

NC DWQ Laboratory Section Sample Submission Guidance Document

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1.0 Introduction

This document is designed to provide a uniform set of procedures for sample submission to the NC DENR/DWQ Laboratory Section – Central Laboratory. The protocols outlined in this document are applicable to parties responsible for the collection, handling, field screening, documentation, packaging, shipping and receipt of samples. Efforts to improve current procedures will continue and this document will be revised, as needs dictate. Similar procedures should be followed when submitting samples to the DWQ laboratories located in the Asheville and Washington Regional Offices.

The primary goal of this document is to promote the use of procedures that will ensure data reported by the laboratory is accurate and able to withstand legal and technical challenges. Many of the errors in environmental analysis result from incorrect sample handling and lack of supporting documentation. Four factors that may ultimately affect the integrity of reported data include: 1) obtaining a representative sample, 2) preventing contamination of the sample, 3) providing legal documentation of the sampling event, and 4) protecting the sample from chemical, physical or biological change prior to analysis.

The DWQ Laboratory Section is committed to providing the highest quality data and the best overall service in environmental testing. To ensure that the results produced and reported meet the requirements of the data users and comply with state and federal regulations, a quality management system must be implemented that is clear, effective, well-communicated, and supported at all levels of the Division.

2.0 Sample Containers, Preservation and Hold Times

Prior to mobilizing, the sampler must decide what samples to collect, which parameters to request, and whenever possible, where to collect them. Containers, preservatives, holding times, sample volumes, and target analytes must be considered. Environmental testing, like the sample collector's time and resources, can be labor intensive and costly. Field personnel must make every effort to follow proper sample handling protocol and limit requests to pertinent analyses.

The parameters to be measured are usually dictated by the purpose of an investigation and should be selected based upon required monitoring conditions or upon the investigator's knowledge of the problem. The volume of sample obtained should be sufficient to perform all the required analyses with an additional amount collected to provide for any quality control needs such as duplicates, matrix spikes, split samples, confirmations or repeat examinations. Determinations for some parameters may be submitted in the same sample bottle if the bottle contains sufficient sample for each analysis, including quality control and repeat analysis, and require compatible collection and preservation techniques. The most common practices include: submission of one 500-ml sample bottle for ammonia, TKN, nitrate+nitrite, and total phosphorous analyses, and one 500-ml container for the following metals: Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr (total), Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Sb, Se, Sn, Tl, V and Zn (Hg may also be included, however, the 28-day holding time would apply). When submitting soil samples, a separate sample container must be collected for each of the analytical groups listed in the preservation and hold time tables (TABLE I – [Surface Water Protection \[SWP\]](#) and TABLE II – [Aquifer Protection and Underground Storage Tank \[APS\]](#)). This will ensure that sufficient sample is submitted for all analyses requested and that sample holding times are not exceeded because a single sample must be transferred between multiple analytical units in the laboratory.

The preservation and hold time tables ([TABLE I - SWP](#) and [TABLE II - APS](#)) list the analytical parameter, the minimum volume or weight of sample required to complete the analysis, the routine container used for sample collection by the DWQ Laboratory Section, the chemical preservative required to maintain the integrity of the sample, and the maximum hold time (including shipping, preparation and quantitative determination). The routine reporting limit for some analyses can be attained when a less than optimum sample volume or weight is supplied. For some analyses (e.g., organic extractions), the final volume of the extract or digest can be adjusted to meet the routine reporting limits. If a data user requires a normal reporting limit from a reduced sample amount, the lab must be alerted prior to sample analysis because the amount of surrogate and/or spiking solution must be adjusted prior to sample preparation. It should also be noted that the container types listed are those commonly used throughout the Division. Other container types may be acceptable. Consult EPA 40 CFR, Chapter 1, Part 136, Table II or the DWQ Laboratory Section [QA/QC Coordinator](#) about the use of proper containers before deviating from those listed in Tables I and II.

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2.1 Containers

The routine containers, used for sampling and analysis, are normally borosilicate glass (G) or plastic (P). High-density polyethylene (HDPE) bottles are also used for a few parameters. Containers that are purchased as pre-cleaned should be certified by the manufacturer or checked to ensure that the parameters tested are below the published laboratory reporting limits. Containers should be stored in a manner that does not leave them susceptible to contamination by dust or other particulates and should remain capped until use. It is recommended that field staff periodically check bottles for contamination attributed to storage conditions by filling representative containers with analyte-free water (available from the Laboratory Section), adding the appropriate preservative(s), and submitting them to the laboratory for metals and wet chemistry analyses. The analysis report should be kept on file and a copy sent to the DWQ Laboratory Section [QA/QC Coordinator](#). Any containers that show evidence of contamination should be discarded. Certificates should be kept on file for glass containers, which are pre-cleaned and certified to be "analyte-free" by the manufacturer.

The Laboratory Section now provides all sample containers to field staff. These containers can be requested by following the directions for ordering supplies found on the Sample Collectors Pages at <http://portal.ncdenr.org/web/wq/lab/staffinfo/supplies>.

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2.2 Sample Preservation

The chemical preservative is the solution added to the sampling containers or supplied as separate solutions or neat materials that preserve the integrity of the sample. They may be chemical additives such as acids or bases used to control pH or reagent solutions such as sodium thiosulfate used to reduce the effect of residual chlorine and other oxidizers. Chemical preservatives are usually analyte-specific.

In addition to chemical preservation, most samples must be thermally preserved at the time of collection (exceptions include metals, chloride, fluoride and field parameters). When collecting samples from multiple sites, ice must be added to the cooler prior to or immediately after the first samples are collected. A temperature range of less than 10°C is acceptable for bacterial samples and less than or equal to 6°C (with no evidence of freezing) is acceptable for other samples requiring thermal preservation during transport (Ref: 40 CFR Part 136 Table II). NOTE: Wet ice (NOT dry ice or 'blue ice' packs) should be used for this purpose. In order to ensure that samples obtain appropriate cooling, they should be nearly covered by, rather than resting on top of the ice. In order to avoid compromising the integrity of samples, sample receipt technicians at the laboratory will measure the temperature of the temperature control sample (also referred to as a temperature blank) in each cooler. The Laboratory Section will furnish the temperature control sample containers. Red 500-ml HDPE bottles have been purchased for this purpose so that the temperature control bottle will stand out from the other samples in the coolers. Other sample containers are acceptable as long as they are representative of the sample containers shipped in the cooler and are clearly labeled as "temperature blanks". Temperature blanks should be handled exactly as the environmental samples are handled. They should be filled with water from the sampling site and added to the cooler when the samples are placed on ice. Samplers must ensure that each cooler submitted to the laboratory contains a labeled temperature blank. NOTE: Do not record temperature blanks on the fieldsheets or COC forms.

Occasionally, samples that are collected and delivered to the lab within a short period of time may not have time to cool to less than or equal to 6°C (or <10°C for bacterial tests) before they arrive at the laboratory. If these samples are placed in ice immediately after collection and are shipped on ice or in ice slurry (as noted above), the sample collectors have complied with these requirements to the best of their ability and the samples will be accepted. Documentation of the actual sample temperature at the time of collection and upon receipt at the laboratory should exhibit a downward trend and will complete the preservation documentation requirements. The effectiveness of icing samples will vary at different times of the year. Adjustments such as pre-icing, adding more ice or fewer bottles to each cooler should be made as necessary. The field temperature should always be documented if there is any question as to whether samples will have time to cool to less than or equal to 6°C (or <10°C for bacterial tests) during shipment.

Proper sample preservation is the responsibility of the sampling personnel, NOT the lab providing the sample containers. It is the responsibility of the field staff to assure that all samples are appropriately preserved. Sample preservation shall be accomplished by obtaining pre-preserved bottles from the Laboratory Section or by adding preservative to the sample in the field. Preservatives shall be reagent-grade or of a higher grade, as necessary. Acids suitable for trace metals analysis must be used for preserving metals samples. Fresh preservatives should be obtained from parent stocks prior to each sampling event. Any remaining preservatives that are not in sealed ampoules shall not be returned to stock, but must be appropriately discarded. Preservatives should be added with disposable pipettes, auto-pipettors or pre-measured ampoules to each sample container.

The effectiveness of required pH adjustments must be checked in the field. Narrow-range pH paper shall be used to test an aliquot of the preserved sample. This may be accomplished by pouring a small portion into a disposable container. If the measured pH is within the appropriate range, the

sample may be submitted to the laboratory as is. If not, the chemical preservative must be added drop-wise until the proper pH is achieved. The additional amount of chemical preservative added should be documented on the fieldsheet. If an excessive amount of chemical preservative must be added to achieve the desired effect, contact the laboratory for additional preservation guidance (care must be taken not to over-dilute the sample or change the sample matrix with preservative). If there is any question as to the amount of preservative required for a particular sample, the sampler may opt to collect two identical aliquots of a sample and use one to determine the preservation requirements. Addition of the same amount of preservative to the other container will achieve the proper preservation without contaminating the sample to be sent to the laboratory for analysis. Because of the risk of compromising sample integrity, sulfide and volatile organics analysis (VOA) samples cannot be checked in the field. Sample collectors are asked to verify the sample preservation process by initialing the preservative space on the sample tag. This is especially important when pre-printed sample tags are used. Sample preservatives and other reagent preparations shall be traceable to preparation dates and vendor sources and/or lot numbers. These records should be kept on file in the field offices.

Samples subject to chlorine interference (i.e., ammonia nitrogen, total Kjeldahl nitrogen or TKN, total cyanide, total recoverable phenols and organic parameters) should be checked for total residual chlorine at the time of collection. This may be accomplished using test strips designed for this purpose. If chlorine is found in these samples, the sampler must add an appropriate (as specified by the method) dechlorinating agent before chemically preserving the sample. If chlorine is detected in these samples upon receipt in the laboratory, they will be rejected.

Additional detailed guidance for preservation of samples (in a table entitled *Sample Preservation Guidance*) can be found at: <http://portal.ncdenr.org/web/wq/lab/staffinfo/samplesubmit>.

The Laboratory Section now provides all sample preservatives to field staff. These chemicals can be requested by following the directions for ordering supplies found on the Sample Collectors Pages at <http://portal.ncdenr.org/web/wq/lab/staffinfo/supplies>.

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2.3 Hold Times

The holding time is the maximum time from collection that the sample can be held prior to preparation or analysis. Some parameters (e.g., semivolatile organics) have separate published hold times for preparation of the sample and analysis of the extract.

In the preservation and hold time tables, some parameters require analysis “within 15 minutes” of collection. This pertains to filtration immediately followed by preservation (thermal and chemical), where required (e.g., dissolved metals, orthophosphate, etc.). NOTE: Carbon dioxide, pH and sulfite are field determinations and should not be submitted to the laboratory for analysis.

Published hold times must be paid close attention. Samples should be delivered to the lab as soon as possible after collection. When immediate delivery is not possible, the sample collector must be aware of the maximum hold times in order to allow ample time for sample preparation, analysis and reanalysis. The only permissible exception to the hold times published in 40 CFR Part 136 Table II is the extended holding time allowed by DWQ for noncompliance coliform samples. Since it is not possible to transport some samples to the Laboratory Section in less than 6 hours, noncompliance samples delivered within 24 hours of collection will be accepted, analyzed and reported with qualification. The qualification will read, “*Holding time exceeded. These codes shall be used if the value is derived from a sample that was received, prepared and/or analyzed after the approved holding time restrictions for sample preparation and analysis. The value does not meet NPDES requirements.*” NOTE: Coliform samples that may be involved in litigation must be delivered to the lab in less than 6 hours.

In addition to meeting the published hold times, sample collectors must keep in mind courier routes and scheduling and the hours of operation of the three DWQ laboratories (i.e., the Central Laboratory in Raleigh, the Asheville Regional Laboratory in Swannanoa and the Washington Regional Laboratory). General and specific limitations on sample receipt are listed on the intranet portal at: <http://portal.ncdenr.org/web/wq/lab/staffinfo/samplesubmit>.

For additional guidance on proper sample collection and handling techniques, refer to "Standard Operating Procedures Manual for Physical and Chemical Monitoring", August 2003 (or subsequent versions) for Surface Water Protection Section samples (available from the [Environmental Sciences Branch](#)). For Groundwater samples, refer to "Groundwater Section QA/QC and Standard Operating Procedures Manual", the "Groundwater Sampling" video, and "Groundwater Section Guidelines for the Investigation and Remediation of Soil and Groundwater" (available from the [Aquifer Protection Section](#)).

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3.0 Field Reagent Handling

All reagents, cleaning materials and preservatives that are maintained by the sample collector shall be stored, transported and handled in such a way to prevent and/or minimize contamination. All chemicals that are maintained in-house and transported to the field shall be segregated according to reactivity (i.e., acids, bases, etc.). Acids should be stored in an acid storage cabinet and solvents should be stored in a vented solvent storage cabinet.

All chemicals transported to the field shall be stored in bottles that will be packed to avoid breakage. If quantities of reagent chemicals are transferred from the original container, the transport container shall be appropriately pre-cleaned and must be of similar construction type as the original container (e.g., acids and bases may be transported in plastic or Teflon® containers). Chemicals shall be segregated from sample containers so as to avoid reaction and accidental contamination. Analyte-free water shall be segregated from solvents to prevent contamination. The use of field blanks to determine ambient contamination is recommended, but is left to the discretion of the sample collector.

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4.0 Sample Packing and Transport

Samples shall be packed so that they are segregated by site, sampling location or by sample analysis type. Sample segregation may follow this segregation scheme or any other that is reasonable. These schemes are dependent upon the levels of contamination present, the number of bottles to be transported, the size of the bottles, etc. When Chain of Custody (COC) samples are involved, it is best to try to segregate samples in coolers by site as much as possible. Then, each cooler must have an associated COC form and field sheet for each site contained in that cooler. By completing individual field sheets for each cooler, discrepancies pertaining to only the parameters affected in that cooler (distinguishing them from samples collected at that same site which may be packed in other coolers and which conform to all requirements) can be documented. In doing this, samples collected from a similar site and packed in different coolers will be given different lab numbers, but the site information will be on the final reports for tracking. Note: If samples from multiple sites will fit in one cooler, they may be packed in the same cooler with the associated field sheets and a single COC form for all (if they are collected by the same Section - SWP, APS or UST). Grab and composite samples, even from the same location, must be listed on separate field sheets.

It is mandatory that trip blanks accompany any volatile organic sample (aqueous or solid) submitted to the laboratory for analysis. A **trip blank** is defined as a sample of analyte-free media (e.g., well water

with charcoal filtration, non-chlorinated bottled water, deionized water) taken from the laboratory or other point of origin to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures (i.e., diffusion of volatile organics through the septum during shipment and storage). The source of the trip blank water should be identified on the fieldsheet (e.g., X Brand Bottled Water, regional office tap water with charcoal filtration, etc.). The trip blank is then submitted for analysis as any other sample. Three 40-ml vials are required for the trip blank (four 40-ml vials are required for each environmental sample). A trip blank is treated just as any other sample and, consequently, a separate field sheet must be completed and submitted with the trip blank and it must also be recorded separately on the COC form, when used. VOA samples from different locations may be placed in the same cooler to reduce the number of required trip blanks (i.e., one for VOA samples and one for TPH/BTEX samples) provided that the samples are wrapped or containerized (i.e., Ziploc® bag or metal can) separately. NOTE: Even when VOA samples are containerized separately, each of the four vials submitted for a site (or each of the three vials submitted for the trip blank) must be labeled appropriately (location ID or sorting number or other unequivocal identifier) to prevent misidentification should the vials become separated during shipment or in the laboratory. Trip blanks associated with emergency samples should likewise be designated "EMERGENCY" so that these results can be reported with the sample results. Click [here](#) for additional guidance on trip blanks.

Field blanks are also a smart idea for a variety of parameters where possible field contamination may occur. A field blank is a sample of analyte-free water transferred at the sampling site into an appropriate container with appropriate added preservative(s). These blanks distinguish ambient contamination from actual sample content. A separate field sheet and appropriate chain of custody documentation must also be submitted for field blanks.

If possible, routine or background samples should be collected by different field staff. If separate collection is not possible, the background sample should be collected first and placed in separate coolers or shipping containers. Additionally, samples should be collected from the least concentrated samples to the more concentrated samples to avoid cross-contamination. Sample collectors must always wear gloves (to avoid cross-contamination as well as for safety reasons) and change these gloves between sampling sites. Highly contaminated samples shall never be placed in the same cooler as environmental samples. It is a good practice to enclose highly contaminated samples in a plastic bag before placing them in coolers. Coolers or shipping containers with samples suspected of being highly contaminated shall be lined with new, clean, plastic bags. Make note on the field sheet and sample labels identifying samples that appear highly contaminated or exhibit other abnormal characteristics (i.e., foaming, odor, etc.). All samples that require thermal preservation shall be packed in thermally insulated coolers with wet ice. Samples must be surrounded (i.e., completely covered) by ice. Only wet ice shall be used in cooling samples to $\leq 6^{\circ}\text{C}$ ($< 10^{\circ}\text{C}$ for bacterial tests). 'BLUE ICE' OR CHEMICAL COOLING PACKS ARE NOT ACCEPTABLE.

After collection, all samples should be handled as little as possible. Field personnel should use extreme care to ensure samples are not contaminated. If placed in a cooler, ensure the sample container caps are tightly secured and the melted ice doesn't cause sample containers to become submerged, as this may cause cross-contamination. It is strongly recommended that all sample containers be placed in plastic bags (segregated by location) before introducing them into the cooler and then completely surrounded by ice. This is especially important when small sample containers (i.e., VOA and bacteriological) are used.

Additionally, samplers must follow the US Department of Transportation (US DOT) regulations (under the Transportation Safety Act) for shipping small quantities of hazardous materials found in [49 CFR 173.4](#).

After each sample is identified, by affixing the proper label, it is then placed in a cooler along with a

field sheet (and when applicable, a COC form). Each collected sample contained in the cooler is specified on the field sheet. Other field information such as sample type, collection time, date, sample collector's signature, station code, field analysis results and the method of shipment are also entered on the field sheet. The field sheet is then placed in a waterproof bag, sealed and taped under the lid of the cooler along with the samples to which it applies. Shipping containers are then secured with strapping tape to avoid accidental opening. For chain of custody purposes, a tamper-proof seal may also be placed over a bag or container containing the samples inside the shipping cooler. This seal should not be broken until the samples arrive at the laboratory and are checked in by laboratory staff. "COC" or "EMERG" shall be written in indelible ink on the cooler strapping tape to alert sample receipt technicians to priority or special handling samples. The date and sample handler's signature must also be written on the COC seal. COC samples sent through the Central Laboratory and then redirected for analysis by the Washington Regional Laboratory (i.e., total residue and total suspended residue samples received at the Central Laboratory on Mondays, Tuesdays and Wednesdays) should be packaged/containered separately from other samples in the cooler (or better, in a separate cooler) so that the COC seal need not be broken in the Central Laboratory before forwarding the samples to Washington. Place the field sheets and COC form inside of the sealed bag with total and suspended residue samples and identify this on the outside of the bag or cooler so that these can simply be iced and forwarded.

For safety and billing reasons, coolers must be packed so that a maximum weight of 50 pounds is not exceeded. Use additional coolers as necessary.

Packed samples shall be delivered to the laboratory by the sampling team, by State courier or via common carrier. If sent by common carrier, all documentation shall be sealed and placed inside the shipping container prior to sealing it closed. Receiving room staffs normally check in samples in the order the coolers are received or stacked. When coolers contain short hold time samples (e.g., coliform and hexavalent chromium) collected early the previous day, these samples often risk exceeding hold times before they can be identified, checked in, and expedited to appropriate analytical unit for analysis. Collectors can greatly increase the possibility of having these samples recognized and checked in first by identifying these priority contents on the outside of coolers using tape or labels.

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5.0 Sample Identification Requirements

All sample containers must be labeled or tagged to prevent misidentification of samples. (Example [Surface Water Protection tag](#) and [Aquifer Protection tag](#)) At a minimum, the label or tag shall identify the sample with the station number or sample identification (sample ID), date of collection, analysis requested, collector, and preservative(s). **The time of collection must also be documented for the following short hold parameters: coliform and chlorophyll a so that set up deadlines can be readily calculated by the analysts.** Additional information (i.e., a location identification code) may be included as a part of the tag or label. For typed tags or tags with pre-printed labels, EPA Region IV has suggested that the sample collector initial the preservative on the tag to verify the sample was preserved with the appropriate chemical preservative. Note: The Aquifer Protection Section has decided to generally exclude water supply well samples from the 3030C metals preparation procedure requirement. Field staffs are required to check "water supply well" on sample tags and to list "water supply well" on field sheets when appropriate. The information on the sample label or tag must unequivocally link the collected sample to the field sheet (and COC documentation, when used) and must be written legibly and in indelible ink. The label or tag shall be attached so that it does not contact any portion of the sample that is removed or poured from the container. As stated in the previous section, each of the individual vials submitted for a VOA sampling site must be labeled to prevent misidentification of any single vial in the field or in the laboratory.

Environmental samples must always be considered to be hazardous to the health of the person performing the sampling. The samples can have toxic, corrosive, explosive, and flammable properties. As a safety precaution, any sample suspected to be hazardous or heavily contaminated should be identified as such on sample labels, field sheets and COC documentation.

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6.0 Sample Custody and Documentation

A sample transmittal form or field sheet (with the associated COC record, when used) must accompany all samples that are submitted to the DWQ Laboratory Section. This record is designed to be a summary form for each set of samples. The history of a sample must be clearly evident from the retained records and documentation. Copies or originals of all documentation that are associated with the analysis or sample collection event must be retained. This includes the documentation that is sent to or received from all sampling and analysis groups. The records must contain enough information so that excessive clarifications, interpretations or explanations of the data are not required from the originator.

There are two levels of custody: 1) Sample transmittal and 2) Legal or evidentiary chain of custody.

Sample transmittal documentation is required by DWQ. It includes all records and documentation necessary to trace a sample from its point of origin through the final report. Sample custody requires that each event or procedure to which the sample is subjected be documented. These include, but are not limited to: sample collection, field preservation, sample receipt and log in, sample preparation, and sample analysis. In addition, those tasks or activities that relate to each of the above-mentioned events (e.g., reagent preparation, calibration, preventative maintenance, quality control measures, etc.) must be documented. The history of the sample must be readily understood through the documentation. The required documentation associated with sample transmittal is outlined in [Section 6.1](#).

Legal or Evidentiary Chain of Custody (COC) is a special type of sample custody that requires the physical possession, transport and storage of a sample be documented in writing. The records must account for all periods of time from sample container acquisition through sample analysis and reporting. If implemented, the minimum documentation requirements outlined in [Section 6.2](#) must be followed.

All records shall be maintained in a manner that facilitates documentation tracking and allows historical reconstruction of all analytical events and ancillary procedures that produced the resultant sample analytical data. The system shall link all documentation through the final analytical result. This may be accomplished through either direct or cross-references to specific documentation. The system shall be straightforward and shall facilitate the retrieval of all working files and archived records for inspection and verification purposes. Final reports, data summaries, or other condensed versions of data that have been prepared by external parties shall be linked to internal records by an unequivocal cross-referencing mechanism (usually field and/or laboratory ID numbers).

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6.1 Sample Transmittal Documentation

The forms (commonly referred to as "field sheets") that are used by the Department of Environment and Natural Resources to document this type of information are: DM-1 for Surface Water Protection (SWP) samples and GW-54 for Aquifer Protection Section (APS) samples and UST-54 for

Underground Storage Tank Section (UST) samples. A separate form is used for fish tissue and SWP sediment samples. These forms can be downloaded from the intranet portal at <http://portal.ncdenr.org/web/wq/lab/staffinfo/samplesubmit/forms>.

The following information shall be recorded on the appropriate field sheet and/or in a bound notebook with serially numbered pages for all sampling events:

- **Location Code** - A location code is a unique identifier for a single sample location that will house all the data gathered for that particular location. A location code must be recorded on each field sheet. Read more about assigning location codes on the intranet portal at <http://portal.ncdenr.org/web/wq/lab/staffinfo/labworks>.
- **Name of sample collector** - If possible, one member of the field team should take all the notes, fill out tags, etc., while the other member does all of the sampling at each location. To provide consistency, each sampler must use the following format for recording his/her name: the initial of the first name followed by the last name. (For example, Betty Smith would record her name as "BSmith".) **IMPORTANT:** When the collector name is illegible or not written in the proper format, it will be impossible for the collector to retrieve their results electronically. **NOTE:** If more than one collector is listed on the field sheet, the first (legible) name written will be entered into LabWorks™ meaning it will be that collector who will be able to access the report electronically and this is where the hardcopy report will be sent.
- **Date and time (beginning and end, if appropriate) of sample collection** – The date and time collected must be documented for each sample to ensure hold times are met. It is recommended that all time be recorded using 24-hour notation (e.g., 1:00 PM is 1300 hours). Dates should be recorded in the following format - YYMMDD.
- **Specific description of sample location** - including sub-basin (use the proper acronym for these), site name and address. The specific sampling point must be further identified (well number, outfall number, station number, etc.). For Surface Water Protection ambient samples, STORET numbers are to be used to document the station number. Contact [Andrea Thomas](#) at (919) 733-9960 for this information.
- **Analyses requested including analytical method when appropriate** – Assure the parameters requested are accurate and that a properly-preserved sample is submitted for each parameter requested on the field sheet. When field sheets are pre-printed and the decision is made in the field, not to submit a sample for any designated parameter, the collector should write "NS" beside the checked parameter to indicate No Sample submitted for that parameter. For dissolved analyses, the sample must be filtered in the field using a 0.45µm pore filter. The filtered sample is to be submitted to the laboratory and "DIS" should be written in the space to the left of the parameter requested on the appropriate field sheet.
- **Preservative name or abbreviation and preservative strength.** The following abbreviations (these should be written in the space to the right of the analysis requested or may simply be written on the sample tag, however, a permanent record is preferred) may be used:
 - HCl - hydrochloric acid
 - 1+1 HCl – hydrochloric acid, 1+1
 - 25% H₂SO₄ - sulfuric acid, 25%
 - 1:1 H₂SO₄ - sulfuric acid, 1:1
 - 1+1 HNO₃ - nitric acid, 1+1
 - 6N NaOH - sodium hydroxide, 6N
 - 0.008% Na₂S₂O₃ - sodium thiosulfate, 0.008%
 - 2N Zn Acetate - zinc acetate, 2N
 - NaHSO₄ - sodium bisulfate
 - H₃PO₄, conc. – phosphoric acid, concentrated
 - ICE - for samples that are thermally preserved only

- **pH verification** (when applicable) – For samples requiring pH adjustment, the effectiveness of this adjustment should be verified by the sample collector prior to sample transport. Because the buffering capacities of different matrices can vary considerably, the collector must verify adequate preservative was used to achieve the desired pH. This is verified again by laboratory personnel in the appropriate preparation or analytical unit.
- **Types of quality control (QC) samples collected** - Include when and where collected, preservative (if applicable) and type (e.g. trip blank including its source, equipment blank, field duplicate, etc.) QC samples must be documented in the same manner as all other samples and must meet all container, preservation, sample volume and hold time requirements specified for the parameter of interest.
- **Mode of delivery** – common carrier (such as UPS, FedEx, etc.), state courier or hand-delivery
- **Comments section** – a place to record sample conditions, safety, etc.

Additional documentation may include:

- **Ambient field conditions** - to include but not limited to information such as weather, tides, etc.
- **Type of composite** (e.g., flow proportioned, continuous, etc.) - Composite samples (if collected) shall indicate number of sample aliquots in the composite and approximate amount/quantity of each subsample.
- **Temperature of samples** (i.e., water temperature) at the time of collection - This is important for short transport times to document a downward trend during the cooling process.
- **Field measurement data** (e.g., pH, specific conductance, etc.) - records shall indicate when measurements were taken.
- **Calibration information** - to include time of all calibrations or calibration checks, concentration of standards and calibration acceptance (this information may be kept in a separate field calibration log).
- Occasionally, information such as the **reporting limits needed, specific method** that must be used, report deliverables and turnaround times required, etc. are documented on the COC or field sheet.

Entries into all records must be written legibly and must be made with waterproof ink. All documentation entries shall be signed or initialed by responsible staff. Entries in records shall not be obliterated by erasures or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. See instructions for completing [Surface Water Protection](#) and [Aquifer Protection/UST](#) field sheets.

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6.2 Legal or Evidentiary Custody Procedures

Legal Chain of Custody (COC) protocols are procedures designed to document and track all time periods and the physical possession and/or storage of sample containers and sample from point of origin through the final analytical result. The primary objective of sample custody is to ensure the integrity of the sample from collection to data reporting by creating an accurate, written, verified record that can be used to trace the possession and handling of the samples from the moment of collection through sample analyses and data reporting. It can be used to demonstrate that the sample and/or sample containers were handled and transferred in such a manner to eliminate possible tampering. Adequate sample custody will be achieved by means of approved field and analytical documentation, document control and review.

Chain of custody is necessary if there is any possibility that analytical data or conclusion based upon that data will be used in litigation. Discretion should be used when requesting COC. It should be noted that COC samples are not necessarily given priority. The only difference between routine samples and COC samples is the custody documentation trail. Samples that require priority handling should be designated as "Emergency" on the field sheet. Samples are only to be designated as "Emergency" when concurrence with the collector's immediate supervisor is understood since those samples are given priority over all other samples and may cause other samples to exceed hold times.

The COC records shall establish an intact, contiguous record of the physical possession including: collected samples, sample aliquots, and sample extracts or digestates. The COC records shall account for all time periods associated with the sample. The COC records shall include signatures of all individuals who are actively involved with physically handling the samples. A short statement that describes the activity of the signatory (e.g., received by, relinquished by, etc.) shall accompany each signature. In order to simplify record keeping, the number of people who physically handle the sample should be minimized. The COC form should be initiated by the sample collector.

The laboratory COC procedures are valid only when the field COC is properly performed and documented.

Documentation responsibilities for field sampling operations are:

- Preservative
- Recording equipment used in the field
- Calibrations associated with that day's sampling and field analysis
- Recording location, and acquisition information
- Attaching labels
- Filling out COC forms

Tracking records shall include, by direct entry or linkage to other records:

- Time of day and calendar date of each transfer or handling procedure
- Signatures of transferors and transferees
- Location and security conditions of samples (if stored in field)
- Storage conditions for the samples including chemical and thermal preservation
- Unique identification for all samples
- Common carrier documents
- Sampling site name and address
- Date and time of sample collection - It is recommended that all time be recorded using 24-hour notation (e.g., 2:00 PM is 1400 hours).
- Unique field identification code for each sample source and container (assigned by the Laboratory Section upon receipt)
- Name of personnel collecting samples
- Number of sample containers. (Count each of the 4 vials submitted for a single VOA sample, i.e., 1 sample=4 containers)
- Required analyses (by approved method number where applicable)

Transport containers shall be sealed with strapping tape and a tamper-proof custody seal shall be affixed directly to the sample bottle/cap or the bag/container holding the sample bottles in the cooler. The custody seal must have space for the signature of the person who affixed the seal along with the date and time the seal was affixed. The seal shall be placed so that the transport container cannot be opened without breaking the seal. The time, calendar date and signatures of responsible personnel

affixing and breaking all seals shall be recorded on the COC form. Shipping bills (i.e., Federal Express, UPS, etc.) will be retained with the COC or field sheet. Local samples are transported to the lab by DWQ staff or State courier.

Entries into all records must be written legibly and must be made with waterproof ink. All documentation entries shall be signed or initialed by responsible staff. Entries in records shall not be obliterated by erasures or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction.

Access to all evidentiary samples shall be controlled and documented. The number of individuals who physically handle the samples should be limited to those responsible for sample collection, initial laboratory receipt, sample preparation, sample analysis and sample disposal. A sample is considered under custody if it is in your possession, in your view after being in your possession, or placed in a secure area (e.g., sealed container for shipping or an area accessible by authorized personnel only) after being in your possession. When sample containers are shipped by a common carrier, the airbill must be completed and retained with the sample file. If samples are stored in the field prior to delivery to the laboratory, the location and security condition of those samples must be documented on the COC form. Chain of Custody seals may be placed on the outside of sample storage refrigerators or over the caps of individual sample bottles for added precaution. All persons visiting a building where samples are stored must sign a log upon entrance and exit. Only authorized personnel are to be permitted within laboratory areas where samples are stored. All external doors are either visually monitored by DENR staff or kept locked. Visitors are required to sign in and wear a visitor's badge during their visit and are accompanied at all times by a DENR staff member when in the laboratory.

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7.0 Sample Receipt Procedures

Sample receipt technicians are responsible for logging all samples into the lab and verifying all records are complete, correct and are entered into the sample custody records.

Upon receipt, the technician will record all samples into a master log and assign a unique number to each set (a set of samples is defined as all sample containers collected at a single site submitted for various analyses which are reported for that sample number). These numbers are written on sample labels, field sheets and COC records during login and are used to track the sample through the lab. Each number is assigned consecutively starting with the last number of the current year, and then a sample type code ("G" for Aquifer Protection and UST, formerly Groundwater and UST and "W" for Surface Water Protection, formerly Water Quality) followed by a unique sequential number. For example, the tenth set of samples logged in the year 2001 for Aquifer Protection would be assigned the following DWQ lab number: 1G0010.

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7.1 Initial Check of Samples and Login Documentation

If shipping containers arrive intact, they are immediately opened by the custody technician in the receiving area, and the field sheet, COC form and temperature blank container are removed for inspection.

The following checks are also performed upon receipt:

- Verification of the integrity and condition of all sample coolers.
- Verification of the integrity and condition of all sample containers.

- Checks for leakage, cracked or broken closures or containers, evidence of grossly contaminated container exteriors or shipping cooler interiors, and obvious odors, etc.
- Checks for air headspace in VOA and sulfide samples.
- Verification of receipt of complete documentation for each container - the minimum information for each sample container must include the items listed previously.
- Verification that sample identification numbers, on sample transmittal forms, correspond to sample identification numbers on the sample containers.
- Verification that COC procedures, when applied, have been properly carried out and documented.
- For samples that require thermal preservation (i.e., wet ice), sample receipt technicians will verify proper storage temperature by determining that sample containers are in adequate contact with wet ice in the shipping chest and by documenting sample temperatures to be less than or equal to 6°C (or less than 10°C for bacterial tests).

The following techniques may be used to verify the actual sample temperature (listed in procedural order):

1. The temperature may be verified by determining the temperature of a temperature control sample (temperature blank) that has been shipped with the sample.
2. The temperature of submitted samples may be measured by pouring an aliquot of a sample into another container. Failure to include a temperature blank, requiring this procedure, creates the risk that a sample may then have too little volume for successful analysis.

UNDER NO CONDITION SHALL A THERMOMETER OR OTHER TEMPERATURE MEASURING DEVICE BE PLACED INTO THE COLLECTED SAMPLE CONTAINER. Container temperature upon receipt is documented on the field sheet.

Samples received at the Central Laboratory and then shipped to the Washington regional laboratory for analysis (i.e., total and suspended residue samples) are checked for proper thermal preservation upon receipt at the Raleigh laboratory. The temperature is checked and recorded on the field sheet. Samples are stored at 4°C and then repacked with ice before shipment. When COC samples are involved, the temperature is verified using the temperature blank. A transmittal form is then completed with sample location, date, time (field sheets should be folded and placed in the plastic bag so that this information is visible) and the temperature. This form is sealed in a separate plastic bag, taped to the lid of the cooler and shipped with the samples. This way, the COC is not broken or compromised.

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7.2 Verification of Sample Preservation

After addition of the sequential identification number and initial check of condition upon receipt, the samples are distributed to the appropriate laboratory unit sample storage areas. Bound sample storage temperature logs are maintained for all sample storage refrigerators to assure proper temperature maintenance (i.e., less than 10°C for bacterial samples and less than or equal to 6°C for all other samples requiring thermal preservation – with no evidence of freezing) throughout the analytical process.

Sample receipt technicians shall verify proper field preservation of each sample by examination of documentation received from the field-sampling party. There must be clear documentation of any chemical preservation of the sample on sample tags. This documentation must demonstrate proper preservation per approved preservation protocols listed in the preservation and hold time tables ([TABLE I – SWP](#) and [TABLE II - APS](#)).

All samples are checked to verify proper pH adjustment and dechlorination by the appropriate preparation or analytical unit soon after receipt in the lab, however, the sample collector should verify the effectiveness of these processes in the field prior to sample transport.

Laboratory personnel shall verify the pH of all acid-preserved or base-preserved samples as soon after receipt as possible and before any sample preparation or sample analysis procedure (excluding VOAs and coliforms which are checked following extraction or sub-sampling of aliquots for analysis). Additional pH checks and adjustments, where required by the approved method, shall be documented. The proper pH adjustment, as stipulated by approved preservation protocols or approved sample preparation methods, shall follow the method-prescribed procedures. If none are specified, the pH shall be determined as follows:

- Use narrow-range pH paper.
- Do not contaminate the raw environmental sample by contact with pH paper or pH electrode.
- Use non-contaminating transfer implements, if necessary, to obtain a sample portion for use in the pH check procedure.
- Check pH of VOA and coliform samples after taking aliquot for analysis, or check pH on a duplicate sample that can be sacrificed for this purpose.
- Pour a portion of the sample on the pH paper, unless the sample is an analytical portion that cannot suffer significant quantitative loss. In this case, transfer a test specimen with disposable pipette, or other non-contaminating transfer implement, to the pH paper.

Samples subject to chlorine interference are checked for residual chlorine by the appropriate preparation or analytical unit. Laboratory personnel shall check samples for residual chlorine using the above procedures as soon after receipt as possible and before any sample preparation or sample analysis procedure (excluding VOA and coliform samples) and document the process.

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8.0 Rejection of Received Samples

Sample receipt technicians use the "[Sample Condition Upon Receipt](#)" (SCUR) form to document any anomalies observed in sample integrity, sample identification and proper preservation. Documentation of incomplete information is also recorded for each container. Discrepancies that may be noted at login include:

Cooler Condition

Samples were not received on wet ice.
No temperature blank submitted
Temperature >6° C (or ≥10°C for bacterial tests)
Samples frozen

Container Condition

Leaking
Broken
Without labels
VOA vials with headspace (approx. >5 mm bubble)
Sulfide samples with headspace

Container Label Condition

Not the same ID/info. as on COC
Not the same ID/info. as on field sheet
Incomplete. Missing the following:
Station #/Sample ID
Collection date
Collector
Analysis
Preservative
Other: _____

Markings smeared or illegible
Torn

Sample Documentation Discrepancies

Samples not received, but listed on field sheet
Samples received, but not listed on field sheet
Samples not received, but listed on COC
Samples received, but not listed on COC
Mislabelled as to tests, preservatives, etc.
Holding time expired.
Improper container used
Insufficient quantity for analysis

Chain of Custody Discrepancies

No custody seals
Custody seals not intact
Not relinquished
No date/time relinquished
No signature
Incomplete information

General Documentation Discrepancies

Field sheet wet/illegible
Field sheet incomplete
Records not written in indelible ink

If any of the problems outlined above occur, the sample submitter or contact person (usually the Regional Office Supervisor) will always be notified immediately and the sample receipt technician will document the problem on the SCUR form. In addition, the contact person will receive a copy. If possible within operating schedules, the Laboratory Section will obtain concurrence or further instruction from the sample submitter regarding any proposed rejection. All correspondence and/or conversations concerning the final disposition of the samples shall be documented on the SCUR form. The Quality Assurance Coordinator and/or receipt staff will attempt to resolve custody discrepancies expeditiously to avoid holding time compromises.

If the client insists on analysis of a compromised sample, the out-of-control event(s) will be fully documented and kept with the file and the sample data will be appropriately qualified as estimated or invalid on all internal documentation and on the final report. After a decision concerning a sample has been made, the sample receipt technician makes a note on the SCUR form that indicates the person notified, time date, and resolution if applicable. This information is also recorded on the field sheet or COC form and initialed. Copies of this documentation are maintained in the sample file.

For the most part, the DWQ Laboratory Section will reject samples if deficient in any of the items listed in [TABLE 1 - SWP](#) and [TABLE 2 - APS](#). Analysis will only be considered if the samples are designated "Emergency". If an anomaly is reported for COC samples, the samples may be analyzed, however they will be treated as routine samples. The custody chain will be considered broken. Rejected samples shall be logged in the laboratory sequential log with appropriate comment/qualifiers.

Chemical preservation and analytical anomalies are documented on similar forms (i.e., Sample Anomaly Report [SAR] forms) and the corrective action procedures outlined above are followed.

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9.0 Laboratory Records

Hard copies of final reports are kept in folders and filed using the following guidelines:

1. by year
2. by analytical parameter
3. by assigned lab number

Lab numbers are filed in descending order. All COCs, field sheets, anomaly forms, etc. are kept in the file with the reduced data. All final reports can be linked to internal records by lab number. Results may be accessed by the data user via computer after analysis is completed and approved (refer to the [LabWorks Info page](#) on the intranet portal for instructions on accessing your data).

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10.0 References

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40 U.S. Code of Federal Regulations Part 136 "Guidelines for Establishing Test Procedures for the Analysis of Pollutants".

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