#### CONNECTICUT DEPARTMENT OF ENERGY AND ENVIRONMENTAL PROTECTION 19PSX0095 TECHNICAL SPECIFICATIONS APPENDIX A THROUGH F

#### 1. SCOPE

A. The technical specifications and requirements that apply to work performed under the following categories:

Salt Water Nutrient Analysis - Appendix A Surface Water (Fresh& Salt) Chemistry Analysis – Appendix B; Fish Tissue Analysis – Appendix C; Sediment Analysis - Appendix D; Toxicity Testing – Appendix E; Drinking Water, Soil, Air, Groundwater, Waste, and Sludge Analysis- Appendix F;

## 2. GENERAL CONDITIONS

A. Analytical Methods:

All laboratory methods must follow the Contractor's or subcontractors internal standard operating procedures (SOP) as applicable for the analytical methods. The Contractor shall document all analytical laboratory procedures, including the key elements of laboratory quality control, in their SOP. The Contractor shall provide copies of any and all SOPs within 30 days of a written (can be electronic) or verbal request from CTDEEP.

All work will be subject to review by CTDEEP and, therefore, analytical methods detection limits, and reporting limits must be consistent with substance specific criteria specified in the Remediation Standard Regulations Sections 22a-133k-1 to 22a-133k-3 of the R.C.S.A, State of Connecticut Water Quality Standards and State of Connecticut Public Health Code pursuant to Section 19–13-B102. Should State or Federal detection limits requirements change during the course of the program, it will be the responsibility of the Contractor to maintain the new standards.

In order for Bidders to participate in this Contract, they must be certified as per CGS 19a-29a and Sections 19-4-1, 19a-36-A25 through 19a-36-A33 inclusive, and 19a-36-A57 through 19a-36-A63 inclusive, of the R.C.S.A.

All Contractor procedures must be well documented by the appropriate SOPs, and written records must be kept of all activities to ensure consistent and reproducible data.

All analyses must be completed within the holding times specified in each method.

Some samples will contain high concentrations of contamination and result in high detection limits. In these instances, the Contractor shall conduct at least two analytical runs to attain the lowest possible detection limits.

The most recent version of all analytical methods shall be used by the Contractor.

B. Sample Collection:

The Client Agency or its representative shall be responsible for obtaining all samples, and for submitting all samples to the Contractor along with a completed chain of custody (COC) form. The Contractor may be expected to pick up the samples as arranged with the Client Agency or its representative.

The Client Agency may request that drinking water samples, treatment design samples and unknown samples be given special attention. These samples will be appropriately designated by the Client Agency or its representative on the chain of custody form. Should the Contractor analysis for drinking water samples indicate Federal Interim Primary Drinking Water Standards or Maximum Contaminant Levels (MCL) (State of Connecticut Public Health Code pursuant to Section 19–13-B102) or the attached standards are exceeded (or the most recent Action Level List published by the State of Connecticut Department of Public Health pursuant to 22a-471), the Contractor is requested to contact the Client Agency or its representative as soon as possible and perform any confirmatory analysis that may be appropriate for the Contractor.

C. The cost for the transportation of samples shall be included in the price for the analyses of the sample(s) unless the sample is collected by the Contractor.

#### 3. LABORATORY EQUIPMENT

A. Preventative Maintenance

Preventative maintenance for all equipment will be documented in the Contractor SOP and shall be performed as per manufacturer's instructions/recommendations in order to maintain equipment in good working condition and minimize downtime.

B. Laboratory Instrument Calibrations

All Contractor instrument calibrations will be addressed in, and performed according to the procedures contained in the Contractor's SOP manual. For each instrument, an instrument logbook or other document will be used to record all usage, maintenance, calibration, troubleshooting and service information.

C. Inspection of Supplies

Laboratory supplies and stock solution preparations will be documented in the Contractor SOP.

#### 4. FIELD EQUIPMENT

The Contractor shall provide the appropriate sample containers, preservatives, labels, prepared trip blanks, and water to prepare field blanks as required for the sampling program at no additional cost.

The Client Agency or its representative will supply all coolers and ice necessary for maintaining samples during field activities. It will be the responsibility of the Contractor to supply coolers and ice for sample transportation from the pick-up location as pre-arranged by the Client Agency.

#### 5. QUALITY ASSURANCE AND QUALITY CONTROL

Contractors are required to have a formal QA/QC (Quality Assurance and Quality Control) program. This program shall be documented in the form of a Quality Management Plan (QMP), however named, which describes the Contractor's QA/QC program and operation. The QMP shall outline the laboratories quality assurance policies and procedures. Contractors are also required to have formal standard operating procedures (SOP) for each method they perform. From time to time the Client Agency is required to submit formal Quality Assurance Project Plans (QAPP) for a given project to the USEPA. Contractors are required to submit information requested for such plans at no additional cost to the Client Agency.

In some cases, the CTDEEP and the USEPA will require approval of the Contractor's QAPP and SOPs. Upon request the Contractor shall submit in a timely manner, copies of the QAPP and SOP for the CTDEEP's and USEPA's review approval. The Contractor shall revise the QAPP and SOP to meet CTDEEP's and USEPA's comments. No additional charges beyond the costs for the analysis of the samples may be charged for this provision.

The detection limits, reporting limits and applicable regulatory criteria may be specified on the chain of custody form and/or request for sample analysis form by the representative of the Client Agency.

Contractor shall report any specified analytical results and QC data in a format acceptable to CTDEEP.

If any of the QA/QC criteria are not met, the Contractor shall narrate in detail the failed criteria, including which analytes and which samples are affected. This information will be presented in a clear and logical manner. When no detection of an analyte was noted, or when results for analytes were below the reporting limit, the Contractor report will indicate the result as "ND," along with the sample-specific reporting limit. Results for total or mass analysis of soil and sediments results must be reported on a dry-weight basis.

The Contractor is to notify the Client Agency or its representative of any samples where QA/QC requirements, including exceedances of holding times, are not met as a result of the Contractor's performance. The Client Agency reserves the right to have the samples recollected and analyzed at the Contractor's expense if QC requirements are not met due to the Contractor's performance. In addition, the using Client Agency also reserves the right to hold the Contractor to a turnaround time based on original sample submittal. The Contractor will also be responsible for all labor and sampling costs incurred by the Client Agency for such re-sampling.

For RCP methods, the Client Agency requires the deliverables as specified in the respective RCP methods. The most recent version of the RCPs are to be used. The RCPs and information regarding the CTDEEP's laboratory control and quality assurance program are available at: <a href="http://www.ct.gov/deep/cwp/view.asp?a=2715&q=324958&depNav\_GID=1626">http://www.ct.gov/deep/cwp/view.asp?a=2715&q=324958&depNav\_GID=1626</a> .

The RCPs establish minimum QA/QC criteria for select methods. For some analyses and projects specific data quality objectives (DQOs) and QA/QC requirements may be more stringent that the requirements of the RCPs.

For RCP methods only, in order for "Reasonable Confidence" to be achieved, the RCP Laboratory Analysis QA/QC Certification Form and required narrative must accompany each report. A copy of the RCP Laboratory Analysis QA/QC Certification Form available on the CTDEEP website must be included with sample results for RCP results. This form includes a series of questions that the Contractor must answer, and a responsible official of the laboratory must sign and date the form. Failure to include with the sample delivery group a completed, signed and dated RCP Laboratory Analysis QA/QC Certification Form, and required narrative automatically means the data set cannot be presumed to meet the requirements for Reasonable Confidence; and additional documentation will be needed from the laboratory to demonstrate that the quality control for the specific sample delivery group is at least equivalent to, or better than, that specified in the RCPs.

#### 6. SAMPLE DELIVERY/SAMPLE RECEIPT

CTDEEP may require that all samples to be treated on a "lot" basis relevant to the survey period, monthly or otherwise for tracking and reporting purposes. CTDEEP will identify which samples belong to a specific lot. In other cases, a sample lot will be limited to the samples on a chain of custody form.

For some sampling events the collected samples will be picked up by the Contractor at the CTDEEP building in Hartford. If samples are to be shipped to the Contractor, shipping costs will be the responsibility of the Contractor. If the Contractor is located in or close to Connecticut, arrangements for sample delivery by CTDEEP staff immediately following a sampling event may be acceptable. The CTDEEP may also deliver samples to the Contractor.

For delivery or pick up, the Contractor must be able to deliver/accept/pick up samples on weekdays, Monday through Friday, from 8:00 am until 8:00 pm and by appointment during other times.

If samples are to be shipped, shipping will be by overnight delivery and the Contractor must be able to take delivery of samples seven days a week.

## 7. LABORATORY TURNAROUND/REPORTING DEADLINES

- A. Contractors must be able to provide analytical reports, including batch and sample specific QA/QC within five (5) working days, from the receipt date of samples. Contractor turnaround time guidelines do not include submission of verbal results, but requires a complete analytical report to be submitted from the Contractor. This analytical report may be submitted electronically. Guidelines for an analytical report are included below in Section 8. For each sampling event, liquidated damages of up to 10% of the analytical fees may be assessed for each calendar day that turnaround times are exceeded.
- B. The Client Agency may request that preliminary or draft results in verbal or electronic format be submitted as soon as the results become available. No additional surcharge will be payable to the Contractor for preliminary or draft results submitted to the using Client Agency.
- C. The Contractor shall submit additional surcharge rates as a percentage increase over the standard 5-day turnaround. Additional rates should be submitted for the following turnaround times:
  - i. 3 day
  - ii. 48 hours
  - iii. 24 hours
  - iv. Less than 24 hours (emergency situations)
- D. Turnaround times are defined as above from the date/time of sample receipt at the Contractor through to the submission of an analytical report.
- E. Contractor will not be paid any surcharge for rapid turnaround if deadline is not achieved.

#### 8. DELIVERABLES AND ANALYTICAL REPORT INFORMATION

- A. Guidelines and formatting for analytical deliverables
  - 1. Project identification
  - 2. Project location
  - 3. Sample description
  - 4. Sample collector name

- 5. Complete site name and site address as referenced on the chain of custody form
- 6. Complete sample identification number, as referenced on chain of custody form
- 7. Complete sample collection date and time, as referenced on the chain of custody form
- 8. Date and time sample was received at the Contractor laboratory, as referenced on the chain of custody form
- 9. Temperature of samples upon receipt by Contractor, as referenced on the chain of custody form
- 10. Sample results
- 11. Detection limits
- 12. Reporting limits (flag data with a "J" for estimated values greater than the detection limit but less than the reporting limits). For RCP methods only the reporting limit is required and "J" flags are not to be included except as provided for in Appendix D.
- 13. Comparison of the analytical results to the substance specified on the Chain of Custody (COC) or Request for Sample Analysis Form ("Request for Sample Analysis Form") with any exceedances marked or bolded to highlight the results.
- 14. Date sample received, sample extracted, sample analyzed
- 15. Analytical method used for analysis
- 16. Summary of method
- 17. Laboratory control samples (blanks, spiked blanks and duplicates)
- 18. Matrix spikes, batch (concentration added, % recovery)
- 19. Copies of all original chain of custody forms and Request for Sample Analysis Form must be attached to each report.
- 20. State of Connecticut, DEEP, Laboratory QA/QC Certification Form or for all RCP analysis and RCP Laboratory Analysis Certification Form (such forms will be attached to the Contract)
- 21. Surrogate percent recoveries, where applicable
- 22. Copies of all original chain of custody and Request for Sample Analysis Form forms must be attached to each report.
- 23. State of Connecticut, DEEP, Laboratory QA/QC Certification Form or for all RCP analysis an RCP Laboratory Analysis Certification Form
- 24. Detailed narrative describing any QA/QC non-conformances signed by the Contractor's Director or Contractor Director's designee.
- 25. Other information requested by CTDEEP.
- B. The analytical report may be submitted as electronic and/or hard copy as previously arranged between using Client Agency and Contractor.
- C. Soils and sediments should be reported on a dry weight basis with the exception of parameters such as pH, and percent solids.

# 9. SAMPLE TRACKING AND CHAIN OF CUSTODY

All CTDEEP submitted samples will be uniquely identified by sample type and location, along with any other relevant companion data and chain of custody information. Each sample ID format/code will vary and may be unique from other submitted samples.

CTDEEP may submit samples using the chain of custody form provided by the Contractor or use a CTDEEP chain of custody form at its sole option. A chain of custody form will accompany all samples from collection to the analytical facility. Unique sample codes will be assigned by CTDEEP at the time of collection and recorded on the custody forms as well as on the field datasheet and on the sample container. The volume of sample filtered, filter identification number, sample weights, etc., when appropriate, will be recorded on the chain of custody forms by CTDEEP field staff.

Upon receiving samples, the Contractor shall assess and inventory each sample container. After checking of the containers is complete, the Client Agency representative and Contractor personnel shall sign at the bottom under "relinquished by" and "accepted by" on the chain of custody form , noting the date and time of signing. The Contractor will log in all samples when they take possession, and assign a unique in-house Contractor ID number to each sample. Contractor ID numbers will be recorded directly on the chain of custody forms by the Contractor staff and the completed original will be returned to the Client Agency with the analytical report for their records.

The Client Agency chain of custody form has a space provided for "analysis required". This section shall be completed by the using Client Agency or its representative unless an analysis request form is submitted attached to the chain of custody form. The Contractor is responsible for reviewing and accepting the accuracy and completeness of COC forms at the time of accepting samples. If problems arise, such as not enough sample provided to perform analysis or discrepancies of analysis to be performed, the Contractor is to contact the Client Agency or its representative within two (2) working days to resolve such issues. The Client Agency will not be responsible for any additional fees resulting from discrepancies between the chain of-custody form and laboratory analysis conducted and reporting.

Samples may be organized in "lots" by CTDEEP, and although lots may be shipped to the Contractor over the course of several days. CTDEEP requires, where possible, all samples to be treated on a "lot" basis relevant to the survey period, monthly or otherwise for tracking and reporting purposes. CTDEEP will identify which samples belong to a specific lot.

#### 10. SAMPLE ARCHIVING AND LABORATORY DATA VALIDATION

Any sample materials provided in excess of the immediate analytical needs must be properly preserved and retained by the Contractor for possible re-analysis. This material may be needed in the case of Contractor accidents or questionable results. CTDEEP acknowledges that EPA protocols set specific holding times for various sample types, but will require the Contractor to hold excess or archived sample material beyond standard holding dates and until all data are reviewed, verified and accepted by CTDEEP in writing. Upon data acceptance, or 30 days from the date of receipt of the laboratory report by the Client Agency, the Contractor will be required to notify CTDEEP in writing (which may be electronic), of their intent to destroy or dispose of sample materials. Only after the Contractor has received written approval (can be electronic) from CTDEEP to dispose of sample material may they do so. CTDEEP reserves the right to extend the holding period at their discretion. It is the responsibility of the Contractor to dispose of any remaining sample materials in accordance with State and Federal statutes and regulations.

Procedures for Contractor data validation shall be contained in the Contractor SOP. After having undergone all validation procedures at the Contractor, the data will be provided to the CTDEEP Program staff as specified in Section II Technical Specifications & Requirements Section 11 Data Reporting (see below).

#### 11. DATA REPORTING

The Contractor will provide all data in either hardcopy or electronic form per CTDEEP or other using entity request. Data should be provided in a format defined and approved by CTDEEP. The electronic form will allow for direct transfer of data into relevant program databases, format defined by CTDEEP for each program, but will use standard software such as MS Access or Excel or ASCII format, eliminating the need for any manual data entry. The submission will include a cover letter from the Contractor that includes the contents of the data package, a laboratory narrative that includes a

discussion of any QC non-conformances and problems (e.g., lab accidents, QA standards or holding times exceeded, unusual results, etc.) and the process(es) used to confirm such values (e.g., if a sample was re-analyzed, etc.) and any corrective actions made or planned. The complete data package from the Contractor will also include the QA/QC data package, which will include all QC generated, and copies of chain-of-custody forms that accompanied the samples. This complete data package will be submitted to the CTDEEP within 5-working days of the receipt of individual samples or the final samples from an individual lot. The CTDEEP may levy liquidated damages up to the cost of the sample analysis and sample collection on the Contractor will retain raw data files, including notebooks, calibration and calculation records, these documents will be made available for review by the Client Agency if requested.

Upon request of the using Client Agency, the Contractor shall also supply at no additional cost beyond the cost of the analysis, a detailed data package including the results of all laboratory quality control analyses as well as all associated raw data, used to identify and quantitate the results. This data package will be submitted in electronic or hardcopy format per the details of the request from the CTDEEP. The data request will include, but will not be limited to, initial and continuing calibrations, direct instrument outputs, preparation runs, instrument log runs, internal chain of custody documentation, and other pertinent documentation. This information must include a project narrative that details any problems or observations concerning the analysis of the samples.

## 12. DATA REVISIONS

Within 10 working days of data receipt, CTDEEP will review the data and contact the Contractor with any questions about unusual, incomplete or potentially erroneous data not explained within the data package. This 10-day response, however, will in no way constitute full acceptance of the data and any potential errors that are identified at any time during the period of an active agreement with CTDEEP will be subject for review and response to CTDEEP. The Contractor will respond promptly to questions raised by CTDEEP staff with regard to the contents of any data package. Any data revised as a result of CTDEEP and/or Contractor review shall be fully disclosed and a written justification or explanation of revision will be provided by Contractor and will become a permanent part of the data package. A complete copy of any revised data package will be provided to CTDEEP with a cover letter narrative specifying any and all changes made since previous versions.

## **13. APPENDICES**

The following appendices detail analysis requirements:

- Appendix A. Salt Water Nutrient Analysis
- Appendix B. Surface Water (Fresh& Salt) Chemistry Analysis
- Appendix C. Fish Tissue Analysis
- Appendix D. Sediment Analysis
- Appendix E. Toxicity Testing Appendix F. Drinking Water, Soil, Sediment, Air, Groundwater, Waste, and Sludge, Sample Analysis

# APPENDIX A TECHNICAL SPECIFICATIONS AND REQUIREMENTS FOR SALTWATER NUTRIENT ANALYSIS CTDEEP BUREAU of WATER PROTECTION & LAND REUSE

## 1. Background

The Contractor shall provide salt water nutrient and ancillary program analytical services. Contractor shall assign one individual as QA manager for this sample type who will be the primary contact regarding data reporting and data quality. The Contractor shall also assign a nutrient laboratory manager to be responsible for, and serve as the CTDEEP point of contact for, sample handling and custody procedures, sample analyses, and the provision of required supplies to CTDEEP.

## 2. Project Description and Schedule

## 1. Schedule of Surveys

a) Samples may be taken from both surface and bottom waters. In addition, field duplicates and field blanks can be prepared and submitted for analysis. Sample storage/preservation is by freezing (filtrates and filters) with the exception of filtrate for dissolved silicate analysis and whole water for BOD analyses, both of which are kept dark and chilled.

Field sampling will be conducted by CTDEEP. Filtration of samples for nutrient analyses will be conducted in the field and filters and filtrate samples will be delivered to the selected Contracted laboratory for analysis. The Contractor will provide all required sample bottles and filters appropriate for the analyses prior to each field survey as specified in, Appendix A, Table 1.*Estimate of Supplies*.

Collections may take place on an unscheduled basis. The Contractor shall provide additional sample bottles and filters as specified in Appendix A, Table 1 *Estimate of Supplies* at CTDEEP's request to meet these demands, as well as have the capacity to perform these analyses when requested.

#### 3. Parameters

The full parameter list for each saline sample collected, including methods and detection limits, can be found in Appendix A, Table 2 Summary of water quality sample analyses. Since BOD<sub>30</sub> analyses require a significant volume of whole water and the test must be initiated within 24 hours of collection, CTDEEP will require local delivery or pick up of those samples. Therefore, Contractors located in or near Connecticut will be considered for BOD<sub>30</sub> analyses.

## 4. Holding Times

Maximum holding times are specified in Appendix A, Table 2. Holding times are calculated from the time samples are received by the Contractor. Note BOD analyses shall be initiated immediately upon receipt of the sample (within 12 hours of receipt) and dissolved inorganic phosphorus analyses shall be initiated within 24 hours of receipt.

## 5. Analytical Methods

CTDEEP has a long-term monitoring presence on the waters of Long Island Sound and it is of utmost importance that nutrient analytical data generated are consistent with existing methods to ensure a smooth and comparable data transition. The Contractor will conduct all analyses in accordance with specified methods and detection limits, generally accepted Contractor procedures, and in keeping with their approved standard operating procedures (SOP). Appropriate QC samples will be run with each lot of samples, typically all the samples from a single cruise. Appendix A, Table 3 summarizes field and analytical methods and associated detection limits.

#### 6. Data Availability

The Contractor shall deliver a full data package for each submitted lot to the CTDEEP within 5 working days of sample receipt. The 5 working day period may be counted from the receipt of the final samples within a lot, which are typically aggregated from a single survey, which may extend over the course of 3-4 days.

# 7. Quality Criteria

Determination of accuracy will be accomplished, as will be specified in the approved Contractor SOP, by evaluating a continuing series of spiked samples. Percent recovery in the range of 85 to 115% is considered to be acceptable providing all other QC conditions are within acceptable limits. Analysis accuracy will also be assessed by analyzing commercially obtained standard reference materials. The Continuing Calibration Verification (CCV), by analysis of standard reference materials (including, when available, U.S. EPA Quality Control Solutions) must fall within the control limits of 85-115% of the true value for instrument performance to be deemed acceptable.

Determination of precision, as will be specified in the approved Contractor SOP, will be accomplished by evaluating a continuing series of replicated samples. The Relative Percent Difference (RPD) is generally used to evaluate the long-term precision of the method for each parameter. A control limit of +/- 15% RPD shall be used to define acceptable precision.

Target QA/QC goals for measured parameters are provided in Appendix A, Tables 4 and 5, along with quality assurance sample types. If a Contractor cannot meet the listed specifications for one or more parameters, they may submit a proposal specifying limits they are capable of for those parameters. Any deviations from specifications in Appendix A, Tables 4 and 5 will be subject to CTDEEP and EPA approval via the QAPP process.

CATEGORY	<u>SPECIFIC</u>	<u>PARAMETER</u>	MONTHLY <u>NUMBER</u>	ADDITIONAL <u>NUMBER</u>
Filter	47 mm diameter; 0.7um pore size; glass-fiber (GF/F); pre- weighed and uniquely numbered with record referencing weight	TSS/PP	88/month	80/summer
Filter	47 mm diameter; 0.4 <i>u</i> m pore size; polycarbonate	BioSi	88/month	80/summer
Filter	25 mm diameter; 0.7 <i>u</i> m pore size; glass-fiber (GF/F)	Chl-a	88/month	140/summer; extra 30/month Feb & Mar
Filter	25 mm diameter; 0.7 <i>u</i> m pore size; glass-fiber (GF/F); precombusted	PC/PN	88/month	80/summer
Filter	25 mm diameter; 0.5 <i>u</i> m pore size; glass-fiber (GF/F)	Chl-a	20/month	extra 20/month Feb & Mar
Sample container	50-ml poly centrifuge tube with screw lid	BioSi (filters)	80/month	80/summer
Sample container	125 ml poly bottle with lid	SiO2	40/month	40/summer

# **Table 1.** Estimate of supplies to be provided by Contractor for Saltwater Sample Analysis.

Sample container	125 ml poly bottle with lid and containing preservative (see immediately below)	H <sub>2</sub> S	N/A	15/summer
Preservative	Appropriate volume of zinc acetate-sodium hydroxide solution provided in H <sub>2</sub> S sample containers	H <sub>2</sub> S	N/A	(See immediately above) volume sufficient for 15 sample containers
Sample container	250 ml poly bottle with lid	Dissolved N,P,C series	40/month	40/summer
Sample container	2 liter (or 0.5 gallon) poly jug with lid	BOD30	25/month	N/A
	Laboratory chain-of-custody forms; each with space to record a minimum of 16 unique samples	All	4/month	6/summer

**Table 2.** Summary of sample containers, preservation, and holding times for the saltwater sample analyses.

VARIABLE	<u>SAMPLE</u> <u>TYPE</u>	SAMPLE CONTAINER	PRESERVATION METHOD	HOLDING <u>TIME</u>
Ammonia ( <b>NH</b> <sub>3</sub> ) <sup>1</sup>	Filtrate	250-ml poly bottle with lid	Sample frozen	14 days
Nitrate + Nitrite $(NO_3^-)$ + $(NO_2^-)^1$	Filtrate	250-ml poly bottle with lid	Sample frozen	28 days
Total Dissolved Nitrogen (TDN) <sup>1</sup>	Filtrate	250-ml poly bottle with lid	Sample frozen	28 days
Particulate Nitrogen (PN)	25mm 0.7 <i>u</i> m GF/F filter	aluminum foil packet; 2 duplicate filters per packet	Sample frozen	28 days
Orthophosphate ( <b>PO</b> <sub>4</sub> <sup>3-</sup> )/ Dissolved Inorganic Phosphorus ( <b>DIP</b> ) <sup>1</sup>	Filtrate	250-ml poly bottle with lid	Sample frozen	24 hours
Total Dissolved Phosphorus ( <b>TDP</b> ) <sup>1</sup>	Filtrate	250-ml poly bottle with lid	Sample frozen	28 days
Particulate Phosphorus (PP) <sup>3</sup>	47mm 0.7 <i>u</i> m GF/F filter	pre-labeled, filter-specific aluminum cup; two per sample	Sample frozen	28 days
Dissolved Organic Carbon (DOC) <sup>1</sup>	Filtrate	250-ml poly bottle with lid	Sample frozen	28 days
Particulate Carbon (PC) <sup>2</sup>	25mm 0.7 <i>u</i> m GF/F filter	aluminum foil packet; 2 duplicate filters per packet	Sample frozen	28 days
Dissolved Silica (SiO <sub>2</sub> )	Filtrate	125-ml poly bottle with lid	Sample chilled	28 days

VARIABLE	<u>SAMPLE</u> <u>TYPE</u>	SAMPLE CONTAINER	PRESERVATION METHOD	HOLDING <u>TIME</u>
Particulate (Biogenic) Silica ( <b>BioSi</b> )	47mm 0.4 <i>u</i> m polycarbonat e filter	with lid. 7 dunlicate tilters	Sample frozen	28 days
Chlorophyll <i>a</i> ( <b>Chl a</b> )	25mm 0.7 <i>u</i> m GF/F filter	aluminum foil packet; 2 duplicate filters per packet	Sample frozen	24 days
Total Suspended Solids ( <b>TSS</b> ) <sup>3</sup>	47mm 0.7 <i>u</i> m GF/F filter	pre-labeled, filter-specific aluminum cup; two per sample	Sample frozen	7 days
Biological Oxygen Demand ( <b>BOD</b> )	Whole water	1.89 liter poly jug with lid	Sample chilled	12 hours
Hydrogen Sulfide ( <b>H₂S)</b>	Whole water	125-ml poly bottle with lid	zinc acetate- sodium hydroxide solution	7 days

<sup>1</sup>A single 250-ml poly bottle of filtrate is provided per sample for the determination of several dissolved parameters.

 $^{2}$  PN and PC will both be determined from the same pre-combusted 25mm filter.

<sup>3</sup> PP and TSS will both be determined from the same 47mm filters.

**Table 3.** Summary of field and analytical methods for Saltwater Monitoring Program water quality indicatorssample analyses.

VARIABLE	FIELD METHOD	ANALYTICAL METHOD description and Method Ref. No.	DETECTION LIMIT
Ammonia ( <b>NH</b> ₃)	Filtration, generally 500mL; 47mm/0.7 <i>u</i> m GF/F; filtrate frozen	automated ion analyzer/colorimetric; EPA 350.1	0.002 mg/L
Nitrate + Nitrite $(NO_3^-) + (NO_2^-)$	Filtration, generally 500mL; 47mm/0.7 <i>u</i> m GF/F; filtrate frozen	automated ion analyzer/colorimetric; EPA 353.2	0.002 mg/L
Total Dissolved Nitrogen ( <b>TDN</b> )	Filtration, generally 500mL; 47mm/0.7 <i>u</i> m GF/F; filtrate frozen	persulfate oxidation; automated ion analyzer/colorimetric; EPA 353.2	0.040 mg/L
Particulate Nitrogen ( <b>PN</b> )	Filtration, generally 200mL; 25mm/0.7um GF/F; filter frozen	high temperature combustion; CHN elemental analyzer; EPA 440.0	0.010 mg/L
Orthophosphate (PO4 <sup>3-</sup> )/ Dissolved	Filtration, generally 500mL; 47mm/0.7 <i>u</i> m GF/F; filtrate frozen	automated ion analyzer/colorimetric; EPA 365.2	0.002 mg/L

VARIABLE	FIELD METHOD	ANALYTICAL METHOD description and Method Ref. No.	DETECTION LIMIT
Inorganic Phosphorus ( <b>DIP</b> )			
Total Dissolved Phosphorus	Filtration, generally 500mL; 47mm/0.7um GE/E: filtrate	persulfate oxidation; automated ion analyzer/colorimetric: FPA	0.002 mg/L
Particulate Phosphorus ( <b>PP</b> )	Filtration, generally 200mL; 25mm/0.7um GE/E: filter	acid (HCl) extraction; automated ion analyzer/colorimetric: EPA	0.001 mg/L
Dissolved Organic Carbon ( <b>DOC</b> )	Filtration, generally 500mL; 47mm/0.7 <i>u</i> m GF/F; filtrate frozen	high temperature combustion; non- dispersive infrared analyzer; EPA 415.1	0.5 mg/L
Particulate Carbon ( <b>PC</b> )	Filtration, generally 200mL; 25mm/0.7µm GE/E: filter	high temperature combustion; CHN elemental analyzer: FPA	0.010 mg/L
Dissolved Silica ( <b>SiO</b> <sub>2</sub> )	Filtration, generally 200mL; 47mm/0.4 <i>u</i> m polycarbonate filter; filtrate chilled	automated ion analyzer/colorimetric; EPA 370.1	0.025 mg/L
Particulate (Biogenic) Silica ( <b>BioSi</b> )	Filtration, generally 200mL; 47mm/0.4 <i>u</i> m polycarbonate filter; filter frozen	heated NaOH digestion; automated ion analyzer/colorimetric; EPA 370.1	0.010 mg/L
Chlorophyll <i>a</i> ( <b>Chl a</b> )	Filtration, generally 200mL; 25mm/0.7µm GE/E: filter	CH₃COCH₃(MgCO₃) extraction; fluorometric analysis: FPA 445.0	0.075 <i>u</i> g/L
Total Suspended Solids ( <b>TSS</b> )	Filtration, generally 500mL; 47mm/0.7µm GE/E: filter	gravimetric; EPA 160.2	1.0 mg/L
Biological Oxygen Demand ( <b>BOD</b> )	whole water sample chilled	oxygen consumption via dissolved oxygen measurements at intervals from 5 through 30 days	0.5 mg/L
Hydrogen Sulfide ( <b>H₂S)</b>	whole water sample preserved with zinc acetate- sodium hydroxide solution	Hach method 8131 producing methylene blue/colorimetric; EPA 376.2	0.002 mg/L

**Table 4.** Measurement quality objectives and quality assurance sample information for Contractor analyses.

Variable	Accuracy Goal	Precision Goal	QA Sample Type	Frequency of QA	Data Generated
Analytical Labora	ntory Measu	rements			

Ammonia ( <b>NH</b> ₃)	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision
Nitrate + Nitrite ( <b>NO</b> 3 <sup>-</sup> + <b>NO</b> 2)	85-115%	15%	Standards, spikes, lab and field duplicates; QC check against Day 0 whole water BOD sample, 11 stations	Per lot; one cruise	Relative accuracy and precision; secondary NOx measurement on fresh sample
Total Dissolved Nitrogen ( <b>TDN</b> )	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision
Particulate Nitrogen ( <b>PN</b> )	85-115%	15%	Field blanks and field duplicates	Per lot; one cruise	Precision; estimate of field contamination
Orthophosphat e ( <b>PO</b> 4 <sup>3-</sup> ) or ( <b>DIP</b> )	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision
Total Dissolved Phosphorus (TDP)	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision
Particulate Phosphorus ( <b>PP</b> )	85-115%	15%	Standards, spikes, field blanks, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision; estimate of field contamination
Dissolved Organic Carbon ( <b>DOC</b> )	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision
Particulate Carbon ( <b>PC</b> )	85-115%	15%	Field blanks and field duplicates	Per lot; one cruise	Precision; estimate of field contamination
Dissolved Silica ( <b>SiO</b> 2)	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision
Biogenic Silica ( <b>BioSi</b> )	85-115%	15%	Standards, spikes, field blanks, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision; estimate of field contamination
Chlorophyll <i>a</i> ( <b>Chl a</b> )	85-115%	15%	Standards, spikes, field blanks, field duplicates	Per lot; one cruise	Relative accuracy and precision; estimate of field contamination
Total Suspended Solids ( <b>TSS</b> )	NA	15%	Standards, field blanks and duplicates; replicates averaged	Per lot; one cruise	Precision; estimate of field contamination

Biological Oxygen Demand ( <b>BOD</b> )	NA	15%	Field duplicate	Per lot; one cruise	Precision
Hydrogen Sulfide ( <b>H₂S)</b>	85-115%	15%	Standards, spikes, lab and field duplicates	Per lot; one cruise	Relative accuracy and precision

**Table 5.** Measurement quality objectives and quality assurance sample information for Contractor analyses.

Variable	QA Sample Type	Frequency of QA
Analytical Laboratory Measu	rements	
Ammonia ( <b>NH</b> ₃)	Standards, spikes, lab and field duplicates	Minimum10% per lot
Nitrate + Nitrite ( <b>NO</b> 3 <sup>-</sup> + <b>NO</b> 2)	Standards, spikes, lab and field duplicates; QC check against Day 0 whole water BOD sample, 10 stations	Minimum10% per lot
Total Dissolved Nitrogen (TDN)	Standards, spikes, lab and field duplicates	Minimum10% per lot
Particulate Nitrogen (PN)	Field blanks and field duplicates	Minimum10% per lot
Orthophosphate ( <b>PO</b> 4 <sup>3-</sup> ) or ( <b>DIP</b> )	Standards, spikes, lab and field duplicates	Minimum10% per lot
Total Dissolved Phosphorus (TDP)	Standards, spikes, lab and field duplicates	Minimum10% per lot
Particulate Phosphorus (PP)	Standards, spikes, field blanks, lab and field duplicates	Minimum10% per lot
Dissolved Org.Carbon ( <b>DOC</b> )	Standards, spikes, lab and field duplicates	Minimum10% per lot
Particulate Carbon ( <b>PC</b> )	Field blanks and field duplicates	Minimum10% per lot
Dissolved Silica (SiO <sub>2</sub> )	Standards, spikes, lab and field duplicates	Minimum10% per lot
Biogenic Silica ( <b>BioSi</b> )	Standards, spikes, field blanks, lab and field duplicates	Minimum10% per lot
Chlorophyll <i>a</i> ( <b>Chl a</b> )	Standards, spikes, field blanks, field duplicates	Minimum10% per lot
Total Suspended Solids ( <b>TSS</b> )	Standards, field blanks and duplicates; replicates averaged	Minimum10% per lot
Biological Oxygen Demand (BOD)	Field duplicate	Minimum10% per lot
Hydrogen Sulfide ( <b>H</b> <sub>2</sub> <b>S)</b>	Standards, spikes, lab and field duplicates	Every sample

# APPENDIX B TECHNICAL SPECIFICATIONS AND REQUIREMENTS FOR SURFACE WATER (FRESH & SALT) CHEMISTRY ANALYSIS CTDEEP BUREAU OF WATER PROTECTION & LAND REUSE

1. Background

The Connecticut Department of Energy and Environmental Protection (CTDEEP) monitor's surface waters of the State on a regular basis to determine whether waters are supporting their respective designated uses. Periodically, the CTDEEP also collects samples of wastewater from permittees for analysis.

2. Anticipated Frequency of Sampling

Surface water samples are collected as needed and have an irregular distribution. Wastewater samples are also collected irregularly. The CTDEEP will notify Contractors in advance of sample collection regarding the number of samples to be analyzed, and the tests and detection/quantitation limits required. The Cost Unit Schedule includes a more complete list of potential analytes.

3. Potential List of Requested analytes/quantitation limits (Additional parameters and detection limits may be requested by the using Client Agency)

Analyte/Parameter for Surface Water	Project Quantitation Limit
Turbidity	1 NTU2
Total Dissolved Solids	1 mg/l
Total Suspended Solids	1 mg/l
Total Solids	1 mg/l
рН	0.1units
Alkalinity	10 mg/l
Hardness	10 mg/l
Chloride	2 mg/l
Fluoride	0.1 mg/l
Chlorophyll a	0.1 <b>µg/I</b>
BOD 5 day @ 20°C	1.0 mg/l
Nutrients	
Nitrate –N	0.1 mg/l
Nitrite-N	0.1 mg/l
Ammonia-N	0.1 mg/l
Organic Nitrogen	0.1 mg/l
Total Kjeldahl Nitrogen	0.1 mg/l
Total Nitrogen	0.1 mg/l
Orthophosphate	0.01 mg/l
Total Phosphate	0.01 mg/l
Low-level nutrients	
Nitrate –N	0.002 mg/l
Nitrite-N	0.002 mg/l
Ammonia-N	0.002 mg/l
Organic Nitrogen	0.002 mg/l
Total Kjeldahl Nitrogen	0.002 mg/l
Total Nitrogen	0.002 mg/l

Orthophosphate	0.002 mg/l
Total Phosphorus	0.002 mg/l
Metals	
Aluminum, Total	0.05 mg/l
Aluminum, Dissolved	0.05 mg/l
Cadmium, Total	0.5 <b>μg/l</b>
Cadmium, Dissolved	0.5 <b>μg/l</b>
Chromium, Total	0.002 mg/l
Chromium, Dissolved	0.002 mg/l
Copper, Total	0.001 mg/l
Copper, Dissolved	0.001 mg/l
Iron, Total	0.01 mg/l
Iron, Dissolved	0.01 mg/l
Lead, Total	0.001 mg/l
Lead, Dissolved	0.001 mg/l
Nickel, Total	0.002 mg/l
Nickel, Dissolved	0.002 mg/l
Zinc, Total	0.001 mg/l
Zinc, Dissolved	0.001 mg/l
Calcium	0.06 mg/l
Magnesium	0.04 mg/l
Potassium	0.1 mg/l
Sodium	0.1 mg/l
Silica complex	0.1 mg/l
Additional Analytes for	-
Wastewater monitoring	
Solids, Fixed	1 mg/l
Solids, Volatile	1 mg/l
Solids, Settleable	
COD	1mg/l
COD Total Residual Chlorine	1mg/l 5 mg/l
	1mg/l 5 mg/l 0.02 mg/l
Total Residual Chlorine	1mg/l 5 mg/l
Total Residual Chlorine Bromide	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l
Total Residual Chlorine Bromide Sulfate as SO₄	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l
Total Residual Chlorine Bromide Sulfate as SO <sub>4</sub> Sulfide as S	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l
Total Residual Chlorine Bromide Sulfate as SO <sub>4</sub> Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l
Total Residual Chlorine Bromide Sulfate as SO <sub>4</sub> Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease, hydrocarbon	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease, hydrocarbon fraction	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l 2 mg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease, hydrocarbon fraction Surfactants MBAs	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l 2 mg/l 0.025 mg/l
Total Residual Chlorine Bromide Sulfate as SO <sub>4</sub> Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease, hydrocarbon fraction Surfactants MBAs GC/MS Semivolatiles (625)	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l 2 mg/l 0.025 mg/l 1 - 40 μg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease, hydrocarbon fraction Surfactants MBAs GC/MS Semivolatiles (625) GC/MS Volatiles (624)	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l 2 mg/l 0.025 mg/l 1 - 40 μg/l 1 - 10 μg/l
Total Residual Chlorine Bromide Sulfate as SO₄ Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease Oil & Grease, hydrocarbon fraction Surfactants MBAs GC/MS Semivolatiles (625) GC/MS Volatiles (624) Total Phenols	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l 2 mg/l 0.025 mg/l 1 - 40 μg/l 1 - 10 μg/l 0.005 mg/l
Total Residual Chlorine Bromide Sulfate as SO <sub>4</sub> Sulfide as S Cyanide (CN) Total Cyanide amenable to chlorination Formaldehyde Oil & Grease Oil & Grease Oil & Grease, hydrocarbon fraction Surfactants MBAs GC/MS Semivolatiles (625) GC/MS Volatiles (624) Total Phenols Pesticides	1mg/l 5 mg/l 0.02 mg/l 0.1 mg/l 5 mg/l 1 mg/l 0.005mg/l 0.005mg/l 1 mg/l 2 mg/l 2 mg/l 0.025 mg/l 1 - 40 μg/l 1 - 10 μg/l 0.005 mg/l 2 - 40 μg/l

Barium	0.002 mg/l
Beryllium, Total	0.001 mg/l
Boron, Total	0.005 mg/l
Chromium, Total (Hex)	0.01 mg/l
Cobalt	0.005 mg/l
Manganese	0.01 mg/l
Molybdenum	0.01 mg/l
Mercury	0.2 <b>μg/l</b>
Selenium	0.005 mg/l
Silver, Total	0.002 mg/l
Vanadium	0.005 mg/l

Some additional sampling may be required in surface waters for fresh water cyanobacteria in Connecticut waterways. There are toxins that can be released by the cyanobacteria and both identification and cell counts for cyanobacteria in a sample will be requested. Identification of these blue-green algae will be required to the species level.

In addition to the cell counts and identification CT DEEP will also require quantification of the following toxins: microcystin, anatoxin-a, clyindrospermopsin, saxitoxins, and lipopolysaccharides. These results should be from analysis in water by Enzyme-Linked Immunosorbent Assays and liquid chromatography mass spectrometry methods. Other methods may be suggested however the Client Agency reserves the right to refuse analytical methodologies beyond those listed here.

4. Requirements: The following are a list of requirements for each Contractor. SOP, QAPP, and QA/QC documents must be available for submission to CTDEEP as described below. The contractor shall meet the QA/QC requirements listed below.

- (1.) Method to be used: method number and a brief description (*e.g.*, U. S. EPA 365.3, Automated Colorimetric). Refer to 40 CFR, Part 136 for acceptable methods. Also see attached spreadsheet for price listing.
- (2.) Laboratory\_SOP for each method must be available for submission to CTDEEP. Guidance for writing an analytical SOP is available at <u>http://www.epa.gov/QUALITY/qs-docs/g6-final.pdf</u> (U.S. EPA 2001). Contractor sample storage and handling protocols must be addressed in the Contractor SOP.
- (3.) Quality Assurance and Quality Control (QA/QC) procedures must be available for submission upon request. At minimum, QA/QC procedures must include:
  - Initial and routine instrument calibration
  - Routine calibration checks
  - Documentation of detection and quantitation limits
  - Documentation of accuracy and precision control limits (measurement performance criteria)
  - Routine assessment of accuracy and precision. (Type <u>and</u> frequency of quality control samples (*e.g.*, standards, laboratory blanks, duplicates, matrix spikes). Note that a field blank and field duplicate will usually be provided with each batch of samples submitted by CTDEEP.
  - Routine monitoring of interferences and contamination
  - Corrective action when control limits are exceeded

For each type of quality control sample utilized by the Contractor, please specify the sample type/definition (*e.g.*, duplicate, spiked matrix, reagent blank, *etc.*), the purpose of its use (*e.g.*, calibration, precision check, *etc.*), the frequency of the QC analysis, the recommended quality control limits, and the corrective action employed when control limits are exceeded.

- (4.) Please provide an example of description of documentation and reporting and/or a sample data package. Data packages should be provided in both hard copy and Access-ready electronic format (MS Access, Excel or ASCII). Documentation for each sample should at minimum include:
  - A cover letter providing a brief description of the procedures and instrumentation used, including the procedures used to calculate Method Detection Limits ("MDLs"), as well as a narrative explanation of any analytical problems, departures from protocols, or failures to meet required quality control limits or holding times.
  - M DL's & Laboratory Reporting Limit as defined in the Reasonable Confidence Protocols available on the DEEP website at http://www.ct.gov/deep/cwp/view.asp?a=2715&g=324958&deepNav\_GID=1626
  - Dates sample collected, received by Method Detection Limits, extracted (if applicable) and analyzed
  - Analytical method used
  - Results from control samples (*e.g.*, blanks, duplicates, matrix spikes)
  - All data and control results should be checked for accuracy and the hard copy report signed by the Method Detection Limits manager or designee.
  - The original chain of custody and Request for Sample Analysis Form
  - State of CT, DEEP, Laboratory QA/QC Certification Form
- (5.) The type and size of sample container or media (*e.g.,* filter) to be provided by the Request for Sample Analysis Form for each parameter or group of parameters (*e.g.,* dissolved metals) (see Attachment I Unit Cost Schedule to this Appendix).
- (6.) The cost per analysis, and any reduced rates for large numbers of samples or analysis of a suite of parameters (*e.g.*, for all metals) (see Exhibit B) to this Appendix).
- (7.) The cost of any sample preparation not included in analysis cost (*e.g.*, digestion, extraction, or filtration) (see Exhibit B to this Appendix).
- 5. References:
  - CTDEEP. 2002. Water Quality Standards. Connecticut Department of Energy and Environmental Protection, 79 Elm Street, Hartford, CT.
  - Federal Register. October 23, 2002. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Methods Update. Volume 67, Number 205.
  - U.S. EPA. March 2001. Guidance for Preparing Standard Operating Procedures (SOPs). U.S. EPA/240/B-01/004. United States Environmental Protection Agency, Office of Environmental Information, Washington, DC.

## APPENDIX C FISH TISSUE ANALYSIS CTDEEP BUREAU of WATER PROTECTION & LAND REUSE

# 1. Background

The Connecticut Department of Energy and Environmental Protection (CTDEEP) in cooperation with the Connecticut Department of Health (CT DPH) periodically samples fish populations to determine levels of contaminants in fish tissue. The CT DPH issues consumption advisories for "high risk" and "low risk" populations based on results from these analyses. The CTDEEP will provide frozen fillets of fish to be analyzed. The CTDEEP will notify Contractor(s) in advance of sample collection regarding the number of samples to be analyzed, and the tests required. Contractor shall prepare homogenates of fish tissue as well as conducting the chemical analyses.

- 2. Potential requested Fish Tissue Analyses (wet weight)
  - Metals, including but not limited to Arsenic (inorganic), Cadmium, Mercury, Selenium, Tribuyltin, and Pesticide screening
  - Chlorinated Pesticides
  - PCBs: Congeners, Homologs & Aroclor specific
  - Dioxin / furans
  - PBDEs
  - PAHs
  - Percent lipids
  - Percent solids
  - Any additional parameters requested by the Client Agency

# 3. Requirements

Contractor shall provide the following information listed below:

- (1.) For preparation of tissue homogenates, please specify:
  - A. SOP for preparing individual and composite tissue homogenates.
  - B. The type of blender/homogenizer/grinder to be used and the type of blades/probes (titanium or tantalum blades/probes are required).
  - C. Storage and handling procedures.
  - D. QA/QC procedures.
  - E. The cost per sample, per analyte for an individual fish, and for a 5-fish composite.
- (2.) For each analysis, please provide
  - A. Method to be used: method number and a brief description.
  - B. The Contractor SOP to be used. The recommended contents of an SOP for tissue analysis (U.S. EPA 1995) are as follows. Failure to include the following may result in rejection of any bid:
    - Scope and application
    - Method performance characteristics (accuracy, precision, method detection and quantitation limits) for each analyte
    - Interferences
    - Equipment, supplies and materials
    - Sample preservation, holding times and handling procedures
    - Instrument calibration procedures
    - Quality control procedures
    - Corrective action procedures
    - Data reduction and analysis procedures (with example calculations)
    - Record-keeping procedures (with standard data forms if applicable)
    - Safety procedures and/or cautionary notes
    - Disposal procedures

- References
- (3.) Provide documentation regarding QA/QC procedures.
  - A. The minimum requirements for Quality Assurance and Quality Control should include the following (U.S. EPA 1995). Failure to include the following may result in rejection of any bid. Initial demonstration of Contractor capability:
    - Instrument calibration
    - Documentation of detection and quantitation limits
    - Documentation of accuracy and precision control limits
    - Analysis of an accuracy-based performance evaluation sample
    - Ongoing demonstration of acceptable Contractor performance:
      - Routine calibration and calibration checks
      - Routine assessment of accuracy and precision
      - Routine monitoring of interferences and contamination
  - B. For each type of QA/QC control sample utilized by the Contractor, please specify the sample type/definition, the objective for its use (e.g., calibration), the frequency of the analysis, the recommended control limits, and the corrective action employed when control limits are exceeded. (See Table 8-6 in U.S. EPA 1995 for an example).
- (4.) Please provide an example: description of documentation and reporting and/or a sample data package. Documentation for each sample should include the following (U.S. EPA 1995). Failure to include such documentation may result in rejection of any bid.
  - Study identification (e.g., project number, title)
  - Procedure description, including documentation and justification for any deviation from the standard procedure
  - Method detection and quantitation limits for each analyte
  - Method accuracy and precision for each target analyte
  - Discussion of any analytical problems and corrective action taken
  - Sample identification number
  - Sample weight (wet weight & dry weight)
  - Final dilution volume/extract volume
  - Date(s) of analysis
  - Identification of analyst
  - Instrument(s) used (manufacturer, model number, location)
  - Summary calibration data, including identification of calibration materials, dates of calibration and calibration checks, and calibration range(s); for Gas Chromatography/
  - Mass Spectrometry (GC/MS) analyses, include Decafluorotriphenylphosphine (DFTPP) spectra and quantitation report
  - Reconstructed ion chromatograms for each sample analyzed by Gas Chromatography/Electron Capture Device (GC/ECD) and/or Gas Chromatography/Flame Ionization Detector (GC/FID)
  - Raw data quantitation report for each sample
  - Description of all QC sample associated with each sample (e.g., reference materials, field blanks, method blanks, and spiked samples) and results of all QC analyses. QC reports should include quantitation of all target analytes in sample summaries. Contractor s should report all surrogate and matrix spike recovery data for each sample; the range of recoveries should be included in any reports using these data.
  - Analyte concentrations with reporting units identified (as ppm or ppb wet and dry weight, to two significant figures unless otherwise justified. NOTE: Reported data should not be recovery-or blank-corrected.

- Lipid content (as percent of wet weight)
- Specification of all tentatively identified compounds (if requested) and any quantitation data
- Data qualifications (including qualification codes and their definitions, if applicable, and a summary of data limitations).
- State of CT, DEEP, Laboratory QA/QC Certification Form
- (5.) The cost per analysis, plus any reduced rates available based on a suite of parameters (*e.g.*, all metals), or a volume of samples (see Exhibit B to this Appendix).
- (6.) The cost of any sample preparation not included in the analysis cost or cost of homogenate preparation (*e.g.*, digestion, extraction). (see Unit Cost Schedule file)
- 4. References:
  - U.S. EPA. September 1995. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1, Fish Sampling and Analysis, Second Edition. U.S. EPA 823-R-95-007. United States Environmental Protection Agency, Office of Water.
  - U.S. EPA. May 2001. National Coastal Assessment Coastal 2000. Quality Assurance Project Plan -2000. U.S. EPA/620/R-01/002. United States Environmental Protection Agency, Office of Research & Development, Washington, DC.
  - NOAA. 1989. Standard Analytical Procedures of the NOAA National Analytical Facility. 2<sup>nd</sup> ed. NOAA Tech.Mem. NMFS F/NWC-92, 1985-86. National Status and Trends Program, National Oceanic and Atmospheric Administration, US Department of Commerce, Rockville, MD.

# APPENDIX D SEDIMENT ANALYSIS CTDEEP BUREAU OF WATER PROTECTION & LAND REUSE

# 1. Background

The CTDEEP periodically samples sediments of water bodies to determine levels of contaminants. The CTDEEP will notify Contractor in advance of sample collection regarding the number of samples to be analyzed, and the tests required.

2. Potential Sediment Analyses (dry weight)

Aluminum Antimony Arsenic (inorganic) Cadmium Chromium Copper Iron Lead Manganese Mercury Nickel Selenium Silver Tin Zinc Simultaneously Extracted Metals (SEMS) Pesticides screening **Chlorinated Pesticides** PCBs: Congeners, Homologs & Aroclor specific Dioxin / furans PBDEs PAHs **Total Organic Carbon** Acid Volatile Sulfide **Butyltins** Grain Size

#### 3. Requirements

For the indicated parameters, Contractor shall provide the following information:

- A. Method to be used: method number and a brief description, and the Contractor SOP. Guidance for preparation of an analytical SOP is available from <u>http://www.epa.gov/QUALITY/qs-docs/g6-final.pdf</u> (U.S. EPA 2001a). Laboratory sample storage and handling protocols must be addressed in the Contractor SOP.
- B. Data Validation Based on Solids Percentage and Sample Modification to Increase % Solids All sediment data may be accepted when the percent solids is greater than 30 %.
  When sediment samples whose percent solids is less than or equal to 30 % and greater than or equal to 10 %, all positive results are to be considered estimated and are qualified with a "J". For sediment samples whose percent solids is less than 10 %, all positive results are rejected and qualified with an "R". For all sediment samples where percent solids is less than or equal to 30 %, all non-detected results are rejected and qualified with an "R".

When samples with low % solids are collected, larger total amounts of material will be obtained and submitted to utilize appropriate levels of actual sediment for analysis specific parameters.

De-watering of samples is another possible strategy to obtain adequate sample amounts. The Contractor shall be aware of and able to utilize the following options for de-watering of sediment samples:

- decanting excess water,
- centrifuging at high speeds,
- vacuum filtration,
- air drying samples in a hood overnight,
- oven drying samples at low temperatures,
- freeze drying samples,
- rigorous multiple extractions of many aliquots of homogenized sample.

When sampling for volatile organic compounds, de-watering is not possible because the volatile compounds would be lost in the process. The need to implement any de-watering procedures shall be determined in consultation with CTDEEP on a sample by sample basis and consider the % solids in the sample provided to the Contractor and the potential effect of procedures to increase % solids on the analytical results for such sample.

- C. Quality Assurance and Quality Control (QA/QC) procedures. At minimum, QA/QC procedures shall include the following items. Failure to include the following may result in rejection of Contractor bid. (see also Appendix A of U.S. EPA 2001b).
  - Initial and routine instrument calibration
  - Routine calibration checks
  - Documentation of detection and quantitation limits
  - Documentation of accuracy and precision control limits (measurement performance criteria)
  - Routine assessment of accuracy and precision
  - Routine monitoring of interferences and contamination

- Regular analysis of a reference material or Contractor control material.
- D. Provide an example of the following: Description of documentation and reporting and/or a sample data package. Documentation for each sample lot or batch should at minimum include:
  - A cover letter providing a brief description of the procedures and instrumentation used, including the procedures used to calculate MDLs, as well as a narrative explanation of analytical problems (if any), departures from protocols, or failures to meet required quality control limits or holding times.
  - Tabulated results in hard copy and Access-ready electronic format (MS Access, Excel or ASCII), including any concentrations of the analytes of interest. The results should be checked for accuracy and the hard copy report signed by the Contractor manager or designee.
  - Tabulated method detection limits achieved for samples.
  - Results for all QA/QC samples (*e.g.*, calibration check samples, blanks, matrix spike, matrix spike duplicates), the batch number linking the batch of samples to appropriate QA/QC samples. (Only data that have met QA requirements should be submitted by the Contractor. When QA requirements have not been met, the samples should be reanalyzed and the reanalysis results submitted.)
  - State of CT, DEEP, Laboratory QA/QC Certification Form
- E. Containers for each analysis to be provided by Contractor.
- F. The cost per analysis, plus any reduced rates available based on a suite of parameters (*e.g.*, all metals), or a volume of samples. (see Unit Cost Schedule file)
- G. The cost of any sample preparation not included in the analysis cost or cost of homogenate preparation (*e.g.*, digestion, extraction). (see Unit Cost Schedule file)
- 4. References
  - U.S. EPA. March 2001a. Guidance for Preparing Standard Operating Procedures (SOPs). U.S. EPA/240/B-01/004. United States Environmental Protection Agency, Office of Environmental Information, Washington, DC.
  - U.S. EPA. May 2001b. National Coastal Assessment Coastal 2000. Quality Assurance Project Plan 2001-2004. United States Environmental Protection Agency, Office of Research & Development, Washington, DC.

# APPENDIX E TOXICITY TESTING CTDEEP BUREAU OF WATER PROTECTION & LAND REUSE

#### 1. Background

The CT DEEP will occasionally need acute and chronic toxicity studies conducted to monitor environmental conditions or evaluate various releases to the environment, including but not limited to the development of National Pollutant Discharge Elimination System (NPDES) permits and the determination of compliance with permit toxicity limits. Data from these tests can also be used to predict potential acute and chronic toxicity in the receiving water. The CT DEEP could also use toxicity test results during the remediation process or as part of site characterizations and as part of permit requirements that are evaluated during compliance evaluation inspections and performance audit inspections.

#### 2. Requirements

Contractor shall detail the types of tests and testing organisms that are likely to be used:

(A)Methods to be used: The type of test and test organisms will be requested specifically by the CTDEEP and NPDES requirements.

CTDEEP requires Contractor to follow the guidelines and standards for acute tests detailed in EPA's *Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition.* Contractor shall also follow modifications for NPDES permits and if applicable, section 22a-430-3 a&j of the Regulations of CTDEEP. No samples collected for toxicity will be dechlorinated, filtered, pH adjusted or manipulated in any way unless specified by the CTDEEP. This goes for all toxicity tests acute and chronic freshwater and marine chronic.

CTDEEP requires Contractor to follow the guidelines and standards for chronic tests on freshwater organisms detailed in EPA's Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition..

CTDEEP requires Contractor to follow the guidelines and standards for chronic testing of marine and estuarine organisms detailed in EPA's *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, Third Edition.* 

CTDEEP requires Contractor to follow the guidelines and standards for sediment toxicity tests detailed in EPA's Methods for Measuring the toxicity and bioaccumulation of sediment associated Contaminants to Freshwater Invertebrates, 2<sup>nd</sup> edition EPA/600/R-99/064 and in Methods for Measuring the toxicity and bioaccumulation of sediment associated Contaminants with estuarine and Marine Amphipods EPA/600/R-94/025 and Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.- Testing Manual (Draft): Inland Testing Manual EPA823-B-94-002.:

CTDEEP requires Contractor to follow the guidelines and standards for Toxicity Identification Evaluation detailed in *Methods for Aquatic Toxicity Identification Evaluations: Phase 1 Toxicity Characterization Procedures, 2<sup>nd</sup>Edition EPA/600/6-91/003.* 

The methods included in the above manuals are referenced in Table IA, 40 CFR Part 136 regulations and constitute approved methods for chronic and acute toxicity tests.

(B)Quality Assurance and Quality Control (QA/QC) procedures. QA practices within an aquatic toxicology Contractor must address all activities that affect the quality of the final effluent toxicity data, such as: (1) effluent sampling and handling; (2) the source and condition of the test organisms; (3) condition and operation of equipment; (4) test conditions; (5) instrument calibration; (6) replication; (7) use of reference toxicants; (8) record keeping; and (9) data evaluation. (EPA 2002)

(C)Provide an example of the following: Description of documentation and reporting and/or a sample data package. Documentation for each test result shall at minimum include:

- A cover letter providing a brief description of the procedures and instrumentation used, including a narrative explanation of analytical problems (if any), departures from protocols, or failures to meet required quality control limits organism specifications.
- The following list should also be completed and included with any submitted materials: Toxicity Test method used, test endpoint, volume of solution per container, number of organisms per container, feeding frequency and type, name of organism, age, life stage, and source of organisms. (EPA 2002)
- Bench sheets shall include survival at 0, 24, 48, 72, and continued every 24 hours, general chemistry conducted during the test, DO, pH, temperature, conductivity, and salinity. Concentrations run for the test in each container shall also be included. Bench sheets shall also include supplemental chemistry, TRC, alkalinity, hardness, temperature on arrival, sample collection time and test start and end times, comments on health or behavior of organisms in the test, or physical conditions in the test beaker such as floc, sediment, color, fibrous materials etc.
- Reference toxicant
- Tabulated results in hard copy form including the concentrations of effluent used as samples, LC<sub>50</sub>, and % mortality in each sampled concentration or the values for NOAEC (no-observed-adverse-effect-concentration), LOAEC (lowest-observed-adverse-effect-concentration), depending on the objectives of the study. Any graphs or charts of data and statistical analysis should also be included in the report. The results should be checked for accuracy and the report signed by the Contractor manager or designee.
- A description of any statistical analysis used to calculate endpoints of test.
- Any explanation for outlier results in the data set should be detailed and statistical analysis should be run both with and without the outlier value.
- 3. The cost per test procedure, plus any reduced rates available based upon multiple use of the same species of test organisms, or repeats or duplicates of effluent samples.
- 4. The cost of any sample preparation including any costs for culturing organisms for CTDEEP toxicity studies.

- 5. References
  - US EPA October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. US EPA-821-R-02-012. United States Environmental Protection Agency, Office of Water, Washington, DC.
  - US EPA October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition. US EPA-821-R-02-013. United States Environmental Protection Agency, Office of Water, Washington, DC.
  - US EPA October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms, Third Edition. US EPA-821-R-02-014. United States Environmental Protection Agency, Office of Water, Washington, DC.
  - USEPA, March 2000. Methods for Measuring the toxicity and bioaccumulation of sediment associated Contaminants to Freshwater Invertebrates, 2<sup>nd</sup> edition EPA/600/R-99/064 United States Environmental Protection Agency, Office of Water, Washington, DC.
  - USEPA, 1994. Methods for Measuring the toxicity and bioaccumulation of sediment associated Contaminants with estuarine and Marine Amphipods EPA/600/R-94/025 United States Environmental Protection Agency, Office of Water, Washington, DC.
  - USEPA, ACOE .Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S.- Testing Manual (Draft): Inland Testing Manual EPA 823-B-94-002 United States Environmental Protection Agency, Office of Water, Washington, DC.
  - USEPA, May 1992. Methods for Aquatic Toxicity Identification Evaluations. Phase I Toxicity Characterization Procedures, 2<sup>nd</sup> Edition EPA/600/6-91/005F United States Environmental Protection Agency, Office of Research and Development, Washington DC.
  - Federal Register, Volume 55, Number 671, p 11798, Hazardous Waste Management System Identification and Listing of Hazardous Waste Toxicity Characteristics Revision, Final Rule, March 29, 1990.

# DRINKING WATER, SOIL, SEDIMENT, AIR, GROUNDWATER, WASTE, AND SLUDGE SAMPLE ANALYSIS

#### 1. Background:

The CTDEEP occasionally needs to evaluate and measure levels of contamination in drinking water, soil, sediments, air and groundwater. Additional testing of hazardous waste determinations and hazardous materials analysis may be needed. Hazardous material analysis could include testing roofing material for asbestos among several other materials. Data from these samples may be used to determine site specific conditions, disposal options and planning of remediation efforts.

#### 2. Requirements:

Contractor shall detail the types of tests and testing organisms that are likely to be used:

(A) Method used: The CTDEEP will submit a list of parameters to be analyzed along with samples of soil or groundwater. Contractor SOPs must be submitted upon request.

(B) Quality Assurance and Quality Control (QA/QC) procedures: For all soil and groundwater samples Contractor will be expected to at minimum follow the guidelines detailed in section 5 of the Technical Specifications & Requirements found on page 2.

(C) Data Reporting: Documentation for each test result should at minimum include:

- Tabulated method detection limits achieved for samples.
- The original chain of custody and Request for Sample Analysis Form
- The dates of sample collection, sample arrival at lab, sample analysis, and report creation
- For RCP methods only, a State of a CT DEEP, QA/QC Certification Form or completed RCP QA/QC form for Contractor \ analysis (<u>http://www.ct.gov/deep/cwp/view.asp?a=2715&q=324958&depNav\_GID=1626</u>)

(D) Related costs: Contractors should include the cost per analysis, plus any reduced rates available based on a suite of parameters (*e.g.*, all metals), or a volume of samples. Also include, the cost of any sample preparation not included in the analysis cost or cost of homogenate preparation (*e.g.*, digestion, extraction).

- 3. References:
  - US EPA July 1992. *Test Methods for Evaluating Solid Waste, Third Edition (or most recent). SW-846*, United States Environmental Protection Agency, Office of Waste, Washington, DC.
  - Connecticut Department of Energy and Environmental Protection, *Reasonable Confidence Protocols*, for various analytical methods, most recent versions.

- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient, Air Second Edition, Compendium Method TO-13A Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using Gas Chromatography/Mass Spectrometry (GC/MS), Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency Cincinnati, OH 45268, January 1999.
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-14A Determination Of Volatile Organic Compounds (VOCs) In Ambient Air Using Specially Prepared Canisters With Subsequent Analysis By Gas Chromatography, Center for Environmental Research Information Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, January 1999
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-15, Determination Of Volatile Organic, Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), Center for Environmental Research Information Office of Research and Development U.S. Environmental Protection Agency Cincinnati, OH 45268, January 1999
- "Test Methods for Evaluating Solid Waste", USEPA SW846 November 1992; Third Edition (or most recent version)
- "Methods for Chemical Analysis of Water and Wastes", USEPA, EPA 600/4-79-020, revised March 1983 (or most recent version).
- Standard Methods for the Examination of Wastewater, 16th Edition.
- Federal Register, Volume 55, Number 671, p. 11798, Hazardous Waste Management System Identification and Listing of Hazardous Waste Toxicity Characteristics Revision, Final Rule, March 29, 1990.
- American Society for Testing and Materials Standards (ASTM).