9 INORGANIC ANALYSIS, PERFORMANCE QUALITY CONTROL AND ANALYTICAL OPERATION

9.1 Introduction

This chapter presents the analytical operations and procedures used in this laboratory by the Inorganic Chemistry Section (ICS) to measure and record analytical performance. This chapter is intended to include elements of quality control, introduced in Chapter 5 of this manual, in more detail and provide general guidance for inorganic analytical chemistry operations. Specific methods and standard operating procedures provide further detail for QC requirements and generally take precedence over other guidance.

9.2 General

9.2.1 Guidance During the course of generating data on samples for inorganic parameters, it is the policy of the Inorganic Chemistry Section to 1) apply the best laboratory practices 2) use approved methodology when mandated by regulation and use standardized methodology, if possible 3) when approved methodology is not applicable, fully document all operations associated with the generation of data and 4) meet certain quality requirements that will be designated in the following paragraphs. It should be noted, however, that occasionally certain matrices and samples present analytical challenges, or are not amenable to standardized methodology. In these instances modifications to standard protocols may have to be made to produce a high quality analysis. When this occurs, any deviations from standard operating procedures will be documented.

9.2.2 Methods It is the practice of the ICS to use widely accepted methodology if possible and approved methods when mandated. The method versions of established methods are identified in the associated R4LIMS project file. Current Standard Operating Procedures (SOPs) and forms used by the ICS are located on the Region 4 SESD's local network drive (K: drive) in appropriate subdirectories of K:\ASB\SOPs\ICS and K:\ASB\Forms\ICS\ respectively. These are the most current approved procedures and forms. Hard copies of SOPs and any supporting memos documenting approval are retained by the ICS Chief in bound manuals referred to as "Inorganic Chemistry Section Compilation of SOPs for Metals" and "Inorganic Chemistry Section Compilation of SOPs for Nutrients/Classicals." Methods listed in this chapter are for commonly requested parameters.

9.2.2.1 Method Development or Modifications Any new instrumentation installation, development of methods, or major modifications will be performed or guided by Lead Analysts or the Section Chief in consultation with the Branch Quality Assurance Officer (QAO). At a minimum, verification consists of 1) optimizing instruments and methods to perform analyses within the specific method guidelines or QC guidelines of this quality assurance manual 2) performing a demonstration of capability (DOC) and method detection limit

(MDL) (Note: some analyses, such as TVSS, are not amenable to a typical DOC or MDL) and 3) documenting the process and storage of documentation by the Section Chief. The DOC is reviewed and approved by the Section Chief and the QAO. Progression of method development and modifications shall be well documented. The analyst should test matrices and interferences anticipated being relevant to the test method. As a final step, the analyst shall develop a standard operating procedure (SOP) consistent with the format used by the ASB. The SOP is reviewed and approved by the Section Chief, QAO, and Branch Chief. When the method has appropriate approval, a memo will be issued by the analyst to the Section Chief concerning the date of implementation of the new method and any other pertinent information.

9.2.3 Safety Safety precautions associated with the safe handling of toxic chemicals, reagents, solutions and samples will be observed and regarded as a first order responsibility of the analyst. The analyst will take the necessary precautions to prevent exposure or harm both to self and coworkers.

9.2.4 Water Water used to prepare calibration standards, spike solutions, standard reference solutions or any sample dilutions or mixtures must meet or exceed the requirements for Type I grade water as specified by the American Society for Testing and Materials (ASTM); Standard Practice D 1193. This grade water is equivalent to Type I water as specified in Standard Methods 1080. The parameter measured to verify the quality of water is resistivity, with a requirement of 18 megohm-cm at 25°C or better. See also section 2.2 of "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," (EPA 600/4-79-019, March l979), and any future updates of the manual.

9.2.5 Reagents and Standards Reagents must be ACS reagent grade quality or better. Reagents and standards will be dated upon receipt and will be properly disposed of when the shelf life has been reached (see Chapter 4). Working solutions and mixtures made from stock reagents and standards will be used only for the appropriate working life of the solution. In general, this is from one week to no more than six months for materials that tend to degrade. In the absence of specific guidelines for working reagents and standards, the primary analyst may choose to prepare these materials fresh each time the analysis is performed or use the sample holding times as a guide for the useful working life. For other materials that do not degrade easily (e.g., simple salts, solutions for metals analyses) the shelf life may be much longer.

9.2.6 Records of Repair and Maintenance Records for the repair and maintenance of instruments shall be maintained within the Section. The preference is to keep these records with the instrument in a bound log and for the working life of the instrument, consistent with the EPA policy on records retention.

9.3 <u>Reviewing/Verification/Recording</u>

9.3.1 Results will be reviewed/verified by another analyst (secondary analyst) qualified to perform the analysis. The primary analyst shall to the best of his/her ability correct all mistakes and resolve all questionable issues before the results are submitted for verification. The review should at a minimum check to see that all required documentation is included with the raw data, proper QC protocols were followed, documentation of any excursions from analysis requirements (e.g., QC acceptability, method, etc.), and a check for math errors. The check for math errors may be from minimal (for routine analyses, a check of one or more calculations) to 100% verification at the discretion of the secondary analyst. Verification of the data shall be recorded (date and initials of the secondary analyst) on a checklist. Additionally, the Section Chief may perform spot checks of raw data on randomly selected projects. These spot checks are documented on a checklist and filed with the raw data in the project file. Current versions of checklists can be found on the Region 4 SESD's local network drive (K: drive) in appropriate subdirectories of K:\ASB\Forms\ICS\. A general data review guideline for inorganics is found in the Appendix of this chapter which can be found on the Region 4 SESD's local network drive (K: drive) in appropriate subdirectories of K:\ASB\QA Manual - Current Version\.

9.4 General QC

9.4.1 Guidance Quality control results are used for making decisions about analyses. Analysts have guidelines available for accepting, rejecting, and flagging results. These include, but are not limited to acceptance limits based on method requirements or historical data, guidelines developed in-house for the area of interest, and method specific guidelines. Method guidance takes precedence. In the absence of method guidance, use other guidance found in Sections 9.6 and 9.7 of this chapter (general guidance can be found in Chapter 5 of this manual).

9.4.2 QC Requirements Concentrations of elements and compounds are quantified in samples by comparing results of analysis with those of known materials. This is typically done by generating a calibration curve or using calibration standards. The calibration itself is verified by analyzing materials of known concentrations that are from a different source (referred to as "second source"). The following is a general strategy that may be followed to ensure elements of quality control are addressed. More detailed requirements are presented in sections 9.6 and 9.7. Additional quality control measures may be added as necessary.

9.4.2.1 For analyses that include sample preparation (extraction, distillation, digestion, etc.):

9.4.2.1.1 Include a method blank and laboratory control sample(LCS) in the preparation to assess method performance. The LCS should be of a matrix similar to the samples and from a different source than that of the calibration standards. An LCS prepared in reagent water is acceptable for analysis when no LCS materials are available for other matrices. For some parameters,

there are no LCS materials available or some methods may not be amenable to an LCS analysis (e.g., TCLP extraction).

9.4.2.1.2 Prepare the LCS in duplicate for the purpose of determining precision. Follow any additional method specific requirements for duplicates.

9.4.2.1.3 Prepare a matrix spike, where appropriate or required by method, to assess the effect of sample matrix on the method performance.

9.4.2.2 Perform a calibration**.**

9.4.2.3 Check the calibration with a reference material (second source as available). This is the check standard.

9.4.2.3.1 If the samples were treated differently than the calibration standards (e.g., samples were digested, but standards were not), the calibration should be checked with a second source material that is treated the same as the calibration standards (no digestion as in the previous example).

9.4.2.3.2 If the samples were treated the same as the calibration standards (e.g., samples and standards were digested), the LCS may double as the calibration check. If the LCS is prepared from laboratory standards, they should be from a different source and/or lot than the standards used to prepare the calibration curve.

9.4.2.4 Continue to check the calibration periodically (the frequencies are defined by analyses) as well as at the conclusion of the run. This is the continuing calibration verification. This can be done with any material of known concentration such as a calibration standard, check standard, or LCS. **If more than one CCV is required, the concentration should be varied. Other QC measures may double for this requirement (e.g., the MQL check may also be used as a low-end CCV).**

9.4.3 Special Circumstances Occasionally, the primary analyst will encounter a situation that is not addressed by available guidelines. The analyst should consult the Lead Analyst or Section Chief in these cases. When possible, decisions will be based on the data quality objectives of the project. For example, if while performing duplicate analyses one result is above the MQL and the other is below (but above the MDL), it may be determined that the MQL is well below the regulatory level or study level of concern and thus the average of the two results will be reported. If study requirements determine that results at the MQL are of importance, the primary analyst may be required to rerun the analyses to clarify or confirm the results.

9.4.4 Results Failing QC Criteria In the absence of method or lab specific guidelines (in the SOPs), the following is a discussion of general guidelines for the Section.

9.4.4.1 Calibration A correlation coefficient (r) of 0.995 or greater is acceptable for calibration curves. If this criterion is not met, the curve must be repeated. If the criterion is again not met, the primary analyst must find the source of the problem before proceeding with analyses.

9.4.4.2 Check Standard (referred to as Calibration Check in some methods) Acceptance criteria for check standards are specified by method or SOP. The general default when criteria are not specified by method or SOP is ± 15%. When acceptance criteria are not met, the check must be repeated. If the criteria are again not met, the primary analyst must find the source of the problem before proceeding with analyses.

9.4.4.3 LCS In the absence of current acceptance limits, use as guidance the best available estimation of limits from established methods or other sources. Judgements on data quality (e.g., adding qualifier flags, etc.) will not be made solely on the basis of these estimated limits until such time as acceptance limits are appropriately determined. In these instances consult the Section Chief (or designee) and Branch QAO for guidance. Where there are specified limits and these criteria are not met, the LCS and associated batch of samples must be repeated. If the criteria are again not met, the primary analyst must find the source of the problem before proceeding with analyses. When troubleshooting, it is acceptable to confine repeats to the LCS (excluding samples) until the primary analyst has confidence that the samples may be repeated with success.

9.4.4.4 LCS Duplicates In the absence of current acceptance limits, use as guidance the best available estimation of limits from established methods or other sources. Judgements on data quality (e.g., adding qualifier flags, etc.) will not be made solely on the basis of these estimated limits until such time as acceptance limits are appropriately determined. In these instances consult the Section Chief (or designee) and Branch QAO for guidance. Where there are specified limits and these criteria are not met, the LCS dups and associated batch of samples must be repeated. If the criteria are again not met, the primary analyst must find the source of the problem before proceeding with analyses. When troubleshooting, it is acceptable to confine repeats to the LCS (excluding samples) until the primary analyst has confidence that the samples may be repeated with success.

9.4.4.5 Repeats Under normal circumstances, repeats are performed one time only for failure to meet matrix QC criteria. If QC fails on the repeats, data is reported with the appropriate flag(s). Occasionally, repeats may need to be performed more than once to locate the reason for QC failure.

9.4.4.6 QC results that approach the limits of acceptance criteria *may* be an indication of a decline in the quality of the analysis. The primary analyst should try to locate the source of the problem and correct it.

9.4.5 DOC Before a new method may be used for sample analysis, a DOC must be performed according to the method or any requirements set forth in Chapter 5. Method guidelines may be used if the requirements of Chapter 5 are also met. Note: some analyses, such as TVSS, may not be amenable to a DOC.

9.4.6 Continuing MDL Studies MDL studies are performed on an annual basis for the Drinking Water Program. These studies are typically performed once a year and may be performed in conjunction with one of the water supply performance test (WS PT) projects. This ensures the MDL studies are performed routinely and that they are performed on the same instrumentation that is used for the PT studies. Only drinking water parameters are required for these continuing studies (an initial MDL study is more thorough). Although some methods mention performing these studies more frequently, this is guidance and not required by the program. Since these studies are used only for drinking water certification, the ICS has chosen to perform them on an annual basis.

9.4.7 Holding Times

9.4.7.1 Holding times begin when the sample is taken. The ICS has chosen to follow the recommendation that holding time starts at the end of the composite period for composite samples (August 1994 memo from Thomas Clarke, Director, EMSL, Cincinnati ("Inorganic Chemistry Section Compilation of SOPs for Classicals/Nutrients").

9.4.7.2 Results are considered to be within holding times if the preparation or analysis is begun within the recommended period of time. Holding times for aqueous samples are defined in 40 CFR Part 136, Table II by parameter. General guidelines for solid matrices regulated by the RCRA program are defined in Table 3-1 in SW-846 ("Test Method for Evaluating Solid Waste," EPA 1982) for metals. See the summary figure for holding times by parameter in Chapter 3 of this manual. Holding times specified in terms of hours will be evaluated based on the hour of collection. Holding times specified in terms of days will be evaluated based on the day of collection. If analyses are performed outside defined recommended holding times, the results will be "J" flagged and an appropriate remark added to the report.

9.5 <u>Reporting Analytical Results</u>

9.5.1 Less Than Values If the concentration of an analyte is < MQL, report the MQL and flag the result as "U" (e.g., if the MQL is 50 mg/L and the analyte is not detected at or above this level, report 50 U mg/L).

9.5.2 Significant Figures See Chapter 5. Two significant figures are reported for all parameters except pH (report three significant figures). Special requests for other than two significant figures will be considered on a case by case basis.

9.5.3 Rounding See Chapter 5.

9.5.4 Reporting Results for sample analyses are reported through R4LIMS. At a minimum, records within the software include data results, the identification of the method used, and the secondary analyst responsible for verifying the results. Two production copies (one original and one file copy) of the report are submitted to the Section Chief (or designee) for review and release. Three production copies are required if the report is to be delivered outside the Division. After projects are branch released, reports are delivered to the Branch Secretary for distribution.

9.5.5 Raw Data Corrections and Changing Reported Data Corrections to raw data require initials (or signature) and date. The original incorrect data should be legible through the striking line. Corrections will be made to the original. Copies will reflect the original with corrections. When corrections that change the reported value are made after project reports have been branch released in R4LIMS, new production copies and memos will be made with appropriate instructions to replace the previously reported data with the corrected data.

9.6 Metals

9.6.1 General Metals analyses are performed in support of various agency programs. Some programs mandate methods (e.g., Drinking Water at 40CFR Part 141.1 ff. and NPDES at 40 CFR Part 136), while others publish methods strictly as guidance (e.g., RCRA except for the Characteristic Tests in Subpart C of 40CFR Part 261) Subject to the restrictions in 9.2.1, mandated methodology will be used for those analyses requiring them. Guidance methods will be closely adhered to with the possibility of changes which are documented in the SOPs. In the case of one-time use methods, documentation of the procedure will be included in the project file along with the raw-data. Methods and procedures are documented with the data results in R4LIMS.

9.6.1.1 Different programs have quality standards or fail levels that are of interest or that may trigger an action. Figure 9-1 lists these various levels for common analytes and programs.

9.6.2 Drinking Water

9.6.2.1 Regulatory Authority National Primary Drinking Water Regulations are found at 40 CFR Part 141. National Secondary Drinking Water Regulations are found at 40 CFR Part 143. In general these regulations apply to Public Water Systems which are defined as "a system for the provision to the public of water for human consumption through pipes or, after August 5, 1998, other constructed conveyances, if such system has at least fifteen service connections or regularly

serves an average of at least twenty-five individuals daily at least 60 days out of the year" (40 CFR Part 141.2). Historically, this laboratory has analyzed few samples from public water systems as the states have been delegated the authority for monitoring public water supplies within their boundaries. However, this laboratory often analyzes samples from individual private potable wells. Therefore, while not legally obligated to adhere to the requirements of 40 CFR Part 141 for these samples, this lab has chosen to follow the requirements in Part 141 whenever possible when analyzing private potable wells.

9.6.2.2 Identification of Samples Drinking water samples from public water systems will be logged into R4LIMS with the activity code HOH. The requirements of 40 CFR Part 141 must be adhered to for the analysis of these samples. Samples from individual potable wells may be received under any program element, the most common being RCRA (RCRP, RCRE, OR RCRU) or Superfund (SF or SFE). The samples will be identified on the sample log sheets as "Potable Water." Requirements of 40 CFR Part 141 will be met whenever practicable. The most common samples requiring drinking water methods are the performance testing samples.

9.6.2.3 Preparation and Analysis of Drinking Water Samples Any or all of the following methods in Figure 9- 2 may be used by this laboratory for the analysis of drinking water and potable well samples. Prior to analysis samples will be digested using the procedure in the approved method. When using Methods 200.8 or 200.7, the digestion step may be omitted on those samples with a turbidity of less than 1 nephelometric turbidity unit (NTU) and a "direct analysis" may be performed. ("Technical Notes on Drinking Water Methods," EPA 600/R-94-173, October 1994 as referenced in 40CFR 141.23). Direct analyses may be performed on blanks which by definition are prepared in water with less than 1 NTU turbidity. The digestion step for mercury may only be omitted when using Method 200.8.

9.6.3 NPDES Monitoring

9.6.3.1 Regulatory Authority The National Pollutant Discharge Elimination System (NPDES) is the national system for the issuance of permits under section 402 of the Clean Water Act (CWA) of 1977 as amended. Test procedures for the analysis of pollutants are found at 40CFR Part 136. The ICP-MS Method 200.8 is also acceptable for this program (August 31, 1999 memorandum from Russell Wright, Region 4 SESD, on Approval for Use of EPA Method 200.8).

9.6.3.2 Identification of Samples Samples received by this laboratory will be logged into the data system under program elements WQU or WQE with one of the following associated activity codes NPCBI, NPCEI, NPCSI, NPDE, NPPAI or NPXSI.

9.6.3.3 Preparation and Analysis of NPDES samples Samples received in support of the NPDES program will be prepared and analyzed in accordance with the requirements at 40CFR 136. Figure 9- 3 lists approved test procedures for metals analyses that may be used by this laboratory. Digestion is required prior to analysis for all metals.

9.6.4 Other Programs and Matrices This section provides general guidance for various programs including RCRA and Superfund which do not require specific methods (the exceptions are RCRA "Characteristics" found in SW-846 Chapter Eight, e.g., TCLP).

9.6.4.1 Waters Monitoring well, ambient water, effluents, and other water samples are digested with nitric/hydrochloric acids according to Method 200.2. Typically, all digests are scanned by either ICP or ICP-MS. Where the detection/quantitation technique is specified by program requirements, positive elements for the ICP scan will be verified by atomic absorption or ICP-MS to satisfy the appropriate requirement. Mercury analyses are performed according to MCAWW[1]-245.1 (equivalent to PE ENVA-100), 245.7 CVAF, or 1631 CVAF depending on detection limit requirements.

9.6.4.2 Soil and Sediment A 50 g aliquot (approximately) is taken from a well-mixed sample and weighed in a crucible. The sample is dried overnight at 60°C (Method 200.2) for a % moisture determination. The dried sample is ground to fineness and l g of subsample is taken for analysis. Sample digestion is conducted according to Method 200.2 or Method 3050[2] for those samples containing large amounts of organic matter and made up to l00 mLs for analysis. Initial weights and final volumes may be adjusted depending on expected sample concentrations and detection level requirements.

9.6.4.2.1 Mercury analysis of sediments will be conducted according to method 245.5 (water bath digestion) or by SW-846[2] Method 7474 for those samples requiring lower levels of detection.

9.6.4.3 Final low level data for metals is generated by any of the following methods, alone or in combination: ICP (Method 200.7 or 6010), Graphite Furnace (Method 200.9 or 3113B) or ICP-MS (Method 200.8, 6020, or 1638).

9.6.4.4 Ultra Trace Level Mercury UTL mercury is performed according to methods 245.7 or 1631.

9.6.4.5 Fish Whole fish are initially prepared with dry ice grinding followed by preparation by Method 200.3 ("Methods for the Determination of Metals in Environmental Samples, Supplement 1": EPA/600/R-94/111.)

9.6.4.5.1 Requests for analysis of individual organs or tissue can be satisfied by using the sample in its entirety or sub-sampling to obtain the maximum

weight required for the analysis. The tissue should be kept frozen during sub-sampling and weighing to prevent fluid migration or drainage.

9.6.4.5.2 Mercury analyses on fish tissue are performed according to Region 4 modification of Method 245.6.

9.6.4.5.3 Digestion of tissue for multielement analyses is conducted according to Method 200.3 (nitric/peroxide) followed by ICP detection. Detection limit requirements are satisfied by manipulation of sample weight, final volume of digestate and in certain instances, detection by graphite furnace or ICP-MS.

9.6.4.6 Other Tissue Generally, other tissues will be prepared and analyzed the same as fish tissue. New matrices will be observed closely as an additional pre-caution to ensure proper digestion. If a new matrix does not respond to the digestion, consult the lead analyst for guidance.

9.6.4.7 TCLP Extracts TCLP extracts will be prepared per Method 1311, optionally digested by Method 3010 (per Method 1311 section 7.2.14) and analyzed by Method 6010, 6020, or other appropriate method.

9.6.4.8 Oil and Oily Samples Oil emulsions and oil samples will be prepared in one of two ways on a wet weight basis. Those oil samples which are thin enough to disperse on heating, yet not cover the entire surface of the digestion fluid resulting in a superheated solution will be prepared by Method 3050 or 200.2. Those samples not amenable to acid digestion will be prepared by the following method: The oil phase is weighed (l g) into a small crucible. The crucible is transferred to a muffle furnace and brought up to l25°C for l hr. Increase the temperature to l75°C for l hr. Increase to 250°C for l hr. Increase to 350°C for 1 hr, then increase to 450°C. The sample is ashed overnight at 450°C maximum temperature. NOTE: Do not open the furnace during the procedure and until the furnace has cooled to l00°C. One mL of concentrated nitric acid and l mL of concentrated hydrochloric acid is added to the ash and warmed until ash is in solution. This solution is diluted to volume and is ready for analysis by ICP.

9.6.4.9 High Volume Air Filters Air filters are typically prepared for analysis by digesting a 1x8 inch strip of an 8x9 inch 'high vol' filter into a final volume of 50 mL according to the SOP ASB M100.0. Final volume may be adjusted to meet detection limits and DQOs. A minimum of two (2) 1x8 strips are required by the lab for each sample to meet QC requirements. Results for all samples are reported in $\mu g/m^3$. Calculations are performed on the basis that each 1x8 strip represents 1/9 of an 8x9 filter. **It is the responsibility of field personnel to communicate to the laboratory any time there is a deviation from this normal protocol.** Blank filter results are reported as $\mu g/m^3$ using a nominal air volume supplied by the samplers. The nominal air volume is equivalent to the typical volume of air

during the study. For the preparation of 'saturation' filters, typically the entire filter is digested because of its small size.

9.6.4.10 QC Blanks Rinse water from various equipment/operations and sampling bottles are routinely submitted for analyses to check for contamination. Containers with liquid are analyzed and reported as routine samples. Add water with the appropriate preservative determined by the analysis to empty containers (e.g., nitric acid for metals and sodium hydroxide for CN) and record the volume. After analysis, correct for the volume of preservative water. Example:

250 mL of preservative water is added to the empty container and analyzed with a result of less than 2.0 µg/L.

2.0 µg/L x 0.250 L = 0.50 µg

The result is reported as 0.50U µg

9.6.4.11 Special Samples Samples received for analysis which are not amenable to the standard digestion techniques will be prepared according to the best judgement of the primary analyst. These cases will require additional documentation such as details about methodology and quality control.

9.6.5 QC Requirements for Metals:

9.6.5.1 Sample Preparation

9.6.5.1.1 A method blank will be prepared with each batch of samples to monitor for contamination of reagents, glassware, and the laboratory. Detectable method blank levels up to 10 percent of the lowest sample concentration are permissible and are not an indication that the blank is out of control.

9.6.5.1.2 A laboratory control sample (LCS), prepared in duplicate from standard reference materials (or laboratory standards that have been confirmed by SRM) will be prepared with each batch of samples when materials are available and it is practical to perform. If the LCS is prepared from laboratory standards, they should be from a different source and/or lot than the standards used to prepare the calibration curve. The LCS may serve several purposes: the solution verifies instrument calibration and monitors the digestion procedure.

9.6.5.1.3 All projects will have at least one spiked sample. Projects with large numbers of samples will be spiked at the rate of one per 20 samples. While matrix spikes can provide valuable information, they typically would not be used for making QC decisions for batches (unless they reveal an obvious situation that is notable such as incorrect preservation).

9.6.5.2 Calibration Standards Commercial single element or multielement standard solutions will be used for the preparation of instrument calibration solutions. These standards will be dated when received and their concentration verified with standard reference materials from NIST, commercial sources where available, or reference samples from NERL-Cincinnati, QA Branch. All commercial standards will undergo additional examination for trace contamination of elements other than the specified element by consulting the certificate of analysis (or by ICP or ICP-MS if warranted). Mixes of these single element standards are prepared according to the requirements of the instrument being used.

9.6.5.3 Calibration All instruments will be calibrated with working standards diluted from commercial stock solutions that have been verified to contain their stated concentration with an external reference. Instruments will be calibrated to cover the range of concentrations found in the samples or the samples may be diluted to fall within the calibration range.

9.6.5.3.1 The following acceptable alternate technique is used in multielement analyses (ICP or ICP-MS) when a sample analyte exceeds the high standard: A high level single element standard may be run as a sample to demonstrate that the linear calibration range has not been exceeded and that no inter-element interferences are presented by the higher level of the analyte. Recovery of the standard must be ±10% of the true value to be considered linear.

9.6.5.3.2 Check Standard An initial check standard should be run as specified in the method.

9.6.5.3.3 Continuing Calibration Verification Calibration must be verified during each set of samples at a frequency that will validate all data generated for that set. Reference standards may be used to verify calibration.

9.6.5.4 Bracketing Samples Reportable sample results should be bracketed by the calibration curve range, samples diluted to fall within the range of the calibration curve, or the calibration curve linearity shall be verified for samples that do not fall within this range (see 9.6.5.3.1, alternate technique).

9.6.5.5 Materials of known concentration (reference samples, check standards, etc.) will be analyzed at the beginning and conclusion of the run to verify the calibration curve. In other words, an analytical run neither begins nor concludes with a sample.

9.6.5.6 Standards Traceability Upon receipt, stock standards are assigned a unique identifying number which is recorded in a log book along with other pertinent information (e.g., receipt date, lot number, vendor and concentration).

Identification of standards allows for tracking intermediate and working standards back to an original stock.

9.6.5.7 Instrument Log Books These logs will be maintained to record all service and maintenance records (40 CFR 160.63C).

9.6.5.8 Sample Analysis Records Log books will be maintained to record preparation of samples to include records of duplicates, spikes, sample numbers, dates, analyst, etc.

9.6.5.9 Log Books These logs will be maintained at each instrument to record instrumental conditions and settings during the analysis of samples.

9.6.5.10 Data Records All raw data from instrumentation will be retained in a project file for future reference for a period of time consistent with Branch policy. Other raw data that was used in decision making will also be retained in these files (see Chapter 4).

9.6.5.11 Generation of Acceptance Limits In the absence of method defined acceptance limits, historical data are compiled to generate warning and control limits for accepting and rejecting data. A minimum of 20 results will be required for developing acceptance limits. After the initial limits are determined, they should be updated again as needed within the first year and then at a minimum of annually thereafter. All reasonable results are included in the data bases (50% to 150% recovery; no obvious errors such as accounting for a dilution, diluting out a spike, omission of a spike, etc.).

Precision criteria are developed as follows:

Relative Percent Difference (RPD) = (Range ÷ Mean) x 100

Acceptance Limit (as RPD) = 3 x Standard Deviation of RPD Mean

or

Relative Standard Deviation (RSD) = (Std Dev of reps ÷ mean) x 100

Acceptance Limit (as RSD) = 3 x Standard Deviation of RSD Mean

Bias Criteria are developed as follows:

%Recovery = ((spike concentration - unspiked concentration)/true concentration) x 100

Acceptance Limits = %Recovery Mean ± (3 x Std Dev of Recov Mean)

9.6.5.12 QC Data Data generated from duplicates, spikes, preparation blanks, and SRM preparations will be compared to acceptance limits generated from historical data for that particular sample type. It is also permissible to use the method defined acceptance limits. See also "Data Review Guidelines for Metals" found in "ICS Compilation of SOPs For Metals" and at K:\ASB\ICS\.

9.6.5.12.1 If the data are not within acceptable limits, the samples will be re-analyzed (e.g., when the LCS results are outside acceptance limits or the primary analyst suspects a problem with the instrument). In some instances, it is appropriate to "J" flag the result and report with the appropriate remark without redigestion or reanalysis (e.g., the LCS results are acceptable, but the matrix spikes are outside acceptable limits). If, after a second analysis, the data stills remain outside acceptable limits, data will be "J" flagged and reported with the appropriate remark. Certain circumstances may warrant more than one repeat.

9.6.5.12.2 In the absence of current acceptance limits, use as guidance the best available estimation of limits from established methods or other sources. Judgements on data quality (e.g., adding qualifier flags, etc.) will not be made solely on the basis of these estimated limits until such time as acceptance limits are appropriately determined. In these instances consult the Section Chief (or designee) and Branch QAO for guidance.

9.6.5.13 Cleaning All reusable vessels will be placed into a detergent soak immediately after use and must not be allowed to dry while dirty. After thoroughly soaking, all detergent is removed by rinsing, followed by a 20% nitric acid rinse and, finally, a thorough rinsing with DI water. Allow the vessel to drain on its side and seal with parafilm or a glass stopper before storage in an upright position. Pipets are rinsed immediately after use and placed in a detergent soak until moved to an automatic rinser with DI water. Labware used in ultra-trace analyses may require more rigorous specialized cleaning according to individual protocols.

Footnotes:

1 MCAWW - "Methods for Chemical Analysis of Water and Wastewater." EPA 600/4-79-020, March 1979. (Revised March 1983), and any future updates.

2 SW846 - "Test Method for Evaluating Solid Waste," EPA 1982, and any future updates.

9.7 Nutrients and Classicals

9.7.1 General These analyses are performed in support of various agency programs. Some programs mandate methods (e.g., Drinking Water at 40CFR Part 141 ff. and NPDES at 40 CFR Part 136), while others publish methods strictly as guidance (e.g., RCRA except for the Characteristic Tests at 40CFR Subpart C Part 261.20 ff.) Subject to the restrictions in 9.2.1, mandated methodology will be used for those analyses requiring them. Guidance methods will be closely adhered to with the possibility of changes which are documented in the SOPs. In the case of one-time use methods, documentation of the procedure will be included in the project file along with the raw-data. Methods and procedures are documented with the data results in R4LIMS. Figure 9-4 lists parameters routinely analyzed in this laboratory and methods of analysis.

9.7.1.1 Different programs have quality standards or fail levels that are of interest or that may trigger an action. Table 9-1 lists these various levels for common analytes and programs.

9.7.2 Drinking Water

9.7.2.1 Regulatory Authority National Primary Drinking Water Regulations are found at 40 CFR Part 141. National Secondary Drinking Water Regulations are found at 40 CFR Part 143. In general these regulations apply to Public Water Systems which are defined as "a system for the provision to the public of water for human consumption through pipes or, after August 5, 1998, other constructed conveyances, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year" (40 CFR Part 141.2). Historically, this laboratory has analyzed few samples from public water systems as the states have been delegated the authority for monitoring public water supplies within their boundaries. However, this laboratory often analyzes samples from individual private potable wells. Therefore, while not legally obligated to adhere to the requirements of 40 CFR Part 141 for these samples, this lab has chosen to follow the requirements in Part 141 whenever possible when analyzing private potable wells.

9.7.2.2 Identification of Samples Drinking water samples from public water systems will be logged into R4LIMS with the activity code HOH. The requirements of 40 CFR Part 141 must be adhered to for the analysis of these samples. Samples from individual potable wells may be received under any program element, the most common being RCRA (RCRP, RCRE, OR RCRU) or Superfund (SF or SFE). The samples will be identified on the sample log sheets as "Potable Water." Requirements of 40 CFR Part 141 will be met whenever practicable. The most common samples requiring drinking water methods are the performance testing samples.

9.7.3 NPDES Monitoring

9.7.3.1 Regulatory Authority The National Pollutant Discharge Elimination System (NPDES) is the national system for the issuance of permits under section 402 of the Clean Water Act (CWA) of 1977 as amended. Test procedures for the analysis of pollutants are found at 40CFR Part 136. The IC Method 300 is also acceptable for this program (August 31, 1999 memorandum from Russell Wright, Region 4 SESD, on Approval for Use of EPA Method 300).

9.7.3.2 Identification of Samples Samples received by this laboratory will be logged into the data system under program elements WQU or WQE with one of the following associated activity codes NPCBI, NPCEI, NPCSI, NPDE, NPPAI or NPXSI.

9.7.3.3 Preparation and Analysis of NPDES Samples Samples received in support of the NPDES program will be prepared and analyzed in accordance with the requirements at 40CFR 136.

9.7.4 RCRA and Superfund Guidance for analysis of RCRA samples is found in "Test Methods for the Evaluating Solid Waste Physical/Chemical Methods" (US EPA SW-846; Office of Solid Waste). Currently, the only method frequently used by Nutrients and Classicals under this program is pH (40CFR, Part 261.22). Otherwise, the methods used for NPDES and Drinking Water programs are typically used to perform chemical analysis of RCRA and Superfund samples.

9.7.5 Other Programs and Matrices

9.7.5.1 General Water Quality Methods used for NPDES and Drinking Water programs are typically used to perform chemical analysis of Water Quality samples.

9.7.5.2 Sediments and Soils In the absence of EPA methods for the analysis of nutrient and classical parameters for sediments and soils, the ICS has compiled a series of methods used specifically by this laboratory (ICS Compilation of SOPs for Nutrients/Classicals). EPA methods are followed when practicable (e.g., Method 300 and Method 9045).

9.7.5.3 QC Blanks Rinse water from various equipment/operations and sampling bottles are routinely submitted for analyses to check for contamination. Containers received bearing liquid are analyzed and reported as routine samples. Add water with the appropriate preservative determined by the analysis to empty containers (e.g., nitric acid for metals and sodium hydroxide for CN) and record the volume. After analysis, correct for the volume of preservative water. Example:

> 250 mL of preservative water is added to the empty container and analyzed with a result of less than 2.0 µg/L.

2.0 µg/L x 0.250 L = 0.50 µg

The result is reported as 0.50U µg

9.7.5.4 Special Samples Samples received for analysis which are not amenable to the standard digestion techniques will be prepared according to the best judgement of the primary analyst. These cases will require additional documentation such as details about methodology and quality control.

9.7.6 QC Requirements for Nutrients and Classicals

9.7.6.1 Sample Preparation

9.7.6.1.1 A method blank will be prepared with each batch of samples to monitor for contamination of reagents, glassware, and the laboratory. Detectable method blank levels up to 10 percent of the lowest sample concentration are permissible and are not an indication that the blank is out of control.

9.7.6.1.2 A laboratory control sample (LCS), prepared in duplicate from standard reference materials (or laboratory standards that have been confirmed by SRM) will be prepared in duplicate with each batch of samples when materials are available and it is practical to perform. In some cases, it is impractical to perform a duplicate LCS (e.g., parameters that can only be prepped a few at a time). If the LCS is prepared from laboratory standards, they should be from a different source and/or lot than the standards used to prepare the calibration curve. The duplicate LCS preparations may serve several purposes: the solutions verify instrument calibration, monitor the digestion procedure, and supply precision information on each element present in the LCS. In other words, these samples are used to indicate whether an analytical procedure is in control and for analytical precision statements.

9.7.6.1.3 Projects will have at least one spiked sample when spiking can be performed. Projects with large numbers of samples will be duplicated and spiked at the rate of one in 20 samples. While matrix spikes can provide valuable information, they typically would not be used for making QC decisions for batches (unless they reveal an obvious situation that is notable such as incorrect preservation).

9.7.6.2 Calibration Standards Commercial single element or multielement standard solutions will be used for the preparation of instrument calibration solutions. These standards will be dated when received and their concentration verified when practicable with standard reference materials from NIST, commercial sources where available, or reference samples from NERL-Cincinnati,

QA Branch. Mixes of these single element standards are prepared according to the requirements of the instrument being used.

9.7.6.3 Calibration All instruments will be calibrated with working standards prepared from commercial stock solutions or reagents that have been verified to contain their stated concentration. Instruments will be calibrated to cover the range of concentrations found in the samples or the samples may be diluted to fall within the calibration range, when appropriate.

9.7.6.3.1 The following acceptable alternate technique is used in multielement analyses (IC) when a sample analyte exceeds the high standard: A high level single element standard may be run as a sample to demonstrate that the linear calibration range has not been exceeded and that no inter-element interferences are presented by the higher level of the analyte. Recovery of the standard must be ±10% of the true value to be considered linear.

9.7.6.3.2 Check Standard An initial calibration check solution should be run as specified in the method.

9.7.6.3.3 Continuing Calibration Verification Calibration must be verified during each set of samples at a frequency that will validate all data generated for that set. Reference standards may be used to verify calibration.

9.7.6.4 Bracketing Samples Reportable sample results should be bracketed by the calibration curve range, samples diluted to fall within the range of the calibration curve, or the calibration curve linearity shall be verified for samples that do not fall within this range.

9.7.6.5 Materials of known concentration (reference samples, check standards, etc.) will be analyzed at the beginning and conclusion of the run to verify the calibration curve. In other words, an analytical run neither begins nor concludes with a sample.

9.7.6.6 Standards Traceability Upon receipt, stock standards are assigned a unique identifying number which is recorded in a log book along with other pertinent information (e.g., receipt date, lot number, vendor and concentration). Identification of standards allows for tracking intermediate and working standards back to an original stock.

9.7.6.7 Instrument Log Books These logs will be maintained to record all service and maintenance records (40 CFR 160.63C).

9.7.6.8 Sample Analysis Records Log books will be maintained to record preparation of samples to include records of duplicates, spikes, sample numbers, dates, analyst, etc.

9.7.6.9 Log Books These logs will be maintained at each instrument to record instrumental conditions and settings during the analysis of samples.

9.7.6.10 Data Records All raw data from instrumentation will be retained in a project file for future reference for a period of time consistent with Branch policy. Readings taken manually are considered raw data and are recorded in the appropriate log book. Other raw data that was used in decision making will also be retained in these files (see Chapter 4).

9.7.6.11 Generation of Acceptance Limits In the absence of method defined acceptance limits, historical data are compiled to generate warning and control limits for accepting and rejecting data. A minimum of 20 results will be required for developing acceptance limits. After the initial limits are determined, they should be updated again as needed within the first year and then at a minimum of annually thereafter. All reasonable results are included in the data bases (50% to 150% recovery; no obvious errors such as accounting for a dilution, diluting out a spike, omission of a spike, etc.).

Precision criteria are developed as follows:

Relative Percent Difference (RPD) = (Range ÷ Mean) x 100

Acceptance Limit (as RPD) = 3 x Standard Deviation of RPD Mean

or

Relative Standard Deviation (RSD) = (Std Dev of reps ÷ mean) x 100

Acceptance Limit (as RSD) = 3 x Standard Deviation of RSD Mean

Bias Criteria are developed as follows:

%Recovery = ((spike concentration - unspiked concentration)/true concentration) x 100

Acceptance Limits = %Recovery Mean ± (3 x Std Dev of Recov Mean)

9.7.6.12 QC Data Data generated from duplicates, spikes, and SRM preparations will be compared to acceptance limits generated from historical data for that particular sample type. It is also permissible to use the method defined acceptance limits.

9.7.6.12.1 If the data are not within acceptable limits, the samples will be re-analyzed (e.g., when the LCS results are outside acceptance limits or the primary analyst suspects a problem with the instrument) or the data will be "J" flagged and reported with the appropriate remark. If, after a second

analysis, the data still remain outside acceptable limits, data will be "J" flagged and reported with the appropriate remark. Certain circumstances may warrant more than one repeat.

9.7.6.12.2 In the absence of current acceptance limits use as guidance the best available estimation of limits from established methods or other sources. Judgements on data quality (e.g., adding qualifier flags, etc.) will not be made solely on the basis of these estimated limits until such time as acceptance limits are appropriately determined. In these instances consult the Section Chief and Branch QAO for guidance.

9.7.6.13 Reference Materials Sources outside the lab will be used for reference materials when available. As of the last update of this document, the following parameters do not have commercial sources known to this lab: color, settlable solids, acidity, and TVSS.

9.7.7 Acid Addition for Nutrient Analyses Prepared standards for nutrient analyses are a combination of total phosphorus, nitrate/nitrite, and ammonia. Sulfuric acid is added to stock and working standards, aqueous reference materials, distillation receiving solutions, and wash water in order to minimize marked differences in pH between these materials and samples (see discussion on ammonia (4500-NH_3 H) in 18th edition of Standard Methods for the Examination of Water and Wastewater, 1992). The concentration of the acid is 1 mL/L. Samples for nutrient analyses are typically preserved with 0.5 to 1.0 mL/L sulfuric acid.

Figure 9-1 Levels of Concern for Various Programs

PARAMETER	DRINKING WATER 40 CFR 141.11 and 141.62	RCRA TCLP (40CFR 261.24 Table 1) and pH (40CFR 261.22)	RCRA LAND BAN LIMITS 40CFR 268.48 Table UTS		WATER QUALITY STANDARDS *
			Wastewater	Nonwastewater	
Antimony	6 µg/L		1.9 mg/L	1.15 mg/L TCLP	* See publication at www.epa.gov/ost/pc/revcom.pdf
Arsenic	10 µg/L * proposed in 2002	5.0 mg/L	1.4 mg/L	5.0 mg/L TCLP	
Barium	2000 µg/L	100.0 mg/L	1.2 mg/L	21 mg/L TCLP	
Beryllium	4 µg/L		0.82 mg/L	1.22 mg/L TCLP	
Cadmium	5 µg/L	1.0 mg/L	0.69 mg/L	0.11 mg/L TCLP	
Chromium	100 µg/L	5.0 mg/L	2.77 mg/L	0.60 mg/L TCLP	
Copper	1300 µg/L* See 40CFR 141.80				
Cyanides (Total)	200 µg/L		1.2 mg/L	590 mg/kg	
Cyanides (Amenable)			0.86 mg/L	30 mg/kg	
Fluoride	4.0 µg/L		35 mg/L	NA	
Lead	15 µg/L* See 40CFR 141.80	5.0 mg/L	0.69 mg/L	0.75 mg/L TCLP	
Mercury (non wstwtr/retort)			NA	0.20 mg/L TCLP	
Mercury	2 µg/L	0.2 mg/L	0.15 mg/L	0.025 mg/L TCLP	
Nickel			3.98 mg/L	11 mg/L TCLP	
Nitrate	10 mg/L				
Nitrite	1 mg/L				
Nitrate + Nitrite	10 mg/L				
pH		≤ 2.0 and ≥ 12.5			
Selenium	50 µg/L	1.0 mg/L	0.82 mg/L	5.7 mg/L TCLP	
Silver		5.0 mg/L	0.43 mg/L	0.14 mg/L TCLP	
Sulfide			14 mg/L	NA	
Thallium	2 µg/L		1.4 mg/L	0.20 mg/L TCLP	
Turbidity	1 NTU				
Vanadium			4.3 mg/L	1.6 mg/L TCLP	
Zinc			2.61 mg/L	4.3 mg/L TCLP	

Figure 9- 2
Drinking Water Methods

ANALYTE	MCL (mg/L)	ICP[4]	Graphite Furnace[4]	Graphite Furnace[5]	ICP-MS[4]	CVAA
Antimony	0.006[1]		200.9	3113B	200.8	
Arsenic	0.050[2]	200.7	200.9	3113B	200.8	
Barium	2.0[1]	200.7		3113B	200.8	
Beryllium	0.004[1]	200.7	200.9	3113B	200.8	
Cadmium	0.005[1]	200.7	200.9	3113B	200.8	
Chromium	0.10[1]	200.7	200.9	3113B	200.8	
Lead	0.015[3]		200.9	3113B	200.8	
Mercury	0.002[1]				200.8	245.1[6] (ENVA-100[7])
Nickel	0.10[1]	200.7	200.9	3113B	200.8	
Selenium	0.050[1]		200.9	3113B	200.8	
Thallium	0.002[1]		200.9		200.8	

Footnotes:

1 40CFR 141.23.
2 40CFR 141.11.
3 40CFR 141.80.
4 ICP method 200.7, Graphite Furnace Method 200.9, and ICP-MS Method 200.8 are in "Methods for the Determination of Metals in Environmental Samples-Supplement 1", EPA-600/R-94-111, May 1994. Available from NTIS, PB 94-184942; (800) 553-6847.
5 Graphite Furnace Method 3113B is in 18th Edition of "Standard Methods for the Examination of Water and Wastewater," 1992, American Public Health Association. Available from American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C.
6 Mercury CVAA Method 245.1 is available from US EPA, NERL, Cincinnati, OH 45268. The identical method was formerly in "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020, March 1983 which is available from NTIS, PB84-128677; (800) 553-6847.
7 PerkinElmer Method ENVA-100, "The Application of Flow Injection Technology to Automating Cold Vapor Mercury Analyses," January 9, 1997. Equivalent to approved EPA Method 245.1 per January 13, 1997 memo from Dr. M. K. Smith, Director, EERD, NERL, Cincinnati, OH.

Figure 9- 3
NPDES Methods

ANALYTE	ICP	GFAA	ICP-MS	OTHER	CVAA-HG
Aluminum	200.7[1]	3113B[2]	200.8[3]		
Antimony	200.7	3113B	200.8		
Arsenic	200.7	3113B	200.8		
Barium	200.7	3113B	200.8		
Beryllium	200.7	3113B	200.8		
Boron	200.7		200.8		
Cadmium	200.7	3113B	200.8		
Calcium	200.7		200.8		
Chromium VI			200.8	3500-Cr D[2]	
Chromium	200.7	3113B	200.8		
Cobalt	200.7	3113B	200.8		
Copper	200.7	3113B	200.8		
Hardness	200.7		200.8		
Iron	200.7	3113B	200.8		
Lead	200.7	3113B	200.8		
Magnesium	200.7		200.8		
Manganese	200.7	3113B	200.8		
Mercury			200.8		245.1 (ENVA-100[4]), 1631[5]
Molybdenum	200.7	3113B	200.8		
Nickel	200.7	3113B	200.8		
Potassium	200.7		200.8		
Selenium	200.7	3113B	200.8		
Silica	200.7		200.8		
Silver	200.7	3113B	200.8		
Sodium	200.7		200.8		

Figure 9- 3
NPDES Methods

ANALYTE	ICP	GFAA	ICP-MS	OTHER	CVAA-HG
Thallium	200.7	279.2[1]	200.8		
Tin	200.7	3113B[1]	200.8		
Titanium		283.2[1]	200.8		
Vanadium	200.7	286.2[1]	200.8		
Zinc	200.7	289.2[1]	200.8		

Footnotes:

1 "Methods for the Determination of Metals in Environmental Samples-Supplement 1", EPA-600/R-94-111, May 1994.

2 "Standard Methods for the Examination of Water and Wastewater." American Public Health Association, 18th Edition, 1992.

3 August 31, 1999 memorandum from Russell Wright, Region 4 SESD, on Approval for Use of EPA Method 200.8.

4 PerkinElmer Method ENVA-100, "The Application of Flow Injection Technology to Automating Cold Vapor Mercury Analyses," January 9, 1997. Equivalent to approved EPA Method 245.1 per January 13, 1997 memo from Dr. M. K. Smith, Director, EERD, NERL, Cincinnati, OH.

5 "Method 1631, Revision B: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence spectrometry," May 1999, EPA 821-R-99-005. Available from EPA Office of Water.

Figure 9- 4
Nutrients/Classicals METHODS

ANALYTE	NPDES	DRINKING WATER	OTHER PROGRAMS OR MATRICES
Acidity	EPA[1] SM[2] 2310		
Alkalinity	SM 2320		
Ammonia	SM 4500-NH_3 B 780-86t R4M[3,4]		ASB102C[4]
BOD	SM[4] 5210B		
Chloride	Method 300[5,6] or SM 4500-Cl C		
Color, ADMI	EPA 110.1		
Color, Pt Co (True and Apparent)	EPA 110.2		
Conductivity	SM 2510B	SM 2510B	
Cyanide	EPA 335.2	EPA 335.4 or SM 4500CN F	Sedmt- SM p.4-19 2.b.
Fluoride	EPA 300	EPA 300	Sedmt- EPA 300
Nitrate/Nitrite	EPA 353.2		Sedmt- EPA 300 or ASB106C
Nitrate	EPA 353.2 or EPA 300	EPA 353.2 or EPA 300	
Nitrite	EPA 353.2 or EPA 300	EPA 353.2 or EPA 300	
Oil and Grease	EPA 1664[7,8]		EPA 1664[7,8]
% Solids or Moisture			See 9.8.4.2
pH		SM 4500H^+-B	RCRA-SW-846[8] Method 9040 or 9045
Phenols	EPA 420.2		
Phosphorus	EPA 365.1 or	EPA 365.1 or	Sedmt- ASB103C; Low Level- EPA R4 method
Phosphorus, Ortho	EPA 365.1 or EPA 300	EPA 365.1 or EPA 300	Low Level- EPA R4 method
Solids	SM 2540 series	SM 2540 series	
Sulfate	ASTM D516-90[9] EPA 300	ASTM D516-90 EPA 300	Sedmt-EPA 300 EPA

Figure 9- 4
Nutrients/Classicals METHODS

ANALYTE	NPDES	DRINKING WATER	OTHER PROGRAMS OR MATRICES
Sulfide	EPA 376.2		Sedmt- ASB101C followed by EPA 376.2
TKN	786-86T[3]: Cu compound substituted for Hg digestion compound		Sedmt- ASB 104C
TOC	EPA 415.1		
Turbidity	EPA 180.1		

Footnotes:

1 "Methods for Chemical Analysis of Water and Wastewater." EPA 600/4-79-020, March 1979. (Revised March 1983), and any future updates.

2 "Standard Methods for the Examination of Water and Wastewater." American Public Health Association, 18th Edition, 1992.

3 Per Method 4500-NH3 H references to instrumentation and/or September 10, 1986 Memo from Robert Booth, Director, Environmental Monitoring and Support, NERL, EPA, Cincinnati, OH.

4 "Inorganic Chemistry Section Compilation of SOPs for Nutrients/Classicals."

5 "Methods for the Determination of Inorganic Substances in Environmental Samples." EPA/600/R-93/100, August 1993.

6 August 31, 1999 memorandum from Russell Wright, Region 4 SESD, on Approval for Use of EPA Method 300.0.

7 "EPA Method 1664, Revision A: N-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry." Available from NTIS (800)553-6847; Document number PB99-121949 or at http://www.epa.gov/OST.

8 SW846 - "Test Method for Evaluating Solid Waste," EPA 1982, and any future updates. Oil and grease is incorporated by reference.

9 Annual Book of ASTM Standards, Water and Environmental Technology, Section 11, Volumes 11.01, 1990 revision.