



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION I

J.F. KENNEDY FEDERAL BUILDING, BOSTON, MASSACHUSETTS 02203-2211

November 3, 1994

Mr. Robert Krivinskas
U.S. Department of the Navy
Northern Division - NAVFAC
10 Industrial Highway
Code 1811/RK - Mail Stop 82
Lester, PA 19113-2090

Re: Comments on the Draft Work Plans for Ecological Field Work at Site 09, Allen Harbor Landfill at Naval Construction Battalion Center (NCBC), RI

Dear Mr. Krivinskas:

Pursuant to § 7.6 of the NCBC Federal Facility Agreement (FFA), please find attached the Environmental Protection Agency's (EPA) comments on the above referenced documents.

EPA received the Work Plans entitled, "Draft Work/Quality Assurance Project Plan for the Narragansett Bay Ecorisk and Monitoring for Navy Sites Offshore Ecological Risk Assessment for the Lower East Passage Study Area", and the "Addendum [for the NCBC Allen Harbor Landfill Site] to the Draft Work/Quality Assurance Project Plan for the Narragansett Bay Ecorisk and Monitoring for Navy Sites Offshore Ecological Risk Assessment for the Lower East Passage Study Area" on September 22, 1994.

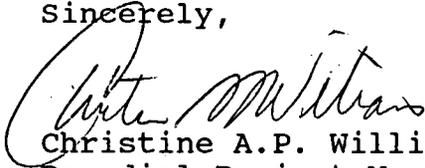
Overall, this workplan and addendum do not adequately explain the objectives, scope of work, or technical approaches proposed for the Allen Harbor Landfill Ecological Risk Assessment (ERA). The technical approaches are overly conceptual/generic and/or insufficiently detailed with respect to tasks, subtasks, and methods needed to perform a comprehensive ERA that addresses contaminant-specific and aggregate risks to ecological indicator species, communities, and ecosystems, while conforming to the EPA guidance on ERA.

As was discussed in a conversation with the Navy and RIDEM on October 25, 1994, these two plans have been rewritten and combined with the ERA workplan for the rest of the NCBC ERA data gaps. This revision was received on November 1, 1994. I will be reviewing the revised workplan and will be providing comments in accordance with the FFA, prior to December 15, 1994.



If you have any questions with regard to this letter, please contact me at (617) 573-5736.

Sincerely,



Christine A.P. Williams
Remedial Project Manager
Federal Facilities Superfund Section

Attachment

cc: Richard Gottlieb, RIDEM
Lou Fayon, NCBC
Tim Prior, USF & WL
Celeste Barr, EPA
Jayne Michaud, EPA
Andy Miniuks, EPA
Mary Sanderson, EPA
Susan Svirsky, EPA

**EPA COMMENTS ON DRAFT WORK / QUALITY ASSURANCE
PROJECT PLAN FOR THE NARRAGANSETT BAY
ECORISK AND MONITORING FOR NAVY SITES.
OFFSHORE ECOLOGICAL RISK ASSESSMENT FOR THE
LOWER EAST PASSAGE STUDY AREA
JULY 12, 1994 version
and
ALLEN HARBOR LANDFILL ADDENDUM**

General Comments

1. Neither document individually, nor both documents taken together, adequately explains the objectives, scope of work, or technical approaches proposed for the Allen Harbor Ecological Risk Assessment (ERA). The technical approaches are overly conceptual/generic and/or insufficiently detailed with respect to tasks, subtasks, and methods needed to perform a comprehensive ERA that addresses contaminant-specific and aggregate risks to ecological indicator species, communities, and ecosystems, while conforming to the U.S. Environmental Protection Agency Region I (EPA) guidance on ERA.
2. We appreciate the Navy's commitment to provide a single ERA work plan for all terrestrial, wetland, and aquatic exposure zones previously identified at NCBC, as a stand-alone document, that outlines the key linkages among the ERA elements and specifies approaches to site characterization, hazard identification, exposure assessment, toxicity assessment, risk characterization, and uncertainty analysis for the NCBC site as a whole. The revised work plan is scheduled to be received by October 31, 1994. Ideally, the work plan should build upon those ecological exposure models for habitats and indicator species presented in prior ERA reports, while designing approaches capable of resolving the issues/deficiencies identified in EPA's prior critiques of those ERA reports.
3. Copies of analytical Standard Operating Procedures (SOPs) associated with the task are provided and some discussion of quality assurance and quality control (QA/QC) procedures are provided in the work plan portion of the document. A separate document entitled Quality Assurance Project Plan for the Offshore Ecological Risk Assessment at the Naval Education and Training Center Newport, Rhode Island is included as Appendix C. The analytical SOPs provide appropriate methods for the preparation and analysis of waters, sediments, and tissue samples from a marine environment for trace metals. The methods are similar to those used by EPA, but have been modified due to

the salinity of the samples. For example, aqueous samples are not digested due to the interference by salts in the matrix. In the past, EPA has recommended a chelation-extraction preparation for saline samples (EPA-600/4-79-020). This technique required an experienced analyst and was often plagued with contamination and interference problems. Advances in analytical instrumentation have largely eliminated the need for this method of sample preparation and it is no longer widely used. Many marine chemists use direct graphite furnace analysis with Zeeman background correction, optimization of ashing and atomization temperatures and matrix modifiers to overcome the saline interference. It was also noted that sample preservation and holding times for samples being analyzed for metals are not strictly in line with EPA guidelines, however this is not likely to have a significant negative impact on the useability of the data. It is suggested that the SOPs be reviewed by University of Rhode Island (URI), since there are typographical errors and discrepancies. For example, the SOP for Flame Atomic Absorption (ERL-N SOP 2.04.004), Section III B, No. 5 appears to be missing a reference to a specific step ("...repeat the procedures of step (which step)"). Also, the title for the ICP SOP is incorrect, the title indicates that the SOP is for Flame Atomic Absorption Spectrophotometry. An SOP for graphite furnace AAS has not been included nor is there an SOP for mercury, which presumably is done by the cold vapor method; yet there are references to mercury and graphite furnace analyses elsewhere in the document. There are numerous other inconsistencies and errors within the SOPs or between the SOPs and other documents included in this package. It is also suggested that specific QC requirements and limits be included within the appropriate SOP. While the procedures and limits are outlined in Table 1 of Appendix C, it is possible that the analyst may not have this information available.

4. The QA Plan appears to be the document provided in Appendix C, however this document does not follow the typical EPA QA Plan format and addresses only a portion of the requirements of the typical EPA QA Plan. While many of the other QA Plan topics such as data management, sampling, calibration procedures, and audits are touched upon in other appendices or in the main portion of the document, there are inconsistencies and gaps which would likely have been caught, had the organization of the document followed the EPA guidelines.
5. The issue of comparability must be raised. The procedures and documentation of an academic laboratory typically do not match those of a commercial

laboratory, which generally follows a prescribed analytical and reporting format which will be reviewed by a regulatory agency. Analyses performed in academia can use more current methods which may be untested, but are subject to peer review. It appears that URI has made an attempt to incorporate the QA/QC procedures typically used in EPA analyses. However, none of the inorganic procedures fully matches published EPA methods. Another gap is the lack of clearly defined data quality objectives and sample handling procedures such as holding time and sample preservation procedures, for all analyses. In spite of these issues, the metals data produced by the URI methods should be useable and, if the proper documentation exists, defensible. Due to the differences in the analytical procedures and their associated uncertainties, however, results may not match those obtained using a more standardized set of procedures such as the CLP protocols. The organic procedures appear to present gaps, which may impact the useability of the data if further documentation cannot be provided.

6. The analytical parameters discussed in this plan include PAH, Pesticides/PCBs, metals, butyltins, grain size and TOC. The methods specified by this plan include organic extractions for BNAs and PCBs as well as metals analyses by flame atomic absorption spectrophotometry and inductively coupled plasma emission. Detailed SOPs or standard method references must be provided for PAH (analysis), Pest/PCBs(analysis), butyltins, and TOC. Table 6, Target analytes for chemical characterization, must provide the specific SOP or standard test method that will be applied to each group or individual analyte presented. If SOPs are to be used for the above parameters, then they must be of sufficient detail. The SOPs must include, but are not limited to, the number and concentrations of standards, frequency of calibration, blank criteria, materials and equipment, sample clean-up procedures, internal standard requirements, initial and continuing calibration curve criteria expressed in percent RSD and percent D, and other quality control requirements as necessary.
7. The matrices targeted for sampling include seawater, sediment, and fish tissue/biota. The document presented includes sampling procedures for deep cores and box cores but does not present any procedures for the collection of seawater and biota sampling. The document must present sampling methods for all matrices presented in the text. The sampling procedures need to provide details of equipment, preservation procedures, containers, decontamination

procedures and quality control sample requirements. Please note that Región 1 requires sediment samples to be greater than 30 percent solids. If this is not possible, a greater sample weight must be used to prevent elevated detection limits due to dry weight corrections.

8. The seawater matrix could provide interferences during the metals analysis. The SOPs for the analytical methods for the metals must address the potential interferences.

Specific Comments

Draft Work Plan

9. This highly conceptual discussion of existing data and EPA's generic approach to ERA (i.e., conceptual framework for ERA) is much too broadly focussed on the entire Narragansett Bay and its Lower East Passage study area to be very useful in the design of a site-specific ERA Work Plan for Allen Harbor. Most of the discussion in the Technical Approach sections for the four tasks is overly descriptive and conceptual, in that:
 - Existing conditions and prior studies are described for the entire bay, without proposing any specific ecological characterization efforts to supplement these data and without mentioning NCBC in Task 1.
 - An overly broad methodological summary of EPA's generic approach to Problem Formulation and a conceptual model for broad scale contaminant inputs to and ecological exposures throughout the bay appears in Task 2, again without mention of Allen Harbor, other NCBC habitats, or proposed technical approaches to the site-specific problem formulation effort that will be needed for the NCBC ERA.
 - Approaches to sampling and analysis of physical and biological media are proposed in Task 3, for the Lower East Passage, again without mention of NCBC.
 - Another very broad and generic discussion of EPA's ERA framework concept of Risk Characterization, again lacking NCBC/Allen Harbor-specific approaches is presented in Task 4, in which the only mention of Allen Harbor is a proposal to apply similar ERA methods and models to the bay-wide ERA, similar to those used previously in the marine ERA for Allen Harbor.

10. Although this document identifies "nearshore coves" of the bay as habitats at greatest risk from adjacent onshore disposal areas at Naval sites, neither Allen Harbor (one such nearshore cove at risk) nor the associated landfill and wetland habitats at NCBC are mentioned as focal points for the broader, bay-wide ERA. By itself, therefore, this Draft Work Plan is only tangentially pertinent to the ERA requirements for Allen Harbor that should be satisfied in a site-specific ERA Work Plan that also ensures resolution of all issues/deficiencies identified in reviews of the prior NCBC ERA reports.

11. Page 13, Table 1

Figure 5 does not contain benthic habitats as referenced in the title of the table. The correct figure appears to be Figure 6.

12. Page 23, Section 7.4.3, Task 2 - Problem Formulation for the Lower East Passage Study Area, Stressors and Ecological Effects

Among the documented potential stressors to this ecosystem are nutrients and pathogens, however no mention is made in the document as to their analysis. Please explain why they are not being included in this project.

13. Page 25, Section 7.4.3, Task 2 - Problem Formulation for the Lower East Passage Study Area, Conceptual Model

This section implies that the impact of the naval installation be evaluated within the context of the bay as a whole. Under the Superfund Program and consistent with EPA Region I guidance, the impact of the naval installation should be evaluated on its own merit, not as a percentage of the total impact from all sources to the bay. The evaluation must be performed to measure site related contaminants in exceedance of background concentrations for each media and provide evidence that these exceedances adversely impact the ecological integrity of the system.

14. Pages 32 - 39, Section 7.4.4, Task 3 - Plan for Analysis / Data Collection, Sampling Plan

There are no references in this section to the decontamination of field equipment or the collection of field blanks. Sampling equipment must be decontaminated between each sample in order to avoid cross contamination of the samples. It is recommended that methanol be used for decontaminating equipment when collecting organic samples and 1:1 nitric acid

for decontaminating for metals samples. In addition, field blanks should be collected at a rate of 1 per 10 field samples in order to verify the efficiency of the decontamination. This is accomplished by running laboratory pure water across the equipment after decontamination. The rinse water is collected for analysis of the same parameters that were being tested on the associated field samples.

15. Page 35, Section 7.4.4, Task 3 - Plan for Analysis / Data Collection, Sampling Plan

The code HN for Allen Harbor indicated here is not consistent with Figure 13. Please correct.

16. Page 35, Section 7.4.4, Task 3 - Plan for Analysis/Data Collection, Surface Box Cores

The document states that three replicate box cores will be collected. Once a box core is removed from the sediment, the matrix remaining is not the same as the initial sample and are not considered replicates. Replicate samples must be taken from the same sampling device or container. The procedure described in this section yields co-located samples or duplicates. Duplicate samples are required to assess the precision of the sampling event. Replicates measure the homogeneity of the sample and the precision of the analytical method. Change this section to reflect the collection of duplicate samples.

17. Page 36, Table 6

The target analytes selected do not encompass the wide range of reasonable, potential causes of stress to the ecosystem. For example, volatile organics and nutrient analyses are not included in the proposed target analytes, but likely do play a role necessary to understanding the ecosystem. Clarify how the potential impacts of these analytes and other factors be accounted for without some form of analysis or measurement.

Please reference the source or clarify the selection criteria used to determine the congeners selected for analysis. The target list does not include the complete list of co-planar congeners, which are the most toxic. It resembles the National Oceanic and Atmospheric Administration (NOAA) Status and Trends List, but Congener 126 is missing. The target list may be sufficient if the congeners selected are either known to be associated with the site, will be used to extrapolate estimated total polychlorinated biphenyl

(PCB) content, or are interpreted as indicators for other toxic PCB congeners.

Please clarify how the detection limits were obtained. In some cases the detection limits exceed established criteria (such as ambient water quality criteria (AWQC) for surface water samples). For example, the listed detection limit for mercury in water at 5 ug/l is well above the AWQC of 0.025 ug/l.

18. Page 39, Section 7.4.4, Task 3 - Plan for Analysis/Data Collection

Quahogs (Mercenaria mercenaria) are referred to previously as hard shell clams. Please use consistent common naming.

Please clarify whether biota samples will be analyzed on whole body or specific tissue portions (such as fillet and offal). Clarify whether mussels will be shucked and soft tissue analyzed or whether the shell will be included in the analyses.

19. Page 39, Section 7.4.4, Task 3 - Plan for Analysis/Data Collection, Mussel Deployment

This paragraph indicates that deployment time will range from 6 to 8 weeks. It is suggested that consideration be given to the idea of extending this period to 12 weeks. This would provide additional time for equilibration of accumulation to take place and also would allow the added benefit of being able to compare these results with other comparable studies, i.e., NOAA's Mussel Watch Program.

20. Page 42, Section 7.4.5, Task 4 - Plan for Risk Characterization, Risk Characterization, Comparison with Reference Stations

It should be made clear that the reference areas must consist of characteristics as similar as possible to site related areas and comparison should be made between these areas of like characteristics.

Ecological risk evaluation is done on a site by site basis. The use of comparative risk assessment is not appropriate under this program.

21. Page 43, Section 7.4.5, Task 4 - Plan for Risk Characterization, Risk Characterization, Comparison with Reference Stations

The temporal interpretation of the findings is important for many of the data but particularly AVS as

its concentrations can be seasonal and its effect on bioavailability strong. Winter and summer levels should be measured to allow for temporal comparisons before calculating risk.

22. Page 49, Section 9.2, Project Quality Assurance Officer

One of the functions of the Quality Assurance Officer is to verify that projects QA/QC procedures are adequate to meet Data Quality Objectives (DQOs). Although the DQOs have been stated through the defining of precision, accuracy, completeness, representativeness and comparability, EPA requires the identification of analytical levels which will meet the DQOs. These levels range from Level 1 -field screening, through Levels 4 and 5 which require the highest degree of quality control producing legally defensible data. The specifying of these DQO levels will ensure that the laboratory procedures will support the goals of this project. These DQOs are defined in Data Quality Objectives for Remedial Response Activities: Development Process, EPA 540/G-87/003, March, 1987.

23. Page 54, Section 10.3, Completeness

This paragraph essentially states that holding time for sediments is not a concern. This is not the case and a recommendation of no more than 6 weeks prior to initiating the tests is applicable. It is recommended that the sediment and pore water toxicity tests methods referenced be included in the work plan for review prior to acceptance.

24. Page 55, Section 11 - Analytical Procedures

The use of the term "internal standard" can be misleading. In most uses, internal standard specifically refers to compounds added after extraction and used as a reference for quantitation. If the internal standard is added prior to extraction, the term surrogate is normally utilized. If this surrogate is then utilized as a reference for quantitation, the analysis should be termed recovery corrected since the surrogate recovery is reduced comparable to the target analytes. The distinction of whether these results are recovery corrected or not is critical to comparability of the data. Most environmental data is not recovery corrected, i.e., surrogates are added as a measure of extraction efficiency and separate internal standards are added after extraction as a quantitation reference. If this is not how the analysis is to be performed, while the

proposed approach may be technically valid, it must be discussed as an issue in terms of comparability. What will be done to allow comparison of recovery corrected data to other environmental data collected and not recovery corrected? Will it be clear to data users in result summaries that the data has already been recovery corrected?

25. Page 56, Section 11.1, Chemistry Methods

In addition, the section for organic contaminants does not specify the percentage of samples which will be subject to duplicate analysis. One field sample (not a field blank) must be analyzed in as a replicate per every 20 field samples analyzed. Replicate analysis is necessary to verify the precision of the analytical procedure.

26. Page 56, Section 11.1 - Chemistry Methods

Are there limits on the concentration of the standard analyzed for determination of the ILOD/ILOQ relative to the final calculated values? In general, the higher the concentration the more precise the determinations will be and, therefore, the lower the ILOQ/ILOD. However, the principle in statistically basing detection limits on precision requires that the concentration of the standards utilized approach the actual limit of detection. It is recommended that the concentration of the standard be limited to no more than five times the calculated ILOD/ILOQ. This is comparable to the requirements for calculation of method Detection Limits (MDLs) according to 40 CFR, Part 136, Appendix B. If the calculated ILOD/ILOQ is less than five times the concentration of the standard used to determine these values, the ILOQ/ILOD should be considered artificially deflated and the ILOQ/ILOD study repeated with lower concentrations.

Please clarify whether reporting limits are MDLs, MLOQs, or MLODs. Please consistently define these terms since the terms are not used or defined consistently throughout the document. Refrain from use of generic terms such as "detection limit" without clarifying whether this value is the MLOQ, MLOD, MDL, ILOQ, ILOD, or reporting limit. In this section the MDL is said to be determined based on the MLOQ (i.e., "The MLOQ is defined as 10 x MDL"). However, these are not consistent. The MDL is a standard term which is calculated according to 40 CFR, Part 136, Appendix B as is stated in the Quality Assurance Project Plan (QAPjP) portion of this document. This value cannot be mathematically related to the MLOQ/MLOD by a simple

relationship, since each are determined through different processes.

27. Page 56, Section 11.2.1 - Organic Contaminants, Blank Analyses

Allowing contamination to be reported as high as three times the MDL appears excessive. If contamination is detected with any regularity above the MDL, the MDL is unrealistic. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. If blank contamination is present, a truly blank (i.e., zero concentration) sample will falsely be measured and reported with positive results. It is recommended that contamination above the MDL be treated as an exceedance of this QC parameter. EPA data validation policy states that target analytes detected in samples at less than five times the level detected in blanks should be treated as unreliable since the value may be significantly impacted by the same contamination source. It is further recommended that for consistency the blank levels should not exceed more than 20 percent (1/5) of the sample concentration.

28. Page 56, Section 11.2.1 - Organic Contaminants, Extraction Efficiencies

Please clarify whether recoveries of matrix spike (MS) compounds are calculated based on internal standards spiked into the sample prior to extraction or following extraction. If internal standards are spike prior to extraction and used as a reference for quantitation, there is little measurement of the overall extraction efficiency. For example, if the internal standard and the spike compounds are recovered both at 25 percent, the spike compound will appear to indicate 100 percent recovery since relative to the internal standard both were comparably lost.

29. Page 56, Section 11.2.1, Organic Contaminants, Extraction Efficiencies

The text states that extraction efficiency is measured through the use of matrix spikes. For organic extractions, the use of surrogates and matrix spikes is suggested to measure extraction efficiency. A surrogate is added to each sample, whereas a matrix spike is performed usually at a frequency of 1:20 field samples.

30. Page 56, Section 11.2.1 - Organic Contaminants, Instrumental Detection Limits

This definition of MDL conflicts with the earlier definition of the MDL as 1/10 the MLOQ and conflicts with the 40 CFR, Part 136, Appendix B standard definition of MDL. The MDL is not a theoretical detection limit based on ideal instrument sensitivity or even actual measurement of instrument sensitivity and assuming 100 percent extraction efficiency, etc.

31. Page 56, Section 11.2.1 - Organic Contaminants, Instrument Variability

The replicate injection estimates instrument variability, not overall analytical variability. Instrument variability is only a minor portion of the overall analytical variability and is of limited the value in evaluating the precision or accuracy of the overall data set. The discussion of replicates appears to conflict with the frequency of QC listed below, which indicates that "duplicates" will be analyzed once for every 20 samples. It is recommended that duplicate analyses (i.e., independent sample preparation and analysis) be utilized to understand the precision of the overall method and the discussion be modified to describe measurement of the variability of the entire analytical process.

32. Page 57, Section 11.2.1, Organic Contaminants, Instrument Detection Limits

The MDLs stated for organic analyses do not match the detection limits found in Table 1 on pages 36-38. Please correct this discrepancy.

33. Page 58, Section 11.2.2, Inorganic Contaminants, Blank Analysis

According to this paragraph, a blank analysis is required every 20-30 samples, however the first bullet item in the fifth paragraph on the same page states that blanks will be analyzed every 20 samples. Please correct this discrepancy.

34. Page 58, Section 11.2.2, Inorganic Contaminants, Extraction Efficiency

Extraction efficiency for inorganic analyses is to be measured through the use of Standard Reference Materials (SRM). Although SRMs will provide information about the extraction efficiency of the method used, they provide little information regarding the matrix of the field samples. A pre-extraction spike of a replicate of a field sample will provide more information regarding the efficiency of the extraction on the specific matrix sampled. Control criteria should be 75-125%. If the recovery of the

pre-extraction spike does not meet these criteria, the sample should be analyzed by the method of standard additions which would account for the matrix effect of the sample.

In addition, this paragraph specifies the frequency of SRM analysis as one for each 20-30 field samples, whereas according to the forth bullet item in the fifth paragraph on the same page, SRMs will be analyzed every 10 samples. Please correct this discrepancy.

35. Page 61, Section 15, Data Validation

Data validation provides a systematic approach to verifying that the quality control performed with the analyses has met all defined criteria. EPA Region I requires that all data validation is performed in accordance with three Region 1 guidance documents: Tiered Organic & Inorganic DV Guidelines, DV Guidelines for Organic and Inorganic Analyses and the CSF (Complete Sample Delivery Group) File Purge Guidance.

36. Page 62, Section 19.0 - Health and Safety

It is recommended that due to the potential for handling contaminated materials and hazardous materials (for instance, decontaminating fluids or sample preservatives), Occupational Safety and Health Administration (OSHA) training be required of all sampling personnel.

APPENDIX B - STANDARD OPERATING PROCEDURES

37. Page B-2, General

The summary of analyses should be referenced to "below" not as described "above".

Please clarify whether confirmation gas chromatograph (GC) analysis will be performed for PCB/organochlorine pesticides (OCP) analysis, if performed, whether the confirmation column will quantify contaminants or qualitatively confirm presence, whether samples for PCBs/OCPs will be GC/MS verified, and what detection limits may be verified using GC/MS procedures. Considering the complexity of marine matrices, PCB/OCP confirmation is critical to provision of supportable data not impacted by matrix interferences or non-target compound co-elution.

38. Page B-3

Storage of samples at 1-2°C is a narrower range than may be attainable and repeatable. The recommended temperature range is 4°C ±2°C. Sample storage conditions may be critical to quality results. SOPs should address issues such as whether sample container temperatures measured upon sample receipt, how frequently refrigerator temperatures are measured, corrective actions if allowable storage conditions are exceeded, whether samples protected from light due to potentially photoreactive target analytes (e.g., polycyclic aromatic hydrocarbons (PAHs)), etc.

Single-time methylene chloride extraction (350 ml MeCl₂/≈3.5 l water) with minimal agitation may not be sufficient for reliable and repeatable extraction. Analytical methods normally require multiple extractions (separatory funnel shakes) to enhance not only the extraction efficiency but also to minimize impacts of matrix effects on extraction efficiency. Have more extensive extractions been considered particularly for re-extraction of samples which have demonstrated low recovery (<≈60 percent) of internal standards spiked prior to extraction? If the extraction method is not modified, it is recommended that corrective actions for poor recovery of MSs or surrogates include re-extraction and re-analysis by either the same method or an improved extraction procedure.

39. Page B-3, Procedures

The procedures described herein do not specify when internal standards, surrogates or matrix spikes are added to the samples. In addition, the concentrations of the spiking solutions used for fortifying samples must be documented.

40. Page B13, Section III A. Methods, Sample Preparation

The SOP does not discuss when to add the matrix spike to the sample. The matrix spike solution should be added to the sample prior to adding extraction acids.

41. Page B15, Section III A. 2., Methods, Sample Preparation

The texts states that to guard against cross-contamination between samples during excision from shells, the instruments should be rinsed in deionized water. Deionized water may not be stringent enough to remove trace metals left on the titanium instrument. To avoid cross-contamination, rinse the instruments with a nitric acid solution followed by deionized water.

42. Page B16, Section C. Sample Digestion

The procedures should state when to add the matrix spiking solution to the sample as well as the concentration of the spiking solution.

43. Page B17, Section III 2., Methods

The text states that aqueous samples for metals analysis should be preserved to a pH of 2-2.5. In order to keep all of the metals in solution, the pH should be lowered to less than 2 units. The procedure used to verify the pH of the sample must be presented. The procedure must use pH sensitive paper and must not be introduced directly into the sample vial.

44. Page B20, Section III B. 1., Calibration and Sample Analysis

The text states that a minimum of three standards and a blank must be run. In order to verify sensitivity at the detection limit, the lowest standard must be at a concentration at or near the detection limit.

45. Page B20, Section III B. 5., Calibration and Sample Analysis

The text states that the sample absorbance of the samples should be less than 0.7 A, however, section III B. 1. states that the concentrations of the standards should yield absorbances no greater than 0.5 A. If that is the case, then the concentration of the samples could exceed the calibration range. In order to provide accurate values, the concentration of the samples must be within the calibration range.

46. Page B22-B25, SOP for the Analysis of Metals by ICP

The title of the document in the upper margin of the text is not correct and should read by "Inductively Coupled Plasma (ICP) atomic emission spectrometry".

APPENDIX C - Analytical Chemistry Quality Assurance and Quality Control Protocols.

47. Page C-9, Detection Limit

The MDL is correctly defined in this section, although this conflicts with the earlier two references in this document. Are MLOQs in any way related to ILOQs? This text also conflicts with the earlier descriptions.

48. Page C-10, Sample Receipt and Storage

The text states that no holding times apply to these samples. Once a sample is removed from the environment and kept contained, physical, biological and chemical transformations are likely to occur due in part to the biota collected with the sample. Although the samples will remain refrigerated until analyzed, the samples can not be stored for an indefinite period of time. Holding times for the water and sediment samples must follow the guidance specified in the Inorganic (ILM03) and Organic (OLM01.9) Statements of Work (SOW) or the Test Methods for Evaluating Solid Waste (SW-846).

49. Page C-11, Sample Analysis Scheme

This section defines a batch as 20 field samples. However, page 58 of the text requires that some of the inorganic QC procedures be performed at a frequency of 1 per 10 samples. Expand this section to include the correct specifications for both organic and inorganic analyses.

50. Page C-12, Table 1

If control criteria for initial calibration are not met, what corrective action will be taken. The recommended corrective action will include troubleshooting of instrument and/or process to determine source of the problem and re-analysis of initial calibration prior to sample analysis.

MDLs should be performed once per matrix per year.

Intercalibration exercises are not an actual part of QC, but rather part of laboratory QA since there is no immediate feedback to analyst measuring the results. Intercalibration exercises impact the overall process (i.e., QA) rather than reflect upon the quality of sample results. The results support the overall laboratory quality, but not specifically the quality of individual batches of field samples. If results would be released only if intercalibration exercises were acceptable, it could be interpreted as QC.

Allowances of 35 percent of all analyte concentration determinations in standard reference materials (SRMs) to be incorrect seems insufficient. It is recommended that some warning limits, as opposed to control limits, be placed that require determination of the causative problems if more than 10 percent of the determinations are incorrect.

Blank contamination above the MDL, not just three times the MDL, should cause qualifying of field sample data in cases where the sample results may be

attributable to the same source of contamination as the blank result.

MS criteria do not appear to be sufficiently stringent if the quantitations are based relative to the internal standard which has been added prior to extraction. Basic to the reliability of recovery correction, is the target analyte and internal standard reference (spiked prior to extraction) exhibit comparable chemical behavior. If the internal standard is well selected to mimic the chemistry of those components which it used as a quantitation reference, the relative difference in the recovery between the MS compounds and the internal standard should be minimal. If the internal standard and MS compounds (as well as all target analytes) do not exhibit comparable chemical behavior, the reporting of results as recovery corrected must be re-examined.

51. Page C-12 through C-13, Table 1, Key Elements for Quality Control of Chemical Analyses

The table is divided into two parts; part 1 for initial demonstration of QC limits and part 2 for ongoing demonstration capability. In part 1 the control limits for the initial calibration are indicated as percent values. Are these %RSDs for the response factors?

The frequency of establishment of an MDL is stated as at least once per matrix. However, the MDL can change over time. It is recommended that an MDL be performed once every six months for each matrix.

In part 2 of the table, the control limits for continuing calibration checks are listed as percent values. Are those values percent differences from the initial calibration?

The frequency of SRM analysis is stated as one per batch of 20 samples. However, page 58 describes the frequency of inorganic SRM analysis as one per 10 samples. Please correct this discrepancy.

The frequency of analysis of laboratory duplicates is stated as one per batch. Page 57 describes the frequency of organic laboratory blanks as one per 20 samples while page 58 describes the frequency of inorganic samples as once per 10 samples. Please expand this section to account for both organic and inorganic criteria.

APPENDIX D - Draft Data Management Plan

52. Page D-2, QA/QC Protocols

The text of this section states that 100% of the data will be checked for transcription errors from the hard copy to the point of computer data entry. Page 59 states that 10% of the data entries will be checked for transcription errors. Please correct this discrepancy.

ALLEN HARBOR ADDENDUM

53. Since this addendum intended only to propose additional tasks to be performed for the Allen Harbor ERA, a detailed ERA Work Plan is still needed that addresses the other terrestrial, wetland, and aquatic habitats at NCBC, that are not associated with the harbor or adjacent landfill. Whether a revised version of this Addendum is to be resubmitted as a Work Plan specific to Allen Harbor, or will be integrated into a site-wide NCBC ERA Work Plan (preferred), the Addendum should be expanded to:
- Clearly outline the site-specific objectives of the ERA
 - Provide a comprehensive ERA work breakdown structure of discrete tasks/subtasks, and propose approaches and a schedule for each
 - Propose an ecological characterization to identify and map the approximate boundaries of saline, brackish, and freshwater habitats to be sampled
 - Specify what pre-existing ERA analyses/data, and indicate what new physical, chemical, and biological data will be collected for the ERA
 - Propose an approach to determining "background" levels of contaminants and/or identify reference area habitats to be used to assess incremental, site-derived risks to ecological receptors
 - Propose statistical methods for data reduction, including averaging methods for non-detects
 - Propose criteria for the selection of contaminants of concern (COCs) and for choosing ecological indicator species and/or biotic communities representative of all affected habitat types and salinity regimes
 - Propose exposure assessment scenarios/pathways to be assessed, including food chain models (which could be adapted from the prior ERA reports)

- Identify appropriate effects measurement endpoints for different COCs or COC classes, and explain how these data and toxicity testing results will be integrated into the toxicity assessment and risk characterization
- Propose a qualitative and/or quantitative approach to characterize direct and indirect contaminant risks at different levels of ecosystem integration

54. Part III - Specific Tasks

Comments on this section are:

- An ecological characterization task is needed, including a discussion of the different types and salinity regimes of the Allen Harbor wetland and aquatic habitats to be sampled.
- An improved Figure 1 is needed to show boundaries of terrestrial, wetland and open water habitats, before the adequacy of the proposed aquatic and wetland sampling stations can be determined.
- Why is no surface water sampling, analysis, and toxicity testing proposed to complement the sediment testing program?
- How many wetland salinity regimes will be sampled for chemical analysis and toxicity testing? Verify that test species will be chosen based to match the salinity regimes of their natural wetland habitats.

55. Task III - Deep Sediment Cores

To support the aquatic ERA, and risk estimates for terrestrial fauna that may consume benthic biota, surficial sediments should be analyzed from all 10 deep core sampling locations. The field screening parameters proposed to determine depth intervals to be analyzed should be used only to select a subset of these 10 locations at which one or more additional, subsurface sediment samples will be collected to document vertical contaminant concentration gradients.

56. Task IV - Synthesis of Phase I, II and III of the Risk Assessment Pilot Study

This section is too vague to understand what will be done in this task with these existing data, what will constitute a "synthesis" of these prior studies, and what is meant by a "Superfund document in accordance with the CERCLA criteria." How will these prior

studies be supplemented by and integrated with new studies proposed here? Finally, conformance to the format and content for an ERA outlined in EPA's ERA guidance may be preferable to the format (from Environmental Compliance Office (ECO) Update bulletin) proposed.

57. Schedule

A single, integrated ERA Work Plan for all affected areas/habitats at NCBC, that includes a complete ERA project schedule, should be presented for EPA review and approval.