## Section B5. Quality Control Requirements

# LABORATORY QUALITY CONTROL REQUIREMENTS

SWAMP will require all participating laboratories to demonstrate capability continuously through:

1. Strict adherence to common QA/QC procedures.
2. Routine analysis of certified reference materials (CRMs).
3. Regular participation in an on-going series of interlaboratory comparison exercises.

Because SWAMP is specifically designed to provide information on "ambient" conditions in the state's surface waters, the ability to provide low-level contaminant analysis is critical. This is a "performance-based" approach for measurements of low-level contaminant analyses, involving continuous laboratory evaluation through the use of accuracy-based materials (e.g., CRMs), laboratory matrix spikes, laboratory method blanks, calibration standards, laboratory- and field-duplicated samples, and others as appropriate. The definition and use of each of these types of quality control samples are explained further below.

Quality control operates to make sure that data produced are satisfactory, consistent, and dependable. Under SWAMP's performance-based chemistry QA program, laboratories are not required to use a single, standard analytical method for each type of analysis, but rather are free to choose the best or most feasible method within the constraints of cost and equipment that is suitable for meeting the data quality objectives (DQO's), as outlined in **Appendix C** (Data Acceptability Criteria tables). SWAMP has developed specific guidelines for measurement precision, accuracy, and levels of detection that are reflected in sampling, handling, and analysis requirements to satisfy a large spectrum of potential management questions. Each laboratory will continuously demonstrate proficiency and data comparability through routine analysis of accuracy-based performance evaluation samples, split samples, and reference materials representing actual sample matrices. No single analytical method has been officially approved for low-level analysis of organic and inorganic contaminants in water or sediments. Recommended methods are available and are provided in **Appendix C's** Target Reporting Limits section (listing of recommended methods).

All laboratories providing analytical support for chemical or biological analyses will have the appropriate facilities to store, prepare, and process samples, and appropriate instrumentation and staff to provide data of the required quality within the time period dictated by the project. Laboratories are expected to conduct operations in a way that includes:

1. A program of scheduled maintenance of analytical balances, microscopes, and other laboratory equipment and instrumentation.
2. Routine checking of analytical balances using a set of standard reference weights (American Society of Testing and Materials (ASTM) Class 3, NIST Class S-1, or equivalents).
3. Checking and recording the composition of fresh calibration standards against the previous lot. Acceptable comparisons are <5 percent difference from previous value.

4. Recording all analytical data in bound (where possible) logbooks, with all entries in ink, or electronic format.
5. Monitoring and documenting the temperatures of cold storage areas and freezer units once per week.
6. Verifying the efficiency of fume hoods.
7. Having a source of reagent water meeting ASTM Type I specifications (ASTM, 1984) available in sufficient quantity to support analytical operations. The resistivity of the reagent water will not exceed 18 megaohm at 25°C. Alternately, the conductivity of the reagent water will exceed 10 μmhos/cm.
8. Labeling all containers used in the laboratory with date prepared, contents, initials of the individual who prepared the contents, and other information as appropriate.
9. Dating and safely storing all chemicals upon receipt. Proper disposal of chemicals when the expiration date has passed.
10. Having QAPP, SOPs, analytical methods manuals, and safety plans readily available to staff.
11. Having raw analytical data, such as chromatograms, accessible so that they are available upon request.

Laboratories will be able to provide information documenting their ability to conduct the analyses with the required level of data quality. Such information might include results from interlaboratory calibration studies, control charts and summary data of internal QA/QC checks, and results from certified reference material analyses.

**QA/QC Documentation**

All laboratories will have the latest revision of the SWAMP QAMP. In addition, the following documents and information will be current, and they will be available to all laboratory personnel participating in the processing of SWAMP samples, as well as to SWAMP project officials:

1. Laboratory QA Plan: Clearly defined policies and protocols specific to a particular laboratory, including personnel responsibilities, laboratory acceptance criteria and corrective actions to be applied to the affected analytical batches, qualification of data, and procedures for determining the acceptability of results.
2. Laboratory Standard Operating Procedures (SOPs): Containing instructions for performing routine laboratory procedures.
3. Laboratory Analytical Methods Manual: Step-by-step instructions describing exactly how a method is implemented in the laboratory for a particular analytical procedure. Contains all analytical methods utilized in the particular laboratory for SWAMP.
4. Instrument Performance Information: Information on instrument baseline noise, calibration standard response, analytical precision and bias data, detection limits, etc. This information is usually recorded in logbooks or laboratory notebooks.

5. Control Charts: Control charts are useful in evaluating internal laboratory procedures and are helpful in identifying and correcting systematic error sources. Contract laboratories are encouraged to develop and maintain control charts whenever they may serve in determining sources of analytical problems.

**Recommended Typical Laboratory Performance Measurements**

Typical laboratory performance measurements included in the analysis stream and designed to check if data quality criteria are met are recommended and briefly defined below. SWAMP **Data Acceptability Criteria are provided for all analytical groups for all media in Appendix C. Note that not all media may have all of these performance measurements (See App C).**

1. Method Blanks (also called extraction blanks or preparation blanks): These account for contaminants present in the preservative and analytical solutions and equipment used during the preparation and quantification of the parameter.
2. Injection Internal Standards and/or Surrogates: These account for error introduced by the analytical instrument or extraction process.
3. Matrix Spike Samples: These are field samples to which a known amount of contaminant is added and used to measure potential analytical interferences present in the field sample.
4. Replicate Samples: These are replicates of extracted material that measure the instrumental precision.

   a. Laboratory Replicate Samples: These are replicates of the raw material that are extracted and analyzed to measure laboratory precision.

   b. Matrix Spike Replicate Samples: These are used to assess both laboratory precision and accuracy. They are particularly useful when the field samples analyzed do not contain many of the target compounds (measuring non-detects in replicate does not allow the data reviewer to measure the precision or the accuracy of the data in an analytical batch).
5. Certified Reference Materials (CRM): Analysis of CRMs is another way of determining accuracy of the analysis by comparing a certified value of material with similar concentrations as those expected in the samples to be analyzed.

These types of samples serve to check if errors were introduced during the analysis process and if so, at what step(s) and at what magnitude. The remainder of this document will provide RMP guidance for general laboratory requirements and protocols for checking and tracking possible sources of errors (outlined above) in the analytical process.

**Laboratory Quality Control Procedures**

The performance-based protocols utilized in SWAMP for analytical chemistry laboratories consist of two basic elements: initial demonstration of laboratory capability for new laboratories (e.g., documentation that the analyses of samples are within the data quality criteria), and

ongoing demonstration of capability. Prior to the initial analysis of samples, each new laboratory will demonstrate capability and proficiency.

## INITIAL DEMONSTRATION OF CAPABILITY

### Instrument Calibration

Upon initiation of an analytical run, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended DQOs (see **Appendix C**), the system will be calibrated with a full range of analytical standards. Immediately after this procedure, the initial calibration must be verified through the analysis of a standard obtained from a different source than the standards used to calibrate the instrumentation, prepared in an independent manner, and ideally having certified concentrations of target analytes of a certified reference material (CRM) or certified solution. Frequently, calibration standards are included as part of an analytical run, interspersed with actual samples. However, this practice does not document the stability of the calibration and is incapable of detecting degradation of individual components, particularly pesticides, in standard solutions used to calibrate the instrument. The calibration curve is acceptable if it has a $r^2$ of 0.990 or greater for all analytes present in the calibration mixtures. If not, the calibration standards, as well as all the samples in the batch must be re-analyzed. All calibration standards will be traceable to a recognized organization for the preparation and certification of QA/QC materials (e.g., NIST, National Research Council Canada (NRCC), US EPA, etc.).

Calibration curves will be established for each analyte and batch analysis from a calibration blank and a minimum of three analytical standards of increasing concentration, covering the range of expected sample concentrations. Only data which result from quantification within the demonstrated working calibration range may be reported by the laboratory (i.e., quantification based on extrapolation is not acceptable). Alternatively, if the instrumentation is linear over the concentration ranges to be measured in the samples, the use of a calibration blank and one single standard that is higher in concentration than the samples may be appropriate. Samples outside the calibration range will be diluted or concentrated, as appropriate, and reanalyzed.

### Initial Documentation of Method Detection Limits

Analytical chemists have coined a variety of terms to define "limits" of detectability; definitions for some of the more commonly used terms are provided in Keith *et al*. (1983) and in Keith (1991). In SWAMP, the method detection limit (MDL) is used to define the analytical limit of detectability. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition:

> "The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte."

The American Society of Testing and Materials (ASTM) defines the limit of detection as:

> "A concentration of twice the criterion of detection...when it has been decided that the risk of making a Type II error is to be equal to a Type I error."

In order to compare MDLs in quantitative terms by different laboratories participating in SWAMP analyses, MDLs will initially be determined according to 40 CFR 136.2 (f) and Appendix B of 40 CFR 136. Determining the MDL with this procedure is elaborate and need not be determined annually provided that:

1. No process or method changes have been made.
2. Check samples containing an analyte spike at about 2x MDL indicate that the sample is detected. The required frequency of check samples is quarterly.

The matrix and the amount of sample (i.e., dry weight of sediment or tissue) used in calculating the MDL will match as closely as possible the matrix of the actual field samples and the amount of sample typically used.

**Limits of Quantification**

In order to ensure comparability of results among different laboratories, recommended Reporting Limit (quantification) values have been established for SWAMP, termed **Target Reporting Limits, or TRL's (see Appendix C)**. These TRL's have been derived empirically. In most cases, they are 2-5 times the MDL as determined by the above process. Most are considerably lower than water quality objectives or sediment and tissue quality guidelines and provide the foundation for having a high level of certainty in the data.

The laboratory shall confirm the ability to analyze low-level samples with each batch. This shall be accomplished by analyzing a method blank spiked at 3 to 5 times the method detection limit or a reference material in the appropriate range. Recoveries for organic analyses shall be between 50 and 150% for at least 90% of the target analytes.

Taylor (1987) states that "a measured value becomes believable when it is larger than the uncertainty associated with it". The uncertainty associated with a measurement is calculated from the standard deviation of replicate measurements ($s_0$) of a low concentration standard or a blank. Normally, the MDL is set at three times the standard deviation of replicate measurements, as it is at this point that the uncertainty of a measurement is approximately ±100% at the 95% level of confidence. The limit of quantification (LOQ, which SWAMP is referring to as the Reporting Limit, or RL), as established by the American Chemical Society, is normally ten times the standard deviation of replicate measurements, which corresponds to a measurement uncertainty of ±30% (see Taylor, 1987). By these standard definitions, measurements below the MDL are not believable, measurements between the LOQ (RL) and the MDL are only semi-quantitative, and confidence in measurements above the LOQ is high.

**Initial Blind Analysis of Representative Samples**

As appropriate, representative sample matrices which are uncompromised, homogeneous, and contain the analytes of interest at concentrations of interest will be used to evaluate performance of analytical laboratories new to SWAMP prior to the analysis of field samples. The samples

used for this initial demonstration of laboratory capability typically will be distributed blind (i.e., the laboratory will not know the concentrations of the analytes of interest) as part of the SWAMP interlaboratory comparison exercises, with the SWAMP QA Program staff conducting and evaluating the exercise. Based on results that have typically been attained by experienced laboratories, a new laboratory's performance generally will be considered acceptable if its submitted values are within the DQO's **(outlined in Appendix C)** of the known concentration, or the consensus value, of each analyte of interest in the samples. These criteria apply only for analyte concentrations equal to or greater than three times the RL. If the results for the initial analysis fail to meet these criteria, the laboratory will be required to repeat the analysis until the performance criteria are met, prior to the analysis of SWAMP field samples.

### Record of Certified Reference Material

As CRMs are routinely included in analysis of batches of reputable laboratories, the historical record of results may also serve as a suitable performance indicator.

## ONGOING DEMONSTRATION OF CAPABILITY

### Participation in Interlaboratory Comparison Exercises

Through an interagency agreement, NOAA's NIST Program and EPA's EMAP program jointly sponsor an on-going series of interlaboratory comparison exercises (round-robins). All SWAMP analytical laboratories are at this point encouraged to participate in these intercomparison exercises, which are conducted jointly by NIST and NRCC. In the near future, this most likely will become a mandatory participation, with approval from NOAA/NIST. SWAMP would then be conducting its own annual interlaboratory calibration exercise for media types not covered within the NOAA/NIST intercalibration (primarily water media), and it will be mandatory for all participating SWAMP labs. These exercises provide a tool for continuous improvement of laboratory measurements by helping analysts identify and resolve problems in methodology and/or QA/QC. The results of these exercises are also used to evaluate both the individual and collective performance of the participating analytical laboratories on a continuing basis and to insure that ongoing measurements are meeting Data Acceptability Criteria. The SWAMP laboratories will be required to initiate corrective actions if their performance in these comparison exercises falls below pre-determined minimal standards.

It is planned for there to be one exercise conducted over the course of a year. In a typical exercise as planned for SWAMP, the 3rd party (referee) contractor will distribute performance evaluation samples of an "unknown" and a certified reference material (CRM) to each laboratory, along with detailed instructions for analysis. A variety of performance evaluation samples could be utilized, including accuracy-based solutions, sample extracts, and representative matrices (e.g., sediment or tissue samples). Laboratories are required to analyze the sample(s) "blind" and will submit their results in a timely manner to the SWAMP interlaboratory calibration study coordinator (as instructed). Laboratories which fail to maintain acceptable performance may be required to provide an explanation and/or undertake appropriate corrective actions. At the end of each calendar year, coordinating personnel at the 3rd party

(referee) contract QA Program will participate in a QA workshop to present and discuss the comparison exercise results. Additionally, a written summary of the evaluation will be provided.

**Routine Analysis of Certified Reference Materials or Laboratory Control Materials**

Certified reference materials generally are considered the most useful QC samples for assessing the accuracy of a given analysis (i.e., the closeness of a measurement to the "true" value). CRMs can be used to assess accuracy because they have "certified" concentrations of the analytes of interest, as determined through replicate analyses by a reputable certifying agency using two independent measurement techniques for verification. In addition, the certifying agency may provide "non-certified or "informational" values for other analytes of interest. Such values are determined using a single measurement technique, which may introduce unrecognized bias. Therefore, non-certified values must be used with caution in evaluating the performance of a laboratory using a method which differs from the one used by the certifying agency.

A laboratory control material (LCM) is similar to a certified reference material in that it is a homogeneous matrix that closely matches the samples being analyzed. A "true" LCM is one that is prepared (i.e., collected, homogenized, and stored in a stable condition) strictly for use in-house by a single laboratory. Alternately, the material may be prepared by a central laboratory and distributed to others (so-called regional or program control materials). Unlike CRMs, concentrations of the analytes of interest in LCMs are not certified but are based upon a statistically valid number of replicate analyses by one or several laboratories. In practice, this material can be used to assess the precision (i.e., consistency) of a single laboratory, as well as to determine the degree of comparability among different laboratories. If available, LCMs may be preferred for routine (i.e., day to day) analysis because CRMs are relatively expensive.

Routine analysis of CRMs or, when available, LCMs represents a particularly vital aspect of the "performance-based" SWAMP QA philosophy. At least one CRM or LCM must be analyzed along with each batch of 20 or fewer samples (i.e., QA samples should comprise a minimum of 5% of each set of field samples). For CRMs, both the certified and non-certified concentrations of the target analytes will be known to the analyst(s) and will be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs or LCMs **(Appendix C)**; these criteria are discussed in detail in the following paragraphs. If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM or LCM, the data for the entire batch of samples is suspect. Calculations and instruments will be checked; the CRM or LCM may have to be reanalyzed (i.e., reinjected) to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before final data are reported. The results of the CRM or LCM analysis will never be used by the laboratory to "correct" the data for a given sample batch.

**Precision criteria:** Precision is the reproducibility of an analytical method. Each laboratory is expected to maintain control charts for use by analysts in monitoring the overall precision of the CRM or LCM. Upper and lower control chart limits (e.g., warning limits and control limits) will

be continually updated; control limits based on 99% confidence intervals around the mean are recommended. The relative standard deviation (RSD) will be calculated for each analyte of interest in the CRM based on the last 7 CRM analyses. Acceptable precision targets for various analyses are listed in **Appendix C**.

**Laboratory Replicates for Precision**

A minimum of one field sample per batch of SWAMP samples submitted to the laboratory will be processed and analyzed in duplicate or more for precision. The relative percent difference among replicate samples (RPD expressed as percent) will be less than the DQO's listed in **Appendix C** for each analyte of interest. Following are the calculations:

Each measured value is compared against the known value of the standard, and accuracy is expressed as the relative percent difference.

$$RPD = \frac{[V_m - V_k]}{V_k} \times 100\%$$

Where: RPD = the relative percent difference
Vm = the measured value,
Vk = the known value.

If results for any analytes do not meet the DQO's for the RPD, calculations and instruments will be checked. A repeat analysis may be required to confirm the results. Results which repeatedly fail to meet the objectives indicate sample in-homogeneity, unusually high concentrations of analytes or poor laboratory precision. In this case, the laboratory is obligated to halt the analysis of samples and eliminate the source of the imprecision before proceeding.

**Accuracy criteria:** The “absolute” accuracy of an analytical method can be assessed using CRMs only when certified values are provided for the analytes of interest. However, the concentrations of many analytes of interest to SWAMP are provided only as non-certified values in some of the more commonly used CRMs. Therefore, control limit criteria are based on “relative accuracy”, which is evaluated for each analysis of the CRM or LCM by comparison of a given laboratory’s values relative to the “true” or “accepted” values in the LCM or CRM. In the case of CRMs, this includes both certified and noncertified values. The “true” values are defined as the 95% confidence intervals of the mean.

Based on typical results attained by experienced analysts in the past, accuracy control limits have been established both for groups of compounds (**Appendix C**).

**Continuing Calibration Checks (CCC's)**

Calibration check solutions traceable to a recognized organization must be inserted as part of the sample stream. The source of the calibration check solution shall be independent from the standards used for the calibration. Calibration check solutions used for the continuing calibration checks will contain all the analytes of interest. The frequency of these checks is

dependent on the type of instrumentation used and, therefore, requires considerable professional judgment. All organic analyses shall be bracketed by an acceptable calibration check. **Appendix C** provides specific frequencies and other criteria for CCC's. If the control limits for analysis of the calibration check solution (set by the laboratories) are not met, the initial calibration will have to be repeated. If possible, the samples analyzed before the calibration check solution that failed the DQO's in **Appendix C** will be reanalyzed following recalibration. The laboratory will begin by reanalyzing the last sample analyzed before the calibration check solution which failed. If the RPD between the results of this reanalysis and the original analysis exceeds precision DQO's (**Appendix C**), the instrument is assumed to have been out of control during the original analysis. If possible, reanalysis of samples will progress in reverse order until it is determined that the RPD between initial and reanalysis results are within DQO's (**Appendix C**). Only the re-analysis results will be reported by the laboratory. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control check) are suspect. In this case, the laboratory will flag the data and prepare a narrative explanation to accompany the submitted data.

### Laboratory Method Blank

Laboratory method blanks (also called extraction blanks, procedural blanks, or preparation blanks) are used to assess laboratory contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one laboratory method blank will be run in every sample batch. The method blank will be processed through the entire analytical procedure in a manner identical to the samples. Method blank criteria are provided in **Appendix C**. If eliminating the blank contamination is not possible, all impacted analytes in the analytical batch shall be flagged. In addition, a detailed description of the contamination source and the steps taken to eliminate/minimize the contaminants shall be included in the transmittal letter. Subtracting method blank results from sample results is not permitted.

### Completeness

Completeness is defined as “a measure of the amount of data collected from a measurement process compared to the amount that was expected to be obtained under the conditions of measurement” (Stanley and Verner, 1985). Field personnel will always strive to achieve or exceed the SWAMP completeness goals of 90% (85% for fish, clam, and mussel tissues) for water, sediment, or biota (biological assessment) samples.

### Surrogates

The usage of the terms “surrogate”, “injection internal standard”, and “internal standard” varies considerably among laboratories and is clarified here.

Surrogates are compounds chosen to simulate the analytes of interest in organic analyses. Surrogates are used to estimate analyte losses during the extraction and clean-up process and must be added to each sample, including QA/QC samples, prior to extraction. The reported concentration of each analyte is adjusted to correct for the recovery of the surrogate compound, as done in the NOAA NS&T Program. The surrogate recovery data will be carefully monitored; each laboratory must report the percent recovery of the surrogate(s) along with the target analyte

data for each sample. If possible, isotopically-labeled analogs of the analytes will be used as surrogates.

Each laboratory will set its own warning limit criteria based on the experience and best professional judgment of the analyst(s). It is the responsibility of the analyst(s) to demonstrate that the analytical process is always "in control" (i.e., highly variable surrogate recoveries are not acceptable for repeat analyses of the same certified reference material and for the matrix spike/matrix spike duplicate). The warning limit criteria used by the laboratory will be provided in the standard operating procedures submitted to SWAMP.

Data will be reported as surrogate-corrected values.

### Internal Standards (if they are used)

For gas chromatography (GC) analysis, internal standards (also referred to as "injection internal standards" by some analysts) may be added to each sample extract just prior to injection to enable optimal quantification, particularly of complex extracts subject to retention time shifts relative to the analysis of standards. Internal standards are recommended if the actual recovery of the surrogates added prior to extraction is to be calculated. The internal standards can also be used to detect and correct for problems in the GC injection port or other parts of the instrument. The compounds used as internal standards will be different from those already used as surrogates. The analyst(s) will monitor internal standard retention times and recoveries to determine if instrument maintenance or repair, or changes in analytical procedures, are indicated. Corrective action will be initiated based on the judgment of the analyst(s). Instrument problems that may have affected the data or resulted in the reanalysis of the sample will be documented properly in logbooks and internal data reports and used by the laboratory personnel to take appropriate corrective action.

### Dual-Column Confirmation

Dual-column chromatography is required for analyses using GC-ECD due to the high probability of false positives arising from single-column analyses.

### Matrix Spike and Matrix Spike Duplicate

A laboratory fortified sample matrix (commonly called a matrix spike, or MS) and a laboratory fortified sample matrix duplicate (commonly called a matrix spike duplicate, or MSD) will be used both to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest and to provide an estimate of analytical precision. Frequencies and specifications for MS and MSD's are provided in **Appendix C**. A field sample is first homogenized and then split into three subsamples. Two of these subsamples are fortified with the matrix spike solution and the third subsample is analyzed to provide a background concentration for each analyte of interest. The matrix spike solution should contain as many representative analytes from the SWAMP analyte list as feasible. The final spiked concentration of each analyte in the sample will be at least 5 times the MDL for that analyte, as previously calculated by the laboratory. Additionally, the total number of spikes should cover the range of expected concentrations.

Recovery is the accuracy of an analytical test measured against a known analyte addition to a sample. Recovery is calculated as follows:

$$\text{Recovery} = \frac{(\text{Matrix plus spike result} - \text{Matrix result}) \times 100}{\text{Expected matrix plus spike result}}$$

Recovery data for the fortified compounds ultimately will provide a basis for determining the prevalence of matrix effects in the samples analyzed during the project. If the percent recovery for any analyte in the MS or MSD is less than the recommended warning limit, the chromatograms (in the case of trace organic analyses) and raw data quantitation reports will be reviewed. If an explanation for a low percent recovery value is not discovered, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interferences and further instrument response checks may not be warranted, especially if the low recovery occurs in both the MS and MSD, and the other QC samples in the batch indicate that the analysis was "in control". An explanation for low percent recovery values for MS/MSD results will be discussed in a cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response will be included. Analysis of the MS/MSD is also useful for assessing laboratory precision. The RPD between the MS and MSD results should be less than the target criterion listed in **Appendix C** for each analyte of interest.

## FIELD QUALITY CONTROL REQUIREMENTS

**Travel Blanks** - The purpose of the travel blank is to determine if there is any cross-contamination of volatile constituents between sample containers. One VOA sample vial (volatile organic analytes= MTBE, BTEX, and VOC's), with deionized (DI) water free of volatile contaminants, is transported to the site, handled like a sample (but never opened up), and returned to the lab for analysis. One travel blank for each batch of VOA samples shipped to the laboratory is required. Travel blanks are not required for other analytes, but are encouraged to be utilized for other analytes as possible and appropriate.

**Equipment Blanks (done in lab prior to field work) -** To ensure that equipment used during sampling does not contaminate samples, the device is filled with DI water or DI water is pumped through the device, transferred to sample bottle(s), preserved (if appropriate) and analyzed by the lab. Equipment blanks are run when new equipment, equipment that has been cleaned after use at a contaminated site, or equipment that is not dedicated for surface water sampling, is used. An equipment blank is prepared for metals in water samples whenever a new lot of filters is used.

**Field Duplicates** - Duplicate samples will be collected for all parameters (including toxicity and bioassessment samples) at an annual rate of 5% of total samples to be collected within a given year's Work Plan. The duplicate sample will be collected in the same manner and as close in time as possible to the original sample. This effort is to attempt to examine field homogeneity as

well as sample handling, within the limits and constraints of the situation.

**Field Blanks** - A field blank is designed to assess potential sample contamination levels that could occur during field sampling and sample processing. Field Blanks (DI water) are taken to the field, transferred to the appropriate container, preserved (if appropriate), and otherwise treated the same as the corresponding sample type during the course of a sampling event. Field blanks are to be collected at a 5% rate for the following constituents: trace metals in water (including mercury), VOA samples in water and sediment, DOC samples in water, and bacteria samples. Field blanks for other media and analytes should be conducted upon initiation of sampling, and if field blank performance is acceptable, further collection and analysis of field blanks for these other media and analytes need only be performed on an as-needed basis, or during field performance audits.