.04 U
4,4'-DDT	0.45 U	0.45 U	0.89 U
aldrin	0.3 U	0.3 U	0.6 U
a-chlordane	0.29 U	0.29 U	0.57 U
g-chlordane	0.31 U	0.31 U	0.61 U
a-BHC	0.26 U	0.26 U	0.52 U
b-BHC	0.36 U	0.36 U	0.72 U
d-BHC	0.29 U	0.29 U	0.59 U
Lindane	0.37 U	0.37 U	0.75 U
cis-nonachlor	0.49 U	0.49 U	0.98 U
trans-nonachlor	0.31 U	0.31 U	0.61 U
Chlorpyrifos	0.39 U	0.39 U	0.77 U
oxychlordane	0.3 U	0.3 U	0.59 U
dieldrin	0.58 U	0.58 U	1.16 U
endosulfan I	0.21 U	0.21 U	0.42 U
endosulfan II	0.52 U	0.52 U	1.05 U
endosulfan sulfate	0.49 U	0.49 U	0.98 U
endrin	0.57 U	0.57 U	1.14 U
endrin aldehyde	0.64 U	0.64 U	1.29 U
endrin ketone	0.67 U	0.67 U	1.34 U
heptachlor	0.44 U	0.44 U	0.89 U
heptachlor epoxide	1.19 U	1.19 U	2.39 U
Hexachlorobenzene	0.63 U	0.63 U	1.25 U
methoxychlor	0.74 U	0.74 U	1.48 U
Mirex	0.47 U	0.47 U	0.94 U
Surrogate Recoveries (%)			
C12(14)	70	64	77
C13(34)	96	87	79
C15(104)	90	76	69
C15(112)	79	90	71

PESTICIDES QA/QC

PROJECT: Task Order TO0015 –Contaminant Analysis of Stormwater and San Diego Bay Seawater

PARAMETER: Pesticides

LABORATORY: Battelle, Duxbury, MA

MATRIX: Water

SAMPLE CUSTODY: Water samples were collected over three days 2/2/04 – 2/4/04. Samples were shipped in three containers to Battelle Duxbury via Federal Express. The samples were received on 2/5/04. Upon arrival, the cooler temperatures were recorded at 2.1°C, 2.6°C, and 3.4°C. No custody issues were noted. Samples were stored in the upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 04-0039. Selected samples were chosen for pesticide analysis.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PESTICIDE	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery	≤30% PD on average	≤30% RPD	MDL: -0.34 – 1.58
				(target spike must be >5 x native conc.)	(for analytes ≥5x MDL)	(calculated between the MS and MSD samples)	

METHOD: Water samples were extracted for pesticide following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts intended for pesticide analysis were solvent exchanged into hexane and analyzed using a gas chromatography/electron capture detector (GC/ECD). Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
04-0039	2/6/04	2/27/04

BLANK: Two blank (PB) samples were prepared with the analytical batch. One procedural blank BD935PB was analyzed to ensure the sample extraction and analysis methods were free of contamination. The other blank, BD938PB, was analyzed to give a background value for "clean" seawater. All analytical data has been qualified according to the concentrations detected in BD935PB.

04-0039 – No exceedences noted.

Comments – No target analytes were detected in sample BD935PB. Lindane, a-BHC, and Heptachlor were detected in sample BD938PB, however these analytes were both below the RL. These detections were qualified with a "J". No further corrective action was taken.

LABORATORY CONTROL SAMPLE: A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

04-0039 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE: A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target pesticides and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

04-0039 – Six percent recovery exceedences noted.
No RPD exceedences noted.

Comments – Chlorpyrifos, endrin, and heptachlor were over-recovered in both samples S0892MS and S0892MSD (background sample OF23CE-SDB3-Comp). In

SRM: A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy.

04-0039 – No exceedence noted.

Comments – None.

SURROGATES: Four surrogate compounds were added prior to extraction, including PCB 14, PCB 34, PCB 104, and PCB 112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

04-0039 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

[illegible]

TSS

SAMPLE LABEL	TSS (mg/L)
SUB-OF11B-SDB3-FF	37.15
SUB-OF11B-SDB3-COMP	96.58
SUB-BAY11B-SDB3-DUR	2.33
SUB-BAY11B-SDB3-AFT	3.52
SUB-OF23CE-SDB3-FF	45.10
SUB-OF23CE-SDB3-COMP	54.67
SUB-BAY23CE-SDB3-PRE	3.43
SUB-BAY23CE-SDB3-DUR	3.19
SUB-BAY23CE-SDB3-AFT	2.37
SUB-OF26-SDB3-FF	38.79
SUB-OF26-SDB3-COMP	21.18
SUB-BAY26-SDB3-PRE	2.18
SUB-BAY26-SDB3-DUR	2.46
SUB-BAY26-SDB3-AFT	2.42
SUB-BAY26A-SDB3-PRE	2.99
SUB-BAY26A-SDB3-DUR	2.05
SUB-BAY26A-SDB3-AFT	3.67

DOC

CLIENT SAMPLE ID	MEAN DOC (mg/L)
SUB-OF11B-SDB3-FF	11.40
SUB-OF11B-SDB3-COMP	11.32
SUB-BAY11B-SDB3-PRE	0.72
SUB-BAY11B-SDB3-DUR	0.47
SUB-BAY11B-SDB3-AFT	0.80
SUB-OF23CE-SDB3-FF	8.97
SUB-OF23CE-SDB3-COMP	13.00
SUB-BAY23CE-SDB3-PRE	0.71
SUB-BAY23CE-SDB3-DUR	0.57
SUB-BAY23CE-SDB3-AFT	0.61
SUB-OF26-SDB3-FF	4.47
SUB-OF26-SDB3-COMP	12.43
SUB-BAY26-SDB3-PRE	0.83
SUB-BAY26-SDB3-DUR	0.70
SUB-BAY26-SDB3-AFT	0.58
SUB-BAY26A-SDB3-PRE	0.52
SUB-BAY26A-SDB3-DUR	0.59
SUB-BAY26A-SDB3-AFT	0.49

SDB4- 10/17/2004

METALS

SAMPLE ID	DISSOLVED COPPER (µg/L)	TOTAL COPPER (µg/L)	DISSOLVED ZINC (µg/L)	TOTAL ZINC (µg/L)
SUB-OF11B-SDB4-FF	93	149	1255	1291
SUB-BAY11B-SDB4-DUR	5	10	53	71

TSS

SAMPLE LABEL	TSS (mg/L)
SUB-OF11B-SDB4-FF	152.94
SUB-BAY11B-SDB4-DUR	8.60

Appendix D3

NAB

SDB4- 10/17/2004

SDB6- 2/10/2005

SDB7- 4/27/2005

SDB4- 10/17/2004

METALS

SAMPLE ID	DISSOLVED Cu (µg/L)	TOTAL Cu (µg/L)
NAB-OF9-SDB4-FF	172	668
NAB-BAY9-SDB4-DUR	17	23

SAMPLE ID	DISSOLVED Zn (µg/L)	TOTAL Zn (µg/L)
NAB-OF9-SDB4-FF	7134	8051
NAB-BAY9-SDB4-DUR	176	256

TSS

SAMPLE LABEL	TSS (mg/L)
NAB-OF9-SDB4-FF	130.40
NAB-BAY9-SDB4-DUR	12.12

SDB6- 2/10/2005

METALS

MSL Code	Rep	Sponsor I.D.	Al (µg/L) ICP-OES	Fe (µg/L) ICP-OES	Cr (µg/L) ICP-OES	Mn (µg/L) ICP-OES	Ni (µg/L) ICP-MS	Cu (µg/L) ICP-MS	Zn (µg/L) ICP-OES
2157*13		NAB-SDB6-OF9-COMP (T)	192	847	2.11	71.3	4.37	59.5	522
2157*10		NAB-SDB6-OF9-COMP (D)	13.9	31.5	1.18	59.6	3.87	40.0	356
2157*14		NAB-SDB6-OF18-COMP (T)	507	832	2.30	26.1	2.45	44.4	214
2157*11		NAB-SDB6-OF18-COMP (D)	15.0	29.5	0.574	8.58	1.27	26.2	101

MSL Code	Rep	Sponsor I.D.	As (µg/L) ICP-MS	Se (µg/L) ICP-MS	Ag (µg/L) ICP-MS	Cd (µg/L) ICP-MS	Sn (µg/L) ICP-MS	Pb (µg/L) ICP-MS	Hg (µg/L) CVAF
2157*13		NAB-SDB6-OF9-COMP (T)	4.93	14.1	0.040	0.551	0.50	3.21	0.00838
2157*10		NAB-SDB6-OF9-COMP (D)	4.80	14.1	0.040	0.414	0.50	0.132	0.00309
2157*14		NAB-SDB6-OF18-COMP (T)	2.28	1.47	0.0411	0.794	0.550	5.74	0.00711
2157*11		NAB-SDB6-OF18-COMP (D)	1.77	1.47	0.040	0.322	0.50	0.291	0.00410

SAMPLE ID	DISSOLVED COPPER (µg/L)	TOTAL COPPER (µg/L)	DISSOLVED ZINC (µg/L)	TOTAL ZINC (µg/L)
NAB-OF9-SDB6-FF	37.5	39.8	197	315
NAB-BAY9-SDB6-PRE	2.4	3.6	6.2	8.5
NAB-BAY9-SDB6-DUR	3.5	6.2	32	44
NAB-OF18-SDB6-FF	38.2	43.7	134	137
NAB-BAY18-SDB6-PRE	2.0	3.2	8.3	8.7
NAB-BAY18-SDB6-DUR	7.9	14.9	55	85

METALS QA/QC

PROGRAM: SPAWAR, Task 19
PARAMETER: Metals
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Stormwater

QA/QC DATA QUALITY OBJECTIVES

	Reference Method	Range of Recovery	SRM Accuracy	Relative Precision	Target Detection Limit (µg/L)
Aluminum	ICP/OES	50-150%	±20%	±50%	50.0
Iron	ICP/OES	50-150%	±20%	±50%	10.0
Manganese	ICP/OES	50-150%	±20%	±30%	0.5
Chromium	ICP/MS	50-150%	±20%	±30%	1.0
Nickel	ICP/MS	50-150%	±20%	±30%	0.05
Copper	ICP/MS	50-150%	±20%	±30%	0.05
Zinc	ICP/MS	50-150%	±20%	±30%	0.5
Arsenic	FIAS	50-150%	±20%	±30%	0.5
Selenium	FIAS	50-150%	±20%	±30%	0.2
Silver	GFAA	50-150%	±20%	±30%	0.5
Cadmium	ICP/MS	50-150%	±20%	±30%	0.05
Tin	ICP/MS	50-150%	±20%	±30%	0.5
Lead	ICP/MS	50-150%	±20%	±30%	0.05
Mercury	CVAF	50-150%	±25%	±30%	0.01

METHOD

Three (3) samples were analyzed for fourteen (14) metals: nickel (Ni), copper, (Cu), arsenic (As), selenium (Se), silver (Ag), cadmium (Cd), tin (Sn) and lead (Pb) by inductively coupled plasma mass spectroscopy (ICP/MS) following EPA Method 1631m, aluminum (Al), iron (Fe), chromium (Cr), manganese (Mn), and zinc (Zn) by inductively coupled plasma optic emission spectroscopy following EPA Method 200.7 and mercury (Hg) by cold vapor atomic fluorescence (CVAF) following EPA Method 1631e.

Samples were preserved with nitric acid prior to arrival at MSL. Samples analyzed for Hg by CVAF were pre-treated with bromine chloride and stannous chloride to oxidize and convert all Hg compounds to volatile Hg, which is subsequently trapped onto a gold-coated sand trap.

HOLDING TIMES

Three (3) samples were received on 2/11/2005 and were logged into Battelle's sample tracking system. The samples were analyzed within the six month holding time for metals and 90 days for Hg. The following list summarizes all analysis dates:

Task	Date Performed
Hg	2/23/05
ICP-MS	2/22/05
ICP-OES	3/1 & 4/05

DETECTION LIMITS

The target detection limit was met for all metals, except Ni, Cu, Se and Cd. The MDL for seawater analysis by dilution is somewhat higher than

our typical MDL's for direct analysis. Sample concentrations were substantially greater than the MDL, except Se. All Se results were less than our MDL for this method. The method detection limit was met for all metals. An MDL is determined by multiplying the standard deviation of the results of a minimum of 7 replicate low level spikes by the Student's t value at the 99th percentile.

METHOD BLANKS

One method blank was analyzed with this batch of samples. Results were less than 3 times the MDL for all metals, except the TRM blank for Zn. The TRM field sample was greater than 10 x the blank concentration and therefore was not impacted by the blank contamination.

BLANK SPIKES

One sample of reagent water was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

MATRIX SPIKES

One sample was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

REPLICATES

One sample was analyzed in duplicate. All results were within the QC limits of $\pm 30\%$ ($\pm 50\%$ for Al and Fe).

SRM

One matrix-appropriate standard reference material (SRM) was analyzed for each method; 1641d, river water, and 1640, natural water, obtained from the National Institute of Science and Technology.

SRM 1640 has 22 certified and reference metals. Recovery for all metals reported were within the control limit of $\pm 20\%$ of the certified or reference value. Tin and Hg are not certified in 1640. SRM 1641d is certified for Hg. Recovery for Hg was within the control limit of $\pm 25\%$ of the certified value.

REFERENCES

EPA. 1991. Methods for the Determination of Metals in Environmental Samples. EPA-600/4-91-010. Environmental Services Division, Monitoring Management Branch.

METALS QA/QC (CONT.)

MSL Code	Rep	Sponsor ID	Al (µg/L)	Fe (µg/L)	Cr (µg/L)	Mn (µg/L)	Ni (µg/L)	Cu (µg/L)	Zn (µg/L)	As (µg/L)	Sr (µg/L)	Ag (µg/L)	Cd (µg/L)	Sn (µg/L)	Pb (µg/L)	Hg (µg/L)
			ICP-OES	ICP-OES	ICP-OES	ICP-OES	ICP-MS	ICP-MS	ICP-OES	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF
PROCEDURAL BLANK																
		Dissolved	3.36	2.51	0.155	0.025	0.074	0.883	0.283	0.158	1.47	0.04	0.054	0.50	0.009	0
		Dissolved - OES reanalysis	3.36	2.51	0.113	0.025	0.074	0.883	0.105	0.158	1.47	0.04	0.054	0.52	N/A	0.00017
		TRM	3.36	2.51	0.113	0.025	0.074	0.883	0.113	0.158	1.47	0.040	0.054	N/A	0.003	0
METHOD DETECTION LIMIT																
		Project Target Detection Limit	3.36	2.51	0.113	0.025	0.074	0.883	0.113	0.158	1.47	0.040	0.054	N/A	0.003	0.00012
STANDARD REFERENCE MATERIAL																
		Dissolved	52.8	38.3	37.4	125	26.9	83.9	54.7	28.9	26.2	7.57	24.1	1.63	29.0	N/A
		Dissolved - OES reanalysis	54.6	34.4	39.0	123	26.7	82.3	54.1	25.7	21.1	7.43	22.2	1.71	31.1	N/A
		TRM	N/A	N/A	N/A	N/A	26.7	85.2	53.2	26.7	22.0	7.63	22.6	NC	27.9	NC
		certified/reference value	52.0	34.3	38.6	122	27.4	85.2	53.2	26.7	22.0	7.63	22.6	NC	27.9	NC
		range	±1.5	range	±1.6	±1.1	±0.8	±1.2	±0.7	±0.73	±0.51	±0.25	±0.96	NC	±0.14	NC
		% difference	2%	6%	3%	2%	2%	2%	3%	6%	19%	1%	6%	N/A	4%	N/A
		% difference	5%	0%	1%	1%	N/A	N/A	2%	N/A	4%	N/A	N/A	N/A	N/A	N/A
		% difference	N/A	N/A	N/A	N/A	3%	3%	N/A	4%	4%	3%	N/A	N/A	13%	N/A
		certified value	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1497
		range	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	1590
		% difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	6%
ICV/CCV RESULTS																
		ICV	98%	101%	98%	100%	100%	101%	100%	98%	100%	101%	100%	104%	101%	95%
		CCV	99%	102%	99%	101%	101%	101%	100%	99%	99%	102%	99%	104%	105%	98%
		ICV	101%	105%	98%	98%	98%	98%	100%	97%	97%	100%	97%	101%	107%	N/A
		CCV	100%	104%	98%	98%	95%	95%	100%	96%	96%	99%	99%	109%	109%	N/A
		ICV	N/A	N/A	N/A	N/A	95%	97%	N/A	N/A	98%	100%	100%	102%	108%	N/A
		OES reanalysis	98%	100%	102%	101%	100%	101%	103%	98%	100%	101%	100%	104%	101%	N/A
		OES reanalysis	100%	102%	99%	98%	101%	101%	100%	99%	99%	102%	99%	104%	105%	N/A
		OES reanalysis	99%	97%	100%	97%	98%	98%	100%	97%	97%	100%	98%	101%	107%	N/A
		OES reanalysis	99%	100%	100%	97%	95%	95%	100%	97%	95%	99%	97%	99%	109%	N/A
BLANK SPIKE RESULTS																
		Amount Spiked	100	100	50.0	100	10.0	50.0	50.0	10.0	10.0	10.0	10.0	10.0	10.0	0.00472
		Blank + Spike	3.36	2.51	0.155	0.025	0.074	0.883	0.283	0.158	1.47	0.04	0.054	0.50	0.009	0.000407
		Amount Recovered	95.8	108	53.7	125	9.80	50.1	96.4	9.86	9.96	10.3	10.1	10.2	11.2	0.00484
		Percent Recovery	95%	107%	107%	125%	95%	100%	113%	99%	100%	103%	101%	102%	112%	94%
MATRIX SPIKE RESULTS																
		Amount Spiked	100	50.0	50.0	50.0	NS	NS	50.0	NS	NS	NS	NS	NS	NS	NS
		NI-SDB6-OF23A-AFF (D) + Spike	17.1	1.02	1.02	1.54	N/A	N/A	134	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		NI-SDB6-OF23A-AFF (D) + Spike	119	74.2	55.9	55.0	N/A	N/A	55.0	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Recovered	102	53.8	53.8	53.8	N/A	N/A	55.0	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	102%	108%	108%	108%	N/A	N/A	110%	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Amount Spiked	NS	NS	NS	NS	10.0	50.0	NS	10.0	10.0	0.0308	0.552	10.0	10.0	0.0102
		NI-SDB6-OF23A-AFF (T)	N/A	N/A	N/A	N/A	3.83	49.4	N/A	1.22	1.47	0.0308	0.552	10.0	10.0	0.0118
		NI-SDB6-OF23A-AFF (T) + Spike	N/A	N/A	N/A	N/A	13.6	102	N/A	11.3	11.5	9.72	10.4	95.3	3.78	0.0196
		Amount Recovered	N/A	N/A	N/A	N/A	10	52.6	N/A	10.1	11.5	9.69	9.85	95.0	11.1	0.00760
		Percent Recovery	N/A	N/A	N/A	N/A	98%	105%	N/A	101%	115%	97%	98%	95%	111%	76%
REPLICATE RESULTS																
		2360*1 1	17.1	20.4	1.02	0.154	3.45	42.6	134	0.968	1.47	0.04	0.369	0.50	0.201	0.00593
		2360*1 2	17.6	19.4	1.08	0.153	N/A	42.6	133	N/A	N/A	N/A	N/A	N/A	N/A	0.00600
		RPD	3%	5%	6%	1%	N/A	N/A	1%	N/A	N/A	N/A	N/A	N/A	N/A	1%
		2360*3 1	290	388	1.47	15.1	3.83	49.4	185	1.22	1.47	0.04	0.552	0.50	3.78	0.0118
		2360*3 2	N/A	N/A	N/A	3.71	N/A	48.6	1.15	0.0444	1.47	0.0444	0.541	0.50	3.85	N/A
		RPD	N/A	N/A	N/A	N/A	3%	N/A	N/A	6%	N/A	N/A	2%	N/A	2%	N/A

U = not detected at or above detection limit. NC = not certified; NA = not analyzed or available; N/A = not applicable; b = Sample results are less than 3 x the blank.

PAHS

CLIENT ID	NAB- OF 8-SD86-FF	NAB- OF 8-SD86- COMP	NAB- BAY 9-SD86- PRE	NAB- BAY 18-SD86- DUR	NAB- OF 8-SD86-FF	NAB- OF 18-SD86- COMP	NAB- BAY 18-SD86- PRE	NAB- BAY 18-SD86- DUR
Battelle ID	S7118-P	S7119-P	S7120-P	S7121-P	S7122-P	S7123-P	S7124-P	S7125-P
Sample Type	SA	SA	SA	SA	SA	SA	SA	SA
Collection Date	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05
Extraction Date	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05
Analysis Date	03/05/05	03/05/05	03/05/05	03/05/05	03/05/05	03/05/05	03/05/05	03/05/05
Analytical Instrument	MS	MS	MS	MS	MS	MS	MS	MS
% Moisture	NA	NA	NA	NA	NA	NA	NA	NA
% Lipid	NA	NA	NA	NA	NA	NA	NA	NA
Matrix	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER
Sample Size	2.62	2.60	2.61	2.61	2.60	2.61	2.61	2.60
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
Naphthalene	4.14 B	7.85	2.31 J	1.42 J	4.59 B	5.46 J	1.93 J	3.54 B
C1-Naphthalenes	2.88 J	2.08 J	0.51 U	0.51 U	3.29 J	0.51 U	0.51 U	1.48 J
C2-Naphthalenes	0.51 U	0.51 U	0.51 U	0.51 U	0.51 U	1.33 U	0.51 U	0.51 U
C3-Naphthalenes	0.51 U	0.51 U	0.51 U	0.51 U	0.51 U	1.33 U	0.51 U	0.51 U
C4-Naphthalenes	0.51 U	0.51 U	0.51 U	0.51 U	0.51 U	1.33 U	0.51 U	0.51 U
2-Methylnaphthalene	2.84 J	2.16 J	0.36 U	0.36 U	2.82 J	4.01 J	0.36 U	1.51 J
1-Methylnaphthalene	2.04 J	2.02 J	0.38 U	0.38 U	2.49 J	4.38 J	0.38 U	0.99 J
Biphenyl	2.39 J	4.52	0.48 U	0.48 U	3.24	1.24 U	0.48 U	0.48 U
2,6-dimethylnaphthalene	0.83 U	0.64 U	0.63 U	0.63 U	0.64 U	1.65 U	0.63 U	0.64 U
Acenaphthylene	0.54 U	0.54 U	0.54 U	0.54 U	0.54 U	1.4 U	0.54 U	1.35 J
Acenaphthene	0.57 U	2.29 J	0.57 U	0.57 U	0.57 U	1.49 U	0.57 U	0.57 U
2,3,5-trimethylnaphthalene	0.44 U	0.45 U	0.44 U	0.44 U	0.45 U	1.16 U	0.44 U	0.45 U
Dibenzofuran	1.39 J	2.77 J	0.23 U	0.23 U	1.52 J	0.23 U	0.23 U	0.23 U
Fluorene	1.52 J	2.93 J	0.52 U	0.52 U	1.85 J	1.36 U	0.52 U	0.52 U
C1-Fluorenes	0.52 U	0.52 U	0.52 U	0.52 U	0.52 U	1.36 U	0.52 U	0.52 U
C2-Fluorenes	0.52 U	0.52 U	0.52 U	0.52 U	0.52 U	1.36 U	0.52 U	0.52 U
C3-Fluorenes	0.52 U	0.52 U	0.52 U	0.52 U	0.52 U	1.36 U	0.52 U	0.52 U
Anthracene	1.4 J	2.49 J	0.39 U	0.39 U	1.36 J	1.01 U	0.39 U	0.39 U
Phenanthrene	5.59	11.38	1.32 J	0.92	8.98	10.5	0.93 J	13.64
C1-Phenanthrenes/Anthracenes	3.3	8.2	0.83 U	0.83 U	5.65	2.16 U	0.83 U	7.55
C2-Phenanthrenes/Anthracenes	0.82 U	11.28	0.83 U	0.83 U	5.13	2.16 U	0.83 U	14.31
C3-Phenanthrenes/Anthracenes	0.82 U	8.07	0.83 U	0.83 U	5.13	2.16 U	0.83 U	8.08
1-Methylphenanthrene	1.45 J	2.95 J	0.47 U	0.47 U	1.92 J	1.22 U	0.47 U	2.86 J
Dibenzothiophene	8.83	14.57	0.39 U	0.39 U	8.41	3.56 J	0.39 U	2.05 J
C1-Dibenzothiophenes	9.15	13.85	0.39 U	0.39 U	12.94	8.2 J	0.39 U	2.35 J
C2-Dibenzothiophenes	24.2	47.41	0.39 U	0.39 U	37.14	28.72	0.39 U	12.9
C3-Dibenzothiophenes	21.76	32.51	0.39 U	0.39 U	31.41	26.22	0.39 U	15.06
C4-Dibenzothiophenes	8.38	16.7	0.39 U	0.39 U	14.14	12.58	0.39 U	7.67
Fluoranthene	6.56	19.95	0.39 U	0.39 U	9.62	6.71 J	0.39 U	20.94
Pyrene	4.98	14.35	0.39 U	0.39 U	10.23	9.04	0.39 U	18.51
C1-Fluoranthenes/Pyrenes	0.68 U	6.31	0.68 U	0.68 U	4.51	1.79 U	0.68 U	6.96
C2-Fluoranthenes/Pyrenes	0.68 U	0.69 U	0.68 U	0.68 U	0.69 U	1.79 U	0.68 U	6.94
C3-Fluoranthenes/Pyrenes	0.68 U	0.69 U	0.68 U	0.68 U	0.69 U	1.79 U	0.68 U	6.94
Benzo(a)anthracene	1.21 J	1.02 J	1.04 U	1.04 U	2.11 J	2.73 U	1.04 U	0.69 U
Chrysene	1.91 J	7.74	0.89 J	0.89 J	4.48	5.67 J	0.89 J	1.15 J
C1-Chrysenes	0.45 U	4.84	0.45 U	0.45 U	4.48	5.67 J	0.45 U	8.45
C2-Chrysenes	0.45 U	0.45 U	0.45 U	0.45 U	0.45 U	1.18 U	0.45 U	6.52
C3-Chrysenes	0.45 U	0.45 U	0.45 U	0.45 U	0.45 U	1.18 U	0.45 U	11.38
C4-Chrysenes	0.45 U	0.45 U	0.45 U	0.45 U	0.45 U	1.18 U	0.45 U	0.45 U
Benzo(b)fluoranthene	0.85 U	4.22	0.89 U	0.89 U	2.1 J	2.32 U	0.89 U	5.42
Benzo(k)fluoranthene	1 U	1.53 J	1 U	1 U	1.5 J	2.62 U	1 U	3.25 J
Benzo(e)pyrene	0.39 U	0.4 U	0.39 U	0.39 U	2.15 J	1.03 U	0.39 U	6.36
Benzo(a)pyrene	0.77 U	0.77 U	0.77 U	0.77 U	0.77 U	2.01 U	0.77 U	0.77 U
Pyrene	1.47 U	1.48 U	1.48 U	1.48 U	1.48 U	3.86 U	1.48 U	1.48 U
Indeno(1,2,3-cd)pyrene	0.76 U	0.76 U	0.76 U	0.76 U	0.76 U	1.98 U	0.76 U	2.7 J
Dibenz(a,h)anthracene	0.64 U	0.64 U	0.64 U	0.64 U	0.64 U	1.68 U	0.64 U	1.98 J
Benzo(g,h,i)perylene	1.39 J	3.81	0.76 U	1.18 J	1.73 J	4.82 J	0.76 U	10.46

PAHs (CONT.)

CLIENT ID	NAB- OF9-SDB6-FF	NAB- OF9-SDB6- COMP	NAB- BAY9-SDB6- PRE	NAB- BAY9-SDB6- DUR	NAB- OF18-SDB6-FF	NAB- OF18-SDB6- COMP	NAB- BAY18-SDB6- PRE	NAB- BAY18-SDB6- DUR
Surrogate Recoveries (%)								
Naphthalene-d8	32 N	63	56	41	59	40	62	49
Phenanthrene-d10	45	80	74	52	71	64	68	76
Chrysene-d12	39 N	69	72	61	59	56	66	67

PAHs QA/QC

PROJECT: Task Order TO0015/TO0019 -- Contaminant Analysis of Stormwater
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 2/11/05. The samples were received at Battelle Duxbury on 2/15/05. Upon arrival, the cooler temperatures ranged from 0.8°C – 3.7°C. No custody issues were noted. Samples were logged into the Battelle LIMS and received unique IDs. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 05-0056, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PAH	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.47 – 1.93

METHOD: Water samples were extracted for PAH following general NS&T methods. Approximately 1 liter of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts intended for PAH were analyzed using gas chromatography/mass spectrometry (GC/MS), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
05-0056	2/17/05	2/25/05 – 3/6/05

BLANK: A procedural blank (PB) sample was prepared with the analytical batch. Procedural blank samples are analyzed to ensure the sample extraction and analysis methods are free of contamination.

05-0056 – No exceedences noted.

Comments – No target analytes were detected above the laboratory control limit (>5 x MDL), however naphthalene was detected in the procedural blank at a concentration less than the reporting limit (RL). The data was qualified with a “J” in the procedural blank. Any authentic field sample naphthalene concentrations that are greater than the reporting limit but less than five times the concentration detected in

**LABORATORY
CONTROL
SAMPLE:**

the associated blank, were qualified with a "B". This resulted in three samples having "B" qualified naphthalene data; S7118 (OF-NAB9-SDB6-FF), S7122 (OF-NAB18-SDB6-FF), and S7125 (BAY-NAB18-SD86-D). No further corrective action was taken.

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PAH were calculated to measure data quality in terms of accuracy.

05-0056 – All target analytes were recovered within the laboratory control limits (40-120%).

Comments – None.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target PAH and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

05-0056 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%). All calculated RPDs were within the laboratory control limit ($\leq 30\%$).

Comments – None

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Surrogate corrected data has been reported for the SRM only.

05-0056 – All target analytes were recovered within the laboratory control limits specified by the client (≤ 30 PD).

Comments – None.

SURROGATES:

Three surrogate compounds were added prior to extraction, including naphthalene-d8, phenanthrene-d10, and chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

05-0056 – Two exceedences noted.

CALIBRATIONS:

Comments – Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (40 – 120% recovery), except for naphthalene-d8 and chrysene-d12 in sample S7118 (OF-NAB9-SDB6-FF). The recoveries for these compounds were calculated to be 32% and 39%, respectively. Chromatography and calculations were reviewed. No discrepancies were found. The exceedences were qualified with an "N". No further corrective action taken. The GC/MS is calibrated with a minimum of a 5 level curve. The RSD between response factors for the individual target analytes must be $<25\%$. Each batch of samples analyzed is bracketed by a calibration check sample, run at a frequency of minimally every 10 samples. This PD between the initial calibration RF and the check should be $<25\%$ for individual analytes.

04-0103 – No calibration exceedences.

Comments – None.

PAHs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE	MATRIX SPIKE NAB-OF16-SDBA-COMP				MATRIX SPIKE OF18-SDBA-COMP				PROCEDURAL BLANK	6071: POSITIVE PARTICULATE SOLN SOLUTION	
		Target	% Recovery	NG/L LIQUID	L LIQUID	Target	% Recovery	NG/L LIQUID	L LIQUID			
Battle ID	BF876LCS-P					S7123MS-P				BF875PB-P		
Sample Type	LCS					MSD				PE		
Collection Date	02/17/05					2/11/2005				02/17/05		SRM
Extraction Date	02/17/05					2/17/2005				02/17/05		
Analysis Date	02/25/05					3/6/2005				02/25/05		
Analytical Instrument	MS					MS				MS		
% Moisture	NA					NA				NA		
% Lipid	NA					NA				NA		
Matrix	LIQUID					WATER				LIQUID		
Sample Size	2.00					0.825				2.00		
Size Unit-Basis												
Naphthalene	NG/L LIQUID	Target	% Recovery			NG/L LIQUID				NG/L LIQUID		
C1-Naphthalenes	579.24	1000.60	56		57	1415.95				0.94	J	1064.46
C2-Naphthalenes	0.86					1756.57				0.66	U	0.66
C3-Naphthalenes	0.86					1.61				0.66	U	0.66
C4-Naphthalenes	0.86					1.61				0.66	U	0.66
2-Methylnaphthalene	604.52	1002.00	60		64	1622.32				0.47	U	891.98
1-Methylnaphthalene	578.63	1001.20	58		59	1524.17				0.55	U	855.14
Biphenyl	567.69	1000.20	59		61	1683.06				0.62	U	861.16
2,6-Dimethylnaphthalene	514.44	1001.00	61		62	1620.23				0.63	U	949.31
Acenaphthylene	597.78	1000.65	60		62	1497.71				0.71	U	877.83
Acenaphthene	516.18	1000.75	62		62	1505.37				0.73	U	918.77
2,3-Dimethylnaphthalene	602.89	1000.30	60		60	1629.66				0.39	U	890.92
Benzo[a]anthracene	621.92	1002.20	77		77	1865.44				0.33	U	953.56
Fluorene	620.35	1000.70	62		64	1697.36				0.66	U	916.11
C1-Fluorenes	0.66					1.65				0.66	U	0.66
C2-Fluorenes	0.66					1.65				0.66	U	0.66
C3-Fluorenes	0.66					1.65				0.66	U	0.66
Anthracene	703.81	1000.65	70		75	1819.36				0.51	U	1037
Phenanthrene	677.51	1000.65	68		75	2059.16				0.51	U	1005.31
1-Methylanthracenes/Anthracenes	1.08					1327.76				1.08	U	1.08
2-Methylanthracenes/Anthracenes	1.08					1292.48				1.08	U	1.08
3-Methylanthracenes/Anthracenes	1.08					35.44				1.08	U	1.08
4-Methylanthracenes/Anthracenes	1.08					30.03				1.08	U	1.08
1-Methylphenanthrene	693.54	1000.30	69		78	1890.47				1.08	U	1.08
Dibenz[ah]pore	667.95	1001.00	69		75	1834.13				0.61	U	1021.46
C1-Dibenzothiohenes	0.51					12.59				0.51	U	1019.19
C2-Dibenzothiohenes	0.51					48.6				0.51	U	0.51
C3-Dibenzothiohenes	0.51					43.67				0.51	U	0.51
C4-Dibenzothiohenes	0.51					53.46				0.51	U	0.51
Fluoranthene	703.26	1000.50	70		77	1862.97				0.77	U	1041.81
Pyrene	718.96	1000.50	72		77	2089.62				0.9	U	1067.39
C1-Fluoranthenes/Pyrenes	0.9					17.67				0.9	U	0.9
C2-Fluoranthenes/Pyrenes						2.17				0.9	U	0.9
C3-Fluoranthenes/Pyrenes	0.9					2.17				0.9	U	0.9
Benz[ah]anthracene	621.47	1000.60	62		64	1604.23				1.36	U	856.76
C1-Fluoranthene	730.19	1000.75	73		60	1556.53				0.59	U	1045.65
C1-Chysenes	0.59					26.53				0.59	U	0.59
C2-Chysenes	0.59					38.76				0.59	U	0.59
C3-Chysenes	0.59					3.54				0.59	U	0.59
C4-Chysenes	0.59					1.43				0.59	U	0.59
Benz[ah]fluoranthene	673.96	1000.75	67		75	2085.1				1.16	U	935.5
Benz[ah]fluoranthene	777.31	1000.65	78		78	2136.71				1.31	U	1086.15
Benz[ah]pyrene	702.15	1001.80	79		85	1979.25				0.51	U	979.25
Benz[ah]pyrene	629.4	1000.65	63		71	1960.11				1.1	U	876.77
Pyrene	656.25	1000.60	66		72	1955.41				1.93	U	909.5
Indeno[1,2,3-cd]pyrene	723.98	1000.20	72		69	1676.14				0.99	U	1033.73
Dibenz[ah]anthracene	685.03	1000.55	82		80	1982.27				0.84	U	916.9
Benz[ah]pyrene	705.99	1000.70	71		80	2256.51				0.89	U	971.43

CUSTOMER ID	LABORATORY CONTROL SAMPLE	MATRIX SPIKE NAB-OF-1A-SDBS-COMP	MATRIX SPIKE DUPLICATE-NAB- OFR8-SDBS-COMP		PROCEDURAL BLANK	GG73: PCBPESTICIDE SRM SOLUTION
Surrogate Recoveries (%)						
Naphthalene-d8	61	55	56		42	51
Benzothiadiazole-ctd0	71	77	84		44	93
Chrysene-d12	72	95	95		43	86

PCBS

CLIENT ID	NAB- OF9-SDB6-FF	NAB- OF9-SDB6- COMP	NAB- BAY9-SDB6- PRE	NAB- BAY9-SDB6- DUR	NAB- OF18-SDB6-FF	NAB- OF18-SDB6- COMP	NAB- BAY18-SDB6- PRE	NAB- BAY18-SDB6- DUR
Battelle ID	S7118-P	S7119-P	S7120-P	S7121-P	S7122-P	S7123-P	S7124-P	S7125-P
Sample Type	SA	SA	SA	SA	SA	SA	SA	SA
Collection Date	2/11/2005	2/11/2005	2/11/2005	2/11/2005	2/11/2005	2/11/2005	2/11/2005	2/11/2005
Extraction Date	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005
Analysis Date	3/6/2005	3/6/2005	3/6/2005	3/6/2005	3/6/2005	3/6/2005	3/6/2005	3/6/2005
Analytical Instrument	MS	MS	MS	MS	MS	MS	MS	MS
% Moisture	NA	NA	NA	NA	NA	NA	NA	NA
% Lipid	NA	NA	NA	NA	NA	NA	NA	NA
Matrix	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER
Sample Size	2.62	2.60	2.61	2.61	2.60	1.00	2.61	2.60
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
C12(8)	0.07 U	0.07 U	0.07 U	0.07 U	0.07 U	0.18 U	0.07 U	0.07 U
C13(18)	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.22 U	0.08 U	0.08 U
C13(28)	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.22 U	0.08 U	0.08 U
C14(44)	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.38 U	0.15 U	0.15 U
C14(49)	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.38 U	0.15 U	0.15 U
C14(52)	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.38 U	0.15 U	0.15 U
C14(66)	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.38 U	0.15 U	0.15 U
C14(77)	0.14 U	0.14 U	0.14 U	0.14 U	0.14 U	0.37 U	0.14 U	0.14 U
C15(87)	0.23 U	0.24 U	0.24 U	0.24 U	0.24 U	0.61 U	0.24 U	0.24 U
C15(101)	0.23 U	0.24 U	0.24 U	0.24 U	0.24 U	0.61 U	0.24 U	0.24 U
C15(105)	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.28 U	0.11 U	0.11 U
C15(114)	0.23 U	0.24 U	0.24 U	0.24 U	0.24 U	0.61 U	0.24 U	0.24 U
C15(118)	0.07 U	0.07 U	0.07 U	0.07 U	0.07 U	0.19 U	0.07 U	0.07 U
C15(123)	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.21 U	0.08 U	0.08 U
C15(126)	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.31 U	0.12 U	0.12 U
C16(128)	0.27 U	0.27 U	0.27 U	0.27 U	0.27 U	0.71 U	0.27 U	0.27 U
C16(138)	0.27 U	0.27 U	0.27 U	0.27 U	0.27 U	0.71 U	0.27 U	0.27 U
C16(153)	0.27 U	0.27 U	0.27 U	0.27 U	0.27 U	0.71 U	0.27 U	0.27 U
C16(156)	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.2 U	0.08 U	0.08 U
C16(157)	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.38 U	0.15 U	0.15 U
C16(167)	0.27 U	0.27 U	0.27 U	0.27 U	0.27 U	0.71 U	0.27 U	0.27 U
C16(169)	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.29 U	0.11 U	0.11 U
C17(170)	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.49 U	0.19 U	0.19 U
C17(180)	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.28 U	0.11 U	0.11 U
C17(183)	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.49 U	0.19 U	0.19 U
C17(184)	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.49 U	0.19 U	0.19 U
C17(187)	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.49 U	0.19 U	0.19 U
C17(189)	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.21 U	0.08 U	0.08 U
C18(195)	0.36 U	0.37 U	0.37 U	0.37 U	0.37 U	0.95 U	0.37 U	0.37 U
C19(206)	0.34 U	0.34 U	0.34 U	0.34 U	0.34 U	0.89 U	0.34 U	0.34 U
C10(209)	0.41 U	0.41 U	0.41 U	0.41 U	0.41 U	1.07 U	0.41 U	0.41 U
Surrogate Recoveries (%)								
C12(14)	41	77	63	51	64	56	63	70
C13(34)	43	76	67	57	67	61	64	74

PCBs QA/QC

PROJECT: Task Order TO0015/TO0019 – Contaminant Analysis of Stormwater
PARAMETER: PCB
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 2/11/05. The samples were received at Battelle Duxbury on 2/15/05. Upon arrival, the cooler temperatures ranged from 0.8°C – 3.7°C. No custody issues were noted. Samples were logged into the Battelle LIMS and received unique IDs. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 05-0056, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PCB	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD on average (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.09 – 0.53

METHOD:

Water samples were extracted for PCB following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate and concentrated. The extract was then fortified with RIS and split quantitatively for the required analyses. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS). The method is based on key components of the PCB congener analysis approach described in EPA Method 1668A. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds

HOLDING TIMES:

Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
05-0056	2/17/05	3/5/05 – 3/7/05

BLANK:

A procedural blank (PB) sample was prepared with the analytical batch. Procedural blank samples are analyzed to ensure the sample extraction and analysis methods are free of contamination.

05-0056 – No exceedences noted.

Comments – No target analytes were detected in the procedural blank.

**LABORATORY
CONTROL
SAMPLE:**

A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB were calculated to measure data quality in terms of accuracy.

05-0056 – All target analytes were recovered within the laboratory control limits (40-120%).

Comments – None.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target PCB and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

05-0056 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%). All calculated RPDs were within the laboratory control limit ($\leq 30\%$).

Comments – None

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Surrogate corrected data has been reported for the SRM only.

05-0056 – All target analytes were recovered within the laboratory control limits specified by the client (≤ 30 PD).

Comments – None.

SURROGATES:

Two surrogate compounds were added prior to extraction, including PCB 14 and PCB 34. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

05-0056 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

CALIBRATIONS:

The GC/MS is calibrated with a minimum of a 6-point curve. The co-efficient of determination must be ≥ 0.995 for each target analyte. Each batch of samples analyzed is bracketed by a calibration check sample, run at a frequency of every 12 hours (minimally). This PD between the initial calibration RF and the check should be $<20\%$ for individual analytes; 15% on average. Additionally an ICC check was run with the initial calibration. The PD for the ICC should be $< 15\%$, for each analyte.

05-0056 – No calibration exceedences.

Comments – None.

PCBs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE	MATRIX SRK- NAB-OFF- SDB4-COMP	MATRIX SRK- NAB-OFF- SDB1-COMP	PRODEGRAL BLANK	PCBPESTICIDE SOLUTION	GG7:	Passing % Difference
Sample ID	BF676/CS-P	S7123MS-P	S7123MS-P	BF676/BL-P	BF676/BL-P	BF676/BL-P	
Collection Date	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	
Collection Date	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	2/17/2005	
Collection Date	3/5/2005	3/5/2005	3/5/2005	3/5/2005	3/5/2005	3/5/2005	
Analytical Instrument	MS	MS	MS	MS	MS	MS	
% Moisture	NA	NA	NA	NA	NA	NA	
Matrix	NA	WATER	WATER	NA	NA	NA	
Sample Size	2.00	0.325	0.325	2.00	2.00	2.00	
Units	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	
NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	
Target	Target	Target	Target	Target	Target	Target	
% Recovery	% Recovery	% Recovery	% Recovery	% Recovery	% Recovery	% Recovery	
Q	Q	Q	Q	Q	Q	Q	
40.12	40.12	40.12	40.12	40.12	40.12	40.12	
52	52	52	52	52	52	52	
20.94	20.94	20.94	20.94	20.94	20.94	20.94	
58	58	58	58	58	58	58	
21.16	21.16	21.16	21.16	21.16	21.16	21.16	
23.36	23.36	23.36	23.36	23.36	23.36	23.36	
26.89	26.89	26.89	26.89	26.89	26.89	26.89	
22.65	22.65	22.65	22.65	22.65	22.65	22.65	
16.82	16.82	16.82	16.82	16.82	16.82	16.82	
17.85	17.85	17.85	17.85	17.85	17.85	17.85	
25.33	25.33	25.33	25.33	25.33	25.33	25.33	
23.54	23.54	23.54	23.54	23.54	23.54	23.54	
23.84	23.84	23.84	23.84	23.84	23.84	23.84	
0.31 U	0.31 U	0.31 U	0.31 U	0.31 U	0.31 U	0.31 U	
16.86	16.86	16.86	16.86	16.86	16.86	16.86	
0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	
21.05	21.05	21.05	21.05	21.05	21.05	21.05	
20.67	20.67	20.67	20.67	20.67	20.67	20.67	
23.4	23.4	23.4	23.4	23.4	23.4	23.4	
23.31	23.31	23.31	23.31	23.31	23.31	23.31	
0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	
0.35 U	0.35 U	0.35 U	0.35 U	0.35 U	0.35 U	0.35 U	
23.83	23.83	23.83	23.83	23.83	23.83	23.83	
20.19	20.19	20.19	20.19	20.19	20.19	20.19	
23.14	23.14	23.14	23.14	23.14	23.14	23.14	
24.37	24.37	24.37	24.37	24.37	24.37	24.37	
24.69	24.69	24.69	24.69	24.69	24.69	24.69	
20.53	20.53	20.53	20.53	20.53	20.53	20.53	
20.51 U	20.51 U	20.51 U	20.51 U	20.51 U	20.51 U	20.51 U	
20.95	20.95	20.95	20.95	20.95	20.95	20.95	
22.87	22.87	22.87	22.87	22.87	22.87	22.87	
26.36	26.36	26.36	26.36	26.36	26.36	26.36	
Surrogate Recoveries (%)							
C21(14)	58	67	78	40	40	56	
C23(41)	59	69	79	40	40	56	
Value	Value	Value	Value	Value	Value	Value	
34.24	34.24	34.24	34.24	34.24	34.24	34.24	
32.93	32.93	32.93	32.93	32.93	32.93	32.93	
31.4	31.4	31.4	31.4	31.4	31.4	31.4	
31.8	31.8	31.8	31.8	31.8	31.8	31.8	
31.9	31.9	31.9	31.9	31.9	31.9	31.9	
32.07	32.07	32.07	32.07	32.07	32.07	32.07	
33.82	33.82	33.82	33.82	33.82	33.82	33.82	
33.55	33.55	33.55	33.55	33.55	33.55	33.55	
35.34	35.34	35.34	35.34	35.34	35.34	35.34	
33.1	33.1	33.1	33.1	33.1	33.1	33.1	
32.96	32.96	32.96	32.96	32.96	32.96	32.96	
34.77	34.77	34.77	34.77	34.77	34.77	34.77	
32.67	32.67	32.67	32.67	32.67	32.67	32.67	
32.74	32.74	32.74	32.74	32.74	32.74	32.74	
33.22	33.22	33.22	33.22	33.22	33.22	33.22	
32.94	32.94	32.94	32.94	32.94	32.94	32.94	
32.43	32.43	32.43	32.43	32.43	32.43	32.43	
32.64	32.64	32.64	32.64	32.64	32.64	32.64	
15	15	15	15	15	15	15	
0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	
0.35 U	0.35 U	0.35 U	0.35 U	0.35 U	0.35 U	0.35 U	
32.72	32.72	32.72	32.72	32.72	32.72	32.72	
32.96	32.96	32.96	32.96	32.96	32.96	32.96	
0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	
31.89	31.89	31.89	31.89	31.89	31.89	31.89	
0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	
28.04	28.04	28.04	28.04	28.04	28.04	28.04	
32.83	32.83	32.83	32.83	32.83	32.83	32.83	
32.02	32.02	32.02	32.02	32.02	32.02	32.02	
32.94	32.94	32.94	32.94	32.94	32.94	32.94	
40.51	40.51	40.51	40.51	40.51	40.51	40.51	
15	15	15	15	15	15	15	
0.30	0.30	0.30	0.30	0.30	0.30	0.30	
0.66	0.66	0.66	0.66	0.66	0.66	0.66	
0.59	0.59	0.59	0.59	0.59	0.59	0.59	
0.45	0.45	0.45	0.45	0.45	0.45	0.45	

PESTICIDES

CLIENT ID	NAB- OF9-SDB6-FF	NAB- OF9-SDB6- COMP	NAB- OF9-SDB6-PRE	NAB OF9-SDB6-DUR	NAB- OF18-SDB6-FF	NAB- OF18-SDB6- COMP	NAB- BAY18-SDB6- PRE	NAB- BAY18-SDB6- DUR
Battelle ID	S7118-P	S7119-P	S7120-P	S7121-P	S7122-P	S7123-P	S7124-P	S7125-P
Sample Type	SA	SA	SA	SA	SA	SA	SA	SA
Collection Date	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05	02/11/05
Extraction Date	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05	02/17/05
Analysis Date	02/22/05	02/22/05	02/22/05	02/22/05	02/22/05	02/22/05	02/22/05	02/22/05
Analytical Instrument	ECD	ECD	ECD	ECD	ECD	ECD	ECD	ECD
% Moisture	NA	NA	NA	NA	NA	NA	NA	NA
% Lipid	NA	NA	NA	NA	NA	NA	NA	NA
Matrix	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER
Sample Size	2.62	2.60	2.61	2.61	2.60	1.00	2.61	2.60
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
2,4'-DDD	0.62 U	0.63 U	0.62 U	0.62 U	0.63 U	1.63 U	0.62 U	0.63 U
2,4'-DDE	0.41 U	0.76 U	0.53 U	0.53 U	0.53 U	1.37 U	0.53 U	0.53 U
2,4'-DDT	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U
4,4'-DDD	0.73 U	0.73 U	0.73 U	0.73 U	0.73 U	1.9 U	0.73 U	0.73 U
4,4'-DDE	0.52 U	0.53 U	0.52 U	0.52 U	0.52 U	1.37 U	0.52 U	0.53 U
4,4'-DDT	0.45 U	0.45 U	0.45 U	0.45 U	0.45 U	1.18 U	0.45 U	0.45 U
aldrin	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.79 U	0.3 U	0.3 U
a-chlordane	0.29 U	0.29 U	0.29 U	0.29 U	0.29 U	0.76 U	0.29 U	0.29 U
g-chlordane	0.31 U	0.31 U	0.31 U	0.31 U	0.31 U	0.81 U	0.31 U	0.31 U
a-BHC	0.26 U	0.26 U	0.26 U	0.26 U	0.26 U	0.69 U	0.26 U	0.26 U
b-BHC	0.36 U	0.36 U	0.36 U	0.36 U	0.36 U	0.95 U	0.36 U	0.36 U
d-BHC	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.78 U	0.3 U	0.3 U
Lindane	0.38 U	0.38 U	0.38 U	0.38 U	0.38 U	0.99 U	0.38 U	0.38 U
cis-nonachlor	0.49 U	0.5 U	0.5 U	0.5 U	0.5 U	1.29 U	0.5 U	0.5 U
trans-nonachlor	0.31 U	0.31 U	0.31 U	0.31 U	0.31 U	0.81 U	0.31 U	0.31 U
Chlorpyrifos	0.39 U	0.39 U	0.39 U	0.39 U	0.39 U	1.02 U	0.39 U	0.39 U
oxychlordane	0.3 U	0.3 U	0.3 U	0.3 U	0.3 U	0.78 U	0.3 U	0.3 U
dieldrin	0.58 U	0.59 U	0.59 U	0.59 U	0.59 U	1.53 U	0.59 U	0.59 U
endosulfan I	0.21 U	0.21 U	0.21 U	0.21 U	0.21 U	0.55 U	0.21 U	0.21 U
endosulfan II	0.53 U	0.53 U	0.53 U	0.53 U	0.53 U	1.38 U	0.53 U	0.53 U
endosulfan sulfate	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1.3 U	0.5 U	0.5 U
endrin	0.57 U	0.58 U	0.58 U	0.58 U	0.58 U	1.5 U	0.58 U	0.58 U
endrin aldehyde	0.65 U	0.65 U	0.65 U	0.65 U	0.65 U	1.7 U	0.65 U	0.65 U
endrin ketone	0.68 U	0.68 U	0.68 U	0.68 U	0.68 U	1.78 U	0.68 U	0.68 U
heptachlor	0.45 U	0.45 U	0.45 U	0.45 U	0.45 U	1.17 U	0.45 U	0.45 U
heptachlor epoxide	1.2 U	1.21 U	1.21 U	1.21 U	1.21 U	3.15 U	1.21 U	1.21 U
Hexachlorobenzene	0.63 U	0.64 U	0.63 U	0.63 U	0.64 U	1.65 U	0.63 U	0.64 U
methoxychlor	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	1.76 U	0.75 U	0.75 U
Mirex	0.47 U	0.48 U	0.47 U	0.47 U	0.48 U	1.24 U	0.47 U	0.48 U
Surrogate Recoveries (%)								
Cl2(14)	61	92	80	65	96	74	77	92
Cl3(34)	59	86	78	66	92	65	74	82
Cl5(104)	47	73	77	68	68	67	72	86
Cl5(112)	49	84	79	68	71	67	75	81

PESTICIDES QA/QC

PROJECT: Task Order TO0015/TO0019 – Conataminant Analysis of Stormwater
PARAMETER: Pesticides
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 2/11/05. The samples were received at Battelle Duxbury on 2/15/05. Upon arrival, the cooler temperatures ranged from 0.8°C – 3.7°C. No custody issues were noted. Samples were logged into the Battelle LIMS and received unique IDs. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 05-0056, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PESTICIDE	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery	≤30% PD on average (target spike must be >5 x native conc.)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.38 – 1.58

METHOD: Water samples were extracted for pesticide following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts intended for pesticide analysis were solvent exchanged into hexane and analyzed using a gas chromatography/electron capture detector (GC/ECD). Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
05-0056	2/17/05	2/25/05 – 2/28/05

BLANK: A procedural blank (PB) was prepared with the analytical batch. Blanks are analyzed to ensure the sample extraction and analysis methods were free of contamination.

05-0056 – No exceedences noted.

Comments – No target analytes were detected in the procedural blank.

LABORATORY CONTROL SAMPLE: A laboratory control sample (LCS) was prepared with the analytical batch. The percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

05-0056 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target pesticides and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

05-0056 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%). All calculated RPDs were within the laboratory control limit ($\leq 30\%$).

Comments – None

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Surrogate corrected data has been reported for the SRM only.

05-0056 – Two exceedences noted.

Comments – All target analytes were recovered within the laboratory control limits specified by the client (≤ 30 PD), except for 2,4-DDD and 2,4-DDT. The percent differences calculated for these two compounds are 58.5% and 51.0%, respectively. Chromatography and calculations were reviewed. No discrepancies were found. The data has been qualified with an "N". Accuracy for this compound has adequately been demonstrated in the LCS, MS, and MSD QC samples.

SURROGATES

Four surrogate compounds were added prior to extraction, including PCB 14, PCB 34, PCB 104, and PCB 112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

05-0056 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

CALIBRATIONS:

The instrument is calibrated with a 5-level (minimum) calibration, ranging in concentration from ~ 0.001 ng/uL to ~ 0.125 ng/uL. Calibration checks are analyzed minimally every 10 samples. The samples must be bracketed by passing calibrations.

04-0275 – No exceedences noted.

Comments – All calibration criteria were met except for two percent differences calculated for HCB in two calibration checks. However since this compound was not detected in any field samples, and accuracy for this compound was adequately demonstrated in all other QC samples, no further corrective action was taken.

TSS

SAMPLE LABEL	TSS (mg/L)
NAB-OF9-SDB6-FF	6.30
NAB-OF9-SDB6-COMP	10.00
NAB-BAY9-SDB6-PRE	5.51
NAB-BAY9-SDB6-DUR	8.29
NAB-OF18-SDB6-FF	5.83
NAB-OF18-SDB6-COMP	20.30
NAB-BAY18-SDB6-PRE	2.15
NAB-BAY18-SDB6-DUR	11.47

METALS

SDB7 - 4/27/2005

MSL Code	Rep	Sponsor I.D.	Al (µg/L)	Fe (µg/L)	Cr (µg/L)	Mn (µg/L)	Ni (µg/L)	Cu (µg/L)	Zn (µg/L)
2360*11		NAB-OF9-SDB7-COMP (T)	1085	5394	6.41	159	11.6	108	1832
2360*6		NAB-OF9-SDB7-COMP (D)	13.2	14.3	1.60	95.9	8.68	37.8	709
2360*12		NAB-OF18-SDB7-COMP (T)	4717	6550	11.1	197	9.96	108	752
2360*7		NAB-OF18-SDB7-COMP (D)	46.4	145	0.729	34.2	3.81	31.2	149
2360*8		Field Blank - Filtered	3.36	2.66	0.119	0.025	0.436	0.883	11.9

MSL Code	Rep	Sponsor I.D.	As (µg/L)	Se (µg/L)	Ag (µg/L)	Cd (µg/L)	Sn (µg/L)	Pb (µg/L)	Hg (µg/L)
2360*11		NAB-OF9-SDB7-COMP (T)	23.4	52.4	0.125	1.59	0.896	13.2	0.0127
2360*6		NAB-OF9-SDB7-COMP (D)	20.2	48.8	0.04	1.04	0.50	0.139	0.00192
2360*12		NAB-OF18-SDB7-COMP (T)	2.51	1.47	0.0915	2.91	0.724	23.0	0.0201
2360*7		NAB-OF18-SDB7-COMP (D)	1.20	1.47	0.04	0.507	0.50	0.853	0.00456
2360*8		Field Blank - Filtered	0.158	1.47	0.04	0.054	0.50	0.0602	0.000871

SAMPLE ID	DISSOLVED ZINC (µg/L)	TOTAL ZINC (µg/L)
NAB-BAY9-SDB7-AFF	10	10
NAB-BAY9-SDB7-PRE	12.7	22.7
NAB-BAY9-SDB7-DUR	14.8	17.7
NAB-BAY18-SDB7-AFF	30	46
NAB-BAY18-SDB7-PRE	307.6	519.0
NAB-BAY18-SDB7-DUR	312.6	600.7

SAMPLE ID	DISSOLVED COPPER (µg/L)	TOTAL COPPER (µg/L)
NAB-BAY9-SDB7-AFF	18	33
NAB-BAY9-SDB7-PRE	2.3	3.9
NAB-BAY9-SDB7-DUR	3.1	6.9
NAB-BAY18-SDB7-AFF	32	67
NAB-BAY18-SDB7-PRE	2.1	3.1
NAB-BAY18-SDB7-DUR	2.5	4.3

METALS QA/QC

PROGRAM: SPAWAR, Task 19, batch 2
PARAMETER: Metals
LABORATORY: Battelle/Marine Sciences Laboratory, Sequim, Washington
MATRIX: Stormwater

QA/QC DATA QUALITY OBJECTIVES

	Reference Method	Range of Recovery	SRM Accuracy	Relative Precision	Target Detection Limit (µg/L)
Aluminum	ICP/OES	50-150%	±20%	±50%	50.0
Iron	ICP/OES	50-150%	±20%	±50%	10.0
Manganese	ICP/OES	50-150%	±20%	±30%	0.5
Chromium	ICP/MS	50-150%	±20%	±30%	1.0
Nickel	ICP/MS	50-150%	±20%	±30%	0.05
Copper	ICP/MS	50-150%	±20%	±30%	0.05
Zinc	ICP/MS	50-150%	±20%	±30%	0.5
Arsenic	FIAS	50-150%	±20%	±30%	0.5
Selenium	FIAS	50-150%	±20%	±30%	0.2
Silver	GFAA	50-150%	±20%	±30%	0.5
Cadmium	ICP/MS	50-150%	±20%	±30%	0.05
Tin	ICP/MS	50-150%	±20%	±30%	0.5
Lead	ICP/MS	50-150%	±20%	±30%	0.05
Mercury	CVAF	50-150%	±25%	±30%	0.01

METHOD

Nine (9) samples were analyzed for fourteen (14) metals: nickel (Ni), copper, (Cu), arsenic (As), selenium (Se), silver (Ag), cadmium (Cd), tin (Sn) and lead (Pb) by inductively coupled plasma mass spectroscopy (ICP/MS) following EPA Method 1631m, aluminum (Al), iron (Fe), chromium (Cr), manganese (Mn), and zinc (Zn) by inductively coupled plasma optic emission spectroscopy following EPA Method 200.7 and mercury (Hg) by cold vapor atomic fluorescence (CVAF) following EPA Method 1631e.

Samples were preserved with nitric acid prior to arrival at MSL. Samples analyzed for Hg by CVAF were pre-treated with bromine chloride and stannous chloride to oxidize and convert all Hg compounds to volatile Hg, which is subsequently trapped onto a gold-coated sand trap.

HOLDING TIMES

Nine (9) samples were received on 5/03/2005 and were logged into Battelle's sample tracking system. The samples were analyzed within the six month holding time for metals and 90 days for Hg. The following list summarizes all analysis dates:

Task	Date Performed
Hg	5/20/05
ICP-MS	5/11/05
ICP-OES	5/23/05

DETECTION LIMITS

The target detection limit was met for all metals, except Ni, Cu, Se and Cd. The MDL for seawater analysis by dilution is somewhat higher than

our typical MDL's for direct analysis. Sample concentrations were substantially greater than the MDL, except Se. The method detection limit was met for all metals. An MDL is determined by multiplying the standard deviation of the results of a minimum of 7 replicate low level spikes by the Student's t value at the 99th percentile.

METHOD BLANKS

One method blank was analyzed with this batch of samples. Results were less than 3 times the MDL for all metals.

BLANK SPIKES

One sample of reagent water was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

MATRIX SPIKES

One sample was spiked at several levels with metals. Recoveries were within the QC limits of 50-150% for all metals.

REPLICATES

One sample was analyzed in duplicate. All results were within the QC limits of $\pm 30\%$ ($\pm 50\%$ for Al and Fe).

SRM

One matrix-appropriate standard reference material (SRM) was analyzed for each method; 1641d, river water, and 1640, natural water, obtained from the National Institute of Science and Technology.

SRM 1640 has 22 certified and reference metals. Recovery for all metals reported were within the control limit of $\pm 20\%$ of the certified or reference value. Tin and Hg are not certified in 1640. SRM 1641d is certified for Hg. Recovery for Hg was within the control limit of $\pm 25\%$ of the certified value.

REFERENCES

EPA. 1991. Methods for the Determination of Metals in Environmental Samples. EPA-600/4-91-010. Environmental Services Division, Monitoring Management Branch.

METALS QA/QC (CONT.)

MSL Code	Rep	Sponsor I.D.	Al (µg/L) ICP-OES	Fe (µg/L) ICP-OES	Gr (µg/L) ICP-OES	Mn (µg/L) ICP-OES	Ni (µg/L) ICP-MS	Cu (µg/L) ICP-MS	Zn (µg/L) ICP-OES	As (µg/L) ICP-MS	Sr (µg/L) ICP-MS	Ag (µg/L) ICP-MS	Cd (µg/L) ICP-MS	Sn (µg/L) ICP-MS	Pb (µg/L) ICP-MS	Hg (µg/L) CVAF
PROCEDURAL BLANK																
		Dissolved	3.36 U	2.51 U	0.119 U	0.025 U	0.074 U	0.883 U	0.248 U	0.158 U	1.47 U	0.04 U	0.054 U	0.50 U	0.009 U	0.00017 U
		TRM	3.36 U	2.51 U	0.119 U	0.025 U	0.074 U	0.883 U	0.113 U	0.158 U	1.47 U	0.04 U	0.054 U	0.50 U	0.009 U	0.00017 U
METHOD DETECTION LIMIT																
		Project Target Detection Limit	3.36	2.51	0.119	0.025	0.074	0.883	0.113	0.158	1.47	0.04	0.054	0.50	0.009	0.00017
STANDARD REFERENCE MATERIAL																
		Dissolved	56.3	34.0	37.4	119	26.0	78.1	53.7	26.2	23.2	7.10	22.3	1.58	27.4	NA
1640		TRM	N/A	N/A	N/A	N/A	25.3	81.4	N/A	25.2	20.1	7.38	21.8	1.72	27.4	NA
1640		certified reference value	52.0	34.3	36.6	122	27.4	85.2	53.2	26.3	22.0	7.22	22.8	1.72	27.4	NA
1640		range	±1.5	±1.6	±1.6	±1.1	±0.8	±1.2	±1.1	±0.7	±0.5	±0.5	±0.9	NC	±0.14	NC
		% difference	8%	10%	3%	2%	5%	4%	1%	7%	3%	7%	4%	2%	2%	NC
		% difference	N/A	N/A	N/A	N/A	8%	4%	1%	8%	3%	7%	4%	2%	2%	NC
1641d		NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	1602
1641d		certified value	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	1550
1641d		range	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	NC	±180
		% difference	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	1%
ICV CCV RESULTS																
ICV			95%	95%	95%	95%	101%	101%	98%	98%	96%	102%	100%	103%	102%	97%
CCV			95%	95%	95%	95%	98%	98%	103%	103%	94%	101%	98%	105%	102%	95%
CCV			98%	102%	95%	100%	93%	94%	101%	93%	89%	100%	99%	103%	100%	97%
BLANK SPIKE RESULTS																
		Amount Spiked	500	500	100.0	100	10.0	50.0	100.0	10.0	10.0	10.0	10.0	10.0	10.0	0.00496
		Blank + Spike	3.36 U	2.51 U	0.245 U	0.038 U	0.005 U	0.015 U	0.248 U	0.006 U	0.096 U	0.005 U	0.004 U	0.011 U	0.05 U	0.000379
		Amount Recovered	567.0	499	99.6	97.3	9.60	49.3	98.0	9.66	9.31	10.1	9.99	9.99	9.93	0.00436
		Percent Recovery	117%	100%	100%	97%	96%	99%	98%	97%	93%	101%	100%	100%	99%	97%
MATRIX SPIKE RESULTS																
		Amount Spiked	NS	NS	NS	NS	10.0	50.0	NS	10.0	10.0	10.0	10.0	10.0	10.0	NS
		NI-OF26-SDB7-COMP (D)	N/A	N/A	N/A	N/A	5.95	18.9	N/A	1.15	1.47	0.04 U	0.882	0.50 U	1.50	N/A
		Spike	NA	NA	NA	NA	15.3	65.2	NA	11.5	11.2	9.03	10.9	11.2	11.3	NA
		Amount Recovered	N/A	N/A	N/A	N/A	9.35	46.3	N/A	10.4	11.2	9.03	10.0	11.2	9.80	N/A
		Percent Recovery	N/A	N/A	N/A	N/A	94%	93%	N/A	104%	112%	90%	100%	112%	98%	N/A
		Amount Spiked	500	500	100	100	NS	NS	100	NS	NS	NS	NS	NS	NS	NS
		NI-OF23A-SDB7-FF (D)	11.1	12.4	0.295	2.57	N/A	N/A	33.4	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Spike	583	515	97.8	100	NA	NA	129	NA	NA	NA	NA	NA	NA	NA
		Amount Recovered	572	503	97.5	97.7	N/A	N/A	95.6	N/A	N/A	N/A	N/A	N/A	N/A	N/A
		Percent Recovery	114%	101%	98%	98%	NS	NS	96%	NS	NS	NS	NS	NS	NS	N/A
		Amount Spiked	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	0.0098
		NI-OF23A-SDB7-FF (T)	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.0154
		Spike	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.0249
		Amount Recovered	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	0.00850
		Percent Recovery	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	87%
REPLICATE RESULTS																
2360*4	1	NI-OF26-SDB7-COMP (D)	121	103	1.90	23.6	5.95	18.9	79.5	1.15	1.47	0.04 U	0.882	0.50 U	1.50	0.0547
2360*4	2	NI-OF26-SDB7-COMP (D)	130	107	2.00	23.9	5.94	18.6	81.6	1.14	1.47	0.04 U	0.863	0.50 U	1.54	NA
		RPD	7%	4%	5%	1%	0%	2%	3%	1%	N/A	N/A	2%	N/A	3%	N/A
2360*6	1	NAB-OFF-SDB7-COMP (D)	13.2	14.3	1.60	95.9	8.68	37.8	709	20.2	48.8	0.04 U	1.04	0.50 U	0.139	0.00192
2360*6	2	NAB-OFF-SDB7-COMP (D)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.00177
		RPD	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	NA	NA	N/A	N/A	8%

U = not detected at or above detection limit; NC = not certified; NA = not analyzed or available.

PAHS

CLIENT ID	NAB- OF1-SDBT-FF	NAB- OF1-SDBT- COMP	NAB- BAY18-SDBT- PRE	NAB- BAY18-SDBT- DUR	NAB- OF1-SDBT- FF	NAB- OF1-SDBT- COMP	NAB- BAY18-SDBT- PRE	NAB- BAY18-SDBT- DUR	NAB- OF1-SDBT- FF	NAB- OF1-SDBT- COMP	NAB- BAY18-SDBT- PRE	NAB- BAY18-SDBT- DUR
Bottle ID	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P	S7473-P
Sample Type	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA	SA
Collection Date	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005	4/28/2005
Extraction Date	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005	5/4/2005
Analysis Date	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005	5/18/2005
Analytical Instrument	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS	MS
% Moisture	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
% Lipid	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Matrix	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER	WATER
Sample Size	2.65	2.65	2.65	2.65	2.65	2.65	2.65	2.65	2.65	2.65	2.65	2.65
Size Unit-Basis	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID	NG/L LIQUID
Naphthalene	11.1	11.05	1.26	1.03	6.62	1.11	1.11	1.11	1.11	1.11	1.11	1.11
C1-Naphthalenes	30.17	12.79	0.42	0.81	5.44	0.45	0.45	0.45	0.45	0.45	0.45	0.45
C2-Naphthalenes	92.08	53.04	0.5	0.5	7.28	0.5	0.5	0.5	0.5	0.5	0.5	0.5
C3-Naphthalenes	128.35	106.99	0.5	0.5	30.58	0.5	0.5	0.5	0.5	0.5	0.5	0.5
C4-Naphthalenes	106.87	169	0.5	0.5	31.63	0.5	0.5	0.5	0.5	0.5	0.5	0.5
2-Methylnaphthalene	24.24	9.71	0.4	0.73	4.36	0.39	0.39	0.39	0.39	0.39	0.39	0.39
1-Methylnaphthalene	19.05	8.22	0.23	0.39	2.92	0.23	0.23	0.23	0.23	0.23	0.23	0.23
Biphenyl	25.03	12.32	0.33	0.66	4.82	0.33	0.33	0.33	0.33	0.33	0.33	0.33
2,5-dimethylnaphthalene	34.62	15.6	0.62	0.98	2.09	0.62	0.62	0.62	0.62	0.62	0.62	0.62
Acenaphthylene	1.92	2.41	0.66	1.17	2.01	0.66	0.66	0.66	0.66	0.66	0.66	0.66
Acenaphthene	3.68	6.15	1.2	0.9	0.56	0.56	0.56	0.56	0.56	0.56	0.56	0.56
2,3,5-trimethylnaphthalene	24.89	16.67	0.44	0.44	0.44	0.44	0.44	0.44	0.44	0.44	0.44	0.44
Dibenzofuran	4.22	4.51	0.76	1.09	1.54	0.76	0.76	0.76	0.76	0.76	0.76	0.76
Fluorene	7.46	7.32	0.95	1.07	1.37	0.95	0.95	0.95	0.95	0.95	0.95	0.95
C1-Fluorenes	18.54	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51
C2-Fluorenes	60.11	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51
C3-Fluorenes	49.79	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51	0.51
Anthracene	4.45	6.66	0.87	2.15	4.42	0.87	0.87	0.87	0.87	0.87	0.87	0.87
Phenanthrene	27.9	36.65	4.15	6.97	22.72	4.15	4.15	4.15	4.15	4.15	4.15	4.15
C1-Phenanthrenes/Anthracenes	31.47	42.81	0.81	3.39	27.32	0.81	0.81	0.81	0.81	0.81	0.81	0.81
C2-Phenanthrenes/Anthracenes	54.75	103.67	0.81	0.81	54.96	0.81	0.81	0.81	0.81	0.81	0.81	0.81
C3-Phenanthrenes/Anthracenes	39.51	102.28	0.81	0.81	50.16	0.81	0.81	0.81	0.81	0.81	0.81	0.81
C4-Phenanthrenes/Anthracenes	13.61	38.91	0.81	0.81	21.76	0.81	0.81	0.81	0.81	0.81	0.81	0.81
1-Methylphenanthrene	7.61	11.81	0.46	1.09	7	0.46	0.46	0.46	0.46	0.46	0.46	0.46
Dibenzothiophene	7.15	8.61	0.38	1.2	6.73	0.38	0.38	0.38	0.38	0.38	0.38	0.38
C1-Dibenzothiophenes	15.2	24.15	0.38	1.24	18.75	0.38	0.38	0.38	0.38	0.38	0.38	0.38
C2-Dibenzothiophenes	32.03	53.21	0.38	3.59	45.18	0.38	0.38	0.38	0.38	0.38	0.38	0.38
C3-Dibenzothiophenes	20.04	64.39	0.38	3.19	50.01	0.38	0.38	0.38	0.38	0.38	0.38	0.38
C4-Dibenzothiophenes	20.54	51.97	0.38	0.38	50.82	0.38	0.38	0.38	0.38	0.38	0.38	0.38
Fluoranthene	46.09	97.53	6.42	19.73	34.34	6.42	6.42	6.42	6.42	6.42	6.42	6.42
Pyrene	31.99	85.54	4.97	12.91	53.06	4.97	4.97	4.97	4.97	4.97	4.97	4.97
C1-Fluoranthenes/Pyrenes	16.36	49.87	1.29	3.67	20.74	1.29	1.29	1.29	1.29	1.29	1.29	1.29
C2-Fluoranthenes/Pyrenes	0.68	62.66	0.68	0.68	28.56	0.68	0.68	0.68	0.68	0.68	0.68	0.68
C3-Fluoranthenes/Pyrenes	0.68	43.51	0.68	0.68	38.2	0.68	0.68	0.68	0.68	0.68	0.68	0.68
Benzo(a)anthracene	2.85	8.84	0.55	1.44	3.41	0.55	0.55	0.55	0.55	0.55	0.55	0.55
Chrysene	18.09	66.78	3.17	9	30.02	3.17	3.17	3.17	3.17	3.17	3.17	3.17
C1-Chyrenes	6.7	31.12	0.44	1.31	29.92	0.44	0.44	0.44	0.44	0.44	0.44	0.44
C2-Chyrenes	6.85	39.11	0.44	0.44	33.96	0.44	0.44	0.44	0.44	0.44	0.44	0.44
C3-Chyrenes	0.44	45.38	0.44	0.44	39.23	0.44	0.44	0.44	0.44	0.44	0.44	0.44
C4-Chyrenes	0.44	23.57	0.44	0.44	20.9	0.44	0.44	0.44	0.44	0.44	0.44	0.44
Benzo(b)fluoranthene	8.84	27.03	1.73	5.74	12.78	1.73	1.73	1.73	1.73	1.73	1.73	1.73
Benzo(k)fluoranthene	6.12	17.8	1.48	4.7	8.13	1.48	1.48	1.48	1.48	1.48	1.48	1.48
Benzo(e)pyrene	6.37	24.43	1.18	3.68	16.04	1.18	1.18	1.18	1.18	1.18	1.18	1.18
Benzo(a)pyrene	2.28	10.23	0.76	2.01	9.77	0.76	0.76	0.76	0.76	0.76	0.76	0.76
Perylene	1.46	3.88	1.28	1.21	2.17	1.28	1.28	1.28	1.28	1.28	1.28	1.28
Indeno(1,2,3-cd)pyrene	3.06	13.48	0.82	2.35	9.17	0.82	0.82	0.82	0.82	0.82	0.82	0.82
Dibenz(a,h)anthracene	0.58	2.72	0.63	1.54	6.34	0.63	0.63	0.63	0.63	0.63	0.63	0.63
Benzo(g,h,i)perylene	6.85	36.76	0.94	2.42	38.09	0.94	0.94	0.94	0.94	0.94	0.94	0.94

PAHs (CONT.)

CLIENT ID	NAB- OF1-SDB7-FF	NAB- OF3-SDB7- COMP	NAB- BAY3-SDB7- PRE	NAB- BAY3-SDB7- DUR	NAB- OF1-SDB7-FF	NAB- OF1-SDB7- COMP	NAB- BAY1-SDB7- PRE	NAB- BAY1-SDB7- DUR
Surrogate Recoveries (%)								
Naphthalene-d8	51	58	59	44	54	50	56	47
Phenanthrene-d10	62	78	68	65	73	72	67	65
Chrysene-d12	83	91	83	85	83	82	87	86

PAHs QA/QC

PROJECT: Task Order TO0015/TO0019 – Contaminant Analysis of Stormwater
PARAMETER: PAH
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 4/28/05. The samples were received at Battelle Duxbury on 5/3/05. Upon arrival, the cooler temperatures ranged from 2.2°C – 3.2°C. One sample, BAY-NI26-SDB7-Pr, was broken upon receipt. The project manager was informed of this issue, and relayed it to the client. The lab was instructed to proceed with the remaining samples. No other custody issues were noted. Samples were logged into the Battelle LIMS and received unique IDs. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 05-0129, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PAH	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery	≤30% PD plus variance (target spike must be >5 x native conc.) (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.50 – 1.93

METHOD: Water samples were extracted for PAH following general NS&T methods. Approximately 1 liter of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts intended for PAH were analyzed using gas chromatography/mass spectrometry (GC/MS), following general NS&T methods. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
05-0129	5/04/05	5/17/05 – 5/19/05

BLANK: A procedural blank (PB) sample was prepared with the analytical batch. Procedural blank samples are analyzed to ensure the sample extraction and analysis methods are free of contamination.

05-0129 – No exceedences noted.

Comments – No target analytes were detected above the laboratory control limit (>5 x MDL), however naphthalene and 2-Methylnaphthalene were detected in the procedural blank at a concentration less than the reporting limit (RL). The data was qualified with a “J” in the procedural blank. All authentic field sample concentrations for these compounds were either greater than five times the

LABORATORY CONTROL SAMPLE:	concentration in the associated blank, or less than the RL. A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PAH were calculated to measure data quality in terms of accuracy.
	05-0129 – All target analytes were recovered within the laboratory control limits (40-120%).
MATRIX SPIKE/MATRIX SPIKE DUPLICATE:	Comments – None. A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target PAH and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.
	05-0129 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%). All calculated RPDs were within the laboratory control limit ($\leq 30\%$).
SRM:	Comments – None A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Surrogate corrected data has been reported for the SRM only.
	05-0129 – All target analytes were recovered within the laboratory control limits specified by the client (≤ 30 PD).
SURROGATES:	Comments – None. Three surrogate compounds were added prior to extraction, including naphthalene-d8, phenanthrene-d10, and chrysene-d12. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).
	05-0129 – One exceedence noted.
CALIBRATIONS:	Comments – Percent recoveries for all surrogate compounds were within the laboratory control limits specified by the method (40 – 120% recovery), except for naphthalene-d8 in sample S7468 (OF-NI26-SDB7-FF). The recovery for this compound was calculated to be 38%. Chromatography and calculations were reviewed. No discrepancies were found. The sample prep records indicate an emulsion formed during the extraction of this sample, and that this extract had difficulty passing through the alumina cleanup column. The exceedences were qualified with an “N”. No further corrective action taken. The GC/MS is calibrated with a minimum of a 6 level curve. The RSD between response factors for the individual target analytes must be $<25\%$, the mean RSD $< 15\%$. Each batch of samples analyzed is bracketed by a calibration check sample, run at a frequency of minimally every 10 samples. This PD between the initial calibration RF and the check should be $<25\%$ for individual analytes, and again the mean PD should be $<15\%$.
	05-0129 – No calibration exceedences.
	Comments – None.

[illegible]

PAHs QA/QC (CONT.)

CLIENT ID	LABORATORY CONTROL SAMPLE	MATRIX M-073A SD87-FF	MATRIX DUPLICATE M-073A SD87-FF	PROCEDURAL BLANK	MATRIX M-073A SD87-FF	MATRIX DUPLICATE M-073A SD87-FF	GG72: PCBPESTICIDE SRM SOLUTION	GG72: PCBPESTICIDE SRM SOLUTION
Surrogate Recoveries (%)								
Naphthalene-d8	83	51	43	83	83	52	68	68
Phenanthrene-d10	80	72	68	78	68	71	75	75
Chrysene-d12	102	89	85	98	85	87	95	95

PCBs

CLIENT ID	NAB- OF9-SDB7- COMP		NAB-OF18- SDB7-COMP	
Battelle ID	S7474-P		S7478-P	
Sample Type	SA		SA	
Collection Date	4/28/2005		4/28/2005	
Extraction Date	5/4/2005		5/4/2005	
Analysis Date	5/29/2005		5/30/2005	
Analytical Instrument	MS		MS	
% Moisture	NA		NA	
% Lipid	NA		NA	
Matrix	WATER		WATER	
Sample Size	2.65		2.65	
Size Unit-Basis	L LIQUID		L LIQUID	
Units	NG/L LIQUID		NG/L LIQUID	
CI2(8)	0.07	U	0.07	U
CI3(18)	0.08	U	0.08	U
CI3(28)	0.08	U	0.08	U
CI4(44)	0.14	U	0.14	U
CI4(49)	0.14	U	0.14	U
CI4(52)	0.14	U	0.14	U
CI4(66)	0.14	U	3.52	
CI4(77)	0.14	U	2.15	J
CI5(87)	0.23	U	2.73	J
CI5(101)	0.23	U	3.57	
CI5(105)	0.11	U	4.44	
CI5(114)	0.23	U	0.23	U
CI5(118)	0.07	U	6.05	
CI5(123)	0.08	U	0.08	U
CI5(126)	0.12	U	0.12	U
CI6(128)	0.27	U	0.27	U
CI6(138)	0.27	U	4.18	
CI6(153)	1.83	J	4	
CI6(156)	0.08	U	0.08	U
CI6(157)	0.14	U	0.14	U
CI6(167)	0.27	U	0.27	U
CI6(169)	0.11	U	0.11	U
CI7(170)	0.18	U	0.18	U
CI7(180)	0.1	U	2.57	J
CI7(183)	0.18	U	0.18	U
CI7(184)	0.18	U	0.18	U
CI7(187)	0.18	U	1.86	J
CI7(189)	0.08	U	0.08	U
CI8(195)	0.36	U	0.36	U
CI9(206)	0.33	U	0.33	U
CI10(209)	0.4	U	0.4	U
Surrogate Recoveries (%)				
CI2(14)	87		76	
CI3(34)	89		82	

PCBs QA/QC

PROJECT: Task Order TO0015/TO0019 – Contaminant Analysis of Stormwater
PARAMETER: PCB
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 4/28/05. The samples were received at Battelle Duxbury on 5/3/05. Upon arrival, the cooler temperatures ranged from 2.2°C – 3.2°C. One sample, BAY-NI26-SDB7-Pr, was broken upon receipt. The project manager was informed of this issue, and relayed it to the client. The lab was instructed to proceed with the remaining samples. No other custody issues were noted. Samples were logged into the Battelle LIMS and received unique IDs. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 05-0129, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PCB	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery	≤30% PD on average	≤30% RPD	MDL: ~0.09 – 0.53
				(target spike must be >5 x native conc.)	(for analytes >5x MDL)	(calculated between the MS and MSD samples)	

METHOD: Water samples were extracted for PCB following general NS&T methods. Approximately 1 liter of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate and concentrated. The extract was then fortified with RIS and split quantitatively for the required analyses. Extracts were analyzed using gas chromatography/mass spectrometry (GC/MS). The method is based on key components of the PCB congener analysis approach described in EPA Method 1668A. Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
05-0129	5/4/05	5/28/05 – 5/30/05

BLANK: A procedural blank (PB) was prepared with the analytical batch. Blanks are analyzed to ensure the sample extraction and analysis methods were free of contamination.

05-0129 – No exceedences noted.

LABORATORY CONTROL SAMPLE: **Comments** – No target analytes were detected in the procedural blank. A laboratory control sample (LCS) was prepared with each analytical batch. The percent recoveries of target PCB were calculated to measure data quality in terms of accuracy.

05-0129 – One exceedence noted.

Comments – All target analytes were recovered within the specified laboratory control limits (40-120%), except for PCB 169. This analyte was over-recovered at

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

141%. It was also over-recovered in both the MS and MSD samples. Chromatography and calculations were reviewed. No discrepancies were found. The exceedence has been qualified with an "N". Since PCB 169 was not detected in any field samples, the affect of this exceedence on the data is minimal. No further corrective action is necessary.

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair was prepared with each analytical batch. The percent recoveries of target PCB and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

05-0129 – Three percent recovery exceedences noted.
No RPD exceedences noted.

Comments – All target analytes were recovered within the specified laboratory control limits (40-120%), except for PCB 169 in samples S7470MS and S7470MSD (background OF-NI23A-SDB7-FF) and PCB 209 in sample S7470MS. All exceedences were due to over-recoveries. Chromatography and calculations were reviewed, no discrepancies were found. The exceedences were qualified with an "N". Since PCB 169 was not detected in any field samples, and PCB 209 was not detected above the RL, the affect of these exceedences on the data is minimal. No further corrective action is necessary.

SRM:

A standard reference material was prepared with each analytical batch. The percent difference (PD) between the measured value and the certified range was calculated to measure data quality in terms of accuracy. The MQO criteria of 30% PD was added to the variance of each analyte. The variance of each analyte is determined by dividing the range value by the target.

05-0129 – All PDs were within the specified laboratory control limits.

Comments – None.

SURROGATES:

Two surrogate compounds were added prior to extraction, including PCB 14 and PCB 34. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

05-0129 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

CALIBRATION:

The GC/MS is calibrated with a minimum of a 6-point curve. The co-efficient of determination must be ≥ 0.995 for each target analyte. Each batch of samples analyzed is bracketed by a calibration check sample, run at a frequency of every 12 hours (minimally). This PD between the initial calibration RF and the check should be <20% for individual analytes; 15% on average. Additionally an ICC check was run with the initial calibration. The PD for the ICC should be < 15%, for each analyte.

05-0129 – One exceedence noted.

Comments – In mid C1466.d PCB 105 was over-recovered and had a PD of 31%. Two samples S7468 and S7478 (Samples OF-NI26-SDB7-Comp and OF-NAB18-SDB7-Comp, respectively) had PCB 105 detected in them. Chromatography and calculations were reviewed. No discrepancies were found. The deviation has been documented and the data reviewed. No further corrective action was taken.

[illegible]

PESTICIDES

CLIENT ID	NAB- OF9-SDB7-COMP	NAB- OF18-SDB7-COMP
Battelle ID	S7474-P	S7478-P
Sample Type	SA	SA
Collection Date	4/28/2005	4/28/2005
Extraction Date	5/4/2005	5/4/2005
Analysis Date	5/14/2005	5/14/2005
Analytical Instrument	ECD	ECD
% Moisture	NA	NA
% Lipid	NA	NA
Matrix	WATER	WATER
Sample Size	2.65	2.65
Size Unit-Basis	L LIQUID	L LIQUID
Units	NG/L LIQUID	NG/L LIQUID
2,4'-DDD	0.61 U	0.61 U
2,4'-DDE	0.25 J	0.52 U
2,4'-DDT	0.37 U	0.37 U
4,4'-DDD	0.72 U	0.72 U
4,4'-DDE	0.52 U	0.9 U
4,4'-DDT	1.39 U	0.44 U
aldrin	1.65 U	0.3 U
a-chlordane	0.34 J	0.28 U
g-chlordane	0.3 U	0.3 U
a-BHC	0.26 U	0.26 U
b-BHC	0.36 U	0.36 U
d-BHC	0.99 U	0.67 U
Lindane	0.37 U	0.37 U
cis-nonachlor	0.49 U	0.49 U
trans-nonachlor	1.14 U	0.31 U
Chlorpyrifos	0.39 U	0.39 U
oxychlordane	0.3 U	0.3 U
dieldrin	0.58 U	0.58 U
endosulfan I	0.21 U	0.21 U
endosulfan II	0.52 U	0.52 U
endosulfan sulfate	0.49 U	0.49 U
endrin	0.57 U	0.57 U
endrin aldehyde	0.64 U	0.64 U
endrin ketone	0.67 U	0.67 U
heptachlor	0.44 U	0.44 U
heptachlor epoxide	1.19 U	1.19 U
Hexachlorobenzene	0.62 U	0.62 U
methoxychlor	0.74 U	5.28 U
Mirex	0.47 U	0.47 U
Surrogate Recoveries (%)		
C12(14)	88	88
C13(34)	94	84
C15(104)	94	83
C15(112)	91	90

PESTICIDES QA/QC

PROJECT: Task Order TO0015/TO0019 – Contaminant Analysis of Stormwater
PARAMETER: Pesticides
LABORATORY: Battelle, Duxbury, MA
MATRIX: Water
SAMPLE CUSTODY: Water samples were collected 4/28/05. The samples were received at Battelle Duxbury on 5/3/05. Upon arrival, the cooler temperatures ranged from 2.2°C – 3.2°C. One sample, BAY-NI26-SDB7-Pr, was broken upon receipt. The project manager was informed of this issue, and relayed it to the client. The lab was instructed to proceed with the remaining samples. No other custody issues were noted. Samples were logged into the Battelle LIMS and received unique IDs. Samples were stored in the access-controlled upper cold room refrigerator at 4.0°C until sample preparation could begin. Samples were extracted as one analytical batch, 05-0129, along with the appropriate quality control samples.

	Reference Method	Method Blank	Surrogate Recovery	LCS/MS Recovery	SRM % Diff.	Sample Replicate Relative Precision	Detection Limits (ng/L)
PESTICIDE	General NS&T	<5xMDL	40-120% Recovery	40-120% Recovery (target spike must be >5 x native conc.)	≤30% PD plus variance (for analytes >5x MDL)	≤30% RPD (calculated between the MS and MSD samples)	MDL: ~0.27 – 1.58

METHOD: Water samples were extracted for pesticide following general NS&T methods. Approximately 2 liters of water was spiked with surrogates and extracted three times with dichloromethane using separatory funnel techniques. The combined extract was dried over anhydrous sodium sulfate, concentrated, processed through alumina cleanup column, concentrated, copper cleaned, and further purified by GPC/HPLC. The post-HPLC extract was concentrated, fortified with RIS and split quantitatively for the required analyses. Extracts intended for pesticide analysis were solvent exchanged into hexane and analyzed using a gas chromatography/electron capture detector (GC/ECD). Sample data were quantified by the method of internal standards, using the Recovery Internal Standard (RIS) compounds.

HOLDING TIMES: Samples were prepared for analysis in one analytical batch and were extracted within 7 days of sample collection and analyzed within 40 days of extraction.

Batch	Extraction Date	Analysis Date
05-0129	5/04/05	5/14/05 – 5/16/05

BLANK: A procedural blank (PB) was prepared with the analytical batch. Blanks are analyzed to ensure the sample extraction and analysis methods were free of contamination.

05-0129 – No exceedences noted.

Comments – No target analytes were detected in the procedural blank.

LABORATORY A laboratory control sample (LCS) was prepared with the analytical batch. The

**CONTROL
SAMPLE:**

percent recoveries of target pesticides were calculated to measure data quality in terms of accuracy.

05-0129 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%).

Comments – None.

**MATRIX
SPIKE/MATRIX
SPIKE
DUPLICATE:**

A matrix spike (MS) and a matrix spike duplicate (MSD) sample pair were prepared with each analytical batch. The percent recoveries of target pesticides and the relative percent difference between the two samples were calculated to measure data quality in terms of accuracy and precision.

05-0129 – All target analytes were recovered within the laboratory control limits specified by the client (40-120%). All calculated RPDs were within the laboratory control limit ($\leq 30\%$).

Comments – None

SRM:

A standard reference material (SRM, a certified second source standard was spiked into a natural seawater as an SRM) was prepared with each analytical batch. Surrogate corrected data has been reported for the SRM only.

05-0129 – All percent differences for reported target analytes were within the laboratory control limits ($<30\%$ difference plus variance).

Comments – None.

SURROGATES

Four surrogate compounds were added prior to extraction, including PCB 14, PCB 34, PCB 104, and PCB 112. The recovery of each surrogate compound was calculated to measure data quality in terms of accuracy (extraction efficiency).

05-0129 – Percent recoveries for all surrogate compounds were within the laboratory control limits (40 – 120% recovery).

Comments – None.

CALIBRATIONS:

The instrument is calibrated with a 6-level (minimum) calibration, ranging in concentration from ~ 0.001 ng/uL to ~ 0.125 ng/uL. The initial correlation coefficient must be > 0.995 . Calibration checks are analyzed minimally every 12 hours. The samples must be bracketed by passing calibrations. Calibration checks must have a percent difference $\leq 25\%$.

05-0129 – No exceedences noted.

Comments – None.

[illegible]

TSS

SAMPLE LABEL	TSS (mg/L)
NAB-OF9-SDB7-FF	11.690
NAB-OF9-SDB7-COMP	60.289
NAB-BAY9-SDB7-PRE	3.277
NAB-BAY9-SDB7-DUR	15.239
NAB-OF18-SDB7-FF	45.573
NAB-OF18-SDB7-COMP	234.378
NAB-BAY18-SDB7-PRE	4.280
NAB-BAY18-SDB7-DUR	6.139

DOC

SAMPLE LABEL	DOC (mg/L)
NAB-OF9-SDB7-FF	7.562
NAB-OF9-SDB7-FF	7.770
NAB-OF9-SDB7-FF	7.943
NAB-OF9-SDB7-COMP	14.439
NAB-OF9-SDB7-COMP	15.064
NAB-OF9-SDB7-COMP	15.188
NAB-BAY9-SDB7-PRE	1.919
NAB-BAY9-SDB7-PRE	1.750
NAB-BAY9-SDB7-PRE	1.552
NAB-BAY9-SDB7-DUR	1.709
NAB-BAY9-SDB7-DUR	1.690
NAB-BAY9-SDB7-DUR	1.742
NAB-OF18-SDB7-FF	11.079
NAB-OF18-SDB7-FF	11.584
NAB-OF18-SDB7-FF	11.442
NAB-OF18-SDB7-COMP	14.983
NAB-OF18-SDB7-COMP	15.441
NAB-OF18-SDB7-COMP	15.169
NAB-BAY18-SDB7-PRE	2.070
NAB-BAY18-SDB7-PRE	1.713
NAB-BAY18-SDB7-PRE	1.756
NAB-BAY18-SDB7-DUR	1.775
NAB-BAY18-SDB7-DUR	1.759
NAB-BAY18-SDB7-DUR	1.952

Appendix D4

NI

SDB4- 10/17/2004

SBD6-2/10/2005

SDB7- 4/027/2005

SDB4- 10/17/2004

METALS

SAMPLE ID	DISSOLVED Cu (µg/L)	TOTAL Cu (µg/L)
NI-OF23A SDB4 FF	74	172
NI-BAY23A SDB4 DUR	5.2	8.0

SAMPLE ID	DISSOLVED Zn (µg/L)	TOTAL Zn (µg/L)
NI-OF23A SDB4 FF	778	1125
NI-BAY23A SDB4 DUR	20.8	21.3

TSS

SAMPLE LABEL	TSS (mg/L)
NI-OF23A-SDB4-FF	201.33
NI-BAY23A-SDB4-DUR	9.89

SDB6- 2/10/2005

METALS

MSL	Code	Rep	Sponsor	Al (µg/L)	Fe (µg/L)	Cr (µg/L)	Mn (µg/L)	Ni (µg/L)	Cu (µg/L)	Zn (µg/L)
			I.D.	ICP-OES	ICP-OES	ICP-OES	ICP-OES	ICP-MS	ICP-MS	ICP-OES
	2360*3		NI-OF23A-SDB6-FF (T)	290	388	1.47	15.1	3.83	49.4	185
	2360*1		NI-OF23A-SDB6-FF (D)	17.1	20.4	1.02	0.154	3.45	42.6	134
	2360*2		Field Blank - Filtered	1.64	0.217	0.119	0.025	0.074	0.883	0.274

MSL	Code	Rep	Sponsor	As (µg/L)	Se (µg/L)	Ag (µg/L)	Cd (µg/L)	Sn (µg/L)	Pb (µg/L)	Hg (µg/L)
			I.D.	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF
	2360*3		NI-OF23A-SDB6-FF (T)	1.22	1.47	0.04	0.552	0.50	3.78	0.0118
	2360*1		NI-OF23A-SDB6-FF (D)	0.968	1.47	0.04	0.369	0.50	0.201	0.00593
	2360*2		Field Blank - Filtered	0.158	1.47	0.04	0.054	0.50	0.009	0.000566

MSL	Code	Rep	Sponsor	Al (µg/L)	Fe (µg/L)	Cr (µg/L)	Mn (µg/L)	Ni (µg/L)	Cu (µg/L)	Zn (µg/L)
			I.D.	ICP-OES	ICP-OES	ICP-OES	ICP-OES	ICP-MS	ICP-MS	ICP-OES
	2157*12		NI-SDB6-O26-COMP (T)	540	756	3.65	51.0	5.93	41.0	87.3
	2157*9		NI-SDB6-OF26-COMP (D)	19.8	22.1	1.31	7.12	4.62	29.1	36.6

MSL	Code	Rep	Sponsor	As (µg/L)	Se (µg/L)	Ag (µg/L)	Cd (µg/L)	Sn (µg/L)	Pb (µg/L)	Hg (µg/L)
			I.D.	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	ICP-MS	CVAF
	2157*12		NI-SDB6-O26-COMP (T)	11.5	38.9	0.0719	1.14	0.739	10.8	0.0212
	2157*9		NI-SDB6-OF26-COMP (D)	11.0	38.3	0.040	0.791	0.50	0.512	0.00213

SAMPLE ID	DISSOLVED COPPER (µg/L)	TOTAL COPPER (µg/L)
NI-BAY23A-SDB6-PRE	2.2	2.3
NI-BAY23A-SDB6-DUR	3.3	6.0
NI-OF26-SDB6-FF	22.2	33.4
NI-BAY26-SDB6-PRE	2.2	2.7
NI-BAY26-SDB6-DUR	4.1	9.7

SAMPLE ID	DISSOLVED ZINC (µg/L)	TOTAL ZINC (µg/L)
NI-BAY23A-SDB6-PRE	6.2	6.3
NI-BAY23A-SDB6-DUR	10.7	11.1
NI-OF26-SDB6-FF	101	129
NI-BAY26-SDB6-PRE	5.1	6.7
NI-BAY26-SDB6-DUR	18	29