APPENDIX D
Quality Assurance Report

Analytical data quality was evaluated by both laboratory review and independent review, as specified in the quality assurance project plan (QAPP) for the project. Independent review and validation of laboratory data packages was performed by Validata, LLC, of Seattle, Washington. Additional review of explosives data was performed by D.M.D., Inc., of Vashon, Washington. The validation report and associated memoranda are included as Appendix C. Data associated with organic analyses were evaluated using *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA 1999) as guidance. Data associated with trace elements analyses were evaluated using *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA 2004) as guidance.

Laboratory results were either accepted as received (i.e., unqualified) or were qualified. No results were rejected. Unqualified results are considered valid with respect to the specified procedures and quality control (QC) measures and may be used as intended. Results qualified as UJ and J are considered usable with the understanding that the associated values are estimates. Certain pesticide and PCB results were flagged by the laboratory as JP, indicating the result is less than the method reporting limit, and that gas chromatographic (GC) confirmation criteria were not met. These data have more associated uncertainty and should be used with caution. An explanation of data qualifiers can be found in the data validation report (Appendix C).

The principal components associated with data quality are precision, accuracy, representativeness, completeness, and comparability. In the following discussion, each of these components is addressed to assess analytical performance and data quality. Control criteria for precision and accuracy were not specified in the QAPP. Precision and accuracy are therefore evaluated using control criteria specified in the USEPA *Functional Guidelines* or established by the laboratory. Other data validation criteria, such as sample custody and holding times, are also summarized below.

Precision

Precision was evaluated using laboratory matrix duplicate samples. The measure for precision in organics analyses (pesticides, PCBs, explosives) is the relative percent difference (RPD) between matrix spike (MS) samples and matrix spike duplicate (MSD) samples. For the lipids analysis, the measures for precision are the RPD between matrix duplicate analyses and the relative standard deviation (RSD) between triplicate analyses. For trace elements, the measure for precision is the RPD between matrix duplicate analyses.

Control criteria are not available for matrix duplicate analysis of pesticides, PCBs, and explosive compounds. RPDs for pesticide matrix duplicates are well within laboratory control limits, with the exception of endrin aldehyde in three samples. Overall precision performance for pesticides is considered acceptable. RPDs for PCB matrix duplicates are well within laboratory control limits. Overall precision performance for PCBs is considered acceptable. RPDs for explosive compounds matrix duplicates are well within laboratory control limits, with the exception of one compound. Overall precision performance for explosive compounds is considered acceptable.

Control criteria are not available for matrix duplicate and triplicate analyses of lipids. RPDs for duplicate analyses ranged from 7% to 22% and are considered acceptable. RSDs for triplicate analyses ranged from 4% to 12% and are considered acceptable.

Results of trace element matrix duplicate analyses meet USEPA Contract Laboratory Program (CLP) control limits, with the exception of chromium for two samples. Associated chromium results were qualified as estimated by the data validator.

Accuracy

Accuracy was evaluated through the addition of known concentrations of analytes to sample and laboratory matrices (MS/MSDs, laboratory control samples [LCS], organic surrogate

compounds). Recovery (R) of spiked concentrations is the measure for accuracy. Overall accuracy performance is considered acceptable for all methods.

Matrix Spikes/Matrix Spike Duplicates

Several pesticide compounds in several MS/MSD samples exceeded laboratory-established control limits and were qualified as estimated by the data validator. PCB MS/MSD recoveries were acceptable. MS/MSD recoveries for the explosive compound tetryl were low; associated results were non-detects and were qualified as estimated by the data validator. Several trace elements in several MS samples were outside the control limits, and associated results were qualified as estimated by the data validator.

Laboratory Control Samples

LCS recoveries of chlordane were low in all but one extraction lot, and the associated data were qualified as estimated by the data validator. LCS recoveries for PCBs were acceptable. LCS recoveries for the explosive compound tetryl were generally low; associated results in two sample delivery groups (SDGs) were qualified as estimated by the data validator. LCS performance for trace elements is considered acceptable.

Organic Surrogate Compounds

Recoveries of organic surrogate compounds were within control limits and are considered acceptable.

Representativeness

Representativeness is a qualitative measure expressing the degree to which the data accurately and precisely represent an environmental condition. The QAPP specifies that field replicates will be used to assess representativeness. At each of the 14 sampling locations, five or six land crabs

and three fiddler crab composite samples were collected. For each location, these samples represent field replicates.

Results for PCBs and explosive compounds are non-detects and are therefore not evaluated further. To evaluate field replicates for pesticides and trace elements, the RSD was calculated for individual parameters at each location. The RSD was calculated only for parameters with reported detections at a given location of greater than 50%. Non-detects were used in the calculations at the value of the MDL. Lipids were not evaluated.

Organochlorine Pesticides

For land crab, multiple detections of individual pesticide compounds in individual sampling areas were infrequent, and few RSDs were calculated as a result. Data sufficient to calculate RSDs were generally limited to DDT, DDE, and DDD isomers. Variability as expressed by the RSD is generally high for these compounds and ranges from 25.8% to 189%; most RSDs are over 100%. Average RSDs were calculated by area and range from 25.8% to 148%.

For fiddler crab, multiple detections of individual pesticide compounds were sufficiently frequent to calculate RSDs in all sampling areas. DDT, DDE, and DDD isomers were the most frequently detected pesticides, occurring in multiple samples in individual sampling areas, although sufficient detections were available to calculate RSDs for a few other pesticides. Variability for pesticide compounds as expressed by the RSD is lower than for land crabs. RSDs range from 7.1% to 137% but are mostly in the range of 35% to 50%. Average RSDs were calculated by area and range from 33.4% to 97.8%.

Trace Elements

For both land crab and fiddler crab, multiple detections of individual trace elements were frequent, and RSDs were calculated for most trace elements and all areas sampled. Variability as expressed by the RSD is higher for land crab than for fiddler crab. Land crab RSDs range from 3.5% to 198%. Average RSDs were calculated by area and range from 30.5% to 63.6%.

Variability of trace elements in fiddler crab as expressed by the RSD is lower than for land crab. RSDs range from 0% to 92.9%. Average RSDs were calculated by area and range from 15.8% to 37.2%.

Completeness

For chemical analyses, the objective for completeness is expressed as a percentage and refers to the percentage of analytical requests for which usable analytical data are produced. Per the QAPP, the completeness objective for chemical analyses in the laboratory is 95%. All data produced by the laboratory are considered usable, as qualified. Therefore, completeness for chemical analyses is 100%.

Comparability

Comparability expresses the confidence with which one data set can be evaluated in relation to another data set. For the Vieques Island Biota Sampling Project, comparability of data was established through the use of standardized sampling and analytical methodologies.

Other Validation Criteria

Sample Custody and Holding Times

All samples were delivered to the laboratory under chain of custody. Samples were received at the laboratory at temperatures ranging from -0.7º C to 11.7º C and held frozen at -20º C until analysis. Regional guidance recommends a maximum holding time of one year. Organic extractions were performed within one year; extracts were analyzed within 40 days, per CLP guidance. Trace elements were analyzed within one year.

Calibrations

Initial calibrations were acceptable for all methods. Continuing calibrations were also acceptable for all methods, with the exception of several pesticide compounds. Associated sample results for those compounds were qualified as estimated by the data validator.

Method and Preparation Blanks

No target compounds were reported as detected in method blanks for the pesticide, PCB, or explosive compounds analyses. Target analytes were reported in some preparation blanks for the trace element analyses. Associated results are greater than five times the blank amount, with the exception of silver. Accordingly, seven silver results were qualified as "U" (undetected) by the data validator.

Target Compound Identification

For pesticides, the RPD between the primary and confirmatory column was greater than 40% for several compounds. Accordingly, the results were qualified as estimated by the data validator.

Several explosive compounds were reported as detected in one or more samples and qualified as "JN" (estimated, no confirmation analysis) by the laboratory. D.M.D., Inc., performed an independent review of the raw data and recommended that it would be more appropriate to report these tentative detections as non-detects at the associated levels. Accordingly, these results were qualified as "U" in the final data set. Additionally, the laboratory reported one positive hit for the explosive compound HMX at 1.7 mg/kg. D.M.D., Inc., noted that the target analyte response was not sufficiently resolvable from the chromatographic background on either the primary or secondary column. The reviewer recommended reporting the response on the confirmatory column and qualifying it as a non-detect (1.2 U), and this result is reported in the final data set.

References

U.S. Environmental Protection Agency (USEPA). 1999. *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*. Office of Solid Waste and Emergency Response, EPA-540/R-99-008. October.

U.S. Environmental Protection Agency (USEPA). 2004. *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*. Office of Solid Waste and Emergency Response. EPA 540-R-04-004, OSWER Directive 9240.1-45.