ASSESSMENT OF ATMOSPHERIC DEPOSITION EFFECTS ON NATIONAL FORESTS

Protocols for Collection of Supplemental Stream Water and Soil Composition Data for the MAGIC Model

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1.0 PURPOSE

Numerous National Forests of the USDA Forest Service have initiated assessments to determine if acidic deposition is adversely affecting the health of forested watersheds. The focus of this document is to describe the water and soil sampling protocols for the Chattahoochee, Cherokee, Pisgah, Nantahala, National Forests in Alabama, and Sumter National Forests. An important Forest Service (FS) goal is to identify which watersheds may be experiencing, or are predicted to experience, base-cation depletion. The principal surrogate used to indicate watershed acid-base status and associated soil base saturation is stream water acid-neutralizing capacity (ANC). Stream water data, including ANC, have been obtained from a number of watersheds. Additional work will involve application of MAGIC (Model of Acidification of Groundwater in Catchments; Cosby et al. 1985a, b) to predict future watershed conditions and determine critical loads of future atmospheric deposition that may be required to provide adequate resource protection. Watersheds that currently have, or are predicted in the future to have, stream water ANC below a critical threshold may be candidates for more-intensive study and perhaps intervention with specific fertilization to improve watershed health. This protocol document is designed to support additional water quality and soils data collection that may be needed in association with this modeling and assessment effort. The purpose of this protocol is primarily to provide data for MAGIC modeling, but the resulting data could also be used in more general assessment efforts aimed at understanding acidification and nutrient depletion cause/effect relationships within the study areas.

The general provisions of this protocol were derived from a number of protocol documents associated with acidic deposition effects assessments and other studies of upland watersheds (Appendix A provides a listing of protocols and methods manuals reviewed). The specific provisions of this protocol have been determined by the need for efficiency and the specific data input requirements of the MAGIC model.

2.0 THE NEED FOR SUPPLEMENTAL DATA

Although the FS seeks to assess previous, current and future forest health effects of acidic deposition, water quality and especially soils data are available for only a subset of the watersheds that are potentially of concern. Decisions to collect additional data will need to address the adequacy of existing data as a basis for characterizing current acid-base status (stream water ANC and soil base cation supply) and to reliably model the future acid-base status of the population of acid-sensitive watersheds within the region.

2.1 Water Quality Data

The adequacy of available water quality data can be determined based on the distribution of sampled sites across geologic sensitivity classes (where there have been no previous glaciations), capacity to regionalize information on current status, and on the need for additional data for MAGIC model application. In the absence of probability-based site selection, effective regionalization will depend on the ability to explain variance in the available data as a function of mappable landscape characteristics.

Previous work in the southern Appalachian region has shown the acid-base status of surface waters (and watershed soils) is mainly a function of watershed bedrock type (c.f., Sullivan et al. 2002). Within the model, the importance of bedrock type is reflected in the soils acid-base chemistry data that are associated with a given bedrock type and the drainage water chemistry data that reflect spatial and temporal integration of lithologic and edaphic factors within the watershed. The availability of regional-scale geologic maps thus provides a basis for regionalization of water quality data obtained for a limited number of sites. Statistical analyses can be used to determine the relationship between the available water quality data and defined bedrock classes present in the subject watersheds. Decisions to collect more water quality data or to simply rely on available data should be based, in part, on the strength of the observed relationships and the degree of coverage with respect to the range of bedrock classes in the region. Additional decisions to collect more water quality data may relate to the input needs of MAGIC and to decisions concerning co-location of water quality and soils data collection.

Selection of sites for additional water quality sampling should be based on systematic and documented criteria. The criteria should include representation of bedrock and/or soil classes, geographic distribution, elevation, watershed size, site accessibility, and avoidance of watersheds with disturbance that might influence stream water composition. Approximate sampling site locations can be identified on a preliminary basis prior to initial sampling trips, with specific site selection and further documentation developed in association with sampling, as described below.

Additional decisions will need to be made concerning the frequency of sampling. Springtime samples are usually regarded as the best representation of water quality when only single samples can be collected. Better representation of annual conditions can be obtained with seasonal or other periodic sampling.

2.2 Soil Composition Data

There is very little soil acid-base chemistry data available for FS lands (especially in the Alabama, Georgia, North Carolina, South Carolina, or Tennessee) that are adequate for calibrating the MAGIC model. Initial MAGIC model calibration to a particular stream might therefore depend on the use of soils data from similar landscapes (for example, with similar or closely-related geologies) in the surrounding region. Collection and analysis of soils samples within each of the watersheds (or catchments) to be modeled is the preferred approach. However, if that is not possible, regional-scale geologic maps and defined bedrock classes could provide a basis for assigning available soils data to each of the specific watersheds to be modeled (Caution will need to be used since some area may have a geological outcrop that results in a subtle chemistry changes in the catchments). The adequacy of this approach can be evaluated based on the level of uncertainty (or range of possible ANC values) for model output associated with individual watersheds or groups of watersheds defined by bedrock class. In general, we recommend collection of soils within each watershed to be modeled. Additional soils data will certainly need to be collected if the uncertainty of model output is considered unacceptable or if soils data are not available to adequately represent the range of bedrock classes and soils types on those bedrock classes within the region.

Selection of watersheds, and sites within watersheds, for additional soils sampling should be based on systematic and documented criteria. The criteria should include such factors as representation of bedrock classes, topographic and geographic distributions, watershed size, site accessibility, and avoidance of watersheds with disturbance that might influence soil composition. One approach to selecting specific sites in individual watersheds is to locate samples on midslope positions (in the catchment), with each selected to represent a contiguous unit of single bedrock type, forest cover, and slope class. The preferred approach is to collect samples from a limited number of soils sites (generally two or three soil pits per watershed for MAGIC calibration) that will be selected using a stratification process according to bedrock type, general soils type, and/or vegetation coverage. Resource and sampling logistical considerations may make it necessary to locate multiple sites in a small number of catchments rather than to distribute single sites among a larger number of larger (e.g., 6^{th} level HUC) watersheds. Approximate sampling site locations can be identified on a preliminary basis prior to initial sampling trips, with specific site selection and documentation provided during sampling, as described below.

2.3 How the Data Might Be Used

The resulting soils and water quality data can be used to calibrate the MAGIC model to one or more specific watersheds, each defined as the area of land that drains to a particular stream sampling point. Model calibrations can then form the basis for estimating past acidification and/or projecting future changes in stream water chemistry on a site-specific (c.f., Sullivan et al. 2003) or regional (c.f., Sullivan et al. 2002) basis. The model output data could also be used to estimate the critical load of deposition that would be required to protect water acid-base chemistry against acidification to specified critical levels, such as ANC = 0, 20, or 50 μeq/L (c.f., Sullivan and Cosby 2002, Sullivan et al. 2003).

3.0 STREAM WATER DATA COLLECTION

3.1 Arrangements with the Water Chemistry Laboratory

Appropriate agreements or contracts will need to be made with a qualified water chemistry laboratory well in advance of field sampling. Besides processing the water samples, the laboratory should prepare and provide sample bottles, insulated shipping containers, and refrigerant, as described below (sections 3.2.3 and 3.2.4). Historically, the USFS/USGS Water and Soil Lab in Fort Collins, Colorado has performed the analyses on water chemistry samples collected in the National Forests.

3.2 Sampling Materials

Each person or sample-collection team should be provided the following equipment and materials, except as noted.

More details on equipment and materials follow in sections 3.2.1–3.2.4. Instructions for use are in sections 3.3 and 3.4, and the Standard Operating Procedures in Appendix C, Table C-1.

- Site information folders (including maps and Stream Water Field Data Forms)
- Site documentation materials

- Sampling protocol (this document)
- Sampling bottles (each preferably placed in a plastic bag) and syringes. The syringes should be placed in a light-weight plastic box with snap-on lid, large enough to hold multiple syringes with plunger pulled three-fourths of the way out.
- Plastic gloves stored in a secure plastic bag
- Insulated containers, refrigerant, and backpacks
- Thermometer
- Wrist watch
- Survey grade global positioning system and compass
- 50 or 100 meter tape (and, if available, laser rangefinder) to measure distances
- Labels and waterproof markers
- Number 2 pencils, or write-in-rain pens, and notebooks
- Digital camera with extra memory cards and batteries
- Aluminum tags if the sites are being established for long-term monitoring of water chemistry

3.2.1 Site Information Folders

The water chemistry samples taken on National Forest lands are collected within a Wilderness, or 6th level Hydrologic Unit Code (HUC) to determine if any of the area of interest may have low amounts of bases to neutralize the acids from natural and anthropogenic sources. The 6th level HUC was chosen because the Forest Service may own a large percentage (20 percent or more) of these watersheds. Typically, 10 first or second order streams are chosen in each HUC to be sampled. The criteria taken into consideration for the stream selections include ease of accessing the site, lithology, wet sulfate deposition estimates, elevation, and if the area has special significance (for example, a wilderness). The information documenting the site information, the sample information, and field measurements (see the field data sheet in Appendix B) for each sample site will be found in the HUC folder.

Site documentation information in the site folders will include site maps, narrative access descriptions, site coordinates and elevation, site-tag numbers (for long-term monitoring sites) and locations, and site photographs.

Maps will include 1:24,000 USGS quadrangle maps with sampling sites identified and Forest recreation maps to help navigate to the area. Maps generated using GIS will also be included to show where the project manager has selected potential sites to sample (see Figure 1 below), or where the sites were sampled in a previous survey.

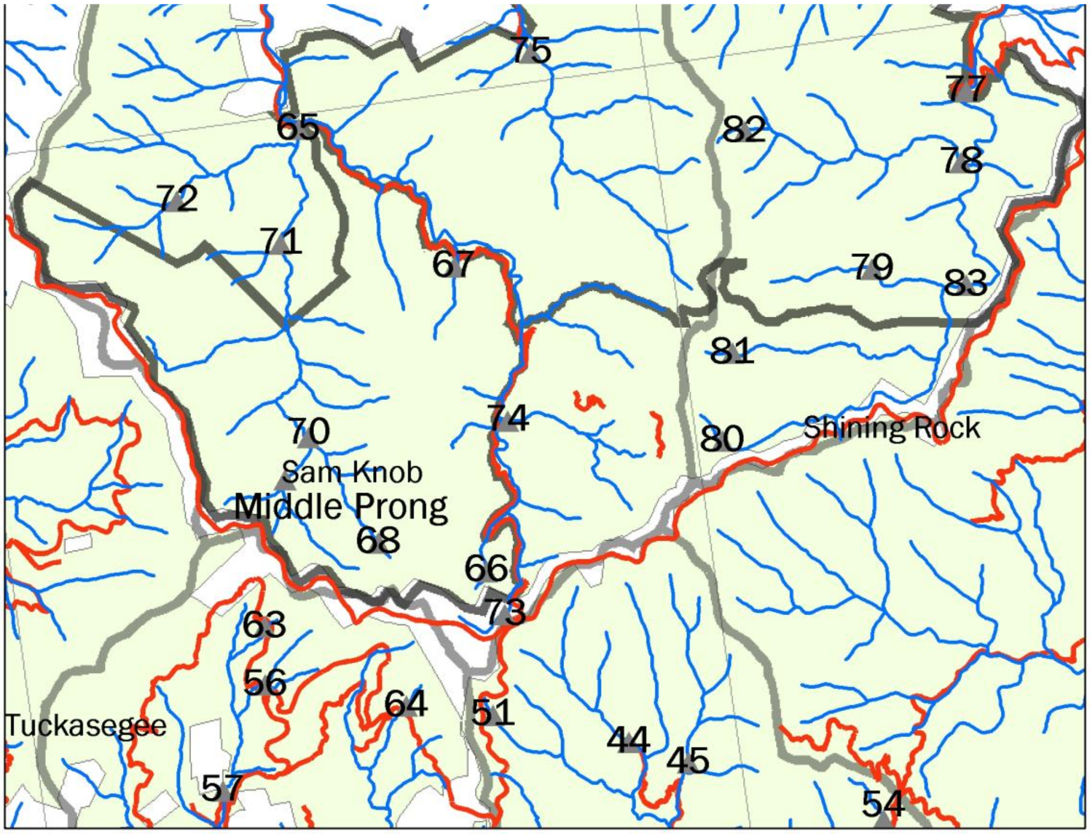

Figure 1. Example of a GIS-generated preliminary sampling site map. The site numbers and triangular symbols indicate preliminary or approximate sampling site locations. Selection of the specific sampling site is based on conditions observed in the field. USGS 1:24,000 quadrangle maps can also be used.

The Surface Water Chemistry Long-Term Monitoring Record data sheets will serve for documenting site information, sample locations and field measurements. These forms should be printed on waterproof paper if rainy weather is expected during the field sampling. A copy of the Surface Water Chemistry Long-Term Monitoring Record data sheet is provided in Appendix B.

3.2.2 Site Documentation Materials

In addition to the material described above for inclusion in site documentation folders, site documentation materials should also include uniquely numbered aluminum tags for sites planned to be sampled repeatedly (i.e. monitoring sites) or replacement of missing tags at previously established sites, nails and a small hammer for tag placement, flagging tape, a camera for site photographs, a GPS unit for determination of geographic coordinates (in decimal degrees). Waterproof pens and pencils (needed in wet conditions) should be provided for completing forms.

3.2.3 Sample Collection Bottles

As indicated previously, the lab that is performing the analysis should normally be responsible for bottle and syringe processing and packaging. Sample collection bottles should be 250 ml (large enough to allow reanalysis, if necessary), wide-mouth HDPE or LDPE Nalgene bottles. Bottle processing should involve multiple rinses with distilled water (see section 3.5). The processed bottles should be stored filled with deionized water, which will be poured out at the time of sample collection. Label tape should be affixed to the bottles prior to packaging.

The field crew will have to take extra precautions to ensure that no bottle mouth or syringe is contaminated with leaking refrigerant, tap water, dirt, or other foreign substance. Each processed bottle should be packaged individually in a zipper-lock type plastic bag if the laboratory analysis indicates that contamination is an issue. The syringes should be placed in a light-weight plastic box with snap-on lid.

3.2.4 Insulated Containers, Refrigerant, and Backpacks

As indicated previously, the lab performing the analysis should be responsible for provision of insulated containers and refrigerant. Field crews will also need backpacks with waterproof covers for transport of samples and other materials when hiking into sites that cannot be accessed by vehicle.

Insulated containers, with chemical refrigerant ("blue-ice"), are needed for transport of collected samples between the field and the lab or other staging location. Small insulated containers that will fit into backpacks can be used to carry and protect the samples in the field. Larger insulated containers can be used for assembly and transport of samples in vehicles.

Ideally, chemical refrigerant packages should be packaged in zipper-lock type plastic bags to minimize the possibility of sample bottle contamination through leakage. The field crews will need to make sure the chemical refrigerant is placed in a freezer two days before sampling.

3.3 Sample Site Location and Documentation

Depending on the objectives for the field data collection, the field crews may be collecting water samples as part of a synoptic survey (inventory), or they may be repeating sampling at the same locations in a monitoring effort to examine changes in water chemistry over a period of time. The extent of documentation required will depend on whether the site is to be sampled once, or numerous times.

3.3.1 Locating Established Sampling Sites

Previously established sample-collection sites can be located by reference to maps provided in the HUC information folder, by reference to access notes provided on the Surface Water Chemistry Long-Term Monitoring Record data sheet, by reference to site photos, and by use of a GPS unit to determine geographic coordinates. It is desirable to enter site coordinates into the GPS unit in advance of sampling trips. For sites with aluminum tree tags, site confirmation is provided by location of the tag.

Forest recreation maps provide orientation and general access information. Specific site location information will be provided by USGS 1:24,000 quadrangle maps with sites indicated. In some cases, multiple maps will be required to provide needed access information.

The Surface Water Chemistry Long-Term Monitoring Record data sheet provides access notes, describing the route to the site and the location of the site. In most cases, the sampling sites will be described as adjacent to, immediately above, or a specified distance and compass heading above or below a particular landmark. Landmarks include bridges, trail crossings, marked boundary lines, confluence points, and other identifiable fixed features. If existing access notes are insufficient, additional notes or clarification should be added to the Surface Water Chemistry Long-Term Monitoring Record data sheet by the field crew. The back of the form can be used if additional space is needed.

The Surface Water Chemistry Long-Term Monitoring Record data sheet will indicate the tag number for any tag previously placed on a tree adjacent to the collection site. For sites with tags, the access notes will indicate the location and provide a description of the tag tree. In some cases, previously placed tags may have disappeared. If the tag is missing, the field crew should replace the missing tag before leaving the site or make a note for future tag replacement.

Ideally, site photos will provide upstream and downstream views of the sampling site, as well as views of the tag tree. If site photos are not available, photos should be obtained by the field crew as described in section 3.3.2.

3.3.2 Establishing New Sampling Sites

Note: avoid disturbance of water upstream of the sampling point before sample collection by performing site documentation steps (placement of site tags and site photography) after sample collection.

Site maps and access notes should indicate general or preliminary sampling site locations for new sites. The 6th level HUC folders for the sample sites will include USGS 1:24,000 quadrangle maps, as described above (section 3.3.1) for location of previously established sites and a GIS maps will be provided (see Figure 1) that indicates approximate sampling site locations. In either case, it will be the responsibility of the field crew to select and document the specific sampling site locations based on conditions observed in the field. The field crew should consider the following guidelines:

- Sample sites should be readily identifiable by reference to permanent-type landmarks such as confluence points of major tributaries, well-marked boundary lines, and stream crossings by permanent roads or well-marked trails.
- Sample sites should be selected to avoid direct runoff from roads and trails, as well as unmixed flow from tributaries. This will be achieved for most sites by sampling sites 25 yards above road or trail crossings, or 25 yards above or below inflowing tributaries.
- The best point to sample will be where the water is flowing fast or falling, where there are no eddies, and where the depth is at least six inches. Ideally the sampling point is one that can be reached during most flow conditions while kneeling on the stream bank or on stable rocks downstream from the sampling point. It should not be necessary to stand in the water to reach the sampling point.

The field crew will need to provide detailed site documentation for new sites. Site documentation consists of maps, access notes, site tags (for permanent plots), site photos, site elevation, and other items described in section 3.3.3 below. Preparation of site documentation for new sites should include the following steps:

1. Record additional information to clarify preliminary site and access descriptions in the space provided on the Surface Water Chemistry Long-Term Monitoring Record data sheet. Use the back of the form if additional space is needed for access description. Be sure to draw a site-sketch map in the box provided on the second page of the forms.
2. At monitoring sites, place site tags on a vigorous streamside tree of at least 6 inches diameter. The tag should be nailed with a small hammer on the downstream side of the tree a few inches above ground level. The nail should not be driven all the way in; leave about ¼ inch to allow for tree growth. Record the tag number, the tag location, the tag tree species, and the tag tree diameter at breast height in the **Field Notes and Observations** portion of the Sample Information section provided on the Surface Water Chemistry Long-Term Monitoring Record data sheet. It will be helpful to draw a map (including bearing and distances) detailing the relationship between the tag tree and the stream sampling site. *Do not place tags on trees in National Forest Wilderness Areas and do not place tags on trees on private land without permission.*
3. Record additional information to clarify preliminary site and access descriptions in the space provided on the Surface Water Chemistry Long-Term Monitoring Record data sheet.
4. Obtain sampling site photos showing the upstream and downstream views from each site. At the sites selected for monitoring the photos should include, in one lower corner of each photo, a large-print card on which is indicated the site ID code for that site. Photos should also be taken to include distinctive streamside features such as particular rocks and trees. Orange flagging should be placed (temporarily) on the tag tree to allow its identification in the photos. More distant views can be taken to provide perspective. Provide picture numbers in the **Field Notes and Observations** portion in the Sample Information section on the Surface Water Chemistry Long-Term Monitoring Record data sheet.
5. Determine site geographic coordinates if a survey grade GPS unit is available. Follow the GPS instructions to obtain the most precise readings that can be obtained with the unit. The units for projection will be a geographic projection (NAD83) recording the outputs in decimal degrees. Record the site coordinates in the **GPS Latitude** and **GPS Longitude** spaces in the Sample Information section provided on the data sheet.

3.3.3 Completing the Site Information Section of the Data Sheet

The field crews need to complete the Basic Site Information, Site Verification, GPS Information and Tagging, and Site Assessment portions of the form (see below) before leaving the site. The **Site ID** is a 13 character code combining the first seven numbers of the longitude and the first six numbers of the latitude. Sites located less than 100 degrees longitude will have a

"0" used in the first character place and there will be no minus character in the Site ID name.

Basic Site Information			
Forest Name:		Wilderness Name (if applicable):	
Stream Name (USGS):		Stream Name (Local):	
Site Name:		Site ID:	
Date of Visit:		Visit: ☐ Initial	☐ Subsequent
Field Team Leader:			
Affiliation:			
Phone:		Email:	
Access: ☐ Vehicle	☐ Short Hike (< 1 hr)	☐ Long Hike (> 1 hr)	☐ Overnight Hike
Travel Directions to Stream Sampling Site and Access Information:			

Site Verification, GPS Information and Tagging					
Stream Verified: ☐ Yes ☐ No	Site has been verified by (check all that apply):	☐ GPS ☐ Roads	☐ Local Contact ☐ Topo Map	☐ Signs ☐ Photos	☐ Vegetation ☐ Other
GPS Information	Latitude (DD) __ __ . __ __ __ __ __ __		GPS Accuracy:	☐ ft	☐ m
Datum:	Longitude (DD) -__ __ __ . __ __ __ __ __ __		Elevation:	☐ ft	☐ m
Site Tag has been Affixed?	☐ Yes ☐ No	☐ New Tag ☐ Existing Tag	Tag Tree Species:		
Describe tag tree location relative to stream sampling site:					

Site Assessment (Observations within 20 m of streambank)		
Streambank Character (use % classes below)	Dominant Age Class (forested areas only)	What are the dominant plant species (if known)?
______% Forest/Shrub		
______% Open Herbaceous	☐ 0 - 10 years	
______% Wetland	☐ 10 - 25 years	
______% Barren (beach/rock)	☐ 25 - 50 years	
______% Agriculture	☐ > 50 years	
______% Developed	Is there beaver activity near the sample site?	
______% Shoreline Mod. (dock, riprap)	Signs of Beaver: ☐ None ☐ Rare ☐ Common	Beaver Flow Modifications: ☐ None ☐ Minor ☐ Major
Rare (< 5%) Moderate (25-75%) Sparse (5-25%) Extensive (> 75%)		

The Watershed Assessment portion of the form will be completed after conducting the fieldwork with the aid of the ArcMap and Spatial Analyst® software. First, the project manager will compare where the sample sites were planned to be established (using ArcView® or ArcMap®) with the location plotted by the field crews on 1:24,000 quadrangle maps (see **Establishing New Sampling Sites** above). Examining the 1:24,000 quadrangle maps will also provide the information needed to complete the **Steam Name** and **Stream Order**. Some proposed sites may have not been sampled and need to be deleted from the spatial coverage; while others sites will need to be moved to where the actual sampling occurred.

WATERSHED ASSESSMENT	
What percent of the watershed above the sample site is?	Primary lithology type:
______% Hardwoods	Note additional significant lithology types:
______% Conifers	
______% Mixed Forest	
______% Exposed Rock	What is the watershed area above the sample site? __________ ☐ ha ☐ ac
______% Herbaceous/Shrubs	What is the watershed aspect (degrees)? ________°
______% Tallus	What is the average slope of the watershed? ________%
______% *Total*	What is the stream order of the sample site (use NHD dataset)? ________

The Select Feature tool in ArcMap can be used to select the HUC containing the sample points. The HUC value (character) can then be copied and pasted into a column (within the site point shape file that has been labeled HUC) using the Calculation Option which appears by selecting the HUC column with a right click of the mouse button. Of course it may be necessary to create the HUC column first in table option of ArcMap before the editing can be completed.

The value for the **Lithology** is determined by using the identify tool to determine what lithology type is present at each of the sample points. Using the Zonal Statistic option in Spatial Analyst along with a 10 or 30 meter digital elevation model (DEM) will provide an estimate of the **Elevation** for each sampling location if the data was not recorded in the field.

Next, it will be necessary to digitize within ArcMap the topographically-defined contributing or source area for that point in the stream (see Figure 2). This will be referred to as the catchment or watershed. Be sure to also include the appropriate HUC number and Stream ID in the catchment shape file database. The catchments are drawn with the aid of a 10 or 30 meter DEM overlaid on a hillshade map, tin coverage, and/or contour interval map developed with Spatial Analyst (see example below).

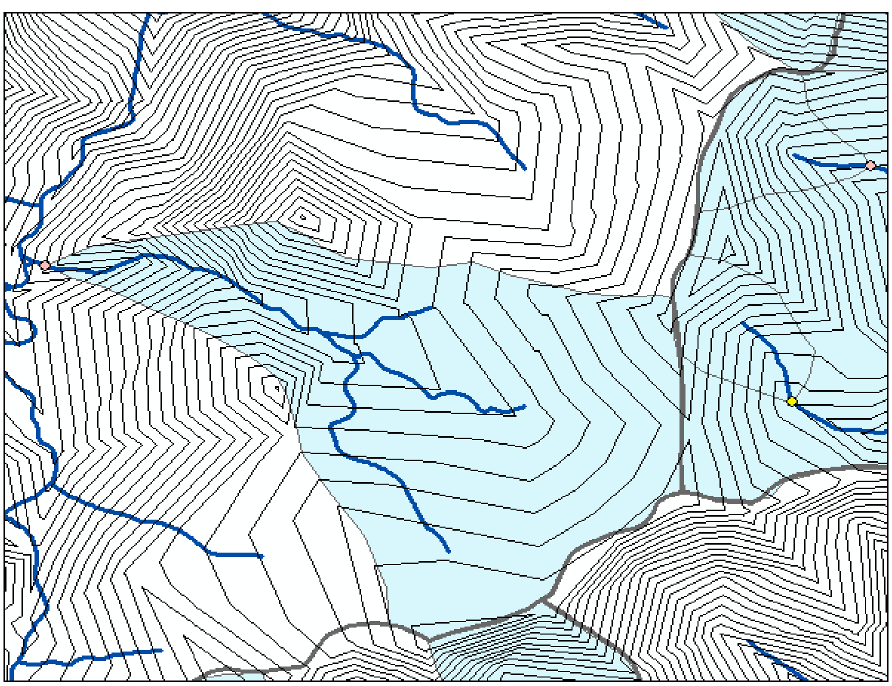

Figure 2. Catchments (light blue polygons) drawn in ArcMap for each sample point with the streams (blue lines), HUC boundary (gray lines), and contours (black lines) derived from a 30 meter DEM.

The remaining values are estimated using the catchment boundaries. The XTool utility is used to estimate the **Catchment Size** in hectares. The **Catchment Slope** is the average (mean) percent slope in the catchment, but first it will be necessary to use Spatial Analyst to derive the percent slope for the area of interest using the 10 or 30 meter DEM. Similarly, the **Catchment Aspect** is also derived using the 10 or 30 meter DEM and the average (mean) aspect is calculated using the Zonal Statistic option in Spatial Analyst. The average **Catchment Aspect** is then placed into one of the following categories:

337.5 – 22.5 = N	22.5 – 67.6 = NE
67.5 – 112.5 = E	112.5 – 157.5 = SE
157.5 – 202.5 = S	202.5 – 247.5 = SW
247.5 – 292.5 = W	292.5 – 337.5 = NW

The catchments can be examined with digital aerial photographs to estimate the percentage of the area above the sample site that is occupied by trees, exposed bedrock, meadow/shrubs, open areas, or other categories.

3.4 Sample Collection, Handling, and Documentation

3.4.1 Preparing for the Stream Sampling Trip

Obtain HUC folders for the sites to be sampled. The Surface Water Chemistry Long-Term Monitoring Record data sheet should be completed to the extent possible prior to the sampling trip. For previously established sites, the Site Information, tag number, and description of the tag tree should be filled out on each form. The following are the additional steps that need to be completed before going to the field:

- Obtain two sample bottles for each site to be sampled. Preprocessed bottles filled with deionized water should be provided by the analytical lab as indicated in Section 3.2.3. Ideally the bottles will have a blank label tape affixed to the bottles.

- Please note two water samples are collected at each site so that the second sample (stored in a refrigerator at the District Office) is available if the first sample is lost or contaminated, or the second sample is submitted as a duplicate. Duplicate samples will be processed at the laboratory for 30% of the sample sites.

- Syringes are used because the concentration of some analytes can change if the water sample equilibrates with atmospheric carbon dioxide partial pressure prior to analysis. Dissolved inorganic carbon (DIC) concentrations and pH are the measurements that are typically analyzed without contact with the atmosphere. This protocol recommends that aliquots of samples be collected in syringes along with the standard bottles, and that the syringe or septum-capped samples be used for analysis of pH and DIC. This precaution is considered to be more important for streams than it is for epilimnetic samples from lakes. Filled syringes should be transported from the field to the lab in plastic containers that minimize disturbance of the seal of the syringe.

- Throughout the water chemistry sampling process it is important to take precautions to avoid contaminating the sample. Many surface waters in regions of the United States considered sensitive to effects of atmospheric deposition have low ionic strength (i.e., low levels of chemical constituents). Samples from such waters can be contaminated quite easily by perspiration from hands, sneezing, smoking, suntan lotion, insect repellent, fumes from gasoline engines or chemicals used during sample collection.
- Obtain frozen refrigerant. Be sure the refrigerant has been in the freezer at least two days prior to the day of sampling. Place the refrigerant in a securely sealed plastic bag in the insulated container(s) that will be used for sample holding and transport. Provide enough refrigerant to keep the samples cold until delivery to the lab or until placement in a refrigerator if samples are to be stored before shipping.
- Transport the two water sample bottles in the cooler that will be used to store the samples.

3.4.2 Stream Sample Collection Procedure

Sampling should be avoided during abnormally low or high runoff conditions in order to obtain samples representative of common watershed conditions. USGS data (for example, North Carolina data are found at: http://nc.water.usgs.gov/info/h2o.html) can be examined prior to going to the field to estimate current stream flow from stream gauges in the area of the sample site. This precaution is more important when watershed characterization will be based on single samples rather than multiple samples collected at different times. MAGIC modeling is generally based either on annual average or spring baseflow water chemistry. The former requires intensive monitoring data collected throughout multiple years. The latter requires only one or a few samples collected during spring. In general, it is preferable, but not necessary, to base the modeling on multiple samples (either collected throughout the annual cycle or just during the spring season) collected over several years. MAGIC can be calibrated to monthly values, but is not an appropriate tool with which to examine episodic (storm-driven) processes. Often, the magnitude and frequency of episodic acidification is considered as an item separate from the modeling. In other words, MAGIC is used to project annual average or spring baseflow chemistry, and the probable extent of episodic acidification is assessed independently for both current and future conditions.

As described above (Sections 3.3.1 and 3.3.2), locate the sampling site and determine the exact sampling point in the stream. Carefully avoid disturbance upstream of the sampling point prior to sample collection. This means not walking in the upstream water or on upstream rocks.

Collect the sample on a step-by-step basis as noted in the Stream Standard Operating Procedures found in Appendix C. The following briefly describes the steps:

1. Open the cooler containing the sample bottles.
2. Remove the gloves from the plastic bag and put them on.
3. Remove the sample bottle from the cooler. Do not put the sample bottle or zipper-lock bag on the ground.
4. The sample bottle will be filled with deionized water. Pour this out at a location below the site selected for sampling and bottle rinsing.

5. If the sample bottle has not been prelabeled, use a permanent ink felt marker to add the site identification code and date to the affixed label tape. Label sample bottles prior to actual sampling, as wet bottles are difficult to label.

6. Walk carefully to a place 2 to 3 feet below the sample site. Rinse the sample bottle in the stream at a location 2-3 feet below the sample collection point. The bottle and cap should be rinsed 3 times. For each rinse, fill up the bottle and then pour the rinse water over the inside of the cap, held bottom-side up in the other hand. Pour the rinse water downstream of the rinsing and sampling points and avoid stirring-up streambed debris during the process.

7. After the rinsing is completed, move to the sampling point and collect the sample. Fill up the bottle as completely as possible, but note that it may be impossible to avoid a small air space in the bottle. While collecting the sample, avoid stirring-up streambed debris that might be collected with the sample. Also avoid collecting water that has come in contact with the gloves or the outside of the bottle. This can be best achieved by sampling rapidly flowing or falling water.

8. Immediately after collecting the sample, place the lid on the bottle (tightly) and return the bottle to the cooler. We recommend placement of the sample bottle back into a zipper-lock bag prior to putting the bottle in the cooler. Do not place the sample bottle on the ground.

9. Immediately repeat the above steps for the second sample. When the second samples are collected, the bottles should be labeled with the **Site ID** and **Duplicate 1**. About 30 percent of the samples will be processed at the laboratory as a duplicate sample.

10. Filled sample bottles should be placed in zipper lock bags prior to transport from the field site. Syringes should be packed in a generic light-weight plastic box with snap-on lid. Each box needs to be long enough to hold syringes that are ⅔ to ¾ filled with sample water, and wide enough to hold multiple syringes. The bagged sample bottles and boxed syringes should be packed with double-bagged ice or frozen refrigerant for transport.

11. Obtain the temperature of the stream water by submersing the thermometer (or pH probe) in the stream water until the reading is stable. Take the reading while the bulb of the thermometer (or pH probe) is in the water. Record the temperature to the nearest 0.1 degree C.

12. If pH and conductivity readings are to be obtained in the field, follow the procedures cited in section 3.5. The pH meter should be calibrated at each sample site with pH 4.0 and 7.0 buffer solutions. Because calibration of the pH meter is time consuming and because pH measurement can be difficult in the field, some investigators choose to not measure pH in the field, but rather to rely on subsequent laboratory analyses. It should be noted, however, that stream impairment listings might require field measurements.

13. Complete the appropriate sections on the data sheet while at the sample site (see Appendix B for further instructions).

3.4.3 Sample Handling

- Place collected samples in an insulated container with refrigerant within 15 minutes of collection.
- For sites that are close to road access, the insulated container for samples can be left in the field team's vehicle. The sample should be placed in the container upon return to the vehicle.
- For sites that require longer hikes (greater than 15 minutes), the collection team should make arrangements to keep the sample cold during the return hike. The recommended approach is to use an insulated container that will fit in a backpack.
- The samples will be stable for most analyses as long as they are kept on refrigerant or in a refrigerator until delivery to the lab. The refrigerant may need to be replenished during sample transit. Do not place the sample bottle in a refrigerator or container with food or in any container that is not clean.
- Samples that are to be shipped to the analytical laboratory will be packed in insulated shipping containers with frozen chemical refrigerant. The refrigerant should be packed in double zipper-lock bags.
- Overnight delivery will be used to ship the samples to the lab as soon as practical after sample collection. Ideally, a cooler full of samples is prepared for shipment each morning before going to the field to collect more samples. Ideally, samples should not be collected on Fridays or on days preceding a holiday when the laboratory will be closed. Samples collected on Friday must be held until Monday before they can be shipped. Check with the laboratory regarding recommended sample holding times.
- The Chain of Custody Form (used to maintain a record of sample custody until receipt at the analytical lab) must be completed for the samples in each shipment and a photocopy of the completed form will accompany the sample. The original will be stored in the HUC folder. An example of the Chain of Custody Form is provided in Appendix B.

3.5 Stream Water Laboratory Analysis

Application of the MAGIC model requires analyses of 10 stream water constituents:

- acid-neutralizing capacity (ANC)
- pH (lab, and if desired, also field measurement)
- 3 acid anions: sulfate, nitrate, and chloride
- 4 base cations: calcium, magnesium, potassium, and sodium
- ammonium

Conductivity should also be measured for purposes of quality assurance. If MAGIC is to be used to estimate future pH, then it will also be necessary to measure dissolved organic carbon, and if pH is below 5.5 to determine total monomeric aluminum and organic monomeric aluminum. If pH is below 5, it is also recommended to measure total fluoride, which will be needed to calculate aluminum speciation.

The following sections describe methods for sample bottle preparation, sample filtration, sample analysis, and quality assurance. These methods are appropriate for analysis of low ionic-strength stream waters associated with forested mountain watersheds in the southern Appalachian Mountain region. Although data obtained by the described methods have been successfully used for other acidification effects assessments and trend monitoring programs conducted in the region, the described methods are specifically proposed here for purposes of supporting an assessment of watershed health on FS lands in the upstate of South Carolina, eastern Tennessee, and western North Carolina. Other methods, as described by other protocols listed in Appendix A, may also be appropriate for this and other purposes.

The critical factors for use of this or other protocols for water quality analysis include documentation of collection and analytical methodology and quantitative determination of accuracy and precision.

3.5.1 Sample Bottle Preparation

Sample bottle preparation will involve a triple rinsing of each 250 ml bottle with deionized water. The bottles will then be stored overnight, or longer, filled with distilled water, followed by a final rinse with deionized water. Finally, each bottle will be filled with deoinized water before shipping to the project location. Treating the sample bottles in this manner maximizes the likelihood of achieving a conductivity for a blank sample that is less than the acceptance criterion of <1.2 uS/cm.

3.5.2 Sample Filtration

All samples are normally filtered using a 0.45 micrometer polyvinylidene fluoride filter as soon as possible after arrival at the laboratory, to remove particulate matter. The ion chromatographs have microbore tubing which can clog easily if even a small particle makes it's way into the tubing. Therefore, samples are kept cold and analyzed as soon as possible after arrival. The samples continue to be kept refrigerated for possible further analysis even after the initial analysis and QA process.

3.5.3 Sample Analysis

Currently, the USFS/USGS Water & Soils Lab in Fort Collins Colorado performs the chemical analysis for the stream water samples (http://www.fs.fed.us/rm/waterlab/Labprotocols.htm). The following is a summary listing of instrumentation and techniques employed for sample analysis. Equivalent instrumentation and techniques can be substituted, such as those employed by the University of Virginia (see Appendix D). A listing of other protocols and methods can be found in Appendix A.

pH (hydrogen ion)

Instrumentation: PC-Titrate (Man-Tech Corp.) Autotitration system for pH and alkalinity
Technique Summary: Gran analysis technique (Gran 1952)

Reporting units: standard pH units

Acid-Neutralizing Capacity

Instrumentation: PC-Titrate (Man-Tech Corp.) Autotitration system for pH and alkalinity (acid-neutralizing capacity)

Technique Summary: Gran analysis technique (Gran 1952)

Reporting units: µeq/L

Conductivity

Instrumentation: PC-Titrate Conductivity Meter Model 4310 for conductivity

Technique Summary: Electrometric (Standard Methods for the Examination of Water and Wastewater, APHA, 20th Edition, 1998)

Reporting units: µS/cm

Sulfate, Chloride, Nitrate

Instrumentation: Waters IC with Dionex AS12 A Separator Column, Model 431 conductivity detector, Model 717 plus autosampler, Model 510 pump

Technique Summary: Ion Chromatograph (IC) with separator column for anions (APHA 1998a)

Reporting units: µeq/L

Calcium, Magnesium, Potassium, Sodium

Instrumentation: Waters IC with Water IC PAK Cation M/D Column, Model 431 conductivity detector, Model 717 plus autosampler, Model 501 pump

Technique Summary: Ion Chromatograph (IC) with separator column for monovalent/divalent cations

Reporting units: µeq/L

Fluoride

Instrumentation: Waters IC with Dionex AS12A Separator Column, Model 431 conductivity detector, Model 717 plus autosampler, Model 510 pump

Technique Summary: Ion Chromatograph (IC) with separator column for anions (APHA 1998a)

Reporting units: µM/L

Aluminum, total monomeric and organic monomeric

Instrumentation: Not analyzed at the Fort Collins lab (see Appendix D)

Dissolved organic carbon

Instrumentation: Not analyzed at the Fort Collins lab (see Appendix D)

3.5.4 Data Quality

The quality control and assurance component of stream water sample collection and analysis should include analysis of reference standards, field duplicates, and process blanks, as well as examination of charge balance and measured versus calculated conductance differences.

Quality control in the field can be achieved by careful site location and close adherence to sample collection and sample handling procedures.

Quality control in the lab can be achieved by close adherence to sample bottle preparation procedures and instrument operation and calibration procedures. In addition, reference standards of known concentration should be distributed among the samples to be analyzed. These should include synthetic mid-range standards and natural-matrix reference standards. When values obtained for the standards deviate from established mean values by unacceptable amounts (typically 5% or 2 standard deviations), analysis results for the associated samples should be disqualified. Additional aliquots should be prepared for reanalysis if necessary. In the event that acceptable reference sample values cannot be obtained, the data records for associated samples should be flagged in the database.

Quality assurance should be provided by analysis of results for standards and duplicates.

Analytical accuracy should be determined by analysis of both synthetic and natural-matrix reference samples with analyte concentrations in the range of the sample concentrations. During the analysis process the reference samples should be distributed among the survey samples. Reference samples should represent about 5 to10 percent of the total number of samples analyzed, with at least one reference sample included in each analysis session. Results of reference sample analysis should be reported for each analyte as the mean and standard deviation of the difference from the nominal concentration values.

Data precision should be determined as combined sampling and analytical precision. Approximately 10% of all samples should be collected in duplicate, with at least one duplicate set included in every sampling trip. The duplicates should be distributed randomly among the samples for analysis. Results should be reported for each analyte as the mean and standard deviation of the absolute difference between duplicate sample pairs.

Quality assurance should also involve analysis of process blanks. These should be sample bottles that have been prepared and filled with deionized water in the same manner as bottles used for sample collection. As indicated in section 3.4.1, two extra bottles should accompany each sample team on each sampling trip. In the field, the deionized water in one will be discarded and replaced with the deionized water in the other. The deionized water sample becomes the process blank. The process blank bottle should be included with the actual sample bottles that are delivered to the lab. Analysis of the deionized water in this bottle will provide a measure of error associated with the complete bottle preparation, sample handling, and analysis process. Results of process blank analysis should be reported for each analyte as the mean and standard deviation of the process blank analyses.

Charge-balance and conductance differences should also be determined for the analysis of all samples. These checks, which provide information about complete sample analysis, are commonly used to indicate analytical error or the presence of unmeasured ions.

Charge balance error is a measure of deviation from charge equivalence for anions and cations. Percent charge balance error for samples is defined as 100 x (sum cations - sum anions)/(sum cations + sum anions). Charge balance error should not exceed 15%, except where dissolved organic carbon concentration is high (> 5 mg/L).

Determination of conductance difference involves comparison of measured conductance (corrected to 25 degrees C) with the sum of the specific conductances of the measured ions. Percent conductance difference is calculated as the 100 x (calculated conductance – measured conductance)/measured conductance. Conductance difference should not exceed 30%.

Analytical records for samples that exceed the criteria for charge balance error or conductance difference should be carefully examined for possible causes. Any analyte that is identified as suspect should be reanalyzed. If the problem cannot be identified or corrected, the sample analysis record should be flagged.

3.6 Stream Water Data Management

The completed datasheet will be entered into the Forest Service corporate database called the Natural Resource Information System (NRIS). A photocopy (make sure all of the entries are clearly legible) of the data sheet is sent (along with any helpful electronic files) to the Air Resource Specialist (currently Bill Jackson) in Asheville, NC. Appendix B provides the FS water chemistry data sheet entitled "Surface Water Chemistry Long-Term Monitoring Record" and instructions for completing the datasheet. Also, included are the "Chain of Custody" forms to be completed and sent with the water samples to the laboratory.

4.0 SOIL DATA COLLECTION

4.1 Arrangements with the Soil Chemistry Laboratory

Appropriate agreements or contracts will need to be made with a qualified soil chemistry laboratory well in advance of field sampling. Currently, the USFS Soils Lab in Grand Rapids, Minnesota has been chosen to perform the analyses on soil chemistry samples collected in the National Forests of western North Carolina, eastern Tennessee and the upstate of South Carolina.

4.2 Sampling Materials

Each field crew should be provided the following equipment and materials, except as noted:

- Site information folders (including maps and Soils Field Data Forms)
- Site documentation materials
- Sampling protocol (this document)
- Sampling equipment (shovel, spade, folding saw, trowel, and/or pruning shears)
- Half-gallon plastic ziplock-type plastic bags
- Backpacks
- Digital camera with extra memory card and battery
- Survey grade global positioning system and compass

- Small tape measure and 50 or 100 meter tape and/or small laser range finder
- Small water bottle
- Hydrogen peroxide to check for the presence of Mn concretions (optional)
- Clinometer
- Golf tees painted bright orange for photographing horizon designation breaks
- Munsell color book
- NSSC Field guide for describing soils (optional)
- Picking knife for cleaning the profile and digging out samples in thin horizons
- Large-print cards with site identification code for photographs

More details on equipment and materials follow in sections 4.2.1–4.2.4. Instructions for use are in sections 4.3 and 4.5.

4.2.1 Site Information Folders

The soil samples taken for acid-base chemistry on National Forest lands in North Carolina, South Carolina and Tennessee will be collected within stream catchments where one or more water chemistry samples have been taken. The catchments will likely be selected for study within a designated Wilderness, or 6th level HUC. The 6th level HUC was chosen as a unit of analysis because the Forest Service may own an appreciable percentage (20 percent or more) of some of these watersheds. Each sampling site's information will be placed in the appropriate HUC folder and will include the Soils Field Data Forms (see Appendix D), site maps, and other site documentation.

Maps will include 1:24,000 USGS quadrangle maps with sampling sites identified and Forest recreation maps to help navigate to the area. Maps generated using GIS will also be included to show where the project manager has selected potential sites to sample. Typically, at least one plot will be planned in each vegetation cover, and soil map unit and/or lithology; and is within 200 meters of the stream and positioned at a mid-elevation in the catchment.

Site documentation may be incomplete at the time of sampling for sites that have not been previously established or for sites that were established following different protocols. Site documentation information will be maintained in the NRIS database for purposes of record keeping, as described in section 4.6.

Site documentation information in the HUC folders should include site maps, narrative access descriptions, site coordinates and elevation, site-tag numbers and locations, site photographs, and sheets or index cards with site identification codes (2 for each site) in large print for use in site photographs.

Soils Field Data Forms serve for documenting sample locations and sample collection. These forms should be printed on waterproof paper. A copy of the Soils Field Data Form is provided in Appendix E.

4.2.1 Site Documentation Materials

Site documentation materials include uniquely numbered aluminum tags, nails and a small hammer for tag placement, flagging tape, a camera for site photographs, a survey grade

GPS unit for determination of geographic coordinates and a large-print site identification label or card for placement in the soil pit when taking the photograph of the soil profile. Pencils and waterproof pens are also required.

4.2.2 Soil Sampling Equipment

Sampling material includes digging, measuring, and soil handling equipment. Digging equipment should include a heavy-duty hand trowel and a “sharpshooter” shovel with a 30-inch steel or fiberglass handle and an approximately 5.5-inch wide, 18-inch long blade. A small folding saw should be included to cut around the perimeter of forest floor samples and/or a pruner to cut large roots.

Measuring equipment should include a tape measure with metric units (and perhaps English units also) and a length of at least five meters.

Soil handling equipment should include a small plastic tarpaulin (about 4’ x 6’), and sample bags. The sample bags should be heavy-duty half gallon sized zipper locked type plastic bags. In addition, dry paper towels should be available for cleaning soil from the tarpaulin and digging equipment between samples.

4.3 Establishing Sampling Sites

All soil sampling sites will be newly established for purposes of the current program. Site maps and access notes should indicate general or preliminary sampling site locations. The site maps may include Forest Recreation maps and USGS 1:24,000 quadrangle maps. GIS maps will be provided that indicate approximate sampling sites locations. In either case it will be the responsibility of the field crew to select and document the specific sampling site locations based on conditions observed in the field. The field crew should consider the following guidelines.

Sample sites should be selected to represent the typical landscape (aspect, slope, and vegetation type). Avoid selecting sites that are:

- Near abrupt changes in landscape condition (aspect, slope, and vegetation type).
- Located where it is apparent that soil has been removed or deposited by erosion or downslope movement.
- Characterized by substantial soil disturbance due to tree-throw or other major disturbances.
- Located in seepage areas, runoff concentration areas, or locations that are otherwise wetter than the surrounding landscape.
- Areas that do not represent the soil type mapped by a recent USDS-NRCS Soil Survey if available (because this may be an inclusion that is not typical for the map unit).

The sample-collection team will need to provide detailed site documentation. Site documentation consists of maps, access notes, site tags, site photos, site elevation, and geographic coordinates. Preparation of site documentation for new sites should include the following steps:

1. Record additional information to clarify preliminary site and access descriptions provided on the Soils Field Data Form. This can include drawing a site-sketch map. Use the back of the form when needed.
2. Access notes should describe the route to the site and the location of the site. In most cases, the access notes should describe a point of departure from an identifiable location on a road, trail, or stream course. The access notes should indicate the compass bearing and approximate distance from the point of departure from an identifiable location to the site location.

   Determine the geographic coordinates of the sampling site. Follow instructions to obtain the most-precise readings that can be obtained with the GPS unit. The units for geographic coordinates (NAD83) will be decimal degrees. The elevation (taken from a 1:24,000 quadrangle map) and geographic coordinates of the sampling site should be recorded on the Soils Field Data Form.

3. Place a site tag a few inches above ground level on a vigorous nearby tree of at least 6-inches diameter. This tag should be affixed to be visible upon approach from the nearest trail or road. The nail should not be driven all the way in; leave about ¼ inch to allow for tree growth. The tag number, the tag tree species, the diameter at breast height of the tag tree, and the distance and compass bearing from the specific sampling site should be recorded on the Soils Field Data Form.
4. Obtain sampling site photos of the site surroundings. One member of the collection team should stand at the sampling site and hold up a large-print site identification label (provided in the site folders) for the photos. Orange flagging should be placed (temporarily) on the tag tree to allow its identification in the photos. Photos should be taken to include distinctive features of the sampling site such as particular rocks and trees. Indicate the number of pictures taken in the space provided on the Soils Field Data Form.
5. Obtain sampling site photos of one face of the soil pit. After the soil pit has been excavated to its maximum depth, square off one representative face using the shovel, and photograph (using the flash unit) the face of the pit to show horizon development. Within the field of view, place an identification label and a tape measure to indicate depth measurements.

The site documentation described above will facilitate return visits to the sampling sites if repeated sampling is contemplated or if decisions are made later to conduct repeated sampling. Given that soil sampling is “destructive,” a sample should not be collected at the same specific location as a previous sample. A simple solution to this problem would involve treating the initial sampling site as the center of a sampling plot. Any repeat sampling can be conducted at fixed distances in an array around the central site.

4.4 Sample Collection, Handling, and Documentation

MAGIC is calibrated using data for mineral soils, generally by designating either one or two soil layers. These do not necessarily correspond with horizon development. The top 10 or 20 cm of mineral soil is commonly collected, and is thought to represent the source areas for relatively quick flow. Deeper mineral soil samples (for example 10 to 50 cm mineral soil depth) are often also used, and are thought to represent the source areas more typical of base flow water chemistry. Soil sample depth is kept constant from site to site and is generally not adjusted for differences in horizon thickness. Soils acid-base chemistry conditions are used to constrain the model behavior, but the principal constraint on model output is the drainage water chemistry to which the model is calibrated.

Data for organic soil horizons are generally not used as part of the modeling effort, but can provide valuable information regarding nutrient dynamics and nitrogen cycling. In addition, the nitrogen components of the model are in flux and may be expected to change in the near future. Thus, collection of O-horizon data is recommended. This material can be archived and analyzed at a later date, if desired.

4.4.1 Sample Collection Procedure

- Avoid sampling during precipitation or when the soil is wet from recent precipitation. (This does not include soils that are naturally wet and have high seasonal water tables.)
- Avoid contact between skin and the soil being collected by being careful in sample collection and/or wearing plastic gloves while conducting the sampling.

Collect samples on a step-by-step basis, as follows:

1. Use the shovel to remove and discard all of the loose forest-floor material (litter) from an area 70 cm in diameter at the sampling site. Do not remove any organic matter that is adhered to the underlying material.
2. Use the shovel and saw or pruners to collect the O-horizon (organic soil horizon) from the 70 cm-diameter sampling circle. Place the organic soil matter on the plastic tarpaulin and thoroughly homogenize manually using the trowel. Place this material in a soil sample bag and seal the bag. Use a permanent-ink marking pen to label this bag, indicating the site identification code, the date, and "O-horizon Sample." If the O-horizon material is too thick to allow collection of all material in one bag, select a sampling circle smaller than 70 cm in diameter, and record this deviation in the sample log.
3. After removal of the forest floor and O-horizon material, wipe off the plastic tarpaulin with a piece of paper towel to remove excess O-horizon material, and use the shovel to dig a 70-cm diameter sampling pit to approximately 10 cm from the top of the mineral soil (interface between the O-horizon and the mineral soil). The material removed from the pit should be placed on the plastic tarpaulin and thoroughly homogenized manually using the trowel. Add homogenized soil, excluding rock fragments, to a sampling bag (approximately 500 grams of soil is needed) until the bag is about three-quarters full. Seal the zip-lock type bag and then place the sample bag inside of another zipper-lock type plastic bag.
4. Label the bag, indicating the site identification code, the date, and "Shallow Sample."
5. Set the remaining soil and rock material on the tarpaulin aside.

6. Dig the soil pit deeper (if possible to 50 cm or deeper from the top of the mineral soil; 40 cm below the bottom edge of the Shallow Sample that you just collected). The material (10 to 50 cm depth mineral soil) removed from the pit should be placed on the plastic tarpaulin and thoroughly homogenized manually using the trowel. Add soil, excluding rock fragments, to a sampling bag until the bag is about three-quarters full. Seal the bag with a twist tie. Label the sample bag, indicating the site identification code, the date, and "Deep Sample."
7. Measure the depth and diameter of the mineral soil sampling pit and record on the Soils Field Data Form.
8. Estimate the percent rock fragments by volume for both the shallow and deep sample depths and record on the Soils Field Data Form.
9. Return all remaining soil and rock fragments to the sample pit and cover with debris. If desired, it might be useful to collect and retain a small sample of the characteristic rock materials (fresh piece(s) a few inches in diameter, or large enough to handle easily).
10. Place each soil sample bag inside a second, outer plastic bag containing the site identification label used in the photographs or other piece of paper on which is written the site identification code.
11. Complete the Soils Field Data Form while at the sample site.
12. Determine if a duplicate soil sample is needed at this site. If so, dig a second pit in close proximity to the first, and repeat all sampling steps.

4.4.2 Sample Processing

Sample processing involves drying the samples and removing material greater than 2 mm in size. Sample drying and sieving of the soil samples can be done by the field crew at a staging area or at the laboratory. In general, we recommend transporting the samples as quickly as possible to the laboratory for processing. Process the samples following these steps:

1. Soil samples in clean plastic bags should be shipped with ice packs to the lab for analysis, via one-day or two-day delivery service. Shipments should not be sent to arrive at the end of the week or on a weekend when laboratory personnel will not be available to begin processing. The lab should be consulted to determine necessary sample amounts. The bags for sample shipment to the lab should be labeled indicating the site identification code, the sample collection date, and either "O-horizon (#299,xxx)", "Shallow Sample (#300,xxx)", or "Deep Sample (#305,xxx)," as appropriate. If the samples will not be shipped to the laboratory within two days of collection, they should be placed on a bench top in a cool room, out of the sunlight, and stored with the bags open at the top to allow for air movement. Reseal the bags and place them in a cooler with ice packs immediately prior to shipping. Some samples might be from states that have restrictions on soil movement because of potential infestation by golden and corn cyst nematodes, imported fire ants, and witchweed. This restriction is under the jurisdiction of the USDA Animal and Plant Health Inspection Service (APHIS) Plant Protection and Quarantine Program. A map of regulated states can be found at the APHIS website

(http://www.aphis.usda.gov/ppq/maps/soil2002.pdf). Field crews sending regulated samples must label the containers "Contents Soil Samples," include a copy of the soil permit, and display a PPQ Form 550 on each sample (the form can be found at: http://www.aphis.usda.gov/ppq/permits/soil/index.html#process).

2. Ten percent of the samples should be selected for duplicate analysis. For these samples, two bags for each of the three soil layers collected should be prepared for shipment to the analytical lab. The duplicates should be collected from separate pits located in close proximity, designated "1" and "2," and labeled accordingly.

3. The receiving lab is required to hold an APHIS permit to receive and handle regulated materials. The preparation crew should check the front entry each day for new samples. Any new samples should be opened and preparation begun on the day of receipt, or placed in the walk-in cooler for preparation on the following work day. Make a photocopy of the shipping form for use in the headhouse and store it in the notebook labeled "Atmospheric Deposition Effects (ADE) Shipping Forms." Give the original to the soils supervisor. *ADE field crews should alert the lab to incoming samples by mailing or emailing a copy of the shipping form prior to shipping samples. The preparation crew should place this form in a "pending" file, then attach it to its mate that should arrive with the samples.* In the headhouse, open boxes and sort new samples by site identification and horizon (O, Shallow, or Deep). Print sample labels using software on the headhouse PC. The sample number scheme is ADE-number (e.g. ADE-300001). Affix labels to each new sample and record the bag label and shipping form information in the hard-copy notebook labeled "ADE Incoming Soils." **Fill all columns to create a complete paper trail**. Make a note if data is missing and, **at least daily**, copy all information from the hard copy into the MS Excel workbook found on Server1 in folder ADE2004. Do not add, delete, or modify spreadsheet columns without approval from the soils supervisor. Select a plastic drying container and label it with sample ID, date, and type of soil (mineral or organic). Zero the balance, then weigh the container and record the weight in "Tare (g)" column. Zero the balance again with the container in place. Empty the sample into the container, weigh, and record the weight in the "Field Moist – Tare (g)" column. Cut bag labels from the sample bags each day and put them in numerical order and store. These are kept permanently.

4. Place sample in soil drying room and, if possible, keep all samples from each state together to facilitate removal. Run the dehumidifier and keep the door closed. Samples are considered air-dry with no more than 5% weight loss overnight. Sweep drying room weekly.

5. Remove from drying room if weight loss is less than 5% overnight. Zero the balance, weigh the container and sample, and record the weight in the "AD + Tare (g)" column.

6. Dry mineral soil should be passed through a 2-mm sieve without forcing material through the sieve. Using a mortar and pestle, break clods (do not grind) so they'll pass through 2 mm screen. Use the NASCO soil grinder only for especially hard clods. Tare a suitable container, weigh, record, and discard the "> 2 mm" portion of sample. Tare a suitable container, weigh and record "< 2 mm" portion. **Do not discard**.

7. The O-horizon samples should be allowed to air dry, but should not be sieved. The O-horizon samples will not be analyzed for MAGIC parameterization, but may be useful in the future for evaluating the nitrogen status of the site. (Note: MAGIC procedures for simulating nitrogen cycling may be updated in the near future and may necessitate analysis of O-horizon samples.) The O-horizon samples should be stored in sealed plastic bags for archiving. The bags should be labeled indicating the site identification code, the sample collection date, and "O-horizon."

8. Prepare a split for oven-drying the mineral soils after air drying. Zero the balance and record the soil can number in the "Soil Can #" column. Weigh and record the soil can weight including its cover in the "Soil Can Wt (g) column. Tare the can with its cover and then add 10-15 g of air dried sample. Record the weight under "AD Soil - Can (g)." Place the can and sample with **cover off** in the oven overnight at 105 ºC. After oven-drying, replace cover, cool the can and sample in a desiccator for 3 to 5 minutes. Zero the balance, weigh and record the oven dry sample with can under "OD soil + can (g)." **Be sure all columns are filled**. After all data is satisfactory and stored in the Excel spreadsheet, dispose of the OD sample and clean the can for next use. The balance of the dry sieved soil should be packed in sealed plastic bags for archiving. The bags for sample archiving should be labeled indicating the site identification code, the sample collection date, and either "Shallow Sample" or "Deep Sample," as appropriate. Before disposing of any portion of regulated soil samples or any material that has been in contact with them, they must be sterilized by exposure to elevated temperature. Place soils in appropriate containers such as aluminum turkey roaster pans and heat to at least 121 ºC. Once the entire mass has reached that temperature, maintain for at least 2 hours. Packaging and other materials can be placed in autoclave bags before heating to 121 ºC. Plastic bags that melt at high temperature might trip the smoke alarm, so must not be placed in oven. These should be securely packaged and stored until incineration can be arranged. Boxes of archived samples from regulated states should be labeled as such. Spilled soil from regulated states must be swept up and heat-treated as described above. The area of the spill must be sprayed with 70% alcohol solution.

9. Prepare pH, cations, carbon, and archive splits. **Split for pH;** label a paper cup with sample ID. Tare the cup and put in 20.0 g of < 2 mm soil. Deliver samples and cups to the chem lab daily. Do each 20th sample in duplicate and include "dup" on the label. **Split for cations;** label a 20 mL scintillation vial with sample ID and "ICP" for cations so it can be distinguished from a similar carbon split. Fill to about ¾ full (leaving shaking room) with < 2 mm soil. Store in storage room next to lab 31 keeping them separate from carbon splits. **Split for carbon;** label the bottom and side of a 20 mL scintillation vial with sample ID and a "C" for carbon so it can be distinguished from a similar cations split. A label on the cap might be worn off as it turns on the vial rotator. Also place clear tape over the side label to prevent wearing off. Add soil to about 1/3 full, insert two steel tumblers, cap securely, and place the vial on the vial rotator. Some samples will be reduced overnight, others might take 3 or 4 days to reduce to a suitable fine grind. *A mesh size has not yet been determined, so grinding is ended based only on the operator's observation.* Store the vials in the storage room next to lab 31 keeping them separate from the cations splits. **Split for archive;** In two places, label a 4 oz.

specimen container and cap with sample ID. The label must remain legible for up to 30 years. Place the container and cap on a balance and zero the balance. Fill container with < 2 mm soil. Replace the cap, weigh, and record the weight under "Archive Wt (g)." Clean all equipment after each sample, and the entire work area at the end of each day.

10. Archived samples should be stored in plastic tubs with secure lids. The plastic tubs should be labeled to indicate the contents. Storage area housing soils from regulated states must be signed: "Contents: Regulated domestic soil to be used in accordance with USDA APHIS PPQ soil permit and compliance agreement." Packages containing soils from regulated states must be so labeled. Each sample must be labeled as "Regulated soil under USDA permit." A SOP describing clean-up of regulated soil spills must be posted in archive and soil preparation areas.

4.5 Soil Sample Analysis

Application of the MAGIC model requires analyses of the following soil properties in both the shallow and deep mineral soil layers. The laboratory performing the analysis needs to be experienced at performing analysis for acid-base chemistry.

- pH (lab measurement)
- Exchangeable bases: calcium, magnesium, potassium, and sodium
- Exchangeable aluminum
- Cation exchange capacity (CEC) or effective CEC for acid soils

Analyses of total nitrogen and total carbon are also recommended.

The following methods are appropriate for analysis of forest soils for assessment of acid-base status and acidification effects in the southern Appalachian Mountains region. All chemical analysis results should be expressed on an oven-dried soil mass basis (70°C for forest floor and 105° C for mineral soils).

4.5.1 Analysis Methods

Currently, the USFS Soils Lab in Grand Rapids, Minnesota will perform the chemical analysis for the soil samples. The following is a summary listing of instrumentation and techniques employed for sample analysis. Equivalent instrumentation and techniques can be substituted, such as those employed by the University of Virginia (see Appendix E). A listing of other possible protocols and methods can be found in Appendix A.

pH (hydrogen ion)

Instrumentation: Mettler Toledo DL53 Titrator with DG111-SC Combination pH Electrode, DT120 Temperature Sensor, Rondo 60 Autosampler, and LabX version 1.0 Software

Technique Summary: Potentiometric measurement in a 1:1 soil to deionized water slurry by mass and in 0.01 M $CaCl_2$ (Cappo et al., 1987)

Reporting units: standard pH units.

Exchangeable Bases (calcium, magnesium, potassium, and sodium), Exchangeable Aluminum, and Cation Exchange Capacity

Instrumentation: Thermo Elemental Iris Intrepid ICP with Timberline IIL Autosampler and TEVA version 1.5.0 Software

Technique Summary: Extraction by 1.0 N ammonium chloride; analysis by inductively-coupled plasma atomic emission spectrometry (Weaver et al., 1982; SSLMM, 1996)

Reporting units: cmol+ / kg soil

Total Nitrogen and Carbon

Instrumentation: Fisons NA1500 Elemental Analyzer

Technique Summary: combustion (Nelson and Sommers, 1996).

Reporting units: percent dry weight

4.5.2 Data Quality

The quality control and assurance component of soil sampling and analysis should include field duplicates (replicated soil pit in close proximity to the original soil pit), repeated analysis of composite samples (if the soil survey will extend for a period of several weeks or more), and evaluation of data internal consistency (relationships among variables).

Quality control in the field will be achieved through careful site location and close adherence to sampling procedures. Quality control in the laboratory will depend on close adherence to holding time criteria, avoidance of sample contamination, and careful following of instrument operation and calibration procedures.

Approximately 10% of all soil samples should be collected in duplicate, by excavating a second soil pit in close proximity to the first. Results for duplicate analysis should be reported for each analyte as the mean and standard deviation of the absolute differences between duplicate sample pairs. Do not assume that the values are incorrect if results for the second pit differ from the first one. Soil variability on the landscape can be large for any number of reasons.

If an extensive soil sampling survey is to be conducted, soil composite samples (both shallow and deep) should be prepared during the first week of field work, each as a large mix of soil from several (n = 5) sites exhibiting a range of expected conditions. This composite sample should be sieved (2 mm), thoroughly homogenized, and air dried. Multiple subsamples should then be drawn from both composites, marked as “deep composite” and “shallow composite”, sealed in zipper-lock style bags, and kept refrigerated. Each week, one of each composite type (shallow and deep) should be dated (date of shipment to laboratory) and submitted to the analytical laboratory for analysis. Time series data illustrating results of repeated results from the same sample will later be used to determine if laboratory procedures were comparable throughout the course of the study, as well as deviations associated with replicate measurements.

4.5 Soil Data Management

The NRIS database will be used to enter the soils protocol document information, site documentation, sample collection record, and analysis record tables. Data entry of the soil information will be accomplished at the Supervisor's Office of the National Forests in North Carolina. Appendix D provides the field data forms for the soil sampling components of the current project. Instructions for collection of the necessary data have been provided above in the text of this document.

5.0 SAFETY ISSUES

For safety reasons, an emergency contact individual, who is not part of the field crew, should always know where the field crew is going each day, and by what route. This person should be contacted by the field crew immediately upon return from the field each day.

Safety equipment should include, but should not necessarily be limited to, the following:

- Two-way radios and/or cellular telephone
- Extra batteries for GPS and radios
- Rain gear
- Space blanket
- Adequate supply of drinking water
- Sunscreen
- First aid kit

Field personnel should have current first aid and CPR certificates. Field personnel should be instructed to not cross a dangerous stream or excessively steep terrain.

6.0 CITATIONS

Cappo, K.A., L.J. Blume, G.A. Raab, J.K. Bartz, and J.L. Engels, 1987. Analytical Methods Manual for the Direct/Delayed Response Project Soil Survey. EPA/600/8-87/020, U.S.E.P.A., Washington, DC.

Cosby, B.J., R.F. Wright, G.M. Hornberger, and J.N. Galloway. 1985a. Modelling the effects of acid deposition: assessment of a lumped parameter model of soil water and streamwater chemistry. Water Resour. Res. 21:51-63.

Cosby, B.J., R.F. Wright, G.M. Hornberger, and J.N. Galloway. 1985b. Modelling the effects of acid deposition: estimation of long-term water quality responses in a small forested catchment. Water Resour. Res. 21:1591-1601.

Gran, G., 1952. Determination of the equivalence point in potentiometric titrations. Part II. Analyst 77:661-671.

Nelson, D.W. and L.E. Sommers. 1996. In D.L. Sparks (ed), Methods of Soil Analysis, Part 3. Chemical Methods. Soil Science Society of America Book Series Number 5. American Society of Agronomy, Madison, WI.

SSLMM (Soil Survey Laboratory Methods Manual), 1996. Soil Survey Investigations Report No.42, Version 3.0. USDA, Natural Resources Conservation Service, National Soil Survey Center, Lincoln, NE.

Sullivan, T.J. and B.J. Cosby. 2002. Critical loads of sulfur deposition to protect streams within Joyce Kilmer and Shining Rock Wilderness Areas from future acidification. Report prepared for USDA Forest Service. E&S Environmental Chemistry, Inc., Corvallis, OR.

Sullivan, T.J., B.J. Cosby, J.A. Lawrence, R.L. Dennis, K. Savig, J.R. Webb, A.J. Bulger, M. Scruggs, C. Gordon, J. Ray, E.H. Lee, W.E. Hogsett, H. Wayne, D. Miller, and J.S. Kern. 2003. Assessment of Air Quality and Related Values in Shenandoah National Park. Technical Report NPS/NERCHAL/NRTR-03/090. U.S. Department of the Interior, National Park Service, Northeast Region, Philadelphia, PA.

Sullivan, T.J., B.J. Cosby, J.R. Webb, K.U. Snyder, A.T. Herlihy, A.J. Bulger, E.H. Gilbert, and D. Moore. 2002. Assessment of the Effects of Acidic Deposition on Aquatic Resources in the Southern Appalachian Mountains. Report prepared for the Southern Appalachian Mountains Initiative (SAMI). E&S Environmental Chemistry, Inc., Corvallis, OR.

Weaver, R.W., S. Angle, P. Bottomley, and D. Bezdiecek (eds). 1982. In D.L. Sparks (ed), Methods of Soil Analysis. Part 2. Chemical and Microbiological Properties. Soil Science Society of America Book Series Number 5. American Society of Agronomy, Madison, WI.

APPENDIX A:

SELECTED LISTING OF PROTOCOLS AND METHODS MANUALS REVIEWED

Blume, L.J., B.A. Shumacher, P.W. Schaffer, K.A. Cappo, M.L. Papp, R.D. Van Remortel, D.S. Coffey, M.G. Johnson, and D.J. Chaloud, 1990. Handbook of Methods for Acid Deposition Studies Laboratory Analyses for Soil Chemistry. EPA/600/4-90/023, U.S.E.P.A., Washington, DC.

Cappo, K.A., L.J. Blume, G.A. Raab, J.K. Bartz, and J.L. Engels, 1987. Analytical Methods Manual for the Direct/Delayed Response Project Soil Survey. EPA/600/8-87/020, U.S.E.P.A., Washington, DC.

Edwards, P.J., and F. Wood, 1993. Field and Laboratory Assurance/Quality Control Protocols and Accomplishments for the Fernow Experimental Forest Watershed Acidification Study. General Technical Report NE-177, USDA Forest Service, Northeastern Forest Experiment Station. Radnor, PA.

Fox, D.G., J.C. Bernabo, and B. Hood, 1987. Guidelines for Measuring the Physical, Chemical, and Biological Condition of Wilderness Ecosystems. USDA Forest Service General Technical Report RM-146, 48 p., Rocky Mountain Forest and Range Experiment Station, Fort Collins, CO.

Morrison, M.L., 1991. Part 1: Quality Assurance Plan for the Long-Term Monitoring Project, in Data User's Guide to the U.S. EPA Long-Term Monitoring Project: Quality Assurance Plan and Data Dictionary. EPA/600/3-91-072, U.S. EPA Environmental Research Laboratory, Corvallis, OR.

NCASI, 1983. Field Study Program Elements to Asses the Sensitivity of soils to Acidic Deposition Induced Alterations in Forest Productivity. Technical Bulletin No. 404, National Council of the Paper Industry for Air and Stream Improvement, Inc. New York, NY.

Shenandoah Watershed Study, Department of Environmental Sciences, University of Virginia, Charlottesville, VA. http://wsrv.clas.virginia.edu/~swasftp/docs/SOP-021303.pdf

Turk, J.T., 2001. Field Guide for Surface Water Sample and Data Collection. Air Program, USDA Forest Service.

U.S.E.P.A., 1989. Handbook of Methods for Acid Deposition Studies: Field Operations Manual for Surface Water Chemistry. EPA/600/4-89/020, U.S.E.P.A., Washington, DC.

U.S.E.P.A., 1987. Handbook of Methods for Acid Deposition Studies: Laboratory Analyses for Surface Water Chemistry. EPA/600/4-87/026, U.S.E.P.A., Washington, DC.

USFS/USGS Water & Soils Lab, Rocky Mountain Research Station, Fort Collins, CO. http://www.fs.fed.us/rm/waterlab/Labprotocols.htm

APPENDIX B:

FOREST SERVICE DATA COLLECTION FORMS AND INSTRUCTIONS

(1) Surface Water Chemistry Long-Term Monitoring Record

(2) Chain of Custody Form

USDA Forest Service
National Protocols for Air Pollution Sensitive Waters

Stream Sampling - Site Documentation
Form 1

Basic Site Information

Forest Name: Wilderness Name (if applicable):

Stream Name (USGS): Stream Name (Local):

Site Name: Site ID:

Date of Visit: Visit: ☐ Initial ☐ Subsequent

Field Team Leader:

Affiliation:

Phone: Email:

Access: ☐ Vehicle ☐ Short Hike (< 1 hr) ☐ Long Hike (> 1 hr) ☐ Overnight Hike

Travel Directions to Stream Sampling Site and Access Information:

Site Verification, GPS Information and Tagging

Stream Verified: ☐ Yes ☐ No	Site has been verified by (check all that apply):	☐ GPS	☐ Local Contact	☐ Signs	☐ Vegetation
		☐ Roads	☐ Topo Map	☐ Photos	☐ Other

GPS Information Latitude (DD) __ __ . __ __ __ __ __ __ GPS Accuracy: ☐ ft. ☐ m

Datum: Longitude (DD) -__ __ __ . __ __ __ __ __ __ Elevation: ☐ ft. ☐ m

Site Tag has been Affixed? ☐ Yes ☐ No | ☐ New Tag ☐ Existing Tag | Tag Tree Species:

Describe tag tree location relative to stream sampling site:

Site Assessment (Observations within 20 m of streambank)

Streambank Character (use % classes below)	Dominant Age Class (forested areas only)	What are the dominant plant species (if known)?
______% Forest/Shrub		
______% Open Herbaceous	☐ 0 - 10 years	
______% Wetland	☐ 10 - 25 years	
______% Barren (beach/rock)	☐ 25 - 50 years	
______% Agriculture	☐ > 50 years	
______% Developed	**Is there beaver activity near the sample site?**	
______% Shoreline Mod. (dock, riprap)	Signs of Beaver: ☐ None ☐ Rare ☐ Common	Beaver Flow Modifications: ☐ None ☐ Minor ☐ Major
Rare (< 5%) Moderate (25-75%) Sparse (5-25%) Extensive (> 75%)		

Watershed Assessment

What percent of the watershed above the sample site is?	
	Primary lithology type:
______% Hardwoods	Note additional significant lithology types:
______% Conifers	
______% Mixed Forest	
______% Exposed Rock	What is the watershed area above the sample site? __________ ☐ ha ☐ ac
______% Herbaceous/Shrubs	What is the watershed aspect (degrees)? ________°
______% Talus	What is the average slope of the watershed? ________%
______% Total	What is the stream order of the sample site (use NHD dataset)? ________

Stream Form 1 - V1.2 - March 2011

Stream Sampling - Site Documentation
Form 2

Stream Name (Local):	Date of Visit:
Site Name:	Site ID:

Photo Log

Photo ID/File Name	Date	Description of Photo

Description and Sketch of Site

Additional Notes

USDA Forest Service
National Protocols for Air Pollution Sensitive Waters

STREAM SAMPLING - SAMPLE RECORD
FORM 3

BASIC SITE INFORMATION

Forest Name:	**Wilderness Name (if applicable):**
Stream Name (USGS):	**Stream Name (Local):**
Site Name:	**Site ID:**
Date of Visit:	**Arrival Time (24hr):** ☐ Standard ☐ Daylight Saving
Field Team Leader:	
Affiliation:	
Phone:	**Email:**

Are there any suggested revisions to the Site Documentation Forms 1 and 2 (select the area of revision below)?

☐ GPS Information ☐ Stream Description ☐ Site Description ☐ Access/Travel Information

Describe suggested revision:

WATER SAMPLES AND REPLICATES

Time Sampled	Sample ID	Sample Type (reg, rep ,blank)	Bottle Type (plastic, glass, syringe)	# of Bottles or Syringes	Bar Code

Collection Location (explain any deviation from targeted sampling location):

GENERAL OBSERVATIONS

Time Obtained (24 hr): **Air Temperature:** ☐ °C ☐ °F

Water Temperature (at sample location): ☐ °C ☐ °F **Instrument Used:**

What is the weather condition on the day of sampling? ☐ Clear ☐ Partly Cloudy ☐ Overcast ☐ Hail ☐ Light Rain ☐ Occasional Rain ☐ Persistent Rain ☐ Snow or Sleet

What was the weather for the 3 days prior to day of sampling? ☐ Generally Dry ☐ Occasional Rain/Snow ☐ Generally Wet ☐ Very Wet

Average stream depth at sample location: ☐ ft ☐ in ☐ m ☐ cm

Average stream width at sample location: ☐ ft ☐ in ☐ m ☐ cm

Discharge Level: ☐ No Flow ☐ Low Flow ☐ Normal Flow ☐ Flood

Deliver method to laboratory: ☐ Vehicle ☐ Overnight Shipping ☐ Other (explain):

Stream Form 3 - V1.2 - March 2011

STREAM SAMPLING - SAMPLE RECORD
FORM 4

Stream Name (Local):	**Date of Visit:**
Site Name:	**Site ID:**

ON SITE WATER DATA (OPTIONAL)

Time Obtained (24 hr)

Parameter	Value	Equipment (make/model)	Method/Reference (EPS/SM/USGS)
Conductivity	uS/cm Corrected to 25° C? ☐ Yes ☐ No		
pH			
Turbidity	____________NTU		
Dissolved Oxygen	mg/l %DO		
Water Temperature	☐ °C ☐ °F		
Other			

PHOTO LOG

Photo ID/File Name	Date	Description of Photo

ADDITIONAL NOTES

Stream Name (Local): **Date of Visit:**

Site Name: **Site ID:**

STAGE AND DISCHARGE DATA

General Information

Time Obtained (24 hr): ☐ **Not obtained in field.**

Method of determining stage and/or discharge (check all that apply below) ☐ **None**

- ☐ Cross section of depth measurements.
- ☐ Velocity-area procedure (number of sets of measurements taken____________).
- ☐ Salt dilution method.
- ☐ Relative stage comparison with nearby fixed gage.
- ☐ Stage measurement with pressure transducer.
- ☐ Stage measurement with staff gage.
- ☐ Timed filling procedure (number of spillways measured____________).

Stage Measurement Only

Stage relative to fixed gage: ____________ ☐ ft ☐ in ☐ m ☐ cm

Location of fixed gage measurement relative to stream sampling site

Stage measurement with staff gage ____________ ☐ ft ☐ in ☐ m ☐ cm

Location of fixed gage measurement relative to stream sampling site

Existing ratings curve? ☐ Yes ☐ No **If yes, referenced to what?**

Mid-channel stream depth at sample location: ☐ ft ☐ in ☐ m ☐ cm

Discharge Measurement by Velocity-Area Procedure

Velocity-area procedure? ☐ Yes ☐ No | **Depth:** _____ ☐ ft ☐ in ☐ m ☐ cm | **Velocity:** ☐ m/s ☐ ft/s

Stream width at measurement location: ☐ ft ☐ in ☐ m ☐ cm

Interval	1	2	3	4	5	6	7	8	9	10
Depth										
Velocity										
Interval	11	12	13	14	15	16	17	18	19	20
Depth										
Velocity										

Discharge Measurement by Timed Filling Procedure

Timed filling procedure? ☐ Yes ☐ No | Time: ☐ min ☐ sec | Volume: ☐ L ☐ gal

Spillway Number	Time to Fill Measure Volume									
	Trial 1		Trial 2		Trial 3		Trial 4		Trial 5	
	Time	Volume	Time	Volume	Time	Volume	Time	Volume	Time	Volume
1										
2										
3										

ADDITIONAL NOTES

Stream Form 5 - V1.2 - March 2011

Stream Sampling Form instructions

There are two types of records used for collecting stream water samples; one documents sample site characteristics and the other documents that actual water samples. Site characteristics are documented on two forms called Stream Sampling – Site Documentation (Form 1 and form 2). These forms are used to document new sampling sites and to clarify and update existing site information. The Stream Sampling – Sample Records (Form 3, Form 4, and Form 5) are used to record each sample site visit. These forms are to be completed each time you visit a sample site and collect data. Site Information is needed at the top of each form, but varies slightly.

Form 1 and Form 3

Basic Site Information

1. Forest Name: document the National Forest where the sample will be collected.
2. Wilderness Name (if applicable): If the sample site is in a wilderness area, document the wilderness name.
3. Stream Name (USGS): Document the stream name where the sample site is located. In some cases the stream will not have a name, in this case document "Unnamed Stream" or "Unnamed Tributary to Name of Stream".
4. Stream Name (Local): Document the local stream name. In most cases this will be the same as the USGS stream name. In some cases an unnamed USGS stream may have a local name associated with it.
5. Site Name: Document the specific site name, as there may be more than one site on a single stream there is a need for a unique sample site name. This unique site name should be developed at the project level. For example, you have a stream named "Mill Creek" with 3 different sample site locations you may call each site "Mill Creek SS1", "Mill Creek SS2", and "Mill Creek SS3".
6. Site ID: There is a need to have a unique identifier for each sample site location and this Site ID should also be developed at the project level. The unique ID can be a mix of letters and numbers in any logical sequence (make sure to document the naming convention). In this case you may want to look bigger than the stream where the samples are being taken to a forest or regional scale. Using the example above, for site name "Mill Creek SS1" on the Smokey Forest, in Region 10. You may abbreviate the names and numbers in the ID, for example, R10SFMILLCKSS1. R10 = Region 10, SF = Smokey Forest, MILLCK = Mill Creek, SS1 = Sample Site 1.
7. Date of Visit: Enter the date you visit the sample site. Check the box whether this is the first "Initial" visit to this site to establish it as part of a survey or monitoring effort, or if this site has been visited and documented previously and therefore this is a "Subsequent" visit.
8. Field Team Leader Information: Enter the name, affiliation, phone number, and email address of the responsible field person.
9. Access Information: Check all boxes that apply to the access of this site. Document any travel directions (attach map if available), Estimated time of travel and any additional information that may be helpful in getting to this site.

Form 2, Form 4, and Form 5

Note that each of these Forms has space near the top to enter information that identifies the stream, sample site and the date of sampling. This is an abbreviated version of the "Basic Site Information" described above. It is important to fully complete this "header" information on each Form to ensure data is kept together and consistent.

The instructions for completing the following four attributes are described under Basic Site Information above.

- Stream Name (Local): see item 4 above.
- Date of Visit: see item 7 above.
- Site Name: see item 5 above.
- Site ID: see item 6 above.

Stream Sampling – Site Documentation Form 1

Site Verification, GPS Information and Tagging

Most of this data will be collected and recorded at the sample site.

1. Stream Verified: Does the available data match conditions observed on the ground sufficiently to verify that the intended sampling site has been located (check Yes/No)? Check all methods used to verify that you have located the correct site.
2. GPS Information: Record the coordinates from the GPS unit —Latitude and Longitude in decimal degrees (if possible to 6 decimal places)—include the Datum of the collected coordinates (e.g., NAD83). Record the Elevation in feet or meters, and if the GPS unit has the capability, record its accuracy in feet or meters.
3. Site Tag: Record whether a metal tree tag has been affixed to a prominent tree in proximity to the sampling site. (Many wilderness areas do NOT allow the use of aluminum tags.) If a tag has been used, indicate the tree species the tag is attached to. If the species is not known, record unknown. Record whether the tag was affixed to the tree on a previous trip (existing tag) or newly placed on this trip (new tag). Describe the tree and its location relative to the sampling site. Include in the description the height above the ground and compass bearing from the tag to the sampling site.

Site Assessment

The site assessment data will be collected from field observations (within 20 meters of the streambank) and recorded at the sample site.

1. Streambank Characteristics: Estimate the percentages of vegetation types along the streambank (i.e., forest/shrub, herbaceous, wetland, barren). Also look at the observed land use in the same area, identify shoreline modifications (e.g., dock, riprap), development and/or agriculture. Use the following classes for these estimates (record the percent range on the form):
    a. Rare (< 5%)
    b. Sparse (5-25%)
    c. Moderate (25-75%)
    d. Extensive (> 75%)

2. Vegetation-Dominant Age Class (forested areas only): If the site is located within a forested area estimate the dominant age class of the trees. Check the appropriate age class box (i.e., 0-10 years, 10-25 years, 25-50 years, or > 50 years).
3. Dominant Plant Species: Observe the dominant plant species within the area of the sample site, if known, record on Form 1.
4. Beaver Activity: Document observed beaver activity, check the appropriate boxes on the form.

Watershed Assessment

Most of this information can be obtained in the office through GIS analysis and aerial photo interpretation prior to visiting the sample site. It can be helpful to have this information completed prior to the first sampling visit. This information will help to locate the targeted sample location. In addition, the information is useful to verify (in the field) if the GIS analysis appears to be a valid identification of watershed characteristics.

1. Vegetation/Cover: Using GIS, ortho and/or aerial photos estimate the different vegetation types including exposed rock and tallus. Document this as a percent of the watershed area above the sample site. Verify in the field if this appears to be a valid identification.
2. Lithology: If GIS information on bedrock lithology is available, indicate the primary lithology (e.g., granitic, volcanic, metamorphic). If there is more than one lithology, make note of additional significant lithology.
3. Watershed area: Using GIS digitize the watershed area above the sample site (the watershed size will vary based on the sample site location). Calculate the watershed area in acres or hectares. It will be helpful to determine the watershed area and boundary prior to determining other watershed assessment attributes.
4. Primary watershed aspect: the primary aspect of the watershed is determined by estimating the direction the watershed is facing based on the direction of stream flow. Indicate the aspect in degrees with:

337.5 – 22.5 = N	22.5 – 67.6 = NE
67.5 – 112.5 = E	112.5 – 157.5 = SE
157.5 – 202.5 = S	202.5 – 247.5 = SW
247.5 – 292.5 = W	292.5 – 337.5 = NW

5. Average watershed slope: A simple way of calculating average slope percent within the watershed boundary is to use DEM GIS files and Zonal Stats in GIS Spatial Analyst.
6. Stream order: Identify the stream order at the sampling site location using the Forest Service NHD dataset.

Photo Log

Take two photos of the sampling site looking both upstream and downstream. On the initial visit to the site it can be helpful to take additional photos to visually record stream and site characteristics (e.g., land use and vegetation). Record the photo ID or file name, date photographed, and description of the photo. It is recommended to attach these photos to the site documentation forms to include in the
Forests hard copy files.

Description and Sketch of Site
Record a description and sketch of the site. This information can help in locating and characterizing the site, it may include access and tree tag information, land marks, and land use indicators.

Additional Notes
Add any additional information that may help to identify, locate, or describe this site.

Stream Sampling – Sample Record Form 3

When collecting water chemistry data care must be taken to protect samples from contamination. It is recommended to collect the water samples prior to the general observations (e.g., stream width and depth); this will ensure stream bottom sediments will not be stirred up causing the possibility of contamination of the water samples.

Basic Site Information
Suggested Revisions: Examine information recorded on the Stream Sampling Site Documentation Forms. Indicate any suggested revisions or updates to the site documentation form. Place a check mark in all boxes requiring revision; explain the suggested revision in the space provided.

Water Samples and Replicates

1. Time Sampled: Indicate time of sample collection, using a 24-hour clock (thus, 4 pm is 1600). Note that the time recorded on the bottle(s) and syringe(s) for the replicates should differ from the time recorded for the normal (regular) sample. This is important! The recommended protocol is to separate the sampling times for normal and replicate samples by one minute.
2. Sample ID: Enter the unique identification code assigned to the sample (developed at the project level). These Sample ID codes are designed to be familiar to the project personnel and can represent the specific site, sample, type, and date, etc. For example, the Site ID combined with the sample type and/or date (e.g., "SITEIDREG" for a regular sample or SITEIDREP for a replicate sample). Note: Bar Codes provided by the ARM Lab are a unique identifier and have a specific location for placement on the form (see item 6 below).

    The sample ID represents a sample of water (bottle(s) and/or syringes) intended to represent conditions at a particular location, on a particular day, at a particular time. Note that multiple containers (bottle and/or syringes) obtained within one time window represent the SAME sample and receive the SAME ID code (and Bar Code). Replicated samples will receive different ID codes (and Bar Codes).

3. Sample Type: Record the type of sample collected (i.e., regular, replicate, or field blank).

4. Bottle Type: Record the type of bottle (or syringe) used for sample collection (i.e., plastic, glass, or syringe).
5. Number of Bottles: Record the number of samples (aliquots) collected for the normal (regular) sample and any replicates that may have been collected.
6. Bar Code: The bar code (a unique number/letter ID) will be prepared and provided by the ARM Lab. Each year the lab provides new bar codes specific for that year. The bar code sheets will have multiple stick-on copies of the same barcode. These can be organized prior to field sampling and subsequently be affixed to the Stream Sampling Record Form, the Chain of Custody Form, and to each container (bottle or syringe) for the sample.
7. Collection Location: If the sample was not collected at the intended (targeted) location, explain the reason for changing the sample location.

General Observations

1. Air Temperature: Record the air and water temperature to the nearest degree and document the time (24hr) when it was measured. Record whether expressed in degrees C or degrees F.
2. Weather Condition (current and previous): Check the boxes that best describe the collection- day weather up to the time of sampling and the average weather over the previous 3 days (if known).
3. Stream Depth: Record the stream depth measured at sample site in mid-channel, check the box for the appropriate measurement units.
4. Stream Width: Record the stream width measured at the sample site; check the box for the appropriate measurement units.
5. Observed Discharge Level: Indicate the observed level of discharge in the stream at the time of sampling. Check the appropriate box (i.e., no flow, low flow, normal flow, or flood).
6. Delivery Method: Record the delivery method type.

Stream Sampling – Sample Record Form 4

On-Site Water Data (optional)

If on-site water data were collected, record the measured values—the time measurements were taken, air temperature and water temperature (at sample site). Check the box for the appropriate measurement units. Note: Field Instrument Data - Express DO in units of mg/L and, if possible, % DO. Correct specific conductance to 25o C. Record the measurements, instruments and methods used for on-site water data collected.

Photo Log

Take two photos of the sampling site looking both upstream and downstream. It can be helpful to take additional photos to visually record stream and site characteristics (e.g., land use and vegetation). Record the photo ID or file name, date photographed, and description of the photo. It is recommended to attach these photos to the site documentation forms to include in the Forests hard copy files.

Additional Notes

Add any additional information that may help to identify, locate, or describe this sample site.

Stream Sampling – Sample Record Form 5

This form is for collecting stream water stage and discharge data.

General information
Record the time this data was obtained and indicate what methods were used to collect an estimate of stream stage or discharge (check appropriate boxes). Complete the appropriate stage or discharge section on the form (Stage Measurement Only, Velocity-Area Procedure, or Timed Filling Procedure).

Stage Measurement Only
If stage measurements (estimates) were made in the field, record the measured value and indicate the unit of measure. Describe the location of measurement. Indicate if a rating curve has been developed with which to estimate discharge from stage measurements at this location, and indicate what the stage is referenced to (i.e., fixed staff gage, permanent landscape feature).

Discharge Measurement by Velocity-Area Procedure
If the velocity-area procedure was used to measure discharge, check "yes" and indicate the units of measurement for water depth and velocity. Record the approximate width of the stream at the sampling location. Record the water depth and velocity in each of up to 20 evenly spaced intervals of the stream cross section.

Discharge Measurement by Timed Filling Procedure
If the timed filling method was used to measure discharge, check "yes" and indicate the units of measurement for time and water volume. Record the time and volume measurements for five separate trials at each of up to three spillway locations.

Chain of Custody Form

Revised:4/2/2004

Forest/Wilderness/Park/Other (Circle one)	***Original Chain of Custody to the attention of:***	***Phone #***
Unit From:		***Fax #***
Address:	***Shipped to by (UPS/Fed Ex/USPO)***	
City, State, Zip:	***Shipping #***	***Page _____of ______***

Date Sampled	***Time Sampled***	***Sample ID***	***Location***	***Sample Collected by***	***Type (regular, blank, dup.)***	***Filtered (Y/N)***	***Preserved (Y/N/ Type)***	***Analyses Requested***	***LabID***

Comments:

Received/Relinquished by:			
Print Name	***Signature***	***Date & Time Received***	***Date & Time Relinquished***

Received at Laboratory by:			
Print Name	***Signature***	***Date***	***Time***

Processed at Laboratory by:			
Print Name	***Signature***	***Date***	***Time***

Please send this form to the contracted laboratory and make a copy for yourself.

Instruction for Completing the Chain Of Custody Form

Forest/Wilderness/Park/Other (Circle one): Circle one of theses options and write the name of the Forest/Wilderness/Park or other in the space below.

Unit From: This could be a separate unit within the Forest, Wilderness, Park or Other (ie. Subcontractor)

Address: Address of the Forest, Wilderness, Park or Other (ie. Subcontractor**)**

City, State, Zip: City, state, zip code of the Forest, Wilderness, Park or Other (ie. Subcontractor)

<u>Original Chain of Custody</u> to the attention of: Name, address, email, etc. of the person the data results and original Chain of Custody form will be sent to.

Phone #: Phone number of the person the data results and original Chain of Custody form will be sent to.

Fax #: Fax number of the person the data results and original Chain of Custody form will be sent to.

Shipped to by (UPS/Fed Ex/USPO): Which carrier you used. (**Remember to consider that on weekends and government holidays there is not anyone to receive samples in the laboratory**)

Shipping #: Tracking number assigned to the shipment by the carrier.

Page ___ of ___: Page number(s) of Chain of Custody forms sent.

Date Sampled: Date sample was taken.

Time Sampled: Time sample was taken.

Sample ID: Unique identification number assigned to sample to for tracking purposes. (ie. Bar code or unique identifier used to identify the sample for connectivity of field analysis with the laboratory analysis)

Location: Location description of sample site (ie. Lake name, stream name.)

Sample Collected by: Person collecting the sample.

Type (regular, blank, dup): Type of sample collected such as regular water, field blank, duplicate, etc.)

Filtered (Y/N): Was this sample filtered in the field?

Preserved (Y/N): Was this sample preserved in the field and with what kind of preservative (ie. H_2SO_4)

Analyses Requested: Instructions for laboratory requesting what type of analysis is to be performed (ie. ANC, pH, Conductivity, Major Cations, Anions, Totals, etc.)

LabID: A unique identifier created by the laboratory to track the samples (if they are not using the Sample ID).

Comments: Any extra remarks or instructions are placed in this space.

Received/Relinquished by:

> **Print Name:** Printed name of sampler relinquishing the samples to another person for shipment to the laboratory or directly to the laboratory**.**
> Signature: **Sampler's signature relinquishing the samples to another person for shipment to the laboratory or directly to the laboratory.**
>
> **Date & Time Received:** Date and time samples were received from the sampler.
> **Date & Time Relinquished**: Date and time relinquished by the sampler or by person shipping samples to the laboratory.

Received at Laboratory by:

> **Print Name:** Printed name of laboratory personnel receiving the samples.
> **Signature:** Signature of the laboratory personnel receiving the samples.
> Date**: Date the samples were received by the laboratory**.
>
> **Time:** Time the samples were received by the laboratory.

Processed at Laboratory by:

> **Print Name:** Printed name of laboratory personnel processing the samples.
> **Signature:** Signature of the laboratory personnel processing the samples.
> Date**: Date the samples were processed by the laboratory**.
>
> **Time:** Time the samples were processed by the laboratory.

APPENDIX C: BASIC STANDARD OPERATING PROCEDURES (SOPS) FOR STREAM FIELD SAMPLING ACTIVITIES (taken from USDA Forest Service National Protocols)

The purpose of this Appendix is to provide basic Standard Operating Procedures (SOPs) for field sampling focused on measurement of stream chemistry on Forest Service land as influenced by atmospheric deposition. The recommended Protocol featured in the main body of this report provides guidelines regarding how to implement a field sampling program for water chemistry, with explanation of some of the reasons why certain steps and precautions are recommended. An SOP is a detailed explanation of sequential steps to be taken in carrying out the water sampling. The SOP is based on the principles outlined in the Protocol. In some cases, there are multiple "correct" ways to carry out a component of the field sampling. The basic principles remain the same, and these are reflected in the Protocol. Nevertheless, the exact steps may differ, and yet still satisfy the aims of the Protocol. Thus, these basic SOPs may be modified by a particular sampling program or field office, as appropriate to local conditions. In modifying aspects of the SOPs, the guidelines represented in the Protocol should always be carefully considered.

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Introduction

This SOP provides guidelines for stream sampling within the FS ARM program. It is intended as a Base SOP, suitable for adoption as a stand-alone procedure, or for modification to fit local program needs. It is divided into individual sections that cover pre-trip activities, sampling site documentation, stream sampling and sample handling, measurement of stream discharge, post-trip activities, and needed equipment and supplies.

Pre-Trip Activities

Background

Field teams conduct a number of activities in their office or at a base site. These include tasks that must be completed both before departure to the sampling site and after return from the site. This section describes pre-trip procedures for office and base site activities that should be carried out in support of stream sampling.

Pre-departure activities include development of sampling itineraries, instrument calibration if appropriate, equipment checks and repair, supply inventories, and sample container preparation. Procedures for these activities are described in the following sections. An example check list for materials and supplies is given in Table C-1. Use this checklist to ensure that equipment and supplies are organized and available at the stream site in order to conduct the activities efficiently. Remember to take any safety equipment required by your unit (hard hats, radios, cell phones, etc.)

Before leaving the base location, package the sample containers (typically two sample bottles and two 60 mL syringes for each site to be sampled, plus backup bottles and syringes in the event that one is lost or contaminated). Take plastic containers to transport filled syringes from the field to the laboratory. Fill out a set of water chemistry sample labels. Attach a completed label to each sample bottle and syringe. Make sure the syringe labels do not cover the volume gradations on the syringe. Place each sample bottle in a separate zipper lock bag. Finally, make sure that ice and or refrigerant for shipment to the lab is frozen or freezing so that it will be ready when you return from the field to the base location with the samples.

Table C-1. Check list of materials and supplies for stream sampling site visits.	
Standard Items	✓
Collection permits and entry permits, if required.	
Site Documentation Forms (for new sites)	
Clipboard	
Site Documentation Reports (compiled as folders for existing sites)	
Stream Water Sampling Record Forms	
Insulated container with ice or frozen refrigerant (packed in sealed plastic bags or other containers)	
Small insulated container (with ice) for hike-in sites	
Watch for recording time	
Digital field camera with free memory and extra charged battery	
GPS unit with extra batteries	
Compass	
Field thermometer (with string attached)	
Pre-processed sample bottle(s) with completed sample label attached. Include a second bottle if sampling at that site is to be replicated. Put each bottle in a clean plastic zip lock bag.	
Plastic gloves in sealed plastic bag	
60 mL plastic syringes (with Luer type tip) with completed sample labels attached. Plastic container with snap-on lid to hold filled syringes	
Syringe valves (Mininert® with Luer type adapter, or equivalent, available from a chromatography supply company)	
Water Chemistry labels (if not already filled out and attached to sample containers at base site)	
Soft-lead pencils and write in rain type pens for filling out field data forms and notebook entries	
Fine-tipped indelible markers for filling out labels	
Roll or box of tape strips	
Field operations and methods documents	
First aid kit	
Backpack	
Extra zip lock bags	
Optional Items (may be required for specific studies)	
60 mL glass bottles with septum caps, and with completed sample labels attached	
Calibrated multiparameter sonde, data logger and cable, with extra batteries	
Calibration standards, quality control check samples, deionized water, rinse bottles, waste	

tray and container, calibration cup, and sensor guard for sonde (multiple sensors combined in a unit that is lowered into the water)	
Sonde calibration and post-calibration record forms	
Measurement tape	
Waders or high-top water proof boots for wading	
Clear packaging tape to cover labels	
Dissolved oxygen/Temperature meter with probe	
DO repair kit containing additional membranes and probe filling solution	
Conductivity meter with probe	

Daily Itineraries

Field sampling efforts should include a Project Leader who guides activities in the field, and a Project Coordinator who remains in the office during the sampling effort. The Project Leader reviews each site folder to ensure that it contains the appropriate maps, contact information, copies of access permission letters (if needed), and access instructions. Additional activities can include confirming the best access routes, calling landowners or local contacts (if applicable), confirming lodging or camping plans and locations (with directions), and coordinating rendezvous locations with individuals who must meet with field teams prior to accessing a site. This information is used to develop an itinerary. The Project Leader should provide the Project Coordinator with a schedule for each day of sampling. Schedules include departure time, estimated duration of sampling activities, routes of travel, and estimated time of arrival at the sampling site(s) and return to the base site. Changes that might be made to the itinerary should be relayed by the Project Leader to the Project Coordinator as soon as possible. Miscommunications can result in the initiation of expensive search and rescue procedures and disruption of carefully planned schedules.

Instrument Checks and Calibration

If appropriate, each field team should test and/or calibrate field instruments prior to departure for the sampling site. Such testing may be appropriate for dissolved oxygen meters, GPS systems, and perhaps other instrumentation. Batteries should be checked prior to departure for field sites. Extra batteries should be carried.

Field personnel should check the inventory of supplies and equipment prior to departure using project-specific site-visit checklists. Meters, probes, and sampling gear should be packed for transport to the field in such a way as to minimize physical shock and vibration during transport. Rafts or float tubes should be packed for transport so as to minimize the potential for puncture by any sharp object.

Site Documentation

Background

This section describes SOPs for establishing and documenting sampling sites on small well-mixed streams or lake outlets. This procedure applies to new sites for which approximate locations have been designated based on program objectives and sampling design. It also applies to previously established sites for which current or updated site documentation is needed.

Objective

The objective of this procedure is to establish and document new sampling sites and to update documentation for established sites, providing:

A. Site descriptions and notes
B. Travel and access descriptions and notes
C. Site coordinates obtained in the field using a GPS unit
D. Site and access-related photos
E. Placement or confirmation of numbered site tags (where applicable)

For established sites, existing site documentation will be evaluated for clarity and improved as needed based on conditions observed in the field.

Material Needed for Use in Field for Site Documentation

A. Available site documentation records for previously established sites:
   a. Site location maps, topographic maps and road maps
   b. Site descriptions and access notes
   c. Site tag numbers and tag tree descriptions (where applicable)
   d. Site coordinates
   e. Site photos
B. Preliminary site documentation for new sites:
   a. Site location maps, topographic maps and road maps, indicating approximate site locations
   b. General site descriptions and access notes
C. General material for site documentation
   a. Regional-scale topographic and road maps
   b. **Stream Water Sampling Site Documentation Forms** on water-proof paper
   c. Clipboard or field notebook and pens for use with water proof paper
   d. GPS unit with replacement batteries
   e. Digital camera with charged battery and charged replacement battery
   f. Site tags, aluminum nails, and hammer (if applicable)
   g. Measuring tape
   h. Blaze orange material for flagging tag trees in photos (if applicable)
   i. Gate keys (if needed)

j. Cell phone with numbers of project staff and management agency offices

Sequence of Site Documentation Activities

A. Initiate the **Stream Sampling Site Documentation Form**.
   a. Enter the Station ID, Station Name, Date Established or Revised, Forest (or other unit), and the name, affiliation, email address, and phone number of the person responsible for site documentation.

B. Select or locate using GPS the specific sampling site (applies to new sites).
   a. The approximate or preliminary location of new sites locations will be indicated on topographic maps. The sample collection team must still determine the exact point on the stream to be sampled.
   b. Avoid establishing sites where streams may not be well mixed, such as locations in close proximity to inflowing tributaries or braided channels. Also avoid locations that may be influenced by runoff from disturbed areas, roads, trails, drainage ditches, or other sources of inflow. Select sites that are upstream rather than downstream of potentially altered inflow. As a general rule select sites that are at least 25 m above or below confluence points or inflow.
   c. The best point to sample will be where the water is flowing fast or falling, where there are no eddies, and where the depth is at least 8 inches (20 cm). Ideally the sampling point is one that can be reached while kneeling on the stream bank or on stable rocks downstream from the sampling point. If possible, avoid standing in the water to reach the sample point.

C. Obtain new coordinates at the site using the GPS.
   a. The unit position format should be set to Decimal Degrees (hddd.ddddd). The Datum should be set to NAD83. Distance and Elevation should be set to meters. WAAS should be enabled.
   b. When “Mark Waypoint” is selected, the default GPS site id (a number) should be changed to the actual Site ID.
   c. Before saving the coordinates note the estimated accuracy of the measurement and enter on the Sampling Site Documentation Form.
   d. Save the coordinates on the GPS and record both the coordinates and the elevation on the Sampling Site Documentation Form. Do not rely solely on the GPS to store the coordinates.
   e. Confirm that the waypoint has been saved in the GPS unit.

D. Enter the approximate stream depth and width on the Sampling Site Documentation Form.
   a. Enter the approximate average values for stream depth and width observed in the sampling site area (about 5 m upstream and downstream of the sampling site) on the sample site documentation date.

E. Enter station description information on the Sampling Site Documentation Form.

a. For existing sites, enter information to improve and update existing site description information.
b. Generally describe the site, referring to proximity to landmarks (trails, bridges, tributaries, trees, landscape features or other relatively permanent features). Add any additional information, including detailed stream bank and stream structure descriptions that will help future sample collectors indentify the site. Also, add any information here that might be relevant to water and stream quality, such as cleared land, roads, construction, logging, development, or any earth disturbance, etc. observed above the site in the watershed or in the stream.
c. As a general convention, the right and left sides of a stream are determined based on looking downstream. When making observations on the form, always indicate whether the observation is made looking downstream or upstream.

F. Enter travel and access directions on the Sampling Site Documentation Form.
a. For existing sites, improve and update existing travel and access information.
b. Travel and access notes should be sufficient to guide future sample collectors to the site without reliance on GPS units. Not all future sample collectors will necessarily have GPS units.
c. Access notes should refer to trails, roads, and permanent landmarks, providing distances and where helpful, compass-based directions. Backtrack if necessary to determine distances. Linear distances and directions from the established site waypoint can be determined using the GPS unit.
d. In cases where a parking location is not immediately adjacent the sampling site, use the GPS unit to obtain the coordinates for the parking location and record in the travel and access information entry area of the Sampling Site Documentation Form.
e. For complicated or long walk-ins, use GPS unit to record and save a track. But again, do not rely on future sample collectors having access to a GPS unit.
f. Sketch the route on the back of the Sampling Site Documentation Form for scanning and saving as a jpg image if that would be helpful.

G. Obtain site and access photos.
a. For site documentation, if the camera allows, set the camera's picture size at 3m. This will create picture files that are about 550-650 kb. Larger, higher resolution files are not needed for site documentation work. Switch to higher resolution if you are taking pictures for other purposes.
b. Photos should be obtained providing downstream-looking and upstream-looking views of the sampling site, views of the tag tree (if applicable, with blaze orange material attached), and views of other distinguishing features in relation to the site (bridges, roads, notable rocks, trees, landforms, signage, etc.) Photos should also be obtained to

show important aspects of site access (parking area, forks in the trail, etc.)

c. All photos should be listed on the form, including the filename, date, and description. Enter this information at the time that the pictures are taken. Do not rely on memory for later entry of photo descriptions. The entered description should serve as the photo caption for site documentation reporting.

Stream Sampling

Background

Water chemistry data are used to characterize acid-base status, trophic condition and to classify steams based on their water chemistry. Samples for analysis of most parameters are collected into plastic bottles. Syringe samples or samples collected into glass bottles with septum caps are preferred for collection of sample aliquots for laboratory analysis of pH and dissolved inorganic carbon (DIC) where practical. Syringes and septum caps are used to protect samples from exposure to the atmosphere because the measured values for these parameters can change if the stream water sample equilibrates with atmospheric CO_2 subsequent to collection.

Stream samples are obtained at a single sampling location below the water surface in the portion of the stream cross section that appears visually to represent the greatest amount of flow, or alternatively at mid-channel in an area of flowing water. Spatial variability across the channel of a single stream is expected to be minimal in relatively small wadeable streams as compared to the variability expected among sites, so a composite water chemistry sample is not required.

At each stream, optional *in situ* and streamside measurements are made using field meters and recorded on standard data forms. Stream water is collected in one or more bottles and two 60 mL syringes or glass bottles with septum caps that are stored on ice in darkness and shipped or driven to the analytical laboratory as quickly as possible after collection. Overnight express mail to the laboratory is required for these samples because the syringe or glass bottle samples need to be analyzed, and some or all of the bottled sample needs to be stabilized (by filtration and/or acidification) within a short period of time (typically 72 hours) after collection. Check with the analytical laboratory in advance of sampling regarding applicable holding times for the parameters to be measured.

This SOP describes standard operating procedures for routine sampling and data collection at water quality monitoring sites on streams. Water samples are collected for lab analysis with optional *in situ* (on-site) measurement of selected water quality parameters (water temperature, specific conductance, pH, dissolved oxygen, and turbidity) using a multi-parameter instrument (sonde).

This section describes procedures to be followed for data collection at established water quality monitoring sites. The sites may be part of a synoptic sampling or fixed long-term sample sites for which water quality data and water samples are collected on a scheduled periodic basis.

Documentation of Data and Sample Collection

The **Stream Water Sampling Record Form** is used to document sample collection and record all field data. The form is used to record the following information:

A. The organization, station ID, and station name.
B. The date and arrival time for the site visit and specific times of measurements obtained.
C. The name, contact information, and affiliation of the individual who is the Collector of Record and responsible for protocol adherence during the site visit.
D. Suggested revisions or amendments to site documentation and travel directions.
E. A listing of site-related photographs taken, including file name, date, and descriptions.
F. Qualitative descriptions of weather, stream discharge level and appearance, and other factors that might influence water quality during the site visit.
G. Air temperature.
H. Results for all water quality data collected *in situ*, including:
   a. Numerical results, units, and measurement time.
   b. Instruments used.
I. Identification of calibration and post-calibration sensor check records.
J. Results for all discharge data collected, including:
   a. Location of measurement site relative to the sampling and data collection site.
   b. Numerical results, units, and specific time of measurement.
   c. Methods identification.
   d. Identification of discharge record files.
K. A listing of all samples collected, including:
   a. Collection time.
   b. Types of samples collected and number of replicates.
   c. Method of delivery to analytical lab.

Sequence of Activities for Data Collection

Collectors are advised to avoid entering or disturbing the stream or stream bank at, or upstream of, the collection site prior to sample collection and completion of water quality data recording. The typical sequence of activities on arrival at the sampling site is as follows:

A. Confirm the site location based on information in the Site Documentation Form, including coordinates, photos, and access notes.
B. Initiate completion of the Stream Water Sampling Record Form.
C. Complete Site Information and General Observations sections of the Stream Water Sampling Record Form.
D. Enter information needed to improve or correct the site description and travel directions provided on the Site Documentation Form.

E. Obtain any photographs needed to improve site documentation and enter file names, dates, and descriptions.

F. Note any factors (other than weather and discharge conditions) that might affect water quality (e.g., bank or upstream disturbance, debris in water).

G. Collect water samples and complete the Water Sample section of the Stream Sampling Record Form. Enter any *in situ* data into that section of the Stream Water Sampling Record Form.

H. Complete the Chain of Custody Form.

I. Check to make sure that all of the information recorded on the sample label(s), Chain of Custody form, and Stream Water Sampling Record Form match.

J. Obtain discharge measurements or stage height data, if required. Indicate method, time of measurement, result, name of the record file, and location of measurement relative to the data and sample collection site in the Stage and Discharge Data section of the Stream Water Sampling Record Form. Note that discharge gauging may be conducted at the same time as other site visit activities if the discharge measurement site is downstream of the water quality and data collection site. **Also note that measurements of discharge or stage height are considered optional.**

> If desired, *in situ* (on-site) measurement of one or more parameters can be made using a multi-parameter water quality sonde (hand held instrument with a probe [containing multiple sensors] that is lowered into the water, and that measures various physical parameters). Such measurements might include temperature, pH, dissolved oxygen, specific conductance, and/or turbidity. The procedures for such *in situ* data collection will vary with the specific field instrument, but in general require the following steps:
>
> - Initiate water quality sonde field calibration and calibration checks. Record results on a water quality instrument calibration and post-calibration record form. Confirm that sensor check criteria are met. If criteria are not met, recalibrate, perform sensor maintenance, or replace sonde or sensors as needed to meet the criteria.
> - Deploy the water quality sonde for the period required to obtain stabilization. Enter the results and time of measurement in the In Situ Water Quality Data section of the Stream Water Sampling Record Form.

Sample Collection

In the field, make sure that the labels all have the same sample ID number (barcode), and that the labels on the bottles (and syringes, if used) are securely attached. Carefully avoid disturbance of water upstream of the sampling point prior to sample collection. This means not walking in the upstream water or on upstream rocks.

Table C.2. Overview of stream sample collection procedures for water chemistry.

Collection into Bottle

1. Select sample location in a flowing portion of the channel near the middle of the stream.
2. Put on gloves provided in the sample bag.
3. Rinse sample bottle and lid three times with stream water.
4. Fill the sample bottle(s) completely, holding the bottle in a tilted position approximately at the midpoint between the water surface and the streambed, being careful not to disturb any sediment prior to or while collecting the sample.
5. If a septum cap is to be used, place the cap on the bottle under water.
6. Put the sample bottle into a clean plastic zipper lock bag.
7. Place the sample bottle(s) in a cooler (on ice or stream water) and shut the lid. This may be a soft cooler for packing out of the field to the vehicle, or a hard cooler in the vehicle. If a cooler is not available, place the bottle(s) in an opaque garbage bag and immerse it in the stream.

Collection into Syringe

8. Rinse the syringe three times with water from the sampling location.
9. Slowly fill the syringe with sample, avoiding generation of air bubbles, until it is two-thirds to three-fourths full. This will help to ensure that the plunger remains inserted far enough into the filled syringe so that it will not be likely to become dislodged during transport.
10. Expel any air.
11. Repeat procedure using a second syringe.
12. Place the filled syringes into plastic container for transport.

Collection from Very Shallow Stream

If the stream is too shallow to collect a sample using standard procedures, the following approach can be used, using a new clean syringe at each site:

13. Rinse the syringe 3 times with stream water, downstream of sample site as usual.
14. Use the syringe to put stream water in the sample bottle and rinse the sample bottle 3 times.
15. Finally, use the syringe to fill the bottle to the brim with stream water at the sample site. Cap the bottle and proceed as normal.

Collect a water chemistry sample as described in Table C.1-2 from the middle of the stream channel at the sampling site, unless no water is present at that location. Throughout the collection process, it is important to take precautions to avoid contaminating the sample. Wear gloves provided in sample bag. Rinse all sample containers three times with stream water before filling them with the sample. Many streams have a very low ionic strength and can be contaminated quite easily by perspiration from hands, sneezing, smoking, insect repellent, sunscreen, or chemicals used when collecting other types of samples (e.g., formalin or ethanol). Make sure that none of the water sample contacts your hands before going into the sample bottle or syringe. The chemical analyses conducted using the syringe samples can be affected by equilibration with atmospheric carbon dioxide; thus it is essential that no outside air contact the syringe samples during or after collection.

Record the information from the sample label on the Stream Water Sample Record Form. Note any problems related to possible contamination in the comments section of the form.

Sample Collection Procedure

The sample should be collected on a step-by-step basis as follows:

a. Remove the gloves from the plastic bag and put them on.
b. Remove the sample bottle from the plastic bag. Do not put the bag on the ground.
c. Check to ensure that the correct labels are affixed to each sample bottle and syringe.
d. Rinse the sample bottle in the stream at a location at least 2-3 feet downstream from the sample collection point. The bottle and cap should be rinsed 3 times. For each rinse, fill up the bottle and then pour the rinse water over the inside of the cap, held bottom-side up in the other hand. Pour the rinse water downstream of the rinsing and sampling points and avoid stirring-up streambed debris during the process.
e. After the rinsing is completed, move to the sampling point and collect the sample by submersing the tilted bottle or syringe to a depth midway between the sediment and the water surface. Fill up the bottle as completely as possible. While collecting the sample avoid stirring-up streambed debris that might be collected with the sample. Try to avoid generating large bubbles in the bottle while it is being filled. Also avoid collecting water that has come in contact with the gloves or the outside of the bottle. This can often be best achieved by sampling rapidly flowing or falling water. If it is deemed that debris may have entered the sample bottle, discard the contents (at a downstream location), re-rinse the bottle (or use a clean back-up bottle), and collect a new sample.
f. Immediately after collecting the sample, place the lid on the bottle (tightly) and return the bottle to its plastic bag. If a septum cap is being used, cap the bottle under the surface of the water to avoid any contact of the sample with air. Seal the bag.
g. If a sample is to be collected into a syringe, submerge a 60-mL syringe halfway into the stream and withdraw a 15-20 mL aliquot. Pull the plunger to its maximum extension and shake the syringe so the water contacts all surfaces. Point the syringe downstream and discard the water by depressing the plunger. Repeat this rinsing procedure two more times.
h. Submerge the syringe into the stream again and slowly fill the syringe with a fresh sample. Try not to get any air bubbles in the syringe. If more than 1-2 tiny bubbles are present, discard the sample and draw another one.

i. Invert the syringe (tip pointing up), and cap it with a syringe valve. Tap the syringe lightly to detach any trapped air bubbles. With the valve open, expel the air bubbles and a small volume of water, leaving the syringe between two-thirds and three-fourths full. Note that the syringe is transported only partially full to avoid dislodging the plunger during transport. Close the syringe valve. If any air bubbles were drawn into the syringe during this process, discard the sample and fill the syringe again (Step h).
j. Repeat Steps g through j with a second (back-up) syringe. Place the syringes together in a separate plastic bag and place in a plastic container, which is then placed into the cooler (or stream water if that method of cooling is used while still in the field).
k. Complete the Stream Water Sample Record Form while at the sample site.
l. Inspect all equipment, and clean off any plant and animal material before moving to the next sample location. This effort ensures that introductions of nuisance species do not occur between streams. Inspect, clean, and handpick plant and animal remains from any footwear or equipment that may have contacted stream water.

Sample Handling

a. Place the bagged sample on double-bagged ice or refrigerant immediately after collection or at least within 15 minutes of collection. Note: do not put ice in the plastic bag that contains the sample bottle or in the plastic container that contains the syringes. Ice or refrigerant should be double bagged in plastic bags to avoid possible leakage and contamination of the samples. Samples can be held in a soft sided cooler until returning to the vehicle.
b. The large sample cooler can be left in the collection team's vehicle. The sample can be transferred to the larger cooler upon return to the vehicle.
c. For sites that are not close to road access, the collection team should make arrangements to keep the sample on ice after collection and during the return hike. One approach would be to use a small soft-pack cooler or other container that will fit in a backpack. Ice, snow, or refrigerant could be placed in a plastic bag in the cooler or container (double bag to avoid leakage and contamination of samples). Samples are transferred to the larger cooler at the vehicle.
d. The samples should be kept in the dark and on ice until delivery to the lab. The ice may need to be replenished during sample transit. Avoid letting the sample bottle float in melted ice water. Do not place the sample bottle in a refrigerator or cooler with food or in any container that is not clean. Ship the samples as soon as possible, preferably within 24 hours of sampling.
e. Note that we do not recommend filtration in the field. If, however, a program filters the samples in the field for chlorophyll *a* measurement, it is important to record on the water sampling record form the volume of water filtered. Record this information in the Notes section of the form. The filter is then sent to the analytical laboratory for determination of chlorophyll *a* content.

Post Sampling Actions

a. Record the Sample ID number (barcode) on the Stream Water Sample Record Form along with the pertinent stream information (stream name, site ID, date, etc.). Note anything that could influence sample chemistry (heavy rain, potential contaminants) in the Comments section. If the sample was collected at the targeted site, record an “X” in the “STATION COLLECTED” field. If you had to move to another part of the reach to collect the sample, place the letter of the nearest transect in the “STATION COLLECTED” field. Record more detailed reasons and/or information in the Comments section. Make sure that the record form and the Chain of Custody Form are completely filled in.
b. Complete the Chain of Custody Form.
c. Check to make sure that all of the information matches on the sample label(s), Chain of Custody Form, and Stream Water Sampling Record Form.
d. Transport the samples back to the vehicle in a soft cooler on ice or snow.
e. After carrying the samples to the vehicles, place the bottle(s) and syringes in a cooler and surround with 1 gallon resealable plastic bags filled with ice. Double bag to avoid getting cooling water into sample bags.

Field Measurements

Determine stream temperature with a field thermometer (one that does not use mercury). Determine specific conductance and dissolved oxygen concentration using field meters (optional). Follow instructions provided in Table C-3. Record the measured values on the Stream Water Sample Record Form.

Table C-4 describes the equipment cleaning procedures. Inspect all equipment, and clean off any plant and animal material. This effort ensures that introductions of nuisance species do not occur between streams.

Post-Trip Activities

Upon return to a lodging or office location after sampling, the team should review all labels and completed data forms for accuracy, completeness, and legibility. A final inspection should be made of all samples. If information is missing from the forms or labels, the Project Leader should attempt, if possible, to fill in the information accurately. The Project Leader should initial all data forms after review. If samples are missing or not properly labeled, it may be necessary to reschedule the site for complete sampling. Other post-sampling activities include inspection and cleaning of sampling equipment, inventory and sample preparation, sample shipment, and communications.

Equipment Cleanup and Check

Inspect, clean, and handpick plant and animal remains from any vehicle, footwear, or equipment that may have contacted stream water.

Table C-3. Procedures for streamside and *in situ* chemistry measurements.

Specific Conductance

1. Check the batteries and electronic functions (e.g., zero, red line) of the conductivity meter as instructed by the operating manual.
2. If you haven't tested the meter at a base location recently, insert the probe into the *RINSE* container of the quality control check sample (QCCS) and swirl for 3 to 5 seconds. Remove the probe, shake it off gently, transfer it to the *TEST* container of QCCS, and let it stabilize for 20 seconds. If the measured conductivity is not within 10% of the theoretical value, repeat the measurement process. If the value is still unacceptable, do not use the meter until it can be inspected, diagnosed, and repaired.
3. Submerge the probe in an area of flowing water near the middle of the channel at the same location where the water chemistry sample was collected. Record the measured conductivity and any pertinent comments about the measurement on the Field Measurement form.

Dissolved Oxygen (DO) and Temperature

1. Inspect the probe for outward signs of fouling and for an intact membrane. Do not touch the electrodes inside the probe with any object. Always keep the probe moist by keeping it inside its calibration chamber.
2. Check the batteries and electronic functions of the meter as described in the operating manual.
3. Calibrate the oxygen probe in water-saturated air as described in the operating manual. Allow at least 15 minutes for the probe to equilibrate before attempting to calibrate. Try to perform the calibration as close to stream temperature as possible (not air temperature) by using stream water to fill the calibration chamber prior to equilibration.
4. After the calibration, submerge the probe in midstream at mid-depth at the same location where the water chemistry sample was collected. Face the membrane of the probe upstream, and allow the probe to equilibrate. Record the measured DO and stream temperature on the Field Measurement form. Record the time the DO and temperature measurement was made in 24 hour units (e.g. 14:23) on the field form. If the DO meter is not functioning, measure the stream temperature with a field thermometer and record the reading on the Field Measurement form along with any pertinent comments.

   NOTE: *Older model dissolved oxygen probes require a* ***continuous*** *movement of water (0.3 to 0.5 m/s) across the probe to provide accurate measurements. If the velocity of the stream is appreciably less than that, agitate the probe in the water as you are taking the measurement.*

Temperature Only (if no field meters are being used)

1. Place a field thermometer (±1 ^{o}C accuracy) beneath the surface of the stream at the approximate depth of sample collection in an area of flowing water at or near where the water chemistry samples were collected.
2. Record the stream temperature (estimated to the nearest 0.1^{o} C) on the Field Measurement form. Record the time the temperature measurement was made in 24 hour units (e.g. *14:23*) on the field form, along with any pertinent comments (e.g., measurement taken in sun or shade).

Table C-4. Post-sampling equipment care. (Modified from Baker et al. 1997)

1. Clean any equipment that may have contacted surface water for biological contaminants. **If you are moving between sites on the same day, do this before moving to the next site.**
2. Clean and dry other equipment prior to storage. Rinse coolers with water to clean off any dirt or debris on the outside and inside.
3. Inventory equipment and supply needs and relay orders to the Project Coordinator.
4. Remove dissolved oxygen meters, other instrumentation, and GPS from carrying cases and set up for pre-departure checks and calibration. Examine oxygen membranes of DO meters for cracks, wrinkles, or bubbles. Replace if necessary.
5. Recharge batteries overnight if possible. Replace other batteries as necessary.
6. Recheck field forms from the day's sampling activities. Make corrections and completions where possible, and initial each form after review.
7. Replenish fuel.

Shipment of Samples and Forms

Upon completion of data and sample collection, the samples and forms should be transported to the analytical laboratory in as short a time as is reasonably possible. Call or email the lab to alert them that samples are in transit and tell them what date to expect delivery. Samples should be maintained in insulated containers with refrigerant after collection and during transport. The Chain of Custody Record form should be maintained and kept with the samples until the samples are logged-in at the analytical laboratory. If samples are to be shipped to the laboratory, an overnight shipping service should be used, and shipping should be avoided when samples would be delayed by transit over a weekend or holiday period.

The field team should ship samples to the lab as soon as possible after collection. Water samples should be shipped in coolers packed with ice. Line each shipping cooler with a large 30-gallon plastic bag. Inside, package the ice separately within numerous (as many as feasible) self-sealing plastic bags and ensure that the ice is fresh before shipment. Use block ice when available, or “blue ice”. Block ice should be sealed in two 30-gallon plastic bags. White or clear bags will allow for labeling with a dark indelible marker. Label all bags of ice as “ICE” with an indelible marker to prevent misidentification by couriers of any leakage of water as a possible hazardous material spill.

To prepare the sample bottles and syringes for shipping, line the shipping cooler with a 30-gal plastic bag. Place another garbage bag in the cooler, and place the samples in the second bag. Place filled syringes and/or glass bottles in sturdy containers to prevent damage during transport. Ensure that all entries are complete and close the bag of samples. Place bags of ice around it. Then close the cooler liner (outer garbage bag). Ship water samples on the day of collection whenever possible. If that is not possible, they should be shipped the next day.

Processing Site Documentation Data and Information

A file system and database with reliable backup should be established for storage of site records and files, map images, and photos. Processing site documentation data and information include the following steps:

A. Retrieve site coordinates (and any tracks) from the GPS unit using the GPS software. Delete any extra coordinate sets (waypoints) and save the file as a *.gdb file.
B. Retrieve photos from the camera.
C. Enter or revise the site record in the site documentation database.
   a. Enter site coordinates obtained in the field.
   b. Enter or revise the site description, travel and access directions.
   c. Enter or revise the tag and tag tree information as needed. If no changes were made, note that the tag placement was confirmed on the particular date. Note that tree tags may not be applied in a wilderness setting.
   d. Add new photos as jpg images with captions to the site record.
D. Create site maps providing both detailed and broader information for access and orientation. Annotate maps and pictures with text and arrows when it would be helpful. Note that the accuracy of maps vary, and the coordinate-based point on the maps, as well as other information, may be misleading. Add clarifying notes. Save these maps as jpg images in the site record. Add captions as appropriate.

Stream Discharge

Background

Stream discharge is equal to the product of the mean current velocity and vertical cross sectional area of flowing water. It reflects the volume of water per unit time that passes a particular location (line drawn at right angle to the stream channel) on the stream. Discharge measurements can be helpful for assessing trends in streamwater chemistry that are sensitive to streamflow differences. Stream discharge information is also useful in interpreting the representativeness of water chemistry data and some physical habitat information.

The location selected for measuring stream discharge should be as close as possible to the location where chemical samples are collected. Variability in stream discharge within the reach of interest is expected to be small compared to variability in stream discharge among streams, so multiple determinations at a site are not required.

No single method for measuring discharge is applicable to all types of stream channels. The preferred procedure for obtaining discharge data for small streams is based on "velocity-area" methods (e.g., Rantz et al. 1982, Linsley et al. 1982). For streams that are too small or too shallow to use the equipment required for the velocity-area procedure, an alternative procedure is presented. It is based on timing the filling of a volume of water in a calibrated bucket.

Velocity-Area Procedure

Because velocity and depth typically vary greatly across a stream, accuracy in field measurements is achieved by measuring the mean velocity and flow cross-sectional area of many increments across a channel (Figure C.1-1). Each increment gives a subtotal of the stream discharge, and the whole is calculated as the sum of these parts. Discharge measurements are made ***at only one*** *carefully chosen channel cross section within the sample reach*. It is important to choose a channel cross section that is as much like a canal as possible. A glide area with a U-shaped channel cross section that is free of obstructions provides the best conditions for measuring discharge by the velocity-area method. You may remove rocks and other obstructions to improve the cross-section before any measurements are made. However, because removing obstacles from one part of a cross-section affects adjacent water velocities, you must not change the cross-section once you commence collecting the set of velocity and depth measurements.

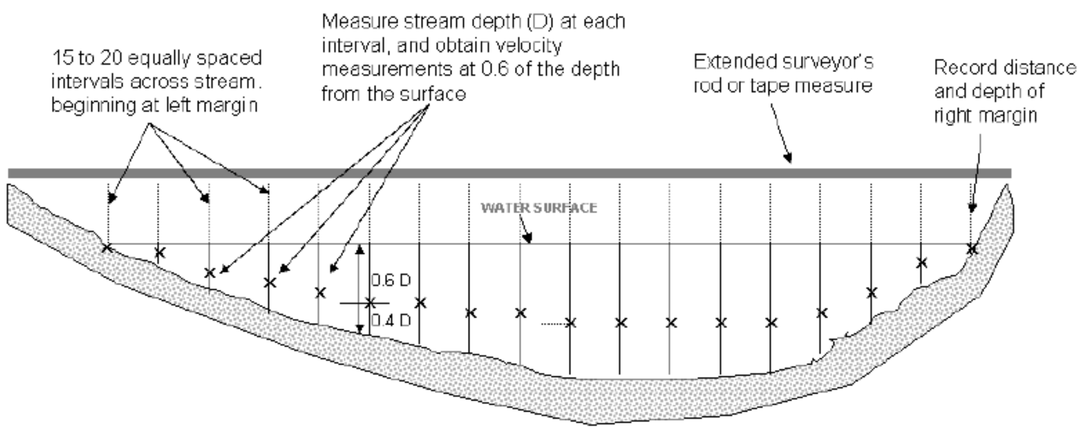

Figure C.1. Layout of a channel cross-section for obtaining discharge data by the velocity-area procedure.

The procedure for obtaining depth and velocity measurements is outlined in Table C.1-5 (based on Rantz et al. 1982). Record the data from each measurement in the Stream Discharge section of the Stream Water Sampling Record Form, giving for each measurement increment the distance from the left bank (facing downstream), water depth, measured velocity, and any required flags or notes.

Timed Filling Procedure

In channels too small for the velocity-area method, discharge can sometimes be determined directly by measuring the time it takes to fill a container of known volume. Small is defined as a channel so shallow that the current velocity probe cannot be placed in the water, or where the channel is broken up and irregular due to rocks and debris, and a suitable cross-section for using the velocity area procedure is not available. The timed filling method can be extremely precise and accurate, but requires a natural or constructed spillway of free-falling water. Because obtaining data by this procedure can result in channel disturbance or stir up a lot of sediment, wait until after all biological and chemical measurements and sampling activities have been completed.

Choose a cross-section of the stream that contains one or more natural spillways or plunges that collectively include the entire stream flow. A temporary spillway can be constructed using a portable V-notch weir, plastic sheeting, or other materials (i.e., rocks, wood) that are available onsite. Choose a location within the sampling reach that is narrow and easy to block when using a portable weir. Position the weir or constructed spillway in the channel so that the entire flow of the stream is completely rerouted through its notch (Figure C.1-2). Impound the flow with the weir, making sure that water is not flowing beneath or around the side of the weir. Use mud or stones and plastic sheeting to get a good waterproof seal. The notch must be high enough to create a small spillway as water flows over its sharp crest.

The timed filling procedure is presented in Table C.1-6. Make sure that the entire flow of the spillway is going into the bucket. Record the time it takes to fill a measured volume on the Stream Discharge section of the Stream Water Sampling Record Form. Repeat the procedure 5 times. Discharge will be calculated as an average of these five measurements. If the cross-section

Table C-5. Velocity-area procedure for determining stream discharge.

1. Locate a cross-section of the stream channel for discharge determination that has most of the following qualities:

   Segment of stream above and below cross-section is straight

   Depths mostly greater than 15 cm, and velocities mostly greater than 0.15 m/s. Do not measure discharge in a pool.

   "U" shaped, with a uniform streambed free of large boulders, woody debris or brush, and dense aquatic vegetation.

   Flow is relatively uniform, with no eddies, backwaters, or excessive turbulence.

2. Lay the surveyor's rod (or stretch a meter tape) across the stream perpendicular to its flow, with the "zero" end of the rod or tape on the left bank, as viewed when looking downstream. Leave the tape tightly suspended across the stream, at the bankfull[1] mark or higher. Adjust the tape with the aid of a small bubble level suspended from the rod or tape so it is, and remains throughout the period of measurement, level.
3. Attach the velocity meter probe to the calibrated wading rod. Check to ensure the meter is functioning properly and the correct calibration value is displayed. Calibrate (or check the calibration) the velocity meter and probe as directed in the meter's operating manual. Place an *X* in the VELOCITY AREA box on the Stream Discharge form.
4. Divide the total wetted stream width into 15 to 20 equal-sized intervals. To determine interval width, divide the width by 20 and round up to a convenient number. Intervals should not be less than 10 cm wide, even if this results in less than 15 intervals. The first interval is located at the left margin of the stream (left when looking downstream), and the last interval is located at the right margin of the stream (right when looking downstream).
5. Stand downstream of the rod or tape and to the side of the first interval point (closest to the left bank if looking downstream).
6. Place the wading rod in the stream at the interval point and adjust the probe or propeller so that it is at the water surface. Place an *X* in the appropriate DISTANCE UNITS and DEPTH UNITS boxes on the Stream Discharge form. Record the distance from the left bank and the depth indicated on the wading rod on the Stream Discharge form.

   Note for the first interval, distance equals 0 cm, and in many cases depth may also equal 0 cm. For the last interval, the distance will equal the wetted width (in cm) and depth may again equal 0 cm.

7. Stand downstream of the probe or propeller to avoid disrupting the stream flow. Adjust the position of the probe on the wading rod so it is at 0.6 of the measured stream depth below the surface of the water. Face the probe upstream at a right angle to the cross-section, even if local flow eddies hit at oblique angles to the cross-section.
8. Wait 20 seconds to allow the meter to equilibrate, then measure the velocity. Place an "X" in the appropriate VELOCITY UNITS box on the Stream Discharge form. Record the value on the Stream Discharge form. Note for the first interval, velocity may equal 0 because depth will equal 0. Note that negative velocity readings are possible, when recording negative values, assign a flag to denote they are indeed negative values.

   For the electromagnetic current meter (e.g., Marsh-McBirney), use the lowest time constant scale setting on the meter that provides stable readings.

   For the impeller-type meter (e.g., Swoffer 2100), set the control knob at the mid-position of DISPLAY AVERAGING. Press *RESET* then *START* and proceed with the measurements.
9. Move to the next interval point and repeat Steps 6 through 8. Continue until depth and velocity measurements have been recorded for all intervals. Note for the last interval (at the right margin), depth and velocity values may equal 0.
10. At the last interval (the right margin), record a *Z* in the FLAG field on the field form to denote the last interval sampled.

[1] Physical indicators of the bankfull stage include: 1) top of highest depositional features, 2) break in the slope of the bank or a change in particle size, 3) staining of rocks, and 4) exposed root hairs below an intact soil.

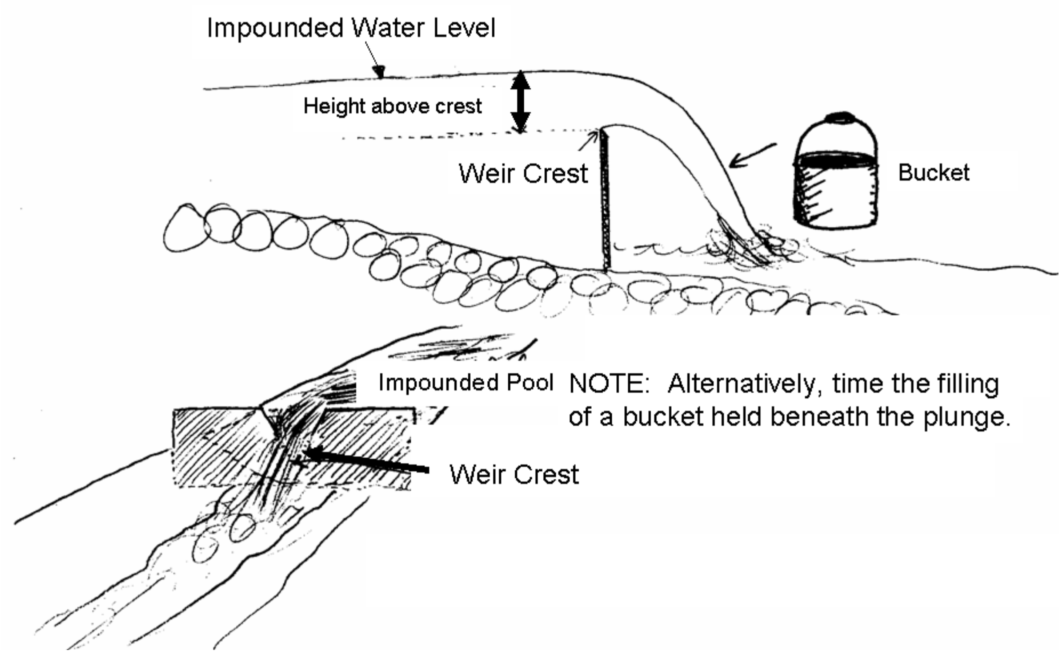

Figure C.2. Use of a portable weir in conjunction with a calibrated bucket to obtain an estimate of stream discharge.

Table C-6. Timed filling procedure for determining stream discharge.

NOTE: If measuring discharge by this procedure will result in significant channel disturbance or will stir up sediment, do not determine discharge until all biological and chemical measurement and sampling activities have been completed.

1. Choose a cross-section that contains one or more natural spillways or plunges, construct a temporary spillway using on-site materials, and/or install a portable weir using a plastic sheet and on-site materials.
2. Place an *X* in the TIMED FILLING box in the stream discharge section of the Stream Discharge form.
3. Position a calibrated bucket or other container beneath the spillway to capture the entire flow. Use a stopwatch to determine the time required to collect a known volume of water. Record the volume collected (in liters) and the time required (in seconds) on the Stream Discharge form.
4. Repeat Step 3 a total of 5 times for each spillway that occurs in the cross section. If there is more than one spillway in a cross-section, you must use the timed-filling approach on all of them. Additional spillways may require additional data forms.

obstacles from one part of a cross-section affects adjacent water velocities, you must not change the cross-section once you commence collecting the set of velocity and depth measurements.

Equipment and Supplies

Table C-7 shows the list of equipment and supplies necessary to measure stream discharge. Use this checklist to ensure that equipment and supplies are organized and available at the stream site in order to conduct the activities efficiently.

Table C.7. Equipment and supply checklist for measuring stream discharge.

Quantity	**Item**	✓
1	Surveyor's telescoping leveling rod (7-m long, metric scale, round cross-section	
1	50-m fiberglass measuring tape and reel	
1	Small bubble level to make sure the tape is level	
1	Current velocity meter, probe, and operating manual	
1-2	Extra batteries for velocity meter	
1	Top-set wading rod (metric or English scale) for use with current velocity meter	
1	Portable weir with 60/ "V" notch (optional)	
1	Plastic sheeting to use with weir (optional)	
1	Plastic bucket (or similar container) with volume graduations	
1	Stopwatch	
1	Covered clipboard	
	Soft (#2) pencils	
	Stream Discharge forms (1 per stream plus extras if needed for timed filling procedure or additional velocity-area intervals)	
1 copy	Field operations and methods documents	
1 set	Laminated sheets of procedure tables and/or quick reference guides for stream discharge	

Acknowledgments

This SOP is based partly on material developed by Kaufmann (2006), Herlihy (2006), and Merritt et al. (1997).

References Cited

Baker, J.R., D.V. Peck, and D.W. Sutton, eds.. 1997. Environmental Monitoring and Assessment Program Surface Waters: Field Operations Manual for Lakes. Report No. EPA/620/R-97/001. U.S. Environmental Protection Agency, Washington D.C.

Herlihy, A. T. 2006. Section 5. Water Chemistry. In: Peck, D.V., A.T. Herlihy, B.H. Hill, R.M. Hughes, P.R. Kaufmann, D.J. Klemm, J.M. Lazorchak, F.H. McCormick, S.A. Peterson, P.L. Ringold, T. Magee, and M. Cappaert. 2006. Environmental Monitoring and Assessment Program-Surface Waters Western Pilot Study: Field Operations Manual for Wadeable Streams. EPA/620/R-06/003. U.S. Environmental Protection Agency, Office of Research and Development, Washington, D.C. pp. 85-98.

Kaufmann, P.R. 2006. Section 6. Stream Discharge. In: Peck, D.V., A.T. Herlihy, B.H. Hill, R.M. Hughes, P.R. Kaufmann, D.J. Klemm, J.M. Lazorchak, F.H. McCormick, S.A. Peterson, P.L. Ringold, T. Magee, and M. Cappaert. 2006. Environmental Monitoring and Assessment Program-Surface Waters Western Pilot Study: Field Operations Manual for Wadeable Streams. EPA/620/R-06/003. U.S. Environmental Protection Agency, Office of Research and Development, Washington, D.C. pp. 99-110.

Linsley, R.K., M.A. Kohler, and J.L.H. Paulhus. 1982. Hydrology for engineers. McGraw-Hill Book Co. New York.

Merritt, G.D.,V.C. Rogers, and D.V. Peck. 1997. Section 3. Base site activities. In: Baker, J.R., D.V. Peck, and D.W. Sutton (eds.). Environmental Monitoring and Assessment Program Surface Waters: Field Operations Manual for Lakes. Report No. EPA/620/R-97/001. U.S. Environmental Protection Agency, Washington D.C.

Rantz, S.E. and others. 1982. Measurement and computation of streamflow: Volume 1. Measurement of stage and discharge. U.S. Geological Survey Water-Supply Paper 2175. U.S. Government Printing Office, Washington, DC. (http://pubs.usgs.gov/wsp/wsp2175/pdf/WSP2175_vol1a.pdf)

APPENDIX D:

A Summary Listing of Instrumentation and Techniques Employed by the University of Virginia for Water Analysis

The following is a summary listing of instrumentation and techniques employed by the University of Virginia (http://www.swas.evsc.virginia.edu/methods). The following is included because it serves as an alternative acceptable laboratory that other people interested in this document can compare against to obtain adequate data for the MAGIC model.

pH (hydrogen ion)

Instrumentation: Beckman Psi 21 pH meter (Part No. 123114) Corning Calomel Combination pH Electrode (Cat. Nos. 476530)

Technique Summary: Potentiometric measurement with open-system samples. Within-aliquot stability (<0.01 units/min.) and sequential-aliquot agreement (<0.03 units difference) obtained for all readings. Lab measurement is recommended due to difficulties associated with field measurement. (USEPA, 1987)

Reporting units: standard pH units

Acid-Neutralizing Capacity

Instrumentation: Beckman Psi 21 pH meter (Part No. 123114) Corning Calomel Combination pH Electrode (Cat. Nos. 476530) Technique

Technique Summary: Two-point Gran titration with 100 ml sample volumes and 0.005 N HCl titrant. Titration endpoints are approx. 4.5 and 4.2. Within-aliquot stability (<0.01 units/min.) should be obtained for endpoint determinations. (Gran, 1952)

Reporting units: µeq/L

Conductivity

Instrumentation: YSI Model 31 Conductivity Bridge; Beckman CEL-GO1 cell

Technique Summary: Standard conductivity bridge and cell, values corrected to 25 degrees C (USEPA, 1987).

Reporting units: µS/cm

Sulfate, Chloride, Nitrate

Instrumentation: Dionex 4000i Ion Chromatograph; HPIC AS4A Separator Column; HPIC AG4A Pre-Column; AMMS Anion Micro-Membrane Suppressor

Technique Summary: Simultaneous determination by ion chromatography. Injection volume: 200 uL. Eluent: 2.2 ml 3.5-4.5 mM Sodium Carbonate/minute. Regenerant: 3-4 ml .035 N Sulfuric Acid/minute. (USEPA, 1987)

Reporting units: µeq/L

Calcium, Magnesium, Potassium, Sodium

Instrumentation: Thermo Jarrell Ash AA/AE Spectrophotometer Model Smith-Hieftje 22

Technique Summary: Flame atomic absorption spectrophotometry. Li/La added to aliquot (Galloway et al., 1982).

Reporting units: µeq/L

Fluoride

Instrumentation: Orion Fluoride Combination Electrode Model 96900

Technique Summary: Determination by ion-selective electrode (USEPA, 1987).

Reporting units: µeq/L

Aluminum, total monomeric and inorganic monomeric

Instrumentation: Technicon Autoanalyzer II

Technique Summary: Colorimetric detection with open-system samples by pyrocatechol violet technique. Fractionation with ion-exchange resin. (McAvoy et al.,1992)

Reporting units: µg/L

Dissolved organic carbon

Instrumentation: Dohrmann Carbon Analyzer

Technique Summary: Persulfate/UV oxidation with infrared detection (USEPA, 1987).

Reporting units: mg/L

APPENDIX E:

Field Data Forms for the Soil Sampling Components of the Assessment Project

SOILS FIELD DATA FORM

(Complete one form for each the O-horizon, shallow and deep samples)

Sample ID (*place on bags*):	Date (*mm/dd/yy*): ________ Time (*hh24mm*): ________	National Forest (*circle one*): Pisgah, Nantahala, Cherokee, or Sumter

Hydrologic Unit Code:	Ranger District:
GPS Location (*decimal degrees*): Longitude: __________________ Latitude: __________________	Wilderness or Other Administrative Name:
Site ID (based upon latitude and longitude):	USGS 7.5 topo map:
Elevation (*m*):	Sketch Profile (describe colors using Munsell chart and measure depth of each horizon):
Tag number and description of tag tree:	
Soils Map Unit:	
General Soil Description (Attach map unit description to this sheet)	
Vegetation Type (*circle one*): hardwood, conifer, open field, hardwood-conifer, conifer-hardwood, or brush	
Local Disturbance:	
Deviations from Sampling Site Protocols (especially depth):	Photograph number(s): _____________

Estimated Percent Coarse Fragments: Indicate Range (*circle*)	<u>Shallow Sample</u>	<u>Deep Sample</u>
	< 15	< 15
	15 to <35	15 to <35
	35 to <60	35 to <60
	60 to < 90	60 to < 90
	≥ 90	≥ 90

Describe Coarse Fragments (i.e., size, shape, aggregation):

Narrative access and site description (Any problems finding or getting to the site?):

Soils layer (*circle*): O-horizon, shallow, deep Type of Sample (*circle*): Regular, Duplicate	Size (diameter) of O-horizon sample collected (cm) ____ Depth of mineral soil sampling pit (cm) ______

Technician Responsible: Last Name	First Name	Phone Number: Area Code ________ Number _________ - _______________

APPENDIX F:

A Summary Listing of Instrumentation and Techniques Employed by the University of Virginia for Soils Analysis

pH (hydrogen ion)

Instrumentation: Beckman Psi 21 pH meter (Part No. 123114) Corning Calomel Combination pH Electrode (Cat. Nos. 476530)

Technique Summary: Potentiometric measurement in a 1:1 soil to deionized water slurry by mass and in 0.01 M $CaCl_2$ (Cappo et al., 1987)

Reporting units: standard pH units.

Exchangeable Bases (calcium, magnesium, potassium, and sodium)

Instrumentation: Thermo Jarrell Ash AA/AE Spectrophotometer Model Smith-Hieftje 22

Technique Summary: Extraction by 1.0 N ammonium chloride; analysis by flame atomic absorption spectrophotometry (SSLMM, 1996).

Reporting units: cmol+ / kg soil

Exchangeable Aluminum

Instrumentation: Thermo Jarrell Ash AA/AE Spectrophotometer Model Smith-Hieftje 22

Technique Summary: Extraction by 1.0 N potassium chloride, analysis by flame atomic absorption spectrophotometry (Weaver et al., 1982).

Reporting units: cmol+ / kg soil

Cation Exchange Capacity

Instrumentation: Thermo Jarrell Ash AA/AE Spectrophotometer Model Smith-Hieftje 22

Technique Summary: Extraction by 1.0 N ammonium chloride, analysis by flame atomic absorption spectrophotometry (SSLMM, 1996).

Reporting units: cmol+ / kg soil

Total Nitrogen and Carbon

Instrumentation: Fisons NA1500 Elemental Analyzer

Technique Summary: combustion (Nelson and Sommers, 1996).

Reporting units: percent dry weight