QUALITY ASSURANCE PROJECT PLAN (QAPP) TO SUPPORT THE ALSEA WATERSHED STUDY REVISITED

A monitoring project to assess the effectiveness of Oregon Forest Practices Rules for watershed protection

Lead Organization:
National Council for Air and Stream Improvement, Inc. (NCASI)
Corvallis, Oregon

Cooperating Organizations

Dr. Stephen Schoenholtz
Department of Forest Engineering
267 Peavy Hall
Oregon State University
Corvallis, OR 97331-5706
541-737-9112
Fax: 541-737-4316
Stephen.Schoenholtz@oregonstate.edu

Dr. John Stednick
Department of Forest, Rangeland, and Watershed Stewardship, College of Natural Resources
327 Natural Resources Bldg.
Colorado State University
Fort Collins, CO 80523-1472
970-491-7248
Fax: 970-491-6307
jds@cnr.colostate.edu

Dr. George Ice
Principal Scientist
NCASI
PO Box 458
Corvallis, OR 97339-0458
541-752-8801
Fax: 541-752-8806
gice@wcrc-ncasi.org

Jeff Light
Forest Hydrologist
Plum Creek Timber Company
PO Box 216
Toledo, OR 97391
541-336-6227
Fax: 541-336-5827
jeff.light@plumcreek.com

Dr. Robert Gresswell
USGS Northern Rocky Mountain Science Center (NRMSC)
P.O. Box 173492
Bozeman, Montana, 59717-3492
(406)994-7085
Fax: (406) 994-6416
bgresswell@usgs.gov

Douglas Bateman
Department of Forest Science
321 Richardson Hall
Oregon state University
Corvallis, OR 97331
(541) 758-8817
doug_bateman@usgs.gov

Quality Assurance Project Plan (QAPP) Prepared By: Terry Bousquet, Project Leader, NCASI, Corvallis, Oregon, to partially fulfill internship requirements for the OSU Environmental Sciences Professional Science Masters Program

This QAPP follows the format and guidelines presented in *Guidance for Quality Assurance Project Plans, EPA QA/G-5* (USEPA 2002) to be consistent with requirements specified in *EPA*

Requirements for Quality Assurance Project Plans EPA QA/R-5 (USEPA 2001). Oregon Watershed Enhancement Board's *Oregon Plan for Salmon and Watersheds, Water Quality Monitoring Guidebook* (OWEB 1999) and Oregon Department of Environmental Quality's *Watershed Assessment Section Mode of Operations Manual (MOMs)* (ODEQ 2004) were used for guidance in developing watershed-specific QA/QC parameters. EPA's *Wadeable Streams Assessment: Integrated Quality Assurance Project Plan* (USEPA 2004a) and *Wadeable Streams Assessment: Field Operations Manual* (USEPA 2004b) also provided information used in developing this plan.

PURPOSE

This QAPP is a supplement to *Best Management Practices (BMPs) Effectiveness for Timber Harvest in Temperate Coastal Forests: The Alsea Watersheds Revisited*. The purpose of the QAPP is to provide reliable, high quality data that addresses the measurement information needs of the Alsea Watershed Study Revisited. The intent is to develop data of known precision and accuracy to characterize water quality parameters. Established analytical procedures will be employed to ensure consistent data quality over the course of the study for use in before and after comparisons. Where feasible, sample collection sites will be comparable to those used during the original study to facilitate data comparisons between studies. This plan documents the planning, implementation, and assessment procedures for the study. Quality assurance (QA) and quality control (QC) activities associated with the sampling and analytical aspects of this study are identified.

APPLICABILITY

This QAPP is applicable to water quality sampling and analytical activities associated with the Alsea Watershed Study Revisited. Data quality components for habitat and biological assessments will be incorporated at a later date or as an additional supplement to the overall study plan. All cooperating investigators shall ensure that the QAPP is implemented as approved and that all personnel under their leadership have access to a copy of the plan and understand its requirements.

QAPP PLAN REVIEW AND REVISION

Due to the length of the study, diversity of sampling locations, and potential unforeseen changes to the watershed, it may become necessary to modify the plan. When such changes are necessary they must be approved by the cooperating investigators. The plan will be reviewed annually and may be modified as needed to be consistent with the project objectives.

Effective Date of the QAPP	Signature:	Date:
Dr. Stephen Schoenholtz	_____	_____
Dr. John Stednick	_____	_____
Dr. George Ice	_____	_____
Jeff Light	_____	_____

CONTENTS

TABLES

FIGURES

Distribution List

Name	Phone/Fax/email	Address
Dr. Stephen Schoenholtz	541-737-9112 Fax: 541-737-4316 Stephen.Schoenholtz@oregonstate.edu	Oregon State University Dept. of Forest Engineering 267 Peavy Hall Corvallis, OR 97331-5706
Dr. George Ice	541-752-8801 Fax: 541-752-8806 gice@wcrc-ncasi.org	NCASI PO Box 458 Corvallis, OR 97339-0458
Dr. John Stednick	970-491-7248 Fax: 970-491-6307 jds@cnr.colostate.edu	Colorado State University Dept. of Forest, Rangeland, and Watershed Stewardship College of Natural Resources 327 Natural Resources Bldg. Fort Collins, CO 80523-1472
Jeff Light	541-336-6227 Fax: 541-336-5827 jeff.light@plumcreek.com	Plum Creek Timber Company PO Box 216 Toledo, OR 97391
Dr. Sherri L. Johnson	541-758-7771 johnsons@fsl.orst.edu	USFS PNW Research Station 3200 SW Jefferson Way Corvallis, OR 97331
Douglas Drake	503-229-5350 Fax: 503-229-6957 drake.doug@deq.state.or.us	Oregon Department of Environmental Quality 2020 SW Fourth Ave. #400 Portland, OR 97201
Dr. Arne Skaugset	541-737 4316 Fax: 541-737 4316 arne.skaugset@oregonstate.edu	Oregon State University Forest Engineering 204 Peavy Hall Corvallis, OR 97331-5706
Terry Bousquet	541-752-8801 Fax: 541-752-8806 tbousquet@wcrc-ncasi.org	NCASI PO Box 458 Corvallis, OR 97339-0458
Cody Hale	706-714-2839 cody.hale@oregonstate.edu	Oregon State University Forest Engineering 204 Peavy Hall Corvallis, OR 97331-5706

Name	Phone/Fax/email	Address
Dr. Robert Gresswell	406-994-7085 bgresswell@usgs.gov	USGS Northern Rocky Mountain Science Center (NRMSC) PO Box 173492 Bozeman, MT 59717-3492
Douglas Bateman	541-758-8817 doug_bateman@usgs.gov	Douglas Bateman Department of Forest Science 321 Richardson Hall Oregon state University Corvallis, OR 97331

QUALITY ASSURANCE PROJECT PLAN (QAPP) TO SUPPORT THE ALSEA WATERSHED STUDY REVISITED

1.0 PROJECT MANAGEMENT

1.1 Project /Task Organization

Dr. George Ice will serve as Project Manager. Cooperating lead investigators (CLIs) have oversight responsibility for different aspects of the study, as shown in Table 1.

Table 1. Key Responsibilities

Investigator	Responsibilities
Project Manager	
George Ice, NCASI	Overall project management, scheduling of events with cooperating investigators, funding coordination
Landowners	
Jeff Light, PCTC	Access to existing Needle Branch sampling locations and gaging stations Coordination of timeline for harvesting Implementation of harvesting activities in compliance with Oregon Forest Practices Act and funding.
USDA Forest Service	Access to Deer Creek and Flynn Creek sampling locations and gaging stations
Habitat Assessment and Fish Population	
Jeff Light, PCTC	Oversight of habitat and fish population assessment
Robert Gresswell, USGS	Field sampling, analysis, data management
Douglas Bateman, OSU	Analysis of fish distribution, abundance, and movement
Field Sampling/Field Water Quality Analyses	
John Stednick, CSU	Hydrology monitoring and stream flow statistics
Stephen Schoenholtz, OSU	Oversight of sample collection and field analyses
Laboratory Water Quality Analyses	
Stephen Schoenholtz, OSU	Oversight of total suspended sediment analyses
Terry Bousquet, NCASI	Oversight of nutrient analyses
George Ice, NCASI	Oversight of COD analyses
Biological Sampling and Analyses (macroinvertebrates)	
TBD	Oversight of biological sampling and analyses
Project QC Officer	
Terry Bousquet	Development of QAPP, review of nutrient analyses data conducted at NCASI

1.2 Background

The original Alsea Watershed Study, conducted from 1958 through 1973 in the Coast Range near Salado, Oregon, involved water quality assessments of three small watersheds. These studies

compared the impacts of forest practices on an unprotected stream (Needle Branch) that was completely clearcut and burned, a stream (Deer Creek) that was patchcut and burned with a riparian buffer left along the main stream channel, and an untreated control stream (Flynn Creek). The results of these studies were used, in part, to develop harvest rules in Oregon's Forest Practices Act (Oregon Revised Statutes (ORS) 527.610-527-770). Information regarding water yields and peakflows, sediment loads, nutrient concentrations, temperature responses, dissolved oxygen impacts, and fisheries responses were generated (Moring and Lantz 1975; Stednick and Hall in prep.).

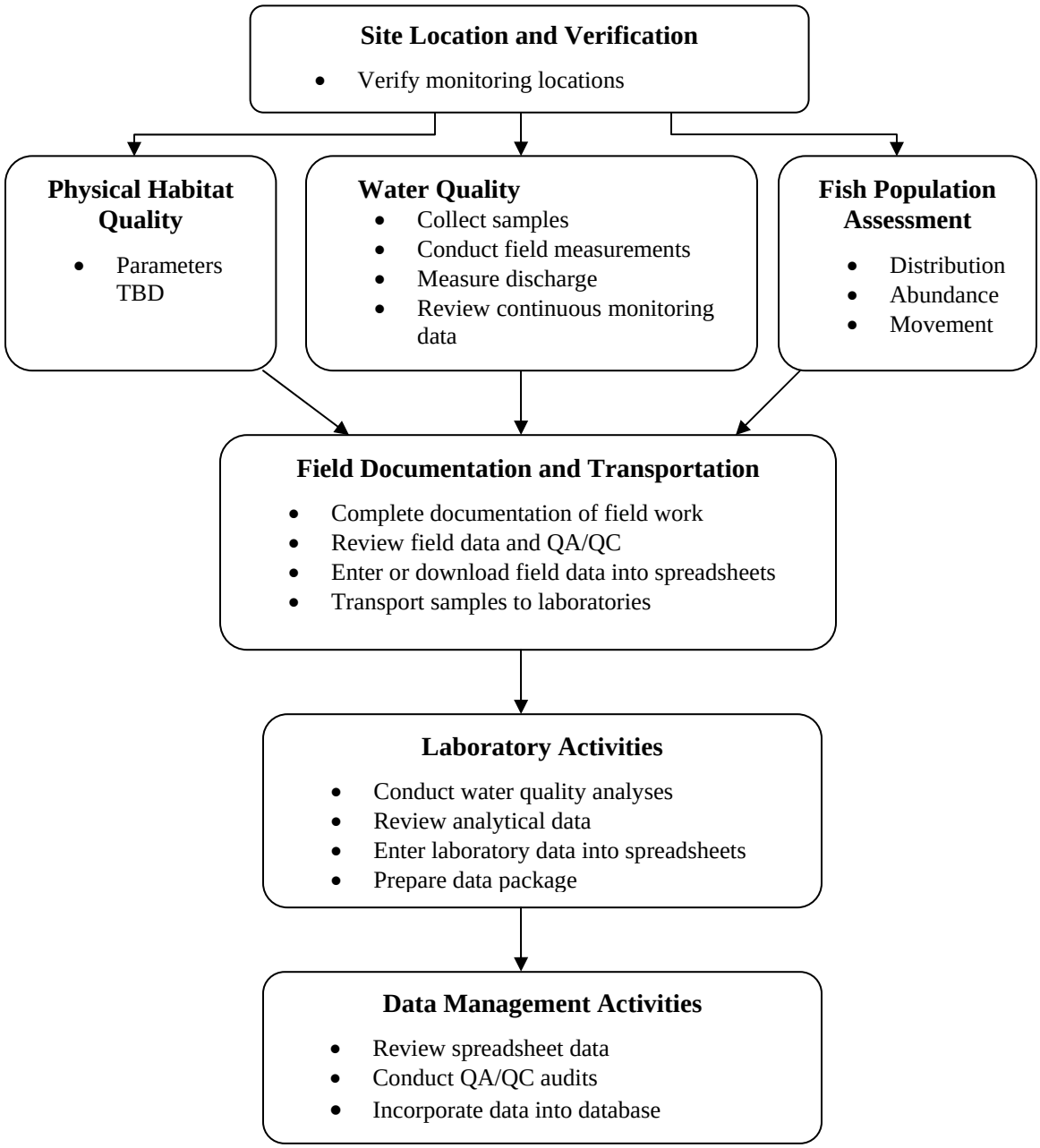
In 1989, stream gages were restarted, temperatures were monitored, and a limited number of water quality measurements were conducted to evaluate the status of these watersheds. The results indicated that streamflow for the control watershed, Flynn Creek, had not changed and dissolved oxygen concentrations and water temperatures had returned to pre-harvest levels at Needle Branch. Development of an alder riparian stand along Needle Branch is thought to be responsible for an observed increase in nitrate-nitrogen as compared to Flynn Creek.

Plum Creek Timber Company (PCTC) manages the upper two-thirds of the watershed, including the Needle Branch stream. PCTC plans to harvest timber in this area, compliant with the Oregon Practices Act, in 2007 or 2008. This will allow an opportunity to revisit the Alsea Watershed to assess improvements in water resource protection due to implementation of new practices such as equipment exclusion zones, riparian buffers around fish-bearing streams, and prevention of slash and debris piling in streams with subsequent cleanout of wood from the channels. To perform these water quality assessments, it will be necessary to conduct sampling and analytical work before, during, and after harvest. It is anticipated that sampling and analytical work will begin in fall of 2005 and end in fall of 2011. The intent is to provide at least two years of water quality data prior to and following harvest.

1.3 Task Descriptions

The original three watersheds will be assessed in the Alsea Watersheds Study Revisited, with Flynn Creek again serving as the control stream. Needle Branch, the stream that was clearcut and burned in the original study, will be clearcut with buffer zones using current forest management practice rules. Harvest on Deer Creek will be managed at the discretion of the USDA Forest Service and may be patchcut or thinned. The study timeline will coincide with forest management activities on the Needle Branch site. To draw comparisons with the data generated in the original study, the same water quality parameters will be evaluated at the same gaging stations or synoptic sampling locations when reasonably possible. Due to the costs associated with reestablishing fish traps, some of the biological parameters evaluated in the original study will not be included in this study. Advances in analytical methods and measurement technology may improve the precision and accuracy of some measurements compared with data that generated in the previous study. Methods to collect and analyze sediment, temperature, and nutrients will incorporate contemporary methods (e.g., turbidity activated pump samplers and electronic temperature data loggers). However, where possible some samples will be collected using original methodologies (e.g., sediment samples collected using a depth integrated DH-48 or a series of single stage suspended-load samplers) to provide a better direct comparison with the original study results. It will be important to characterize the data quality of this study when making comparisons with the original study results. Where applicable, analyses will be conducted using methods approved under EPA's Clean Water Act at 40 CFR, part 136.3 (USEPA 2004c).

Figure 1 summarizes the sampling and measurement activities expected during this study.



*Adapted from EPA's *Wadeable Streams Assessment* (USEPA 2004a,b)

Figure 1. Summary of Sampling and Measurement Activities

The primary activities will involve site location and verification, physical habitat, water chemistry, biological monitoring, additional field activities, laboratory activities, and data management activities. Activities associated with habitat and biological monitoring are to be determined (TBD).

1.3.1 *Sample site selection*

In order to compare results from the Alsea Watershed Study Revisited with data from the original study, sampling sites and gaging stations will be the same as (or as close as reasonably possible to) the original data collection sites on Flynn Creek, Deer Creek, and Needle Branch. A new gaging station immediately below the headwater to the harvest unit on Needle Branch is also planned. Synoptic samples will be collected as appropriate throughout the watershed during specific events to support information needs associated with storm events, seasonal fluctuations, or biological monitoring.

The Flynn Creek watershed (approximately 202 ha) will serve as a control for the study. In the original study, stream length was described as approximately 1433 m, with a mean summer width of 1.74 m and mean summer depth of 13 cm. The stream gradient average was cited as 0.025 m per stream meter. The gaging station location was designated as 0 meters, 0 feet. A fish collection trap was located 305 m (1000 ft) downstream from the gage. Between 305 and 549 m (1800 ft) above the weir a steep canyon restricted access for fish. Four small tributaries to Flynn Creek, evenly spaced along the stream length, were identified, with the final study marker at 1006 m (3300 ft) upstream (Moring and Lantz 1975). The sampling locations identified during the original study will be duplicated for specific parameters where reasonably feasible.

The Deer Creek watershed (304 ha) is managed by the USDA Forest Service and may be patchcut or thinned during the course of the study. It was patchcut in the original study and has had continuing harvest activities since 1972. This provides a measure of watershed response to a shifting mosaic of management activities. The stream length studied in the original assessment was reported to be approximately 2324 m, with a stream gradient of 0.018 m per stream meter, mean summer width of 1.80 m, and mean summer depth of 11 cm. A fish trap was located about 152 m (500 ft) below the stream gage. A steep canyon was noted from the 152 m stream marker to approximately the 427 m (1400 ft) location. The stream was described as slow moving and meandering from 427 to 1219 m. East Fork was noted to be a major tributary of Deer Creek at about 1433 m. Four additional smaller tributaries were noted, two upstream and two downstream of the East Fork tributary. (Moring and Lantz 1975). As with the Flynn Creek site, sampling locations for this study will attempt to approximate the original sampling sites when applicable to the study requirements.

The Needle Branch watershed (75 ha) will be harvested by PCTC in two or more units. The stream length studied in the original assessment was reported to be approximately 966 m, with a stream gradient of 0.014 m per stream meter, mean summer width of 1.10 m, and mean summer depth of 7 cm. A fish trap was located about 61 m (200 ft) below the stream gage. Small waterfalls were noted at 808 m (2650 ft) and 869 m (2850 ft). The final study area stake was placed at 792 m (2600 ft), but biological factors and other surveys were conducted above that point after 1966. The area above the second fall included two small tributary streams, and above 1067 m (3500 ft) flow was described as greatly reduced, with isolated pools present to the headwaters (Moring and Lantz 1975). As with the Flynn Creek site, sampling locations for this

study will attempt to approximate the original sampling sites when applicable to the study requirements.

Water quality monitoring will be conducted before, during, and after harvesting in the Needle Branch watershed. Pretreatment monitoring will be conducted for approximately two years. Harvests will occur in two phases, with a one or two year gap between harvests during which monitoring will continue. Monitoring will be conducted for two to three years following the first year of harvest. Further discussion of field measurements and field sampling activities are included in Sections 2.2 and 2.3, respectively.

1.3.2 *Measurement and analysis*

Water quality monitoring will consist of field and laboratory measurements. Discharge measurements will be conducted using an electronic continuous stage recorder at the original gaging stations of both creeks to measure lowflows and peakflows, and to calculate sediment loads and water yields. A continuous turbidimeter will be employed as an indirect measure of sediment to trigger suspended sediment collection. The turbidity level that triggers sample collection will be based on historical data and may be adjusted based on initial monitoring results. Field measurements will include dissolved oxygen (DO), stream temperature, and conductivity and will be conducted using procedures described in the *Oregon Plan for Salmon and Watersheds, Water Quality Monitoring Guidebook* (OWEB 1999).

Laboratory measurements will include total suspended solids (TSS) or chemical oxygen demand (COD), and nutrients. Analytical methods comparable to those approved by EPA under the Clean Water Act in 40 CFR, part 136.3 will be employed for laboratory measurements. Analytical methods are discussed further in Section 2.6.

Habitat assessment and biological monitoring parameters are still to be determined. It is likely that they will be based on fish and habitat monitoring routinely conducted by the Oregon Department of Fish and Wildlife (ODFW) and macroinvertebrate sampling conducted by the Oregon Department of Environmental Quality (ODEQ). These parameters will either be added to a subsequent update or included in a separate QAPP.

1.3.3 Timeline

Table 2 summarizes the schedule of events and milestones for the Alsea Watershed Study Revisited. These dates are subject to change as the study progresses.

Table 2. Schedule of Events and Milestones

Event	Expected Start Date	Expected Completion Date
Install monitoring stations	June 2005	September 2005
Pretreatment monitoring	October 2005	Spring 2008
First harvest (Needle Branch)	Spring 2008*	TBD
Second harvest (Needle Branch)	TBD	TBD
Post treatment monitoring	Fall 2008	Fall 2011
Annual research summaries	October annually	January annually
Final report	Fall 2011	TBD

* Jeff Light is working with ODFW to test whether an additional pre-treatment year will substantially improve the statistical power to evaluate fish population changes

1.4 Quality Objectives and Criteria

The intended user groups for these data are members of the timber industry, Oregon Department of Environmental Quality, USDA Forest Service, Oregon Department of Forestry, Oregon Department of Fish and Wildlife, and other researchers interested in water quality data. The minimum data quality objectives for water quality field measurements and laboratory measurements are based on the ranges, detection limits, precisions, and accuracy statements of the field or analytical instruments or measurement methods. More stringent data quality requirements may be specified for individual parameters to be consistent with the *Oregon Plan for Salmon and Watersheds, Water Quality Monitoring Guidebook* (OWEB 1999), or when superceded by more stringent requirements specified in field- or laboratory-specific SOPs.

Applicable field check measurements or reference checks will be used periodically to validate continuous field monitoring equipment, and reference materials will be used to validate laboratory data if appropriate materials are available. QA/QC checks such as duplicates and matrix spikes will be conducted at the method-specified frequency (typically 5 to 10% of samples) and must meet the data quality criteria of the specific method unless otherwise stated in this document. Field and laboratory measurements will be compared with EPA-specified water quality criteria where such criteria are published.

Data quality objectives for habitat measurements and biological parameters will be addressed when the parameters, test methods, and equipment are identified. Data quality requirements are further discussed in Section 2.7.

1.5 Training and Certification

Field samplers will be trained in calibration and maintenance of equipment, proper sample collection techniques, and field safety. General field safety considerations are presented in Section 5.0, but may be superceded by more stringent requirements of land owners or cooperating lead investigators (CLIs). Field and laboratory analysts will follow procedures specified in the analytical methods or in field or analytical standard operating procedures (SOPs) unless otherwise specified. No specialized training other than that normally conducted by the laboratory, field sampling organization, or property owner is required. State certification is not required. The use of blind QA/QC samples as a check of field or laboratory capabilities may be conducted for specific parameters.

1.6 Documents and Records

1.6.1 *Sample collection documentation*

Field samplers will document field sampling activities in field notebooks or on designated field data sheets. All field data will be identified by sampling site and station (e.g., Needle Branch gage; Flynn Creek synoptic site 3). Field samplers will record information regarding date and time of collection, instrumentation, field measurements, collection of samples for off site analyses, maintenance, calibrations, field validation checks, and collection of field blanks and field duplicates. Results of any QA/QC testing conducted in the field will be recorded. Field samplers will document observations that may impact field results, such as fallen trees, other debris, or tampering. An SOP describing water quality field sampling to be conducted by OSU is included in Attachment A. Additional documentation information is presented in Section 2.4.

1.6.2 *Sample handling documentation*

Samplers will provide sample collection documentation to the laboratory that indicates the date and time samples were collected, identifies each sample bottle and QA/QC sample bottle by a unique sample code, identifies the analytical parameter(s) to be tested, identifies field preservatives used, and indicates the method of transportation to the laboratory (e.g., hand delivered). Samplers must verify custody of the samples from the time of collection to the laboratory as described in Section 2.5.

1.6.3 *Analytical laboratory documentation*

Analytical laboratories conducting testing will record the date of sample receipt and any discrepancies between the sample label and the documentation provided by the sampler. The laboratory will be responsible for maintaining laboratory bench sheets or notebooks, instrument logs, raw data, and instrument printouts where applicable. Laboratory records will indicate the analytical methods and instrumentation used, the date of sample preparation and analysis, and either the time of analysis or analytical sequence. The laboratory will be responsible for providing a package of data deliverables showing the sample results and QA/QC data specified in Section 2.7.3.

1.6.4 *Data audits and review*

CLI with oversight responsibilities for sampling, laboratory analyses, habitat assessment, or biological parameters are responsible for ensuring that data are reviewed and audited. Laboratories conducting analyses are required to internally review their data and flag any parameters that are not within the quality control criteria specified in Section 2.7. Data management considerations are discussed in Section 2.12 and auditing considerations are discussed in Section 4.1.

1.6.5 *Records retention policy*

All documentation and data files will be retained for no more than five years following the completion of the final report. Records may be maintained for longer than five years provided all cooperating organizations provide written approval.

2.0 DATA GENERATION AND ACQUISITION

2.1 Sampling Process and Design

The sample sites used during the original Alsea Watershed Study were summarized in Section 1.3.1. Where reasonably feasible, the same Flynn Creek, Deer Creek, and Needle Branch sampling locations will be used when the same parameters are being measured. Based on results of the previous study and limited resources, some of the sampling sites or parameters may not be addressed in this study. The sampling locations for each parameter are identified in Section 2.2 for field monitoring and field analyses and Section 2.3 for samples to be analyzed in the laboratory.

Samples were collected during the original study to correspond with peakflows and lowflows, capture seasonal variability, and correspond with biological habitat parameters. Similar sampling frequencies are likely to be employed in this study.

2.2 Field Measurements for Water Quality Parameters

Permission or permits for access to the sampling sites will be obtained from site owners prior to sampling events. To ensure consistency in the conduct of field measurements and sample collection, a field sampling protocol describing the operations of field equipment, equipment checklists, sample collection methods, field preservation, record keeping requirements, and transportation of samples to the laboratory is included in Attachment A. Instances where sampling should be curtailed or postponed due to safety concerns, weather events, or other considerations are described in the protocol.

During the original study, streamflow data were collected using continuous analog recorders housed in 4 ft by 4 ft shelters over 42 inch culvert pipe stilling wells, each of which was connected to the stream by two 2 inch intake pipes. The stage control structure for each station was a concrete V-notch weir. Discharges at the gaging stations were determined from stage-discharge relations defined by a series of discharge measurements. High and medium flow discharge measurements were made using either the standard Price or pygmy current meter. Low flow measurements were made on Needle Branch by timing the filling of a metal container of known volume held below the weir. Each discharge measurement was plotted to the concurrent gage height. Stage-discharge relations were developed from the measurements for each site and were used to obtain the discharge data for the analyses (USGS 1971). Original streamflow data were recorded using 1:0.1 ratios and were reduced in accordance with the techniques described in *Measurement and Computation of Streamflow* (USGS 1982). Data generated after 1989 were recorded using 1.0:1.0 ratios, providing a more accurate stage measurement intended to more accurately estimate discharge. The same gaging stations at the three sites will be used to generate streamflow data using a Campbell Scientific CR10x data logger with CS420-L Druck pressure transducer to collect stage measurements. The data logger will be set to a minimum flow to initiate collection of data approximately every 10 minutes. More or less frequent measurements may be adopted depending on the results of early monitoring. Initially, data from the original study and more recent measurements will be used to establish the baseline data collection stage. The minimum flow used to initiate data collection may be adjusted as new background data become available. Stage-discharge relations will be validated by comparing the value on the data logger with the height measured on the weir weekly or each time the monitor is checked and data are downloaded.

During the original study, continuous recordings of water temperatures were obtained with Partlow recording thermographs. At least one unit was deployed in each watershed. Six thermographs were used on Needle Branch during the early phase of the study, but some units were removed and three units were used for the study duration. One unit was used at the stream gaging station on Flynn Creek (Moring and Lantz 1975). During monitoring initiated in 1989 temperatures were measured first using a Unidata® stage recorder and temperature probe, and later with Vemco® data loggers. In the original study, additional temperature measurements were conducted at locations important to specific behavioral studies and at locations determined by the objectives of short-term investigations conducted by graduate students. Due to advances in temperature logging equipment, CR10X® data loggers with CS547A-L dual temperature and conductivity probes will be deployed immediately below the headwater harvest unit on Needle Branch and at the original gaging stations on each of the three streams to measure temperature and conductivity at approximately 10 minute intervals. More or less frequent measurements may be adopted depending on the results of initial monitoring. Conductivity measurements were not conducted during the original study, but will be monitored in the new study. Additional

temperature monitoring throughout the watersheds may be conducted during this study to support an understanding of spatial responses of stream temperatures to current harvesting.

Sediment samples were collected with a U.S. DH-48 sediment sampler at the concrete control weirs of the original gaging stations. The nozzle of the sampler traversed the entire depth of the stream from the water surface to the crest of the weir. The sediment concentration and load obtained using this sampling technique was termed "suspended sediment." Several years after completion of the Alsea Watershed Study, a recording turbidimeter and pumping sampler were installed at the fish trap on Flynn Creek to obtain nearly continuous measurements of turbidity and suspended sediment concentrations at the mouth of the watersheds during high flows, and a correlation between turbidity and suspended sediment was determined for each watershed. Considerable variability was noted due to seasonal changes, storm dynamics, and land use activities (NCASI 1991). A CR10X data logger with an OBS-3-L turbidity monitor and an activated pump sampler will be deployed at the original site and at the new station immediately below the headwater harvest unit on Needle Branch to develop a correlation between turbidity and suspended sediment. The minimum stage level will trigger collection of turbidity data. Programmed rising and falling turbidity threshold limits will then trigger an ISCO 3700C automated sampler to collect samples for suspended sediment analysis. These samples will be sent to Oregon State University (OSU) for analysis. Existing data will be used to set initial minimum and threshold levels, which will be adjusted as more data become available. In addition, synoptic grab samples may be collected during storm events for comparison with autosampler results. Samples will be collected at the weirs used during the original study.

Figure 2 illustrates the CR10X data logger measurements to be applied for discharge, turbidity, temperature, and conductivity monitoring. Stage is a direct physical measurement that can be compared with a physical stage marker on the weir for validation. As shown in the figure, discharge is calculated from the relationship between stage and velocity over a cross sectional area of the weir. The rising and falling turbidity threshold trigger for collection of total suspended solids (TSS) samples is also illustrated.

Dissolved oxygen (DO) was measured throughout the year at the surface and in the intragravel environment during the original study. Permanent standpipe locations were established for the collection of the intragravel measurements, and surface water measurements were made near the standpipe stations throughout the study. Nine stations were established in Needle Branch, approximately nineteen in Deer Creek, and eleven in Flynn Creek. The Winkler method, the Alsterburg modification of the Winkler method, and a portable Hach dissolved oxygen kit were used to determine oxygen levels during the original study. DO was monitored from November to June throughout the original study, during the time Coho salmon eggs were in the gravel. Additional measurements were made in the summer prior to logging and during the critical years during harvest (Moring and Lantz 1975). Surface waters will be sampled for DO at selected key sites and times during this study and will attempt to overlap with sites used during the original study when reasonably possible. A HACH Hydrolab Quanta® multiparameter probe, or equivalent, calibrated as described in Standard Method (SM) 4500-O G (Standard Methods 1998) or by manufacturer's instruction will be used to conduct the measurements. Validation of probe measurements will be conducted using the Hach digital titration procedure 8215 described in the *Oregon Plan for Salmon and Watersheds, Water Quality Monitoring Guidebook* (OWEB 1999).

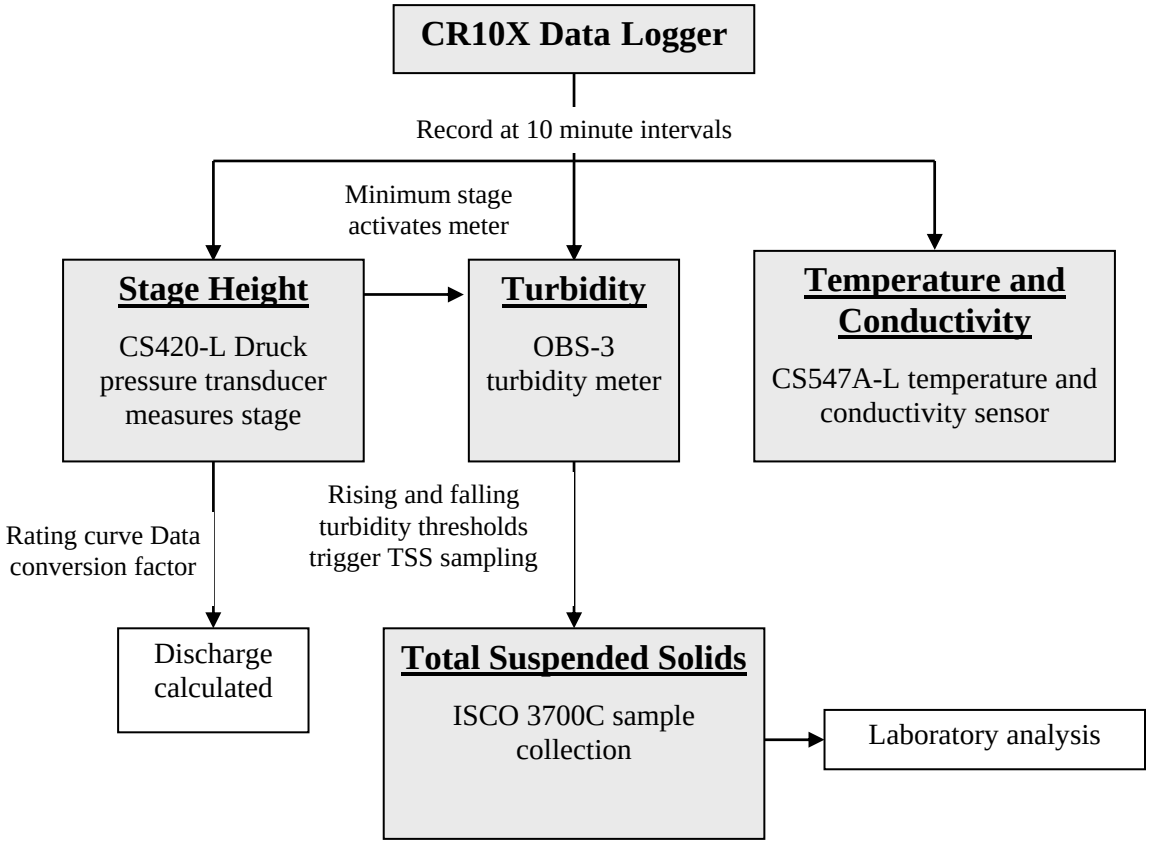


Figure 2. Data Logger Configuration and Monitoring Parameters

Table 3 shows the sampling locations, sample types, and equipment to be used to conduct field measurements. The sensors and electronic data logger (EDL) being used for monitoring are shown, and other sampling equipment that may be used for validation or to collect synoptic samples are included.

Table 3. Equipment Requirements for Water Quality Measurements Conducted in the Field

Parameter	Sampling Locations	Sample Type and Frequency	Equipment
Streamflow (cfs)	Flynn Creek, Deer Creek, Needle Branch gaging stations	Continuous recording at 10 minute intervals	Electronic data logger (EDL) CR10X with liquid level sensor CS420-L
Temperature (°C)	Flynn Creek, Deer Creek, Needle Branch gaging stations and synoptic sites	Continuous recording at 10 minute intervals	EDL CR10X with CS547A-L temperature/conductivity sensor at weirs Synoptic using HACH Hydrolab Quanta multiparameter probe or equivalent
Conductivity (mS/cm or µmhos/cm)	Flynn Creek, Deer Creek, Needle Branch gaging stations and synoptic sites	Continuous recording at 10 minute intervals	EDL CR10X with CS547A-L temperature/conductivity sensor Synoptic using HACH Hydrolab Quanta multiparameter probe or equivalent
Turbidity (NTU)	Flynn Creek, Deer Creek, Needle Branch gaging stations and synoptic sites	Continuous recording at 10 minute intervals	EDL CR10X with OBS-3-L turbidity senso Synoptic using HACH 2100P field turbidimeter
Dissolved oxygen (mg/L) and pH	Flynn Creek, Deer Creek, Needle Branch gaging stations and selected synoptic sites	Synoptic surveys conducted to include lowflow events	HACH Hydrolab Quanta multiparameter probe or equivalent, or superior polarographic or luminescence instrument Hach digital titration procedure 8215 DO validation

2.3 Field Sampling Procedures for the Laboratory Analysis of Water Quality Parameters

During the original Alsea Watershed Study nitrate-nitrogen, potassium, and dissolved phosphorus in stream water were monitored for two years prior to and following harvest for all three watersheds. Samples were collected once a month during the first year and twice a month thereafter. Storm events were sampled occasionally. Results were reported in mg/L, and yield data in kg per ha were derived from discharge measurements to compare yearly fluxes (Brown, Gahler, and Marston 1973). Nutrients associated with sediment loads were not included as part of the original study and are not expected to be included in this study. Total phosphate concentrations were unchanged during the original study in all three watersheds. Potassium concentrations were unchanged in the control (Flynn Creek) and patchcut (Deer Creek) streams. During logging activity the average potassium levels rose in Needle Branch during the first storm after burning, but returned immediately to pre-logging levels. Therefore, potassium will not be

monitored in this study. Significant increases were noted in nitrate-nitrogen yields at Needle Branch, but not at Flynn or Deer Creeks (Brown, Gahler, and Marston 1973). OWEB's watershed sampling guidance recommends monitoring for nitrate/nitrite, total Kjeldahl nitrogen, orthophosphate, and total phosphorus through a laboratory in order to obtain lower detection limits than those that can be obtained with field measurement equipment (OWEB 1999). In addition, ammonia as nitrogen will be measured. Grab samples for nutrient analyses will be collected monthly before, during, and after harvest and will be of sufficient volume to include QA/QC laboratory measurements. An additional grab sample should be collected quarterly at one of the sites to assess field precision.

Total suspended sediment analyses (TSS) were conducted during the original study. Sampling for TSS will be activated by the turbidity meter at each gaging station. Samples will be collected into 1 L bottles using an ISCO 3700 automated sampler. Where it is deemed appropriate during storm events, additional synoptic grab samples may be collected. Auxiliary samples will be collected quarterly for suspended sediment field replicates or to confirm the turbidity measurements collected by the data logger. These auxiliary samples should be collected at one of the sites each quarter, with each site being represented at least once per year during the study.

Neither biochemical oxygen demand (BOD) nor chemical oxygen demand (COD) measurements were conducted during the original study. Grab surface water samples will be collected monthly from each of the four gaging stations for COD measurements. Additional synoptic surface water samples for COD will be collected over a 24 hour period in June. Dissolved oxygen (DO) will be measured in the field monthly at each gaging station and synoptic sampling site.

Unfortunately, sampling procedures, sampling locations, and analytical methods for laboratory measurements were not discussed in the reports by Brown, Gahler, and Marston (1973) or Moring and Lantz (1975). EPA's *Wadeable Streams Assessment: Field Operations Manual* (USEPA 2004b) recommends collecting samples from a flowing section in the middle of the stream channel at each site. Samples will be collected midstream near the gaging stations on each stream when safely accessible. Sample collection activities will probably coincide with routine checks of the continuous monitors and, therefore, the time of day that samples are collected at each station may vary.

Sampling frequencies, sample types, sample containers, and preservation requirements for this study (Table 4) were derived from OWEB's watershed guidance (OWEB 1999) and Table 2 in *Guidelines for Test Procedures for the Analysis of Pollutants Under the Clean Water Act*; in 40 CFR, part 136 (USEPA 2004c). Where either glass or polyethylene containers may be used for sample collection, polyethylene containers are specified to avoid the potential for breakage in the field.

Table 4. Field Sampling Equipment, Containers, and Preservation for Water Quality Parameters

Parameter	Frequency	Type	Containers	Preservation and Shipping
Ammonia as nitrogen Nitrate/nitrite, total Kjeldahl nitrogen Total phosphorus	Monthly	Grab, collect field replicate quarterly	125 to 500 mL acid washed pre-cleaned polyethylene bottle[a]	Ice to <6.0ºC during transport and storage, deliver to the laboratory within 24 hrs; adjust to pH 2 with concentrated H_2SO_4 in the field or in the laboratory if samples are not transported and analyzed within 72 hours
Ortho-phosphates	Monthly	Grab, filter using a 45 µm filter disc and syringe, collect field replicate quarterly	20-50 mL acid washed pre-cleaned polyethylene bottle	Ice to <6.0ºC during transport and storage, deliver to laboratory within 24 hrs
COD	Quarterly	Grab	1 L pre-cleaned polyethylene bottle	12 drops H_2SO_4; ice to <6.0ºC during transport and storage, deliver to laboratory within 24 hrs
Suspended sediment	Determined by turbidity activated pump; quarterly auxiliary samples at one site	ISCO 3700 autosampler, synoptic grabs using a DH-48 depth integrated sampler, auxiliary samples quarterly	1 L pre-cleaned polyethylene bottle	Ice to <6.0ºC during transport and storage, deliver to laboratory within 24 hrs

[a] A single bottle can be used for ammonia, total phosphorus, nitrate-nitrite, and total nitrogen collection and preservation. If samples are to be preserved in the laboratory, an aliquot can be taken for O-phosphate from the other nutrient sample bottle prior to pH preservation.

2.4 Record Keeping and Reporting Requirements for Field Procedures and Measurements

Accurate accounts of activities will be recorded in a field notebook or on preprinted datasheets during each field sampling episode. Standard sample forms will be developed to help ensure that QAPP-identified information is routinely collected. Additional details regarding field records are included in the field sampling SOPs in Attachment A. Minimum general and parameter-specific information to be recorded during each field sampling event is shown here.

Record Keeping Requirements for Field sampling

Minimum reporting information

- Field site and sampling location (e.g., Needle Branch, weir, or distance marker)
- Date and time of sample site visit
- Sampler's name or initials
- Visual changes to the sampling site (e.g., downed trees, signs of tampering, animal activities that may impact results)
- Maintenance conducted (e.g., removal of leaves or branches from weirs, cleaning)
- Calibrations, instrument resets, instrument validation data (if applicable)
- Method or instrument data

Temperature, conductivity, and turbidity using EDL

Prior to visiting sites:

- Equipment identification number, location of deployment, and expected duration of deployment
- Dates, times, and results of pre-deployment accuracy tests

During site visit:

- Times, dates, and site identifications
- Equipment identification number
- Dates, times, and results of any field accuracy checks
- Dates and times data are downloaded to the computer and instrument is reset
- Stream characteristics or environmental parameters; photographs may be used to illustrate shade, width, debris, or vegetation characteristics that might impact results
- Any maintenance or observations that might affect sensor results. (e.g., algae or silt buildup or coating)

Following deployment:

- Dates, times, and results of post-deployment accuracy tests
- Maintenance activities

Dissolved oxygen

When field measurement is conducted:

- Times, dates, and locations of measurements
- Equipment and/or test method used
- Dates, times, and results of pre- and post-deployment accuracy tests if probe is used
- Dates, times, and results of field accuracy checks or calibration
- Measurement results
- Any maintenance or observations that might affect results

Nutrients and chemical oxygen demand (COD)

During each sampling event:

- Times, dates, and locations of sample collection
- Sample volumes, number of samples collected, and preservation
- Custody and laboratory delivery information
- Observations about the sampling event that may impact results

Total Suspended Sediment (TSS)

When samples are retrieved from the autosampler:

- Times, dates, and locations
- Sample volumes and numbers of samples collected

When synoptic grab samples are collected:

- Times, dates, and locations of sample collection
- Sample volumes and numbers of samples collected

Additional General Information

- Custody and laboratory delivery information
- Observations about the sampling event that may impact results

2.5 Sample Handling and Custody

Samples to be transported to OSU or NCASI laboratories will be stored on ice in a cooler and hand delivered to the laboratories within the holding times shown in Table 4. Receiving laboratories must maintain the required storage temperatures, conduct analyses within specified holding times, and have a system in place to track and document sample receipt, sample processing, analytical processes, and reporting.

2.6 Analytical Methods

Contemporary analytical methods will be used for analyses unless a specific method is required for comparison with original Alsea Watershed Study data. Methods approved by EPA at 40 CFR, part 136.3 for the Clean Water Act (USEPA 2004c) will be applied whenever possible. Analysts will be required to follow designated methods or established SOPs. Any deviations must be documented and approved prior to analysis.

Samples collected for suspended sediment will be analyzed at OSU according to the SOP in Attachment B. The SOP was adapted from *Standard Method for the Examination of Water and Wastewater* Method 2540D (Standard Methods 1998) and the USDA Forest Service Redwood Science Laboratory's *Laboratory Procedures for Determining Suspended Sediment Concentration* (USDA 2002).

Ammonia as nitrogen, nitrate-nitrite, total Kjeldahl nitrogen, total Kjeldahl phosphorus or total persulfate phosphorus, and orthophosphate analyses will be conducted at NCASI using an ALPKEM 3000 flow injection analyzer (ALPKEM 1996). NCASI's SOPs were adapted from ALPKEM procedures that were either directly approved or are equivalent to EPA 300 series methods approved by EPA under 40 CFR, part 136 (USEPA 2004c). A list of NCASI's nutrient SOPs and an example are included in Attachment C. COD measurements will be conducted according to HACH Method 8000, which is an approved equivalent to EPA Method 410.4 in 40 CFR, part 136.

Turbidity measurements to confirm the results of the turbidity meter will be conducted on auxiliary samples collected from one of the stream locations each quarter. Each stream location should be represented at least once per year. Turbidity is measured as nephelometer turbidity units (NTU) using SM 2130B (Standard Methods 1998).

Table 5 lists the analytical methods to be employed for each parameter during the course of this study. The calibration ranges or minimum levels, reporting units, and holding times are also shown. Laboratories must be able to conduct analyses within the method-specified calibration range or be able to achieve the minimum levels specified. Laboratories are required to conduct analyses within the specified holding times and report data in the reporting units noted in the table.

Table 5. Analytical Method Requirements

Parameter	Applicable or Equivalent Approved Method	Calibration Range or Minimum Reporting Level (ML)	Reporting Units	Holding Time
Suspended sediment	SM 2540D	0.1 mg/L (ML)	mg/L	7 days
Ammonia	EPA 350.1	0.02 – 2.0 mg/L	mg/L	28 days
Nitrate-nitrite	EPA 351.2	0.01 – 5.0 mg/L	mg/L	28 days
Total Kjeldahl nitrogen	O-I/ALPKEM PAI-DK03	0.05 – 2.0 mg/L	mg/L	28 days
Total Kjeldahl phosphorous	EPA 365.1	0.05 – 2.0 mg/L	mg/L	28 days
Total Persulfate phosphorous	EPA 365.1	0.005 – 2.0 mg/L	mg/L	28 days
Orthophosphates	EPA 365.1	0.01 – 2.0 mg/L	mg/L	48 hrs
COD	HACH 8000 (EPA 410.4)	3 – 150 mg O_2/L	mg /L	28 days
Turbidity	SM 2130 B	1 NTU (ML)	NTU	48 hrs

2.7 Quality Control

This section describes the quality control elements required to assess contamination, precision, and accuracy of water quality data. Each measurement must adhere to the QA/QC requirements specified in the analytical methods or equipment manufacturer's guides. Project-specific requirements or requirements specified by collaborating regulatory agencies may supercede these criteria. Typical quality assurance requirements include field blanks, field duplicates, field validation, calibration checks, laboratory blanks, replicates, and quality control spikes. Data that fail to meet the QA/QC requirements specified in this section must be flagged using the laboratory's standard reporting conventions.

2.7.1 *Field measurements and QA/QC*

Table 6 lists the field parameters, equipment, method performance targets, and corrective action items specified for field measurements. The purpose of these QA/QC measures is to ensure proper functioning of field data loggers and other field measurement parameters and to validate measurements. Data loggers will be deployed year-round except when removed annually for routine maintenance or when significant problems are noted. Equipment will be checked weekly during the rainy season and after storm events and biweekly during the dry season using the maintenance checks described in Section 2.8. Data will be reviewed graphically when downloaded to look for areas of concern that may indicate that equipment is not functioning properly.

Table 6. QA/QC for Field Sampling Parameters

Parameter	Equipment and Method Performance Targets	Corrective Actions
Discharge	CR10X, CS420-L sensor; validate stage measurement of recorder against reference stage at weir marking using a Leopold-Stephens Model A-35 stage recorder; review data in the field to look for anomalies between flow and turbidity that may indicate obstructions or interferences	Conduct routine maintenance; adjust sampling frequencies or adjust for stage-discharge shift as appropriate
Temperature/ conductivity	CS547A temperature probe capable of measuring 0 to 50°C with accuracy of 0.2°C and factory tested using a NIST traceable standard with ±0.25% accuracy; pre-deployment tests not required Data will be checked against HACH Hydrolab *in situ* measurements when equipment is serviced. Field checks every three months during deployment and post-deployment checks using audit thermometers with accuracy of ±0.5°C and resolution of ±0.2°C following procedures described in OWEB's guidebook; conductivity measurements checked quarterly using KCL standard	Conduct maintenance if field accuracy tests indicate equipment not operating properly; inform supervisor if maintenance cannot be conducted in the field
Turbidity	CR10X/OBS-3-L to measure turbidity and trigger collection of threshold suspended sediment samples using ISCO 3700; minimum level to trigger turbidity data collection and threshold values to trigger suspended sediment sampling will be evaluated throughout study Depth integrated (DI) samples (cross-sectional average sediment concentrations) used to correct pumped samples; simultaneous pumped sample collected via data logger program while manual sample is collected in a 1 L sample bottle; samples should be collected every 20 samples or quarterly, whichever is greater, during months equipment is activated	Adjust threshold values as necessary based on sample data; auxiliary suspended sediment sample triggered manually via data logger program should be collected when too few samples have been collected during a storm or when equipment malfunctions; auxiliary samples should be clearly identified on sample label
Dissolved oxygen and pH	YSI DO probe or HACH Hydrolab Quanta probe to conduct monthly measurements; probe will be air calibrated according to SM 4500-O G; confirm DO measurements using Hach Method 8215 every 20 samples or quarterly, whichever is greater	Clean probes, conduct maintenance when Hach method indicates measurements are no longer accurate

2.7.2 *Laboratory QA/QC*

Laboratories are required to conduct the minimum QA/QC specified in the analytical method or the manufacturer's instructions. These specifications may be superceded by project-specific criteria or as stipulated by regulatory collaborators. All laboratory entries are to be recorded in ink. Corrections to data entries are to be initialed and dated by the analyst or auditor. The laboratory's QA/QC plan must address precision, accuracy, and potential sources of field and laboratory contamination. The various steps commonly used to address these issues are described here.

QA/QC Definitions and Terminology

- **Field blanks** are intended to assess the potential for contamination from field equipment or sampling containers. Distilled water is generally used to represent a sample in the field. Collection procedures for field blanks should be addressed in the Field Sampling SOPs in Attachment A.
- **Field duplicates** are samples collected simultaneously in the field from the same location and treated in the laboratory as independent samples. When simultaneous collection is not possible, back to back samples may be employed.
- **Method blanks** are typically distilled water samples taken through the entire analytical process to test for contamination of laboratory glassware, reagents, or instrumentation.
- **Laboratory duplicate** sample analyses are conducted to assess method precision in a sample matrix. Matrix spike and matrix spike duplicates may also be conducted to assess precision and should be employed to assess precision when the sample matrix is not expected to have detectable levels of the target analyte(s).
- **Matrix spikes** are spikes of a standard of known concentration into a sample matrix to determine the procedure's ability to recover the target analyte(s). The background concentration level in the sample matrix is subtracted from the spiked concentration to determine percent recovery of the analyte. Matrix spike levels should be 1 to 3 times the background level in the sample.
- **Calibration checks** are typically daily analyses of a single point on the calibration curve to ensure that the instrument is functioning properly. These may also be called **instrument checks**.
- **Validation or confirmation checks** are spikes of a standard of known concentration from an external source into reagent water. The spiked water is then taken through the entire analytical process to assess laboratory bias.
- **Laboratory control charts** assess the long-term performance of the method; show trends that may indicate deterioration of reagents, standards, or instrument performance; and are indicators of the need for maintenance or preparation of new reagents or standards. They are also used to identify outliers.

System QA/QC such as method blanks and calibration checks are required on each day on which analyses are conducted and must meet the quality control specifications of the method before sample analyses proceed. Sample QA/QC analyses are typically conducted with each analytical batch of samples. An analytical batch is considered by many methods to be a batch of 20 or fewer samples. More frequent QA/QC may be specified in the analytical method, SOPs, or as specified by the CLI. When fewer than 20 samples are analyzed in a given month, QA/QC should be conducted at least quarterly. For instance, if nutrient samples are only collected at the weirs, monthly nutrient sampling will generate three samples. Therefore, duplicates and matrix spike analyses should be conducted at least quarterly. The laboratory should attempt to conduct QA/QC representing each site at least once annually.

The minimum QA/QC parameters for each method and the frequency of analyses are presented in Table 7. In addition, laboratories should maintain control charts of the analytical data. Additional QA/QC information is included in the SOPs in Attachments B and C.

Table 7. QA/QC for Laboratory Analyses

Parameter	QA/QC Parameter	Frequency	Criteria	Corrective Actions
Nutrients	Method blanks	Per batch of 1 in 20 samples	< minimum calibration level	Identify contamination source (e.g., reagents, glassware, instrument carryover); if field blank shows contamination and method blank does not, field activities should be evaluated to identify contamination source; prepare and analyze a new blank; if sample analyses were conducted in the same batch, evaluate sample results against method blank concentration; flag sample concentrations <5 times method blank that cannot be reanalyzed
	Calibration checks	Daily	Recovery 80-120%, relative standard deviation (RSD) of continuing calibration <25%	Conduct instrument maintenance if recovery is outside acceptance range; recalibrate instrument if necessary; samples should not be analyzed until instrument is working properly; flag data associated with analyses conducted when acceptance criteria not met
	Field duplicates	Quarterly	Relative percent difference (RPD) <35%	Check field precision against method precision to determine if field activities impact precision; flag data outside acceptance range
	Matrix spikes MS/MSD	Per batch of 1 in 20 samples	Recovery 80-120%, RPD MS duplicates <25%	Repeat analyses if RPD of results are outside acceptance criteria and sufficient sample is available; note observations that may have caused discrepancies (e.g., algae, film, or foreign objects in samples); flag data outside acceptance range if repeat analyses do not rectify problem; if results fail percent recovery criteria, compare recoveries with a spike in reagent water to determine if there is a matrix effect; flag data outside acceptance range
	Validation checks	Quarterly	Recovery 80-120%	If data fail recovery criteria, determine source of laboratory bias
Suspended sediment	Auxiliary duplicates	Quarterly	±35% of ISCO sampler result	Report duplicate measurements and flag data outside range; report observed differences in samples that may account for differences in results
Turbidity	Validation of turbidity meter	Quarterly	±35% of data logger result	Report duplicate measurements and flag data outside range; evaluate data logger for necessary maintenance or drift
COD	Standard check	Annually	Recovery 80-120%	Evaluate technique and quality of reagents and flag data if they fail to meet criteria

The laboratory must conduct an internal audit of the data prior to reporting. Samples that fail to meet the acceptance criteria in Table 7 should be reanalyzed if analyses can be conducted within holding times. If holding times are past, contact the CLI for further direction. Data that fail to meet QA/QC requirements must be flagged in laboratory reports. The laboratory should review

data for completeness, holding time requirements, proper analytical sequence, initial calibration, calibration verification, blank contamination, precision, and recovery. Analytical data should be reported in a data package to the CLI responsible for ensuring that the data are entered into electronic media. The CLI may request that laboratory data be delivered in electronic format to expedite data transfer.

2.7.3 *Laboratory data package*

After completing an internal review of the data, the laboratory must compile a data package. A copy of the data package and any electronic reports will be submitted to the CLI. The minimum data package must include a summary report of the sample results; results of all QA/QC analyses including calibration checks, method blanks, and duplicate and matrix spike results; and copies of laboratory data entry sheets or notebook pages and instrument run logs. The CLI may also request instrument printouts or raw data to conduct a more thorough audit. Figure 3 shows three levels of data packages that may be requested by the CLI. The minimum data package should include both basic and QA/QC-level reports. The CLI is responsible for ensuring that data audits are conducted and any problems are reported to the laboratory and resolved. Auditing considerations and corrective actions are presented in Section 4.0.

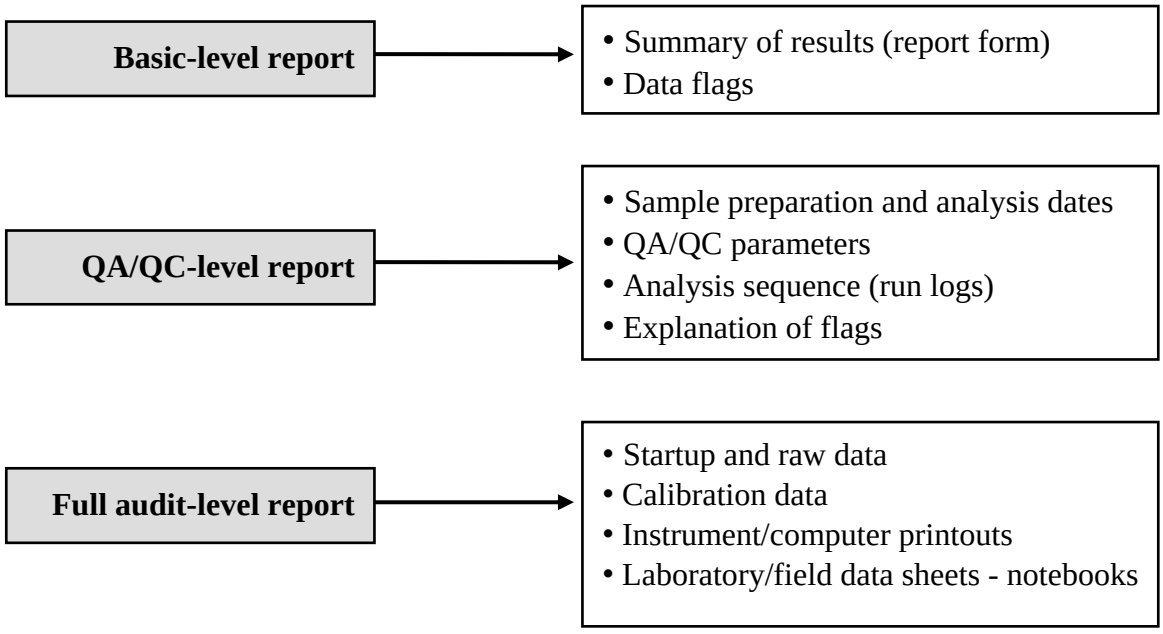


Figure 3. Data Package Requirements

2.8 Instrument Testing, Inspection, and Maintenance

Prior to going into the field, test equipment function. Replace batteries if necessary, or bring backups. Follow manufacturer's instructions and schedules for maintenance of equipment. Log maintenance and repair activities. Periodically check for coatings of algae, silt, or debris that may interfere with proper instrument function. Maintenance check information provided by the EDL

manufacturer and from the Redwood Science Laboratory (USDA 2003) were used to develop this field measurement and field sampling maintenance check list.

Maintenance Check List

- Clean flumes or weirs, remove large debris and obstructions
- Clean and service EDL probes or sensors as described in manufacturer's instructions
- Check battery condition and replace batteries approximately every three months or as needed
- Check for loose connections, damaged cables, or damaged transducers, replace or secure as needed
- Periodically check the desiccant used to keep the vent tube dry; replace every three months or as needed
- Check validity of the calibration and adjust offsets to correct for drift if necessary
- Adjust, troubleshoot, and replace equipment as needed
- Replace pump tubing in the ISCO sampler when the tubing life counter has reached the 500,000 count warning limit, check rollers for silicone buildup, and verify that rubber liners are snug
- Check the position of the ISCO intake mounted in the flume to ensure that it is properly submerged
- Send transducers to the manufacturer for inspection and service at the manufacturer's recommended time intervals, typically every 2 to 3 years

2.9 Instrument/Equipment Calibration and Frequency

Equipment is to be calibrated prior to analysis and anytime quality control measures indicate that the system is out of control. Recalibrate instrumentation when major equipment maintenance is conducted. Follow method requirements for calibration range, number of calibration points, and frequency of calibration. Calibration checks should be conducted at the frequency indicated in Section 2.7 for field and laboratory parameters.

2.10 Inspection/Acceptance of Supplies and Consumables

Material Safety Data Sheets (MSDS) must be obtained for all hazardous chemicals. Record receipt date or preparation date on reagent and standard containers. Inspect all consumable reagents and supplies for expiration dates and prepare new reagents as needed prior to conducting laboratory or field work. Prepare new reagents at least annually if expiration information is not available. Analysts must maintain records of chemical receipts and documentation of chemical purity or lot numbers, if provided. All reagents and standards prepared for the study should be traceable to the original supplier. Equipment consumables should be inspected for breakage, cracks, frays, or other signs of damage in shipping.

2.11 Non-Direct Measurements

This element addresses data obtained from existing data sources and not directly measured or generated. Potential non-direct measurements may include:

- Existing sampling and analytical data from the original Alsea Watershed Project may be compiled for comparison with new data. The reporting units, number of data points available for comparison, identification of sampling locations, sample collection dates, sampling times, and quality of supporting information should be considered when compiling these data.

- Photographs or topographic maps from both the original study and the new study should be obtained for documentation and to illustrate alterations to the site. Photographs of shelters, equipment placement, and sampling locations should be obtained to document study design.

- Meteorological data such as air temperature or precipitation from nearby stations may be used directly to determine local conditions or as a check of measurements conducted during the study.

  Note: Precipitation data were collected using weighing Belfort rain gages just downstream from each of the outflow stream gaging stations and were reduced in accordance with Belfort instructions during the original study. Because the gages were placed at the lowest altitude in each basin, the records were considered unlikely to represent the average precipitation on the basins. Although the records probably indicated less than the basin average and were partly estimated, they were considered adequate to indicate approximate areal distribution and year to year variation of precipitation (USGS 1971). The rain gages measured cumulative rainfall over weekly periods. Rain gage locations used for this study are described in Attachment A.

2.12 Data Management

Field data should be recorded on standardized field data forms and transferred to electronic data files. Example data forms are included in the field sampling SOPs in Attachment A. Data collected using EDLs will be downloaded to the computer. Backup copies of all electronic data will be maintained. Once the electronic download is validated, data will be transferred to the Alsea Watershed Information Management System (AWIMS).

Tracking records documenting sample collection and delivery to contract laboratories must be maintained using either standardized forms or common carrier shipping forms. Copies of these documents will be retained in both laboratory and field files.

Analytical processes will be recorded in laboratory notebooks and instrument logs. A complete data package will be prepared and filed for each analytical batch of samples (Section 2.7) in the laboratory. Upon request, a copy of the data package will be forwarded to the CLI, who is responsible for ensuring that the data are entered into AWIMS and audited. The laboratory may be asked to provide data in electronic form for transfer into AWIMS. From these records an external auditor should be able to determine holding times, who conducted the analyses, what method was employed, any deviations from the method, sample volumes processed, and sample results, in order to evaluate compliance with QA/QC measures. Refer to Section 4.0 for information on conducting data audits.

Data are to be retained by all parties for five years following completion of the study report. Figure 4 illustrates the flow of data.

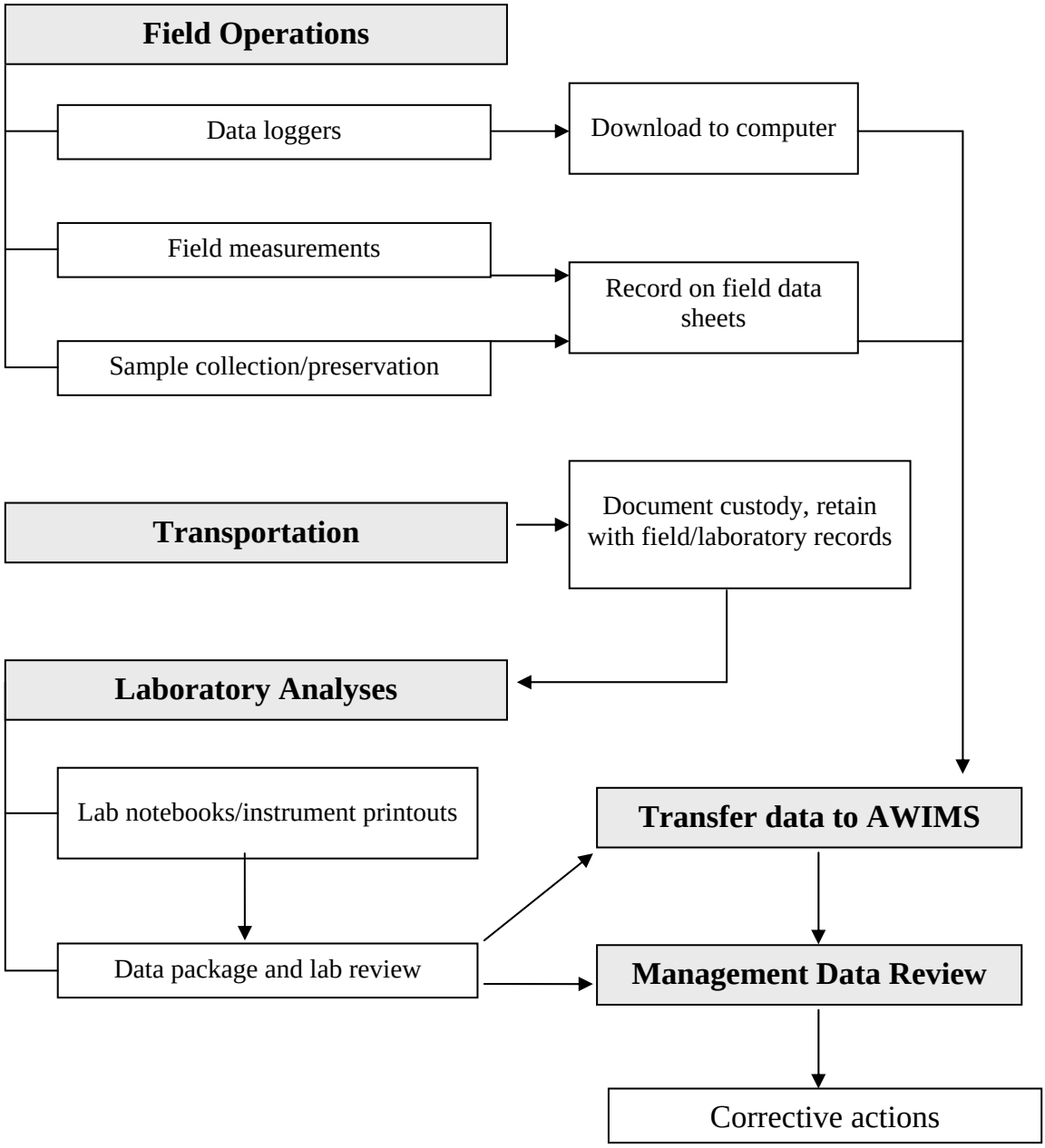


Figure 4. Data Management Flow Chart

3.0 ASSESSMENT AND OVERSIGHT

3.1 Assessments and Response Actions

Activities used to implement the plan will be reviewed by the CLI with oversight responsibility for the parameter group. The CLI is responsible for ensuring that the appropriate equipment is available and maintained, and that sampling and analyses are being conducted at the required

frequency, locations, and conditions specified in the QAPP. When data review indicates the need for corrective action, it is the CLI's responsibility to ensure that the corrective actions are conveyed and followed.

3.2 Reports to Management

Field and laboratory data will be reported at least quarterly or at a frequency specified by the CLI cited in Table 1 of Section 1.0. Summaries of the research findings will be prepared annually for review by the cooperating organizations, and recommendations for study changes will be reviewed.

4.0 DATA VALIDATION AND USABILITY

4.1 Data Review, Verification, and Validation

Field and analytical data will be reviewed by the CLI with oversight responsibility for the parameter group. Although field data and laboratory data are somewhat different, many of the same elements of data review apply. The data review process shown in Figure 5 illustrates the various stages of data review based on the level of data package undergoing review.

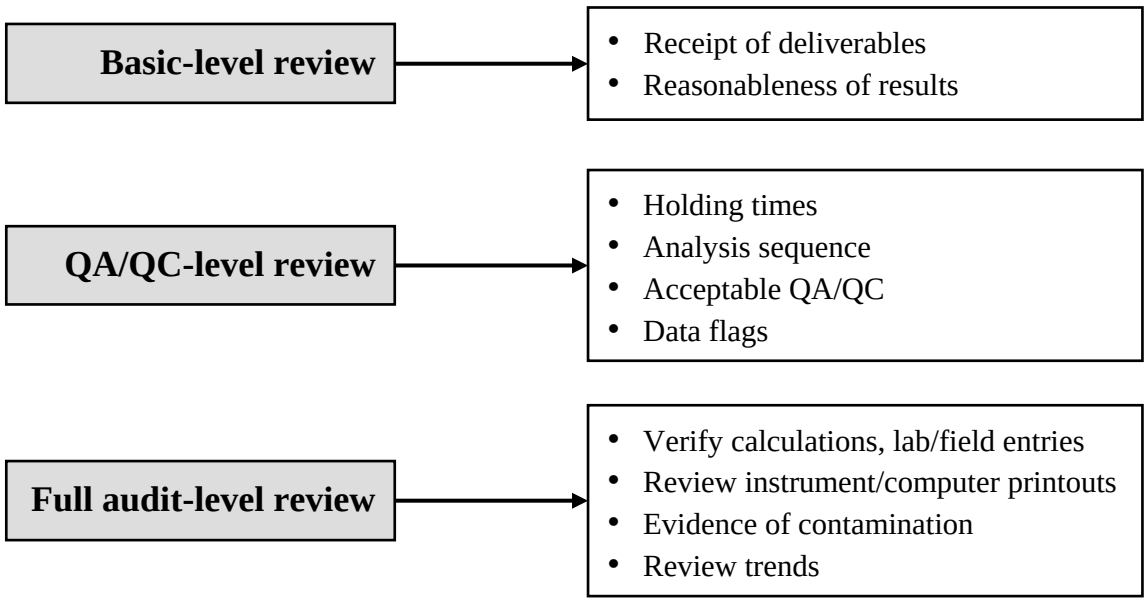


Figure 5. Data Audit Review Process

As illustrated in Figure 4, corrective actions may be necessary if the data review identifies a QA/QC problem. Data review activities and corresponding corrective actions are presented in Table 8. These activities are based on the review process illustrated in Figure 5 and are consistent with those in EPA's *Wadeable Streams Assessment: Integrated Quality Assurance Project Plan* (USEPA 2004a).

Table 8. Quality Control Activities for Review, Verification, and Validation of Results

Data Review Activity	Corrective Action
Review data deliverables	
Were all requested deliverables received?	Request additional information if data package is not complete
Are the results within expected values?	Determine if calculation errors or reporting units explain unusual results; correct data entry if applicable
Review QA/QC Parameters	
Were samples analyzed within holding time?	Resample if possible; require laboratory to meet holding times for future measurements
Were analyses conducted using the analytical sequence specified in the method, if applicable, and is there evidence that samples from other sources that may be a potential source of contamination were analyzed in the batch?	If analyses were not conducted as prescribed by method requirements, direct laboratory to take corrective action; address potential sources of contamination if applicable
Were the results of blank analyses either non-detect or within method-specified values?	If method blank fails criteria, look for potential sources of contamination and take corrective action to eliminate; reject or flag data that are not >5 times blank level
Did duplicates pass the acceptance criteria?	If duplicate results fail acceptance criteria, determine if analytical error or field sampling error may be responsible; take corrective action to improve precision if appropriate
Did matrix spikes or other QC spikes pass the recovery criteria?	If spike data fail acceptance criteria, determine impact to results; attempt to determine if matrix effects are present; if calibration check recovery fails, all data analyzed on that day should be reanalyzed or rejected; reject or flag sample data with recoveries outside acceptance range; typically, recoveries more than 10% outside acceptance range should be rejected
Are there data qualifiers?	Determine if concentration value is suspect or invalid based on qualifiers; determine if suspect results impact outcome of data; request repeat analyses if applicable; qualify data in AWIMS system if applicable
Full Audit Review (if QA/QC failure is noted)	
Were dilution factors and other calculations conducted correctly?	If incorrect calculations are noted, instruct laboratory to correct and reissue report; correct all data in AWIMS
Do notebooks, data sheets, or instrument raw data indicate analytical problems?	If analytical problems are noted during raw data review, contact laboratory or field crew to discuss corrective actions
Prepare control charts. Do control charts indicate trends or outliers?	If unexpected trends become evident, discuss potential corrective actions with laboratory or field crew; evaluate factors that may explain outlier data
Other QA/QC Activities	
Are results consistent with expected research goals?	If deviations from expected research goals can be explained by field or laboratory procedures, take appropriate corrective actions
Does exploratory data analysis utilizing all data indicate potential problems?	Reassess study design or objectives if data analysis reveals problems with data interpretation
Confirm assumptions regarding specific types of statistical techniques being utilized in development of metrics and indicators.	Statistical techniques should be consistent with data being generated; should other techniques be employed?

4.2 Reconciliation and User Requirements

When data fail to meet objectives specified in the QA/QC section of this document, the CLI or data user must determine the impact. Failure to meet a QA/QC objective does not necessarily invalidate data usage, but may limit the extent to which conclusions may be drawn from the data. Therefore, careful consideration must be given to the data quality objectives and end data uses.

5.0 FIELD SAFETY CONSIDERATIONS

It is the responsibility of field sampling personnel and their supervisors to be familiar with potential hazards *prior* to going into the field to conduct watershed sampling and monitoring. Safety considerations in this section are designed to minimize the potential hazards of fieldwork. No sample is worth endangering yourself or your co-workers. Special safety hazards to consider prior to field work include windthrow, high flows, and slippery conditions along paths or banks and at sampling sites. All safety incidents, accidents, and observed hazards should be reported to the CLI to determine if corrective actions are required. When conducting field sampling in wadeable streams use the guidelines herein.

5.1 General Field Safety Guidelines

Employees should survey their working environment at each location. When working outdoors inspect each site for situations that may put an employee at risk, such as a slippery riverbank, a high water area, rotting footholds, or encounters with individuals who cause the employee to feel intimated or harassed. If an employee feels unsafe, he or she is advised to leave the area immediately. Scheduled work can be completed at a later time or omitted if the employee does not feel comfortable returning to the area. CLIs should make sure employees are familiar with these aspects of their surroundings.

Employees should remain alert at all times to the activities around them, and watch for moving objects, vehicles, and machinery. Employees should be aware of and avoid possible hazards such as leaks, spills, and drips of hazardous substances. Employees should notify landowners or CLIs of any unsafe conditions, such as faulty equipment, fires, or chemical spills.

Employees must wear or carry the required personal protective equipment identified in the SOPs in Attachment A at all times while working in the field. Optional safety equipment is available, and should be used when the situation warrants.

Safe travel to field locations should not be neglected. Field staff should check in with expected departure and arrival times. Prior to departure, notice should be given of the expected time of return. Field personnel are required to abide by Oregon motor vehicle laws.

5.2 Personal Protective Equipment

All workers will be provided with personal protective equipment for use in the field, and will be trained in the use of such equipment. All workers are required to use the personal protective equipment the field site specifies as mandatory. Additional required safety equipment may be noted in the field SOPs in Attachment A. More stringent requirements specified by property owners supersede the requirements of this plan.

The personal protective equipment listed here is commonly required for employees engaged in field activities and is based on field safety procedures described in NCASI's *Chemical Hygiene and Safety Plan* (NCASI 2005) and ODEQ's *Watershed Assessment Section Mode of Operations Manual* (ODEQ 2004).

- Personal flotation devices (PFD) are not required, as the stream depths are typically shallow even during winter flows. ODEQ (2004) warns field personnel: "Use caution when wading in streams with swift current. As you get deeper your ability to keep a grip on slick substrate will be reduced and you may be pushed off your feet by slower

velocities. Even shallow water at high velocities can be dangerous. Do not attempt to wade in a stream for which values of depth multiplied by velocity equal or exceed 10 ft^2/sec."

- Wear appropriate footwear and clothing. Shoes with sufficient tread are required when working in and alongside any body of water. Check carefully for unstable substrate or unexpected holes. If it is necessary to wear hip boots or waders, ensure that the equipment is not too tight and can be easily removed in an emergency situation.
- Safety glasses and protective gloves should be carried and used as needed to protect employees from preservatives and stains. Safety glasses and gloves may also be used to protect the eye and hands from scratches caused by the movement of brush or tree limbs.
- A first aid kit should be available to treat cuts, scratches, burns, stings, strains, or other emergencies.
- Steel toed boots and hardhats may be required when working in logging areas.
- Orange high visibility vests should be worn in the field, particularly during hunting season, and are required in logging areas.

Additional personal protective equipment may be needed under certain circumstances. The work area and the nature of the work should be considered in planning any field activity, and personal protective equipment needs should be assessed in light of this information. Optional safety equipment to consider including in the field kits includes:

Optional Safety Equipment Checklist

- Walking stick
- Map to the nearest hospital
- Personal medications (e.g., bee sting kits, allergy medications)
- Sunscreen
- Hardhats
- Steel toed boots

This is not intended to be an exhaustive list of safety equipment, but a reminder of equipment that is often necessary. Any additional equipment that is necessary to ensure worker safety will be made available as needed, and appropriate training will be provided.

5.3 Safety Training and Information Sources

A field safety training program will consist of a general safety orientation prior to the first trip into the field. Training will include the proper use of safety equipment employees are required to use. A field safety check list may be used to act as an ongoing reminder of safety priorities by:

- Providing a routine reminder of field safety considerations
- Making safety awareness a required part of the preparation for each fieldwork assignment
- Providing opportunities for reporting unforeseen safety issues that may have arisen during field sampling activities and making recommendations to prevent future incidents or acquire safety equipment

Individual field locations may require additional safety gear or training. Attention to and prior preparation for these requirements is important. Observe general safety rules at all times while in the field.

5.4 Material Safety Data Sheets and Hazardous Materials Handling

Material Safety Data Sheets (MSDS) must be available to all personnel working with hazardous chemicals. CLIs are responsible for ensuring that safety equipment is available and appropriate training is conducted for all field and laboratory personnel, unless equipment and training are already provided by the contracting laboratory or contributing organization. Personnel are responsible for wearing appropriate safety equipment and following all safety rules. Safety accidents and incidents must be promptly reported to the CLI. Disposal of hazardous materials must comply with all state and federal hazardous waste regulations.

REFERENCES

ALPKEM. 1996. *Flow Solution™ 3000: Advanced technology in flow analysis operations manual*. Document No. A001702, Rev. B. Wilsonville, OR: ALPKEM.

Brown, G.W., Gahler, A.R., and Marston, R.B. 1973. Nutrient losses after clear-cut logging and slash burning in the Oregon Coast Range. *Water Resources Research* 9(5).

Moring, J.R., and Lantz, R. 1975. *The Alsea Watershed Study: Effects of logging on the aquatic resources of three headwater streams of the Alsea River Oregon*, parts 1, 2, and 3. Fishery Research Report No. 9. Project AFS-58. Corvallis, OR: Oregon Department of Fish and Wildlife.

National Council for Air and Stream Improvement, Inc. (NCASI). 1991. *The New Alsea Watershed Study*. Technical Bulletin No. 602. Research Triangle Park, NC: National Council for Air and Stream Improvement, Inc.

———. 2005. *Chemical hygiene and safety plan*. Internal document. Corvallis, OR: National Council for Air and Stream Improvement, Inc. West Coast Regional Center.

Oregon Department of Environmental Quality (ODEQ). 2004. *Watershed Assessment Section Mode of operations manual (MOMs)*. 03-LAB-0036-SOP, ver. 3.1. Portland, OR: Oregon Department of Environmental Quality, Laboratory Division.

Oregon Watershed Enhancement Board (OWEB). 1999. *Oregon plan for salmon and watersheds: Water quality monitoring technical guidebook*, ver. 2.0. Oregon Watershed Enhancement Board.

Standard Methods. 1998. *Standard methods for the examination of water and wastewater*, 20th ed. Washington, DC: American Public Health Association.

Stednick, J.D., and Hall, J.D. in prep. The Alsea Watershed: Hydrological and biological responses to temperate coniferous forest practices. *Ecological Studies*. New York: Springer Verlag.

United States Department of Agriculture (USDA). 2002. *Laboratory procedures for determining suspended sediment concentration*. Rev. 11/25/2002. USDA Forest Service, Redwood Sciences Laboratory. http://www.fs.fed.us/psw/topics/water/tts/index.shtml#tech.

———. 2003. *Turbidity threshold sampling field manual*. Rev. 12/04/2003. USDA Forest Service, Redwood Sciences Laboratory. http://www.fs.fed.us/psw/topics/water/tts/index.shtml#tech.

United States Environmental Protection Agency (USEPA). 2001. *EPA requirements for quality assurance project plans, EPA QA/R-5*. EPA/240/B-01/003. Washington, DC: United States Environmental Protection Agency, Office of Environmental Information.

———. 2002. *Guidance for quality assurance plans, EPA QA/G-5*. EPA/240/R-02/009. Washington, DC: United States Environmental Protection Agency, Office of Environmental Information.

———. 2004a. *Wadeable streams assessment: Integrated quality assurance project plan* (draft). EPA/841/B-04/005. Washington, DC: United States Environmental Protection Agency, Office of Water and Office of Research and Development.

———. 2004b. *Wadeable streams assessment: Field operations manual*. EPA/841/B-04/004. Washington, DC: United States Environmental Protection Agency, Office of Water and Office of Research and Development.

———. 2004c. Guidelines establishing test procedures for the analysis of pollutants. *Federal Register*, 40 CFR, part 136. July 1, 2004. Washington, DC: United States Environmental Protection Agency.

United States Geological Survey (USGS). 1971. *Streamflow, sediment-transport, and water-temperature characteristics of three small watersheds in the Alsea River Basin, Oregon*. Harris, D.D. and Williams, R.C. Geological Survey Circular 642. Washington, DC: United States Geological Survey.

———. 1982. *Measurement and computation of streamflow*, vol. 2, Compuation of discharge. Water Supply Paper 2175. Washington, DC: United States Geological Survey.

ATTACHMENT A

STANDARD OPERATING PROCEDURES FOR SERVICING GAGING STATIONS AND RAIN GAGES AND WATER QUALITY SAMPLING: THE ALSEA WATERSHED STUDY REVISITED

Received via personal communication (email) from Cody Hale, graduate student, Oregon State University, Forest Engineering, May 2006

Standard Operating Procedures for Servicing Gaging Stations and Rain Gages and Water Quality Sampling for the Alsea Watershed Study Revisited

prepared by Cody Hale

May 2006

Introduction

These standard operating procedures (SOPs) have been developed to provide guidance to field personnel responsible for servicing and maintaining the gaging stations and rain gages associated with the Alsea Watershed Study Revisited (AWSR). This document is intended to be used in conjunction with the National Council for Air and Stream Improvement Quality Assurance Project Plan (hereinafter referred to as NCASI and QAPP).

There are currently three gaging sites that were initially installed by the United States Geologic Survey as part of the original Alsea Watershed Study, one each at Flynn Creek, Needle Branch, and Deer Creek. A fourth gaging site will be added in the Needle Branch catchment during the summer of 2006. This document will be appended accordingly upon its installation. Water quality and quantity data are collected at each station. Specifically, stage (the depth of water at the gage), turbidity, temperature, and conductivity are measured *in situ*. Samples to be analyzed for suspended sediment concentration are collected using an auto-sampler. Grab samples are collected for nutrient analysis.

Data are obtained from Leopold-Stevens, Model A-35 stage recorders (Leopold-Stevens Company, Beaverton, OR) and from instrumentation associated with the turbidity threshold sampling (TTS) protocol developed by the USDA Forest Service's Redwood Sciences Laboratory. TTS involves the use of turbidity thresholds in conjunction with stage data to trigger collection of suspended sediment samples via an automated sampler. The TTS equipment includes a Campbell Scientific CR-10X datalogger (Campbell Scientific, Inc., Logan, UT), a D&A Instruments OBS-3 turbidity probe (D&A Instrument Co., Port Townsend, WA), a Druck pressure transducer (Druck Inc., New Fairfield, CT), a temperature and conductivity probe, and an ISCO 3700 Automated Sampler (Teledyne ISCO, Inc., Lincoln, NE). Five tipping bucket rain gages outfitted with HOBO event loggers (Onset Computer Corp., Bourne, MA) are located in the study vicinity.

Field Equipment

Necessary equipment will vary depending on the tasks to be completed in the field. A checklist for typical equipment required when servicing the gaging stations and rain gages is contained herein. Before leaving for the field, stamp the field book with rubber stamp containing necessary headings for data to be collected and print out field forms for gage houses. In addition, make sure a contact person knows your expected destinations and return time.

Directions

The AWSR research catchments are located in the Coast Range of Oregon, Lincoln County (Figure 1). The sites are usually accessed from Corvallis or Toledo.

From Corvallis:

- Take Hwy 20 west to Burnt Woods (approximately 22 miles)
- Take left on Burnt Woods-Harlan Rd (there's a turn lane and a country store on the left)
- Follow Burnt Woods Rd to Harlan (approximately 8 miles)
- In Harlan, Burnt Woods-Harlan Rd more or less dead ends into Harlan Rd; take a right
- Follow Harlan Rd for approximately 1.5 miles, turn left on Grants Creek Rd
- This road immediately crosses Big Elk Creek, veer right onto FS road 31 after the bridge; tune CB to 17

Gaging Stations

Flynn Creek (Figure 2):

Follow FS 31 and continue past the end of asphalt. At this point the road begins to descend. At the bottom of the descent there is a hairpin turn which crosses a creek (culvert, not bridge). This is Flynn Creek (also an open wet meadow on the left side of the road which Flynn Creek meanders through). Parking is best on the right side of the road. Be sure to clear the vehicle of the road to allow for log trucks and other traffic to pass. On foot, cross over the earthen mound and follow the trail to the gage house.

Needle Branch (Figure 3):

Continue past Flynn Creek on FS 31 and take a left at the dead end into FS 59 (also known as 1000 Line Rd); tune CB to 4. Follow 59 for ~1.7 miles. Needle Branch gaging station is located on the left side of the road immediate across from the ranch. Remember that you are on private property, so be respectful and make sure to park appropriately so log trucks can pass.

Deer Creek (Figure 2):

Either take a right on FS 59 (from 31) or turn around from Needle Branch. Follow FS 59 for approximately 1.2 miles from FS 31 (2.9 miles from Needle Branch). Take a right into opening and park near the far end. On foot, walk through the cut in the tree and follow the trail to Deer Creek gage house.

From Toledo:

- Follow Hwy 20 Business to SE Butler Bridge Rd, take left or right depending on direction of approach
- Veer right onto South Bay Rd after first bridge crossing
- Continue on South Bay and cross two bridges, turn left onto 1000 Line Rd
- Deer Creek turnout is on the left at approximately mile 6.8
- Needle Branch is located on the left near mile 10
- FS 31 to Flynn Creek is on the left near mile 8, follow for 1.1 miles park on left at hairpin curve

Rain Gages

Flynn Creek rain gage (Figure 2):

Follow the trail to Flynn Creek gaging station; rain gage is on the right just before the trail turns to the left. A marked trail should be maintained from the main trail as the currently cleared road begins to re-vegetate.

Needle Branch (Figure 3):

Continue past Needle Branch gaging station on FS 59 (1000 Line Rd) and take the first left (approximately 75 m). Follow the forest road and take a left at the first split. The rain gage is at the far end of the old loading dock, immediately opposite the road.

Deer Creek 59 (Figure 2):

Follow Deer Creek trail and drop down to the creek (not marked to avoid vandalism) just before the gaging house. Follow the creek downstream through the riffle section; the rain gage is located on the left bank shelf (look for lots of *Rubus*).

Deer Creek 5083 (Figure 2):

Continue on FS 59 (1000 Line Rd) past Deer Creek entrance heading away from Needle Branch. Take a right on FS 5083. The rain gage is located off the right side of the road embankment.

Meadow (Figure 2):

From Flynn Creek, follow FS 31 approximately 0.9 miles heading towards FS 59. Take a left on Plum Creek 1005. Park after crossing the steel bridge. The rain gage is located in the meadow on the right side of the road. Follow the opening in the *Rubus* and continue nearly parallel to PC 1005.

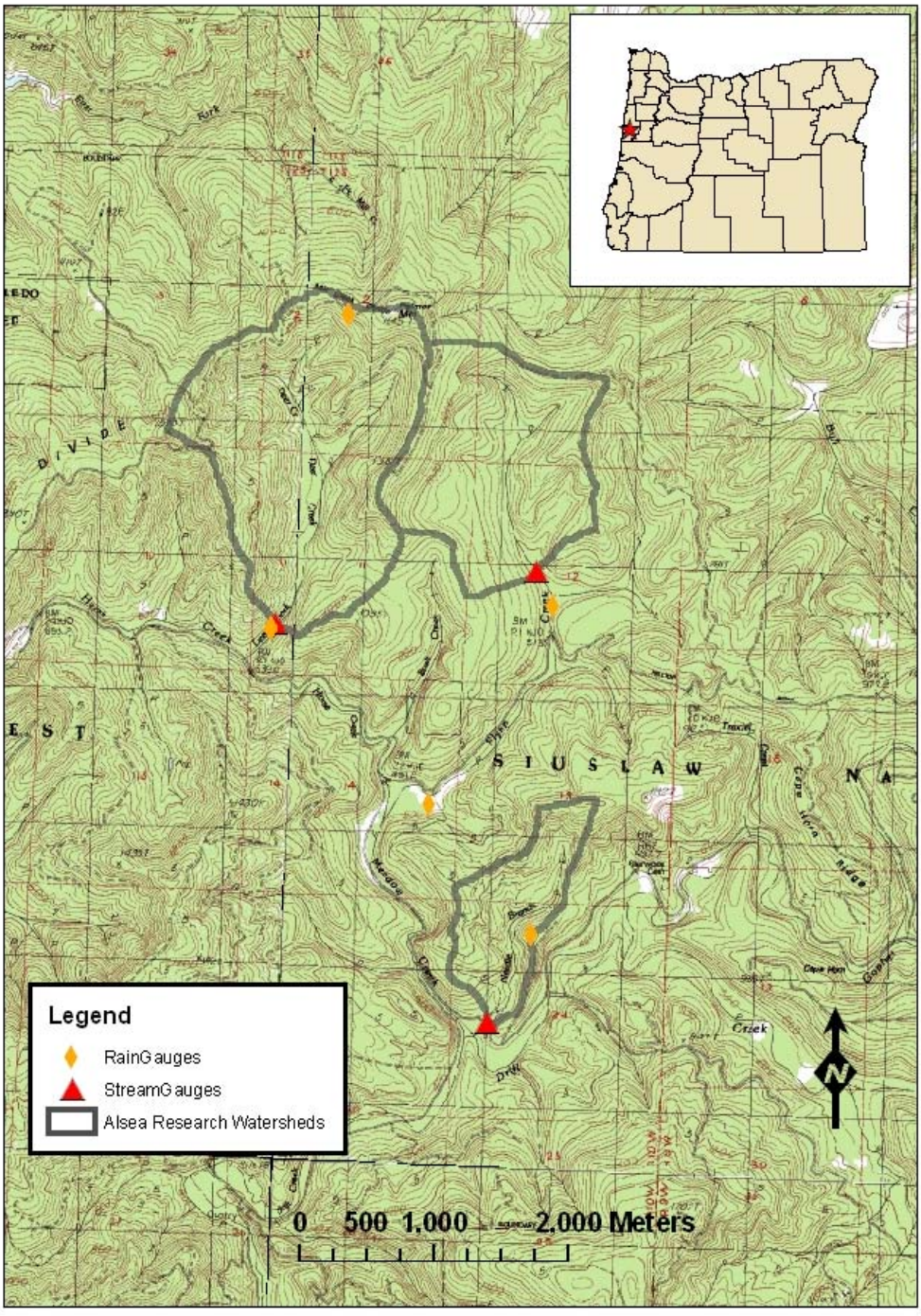


Figure 1. Location map for the Alsea Watershed Study Revisited research catchments and instrumentation.

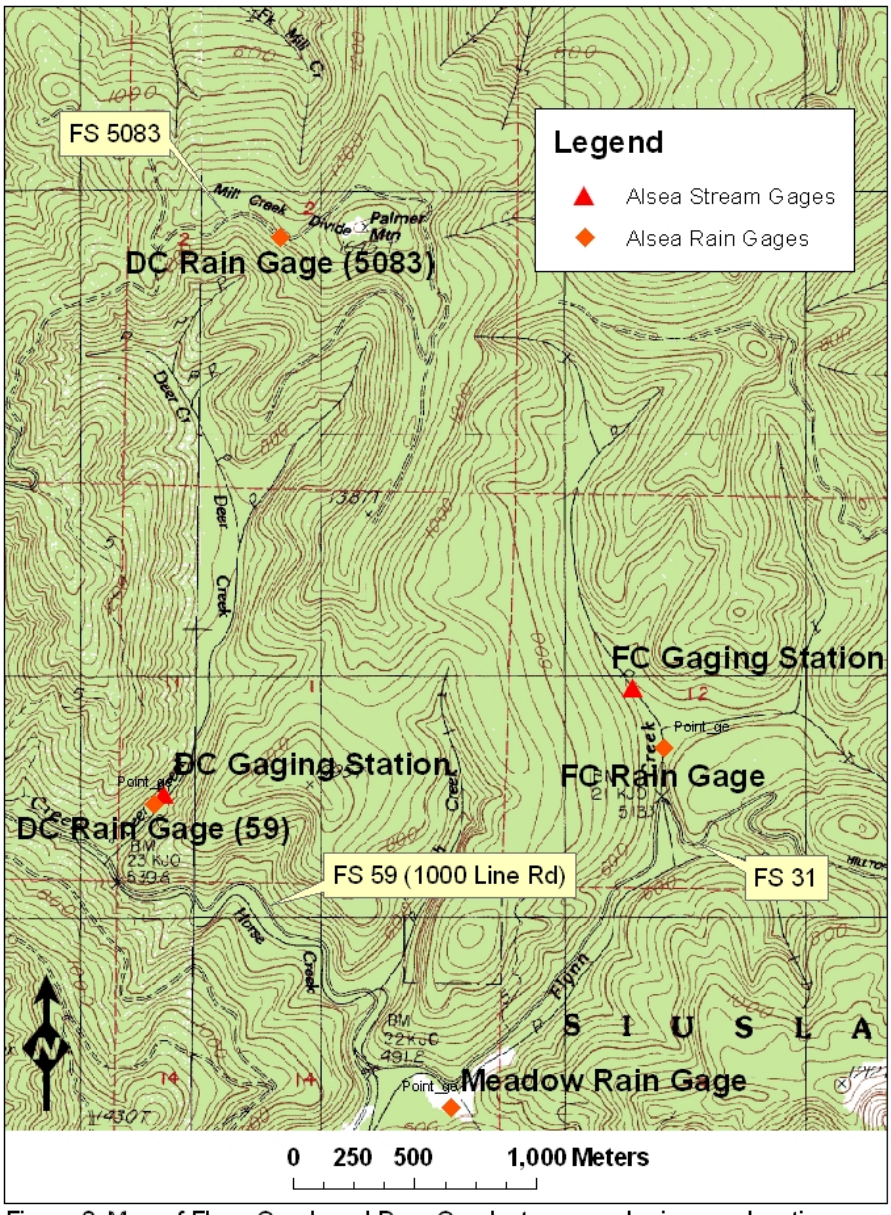


Figure 2. Map of Flynn Creek and Deer Creek stream and rain gage locations. The "Meadow" rain gage is also shown.

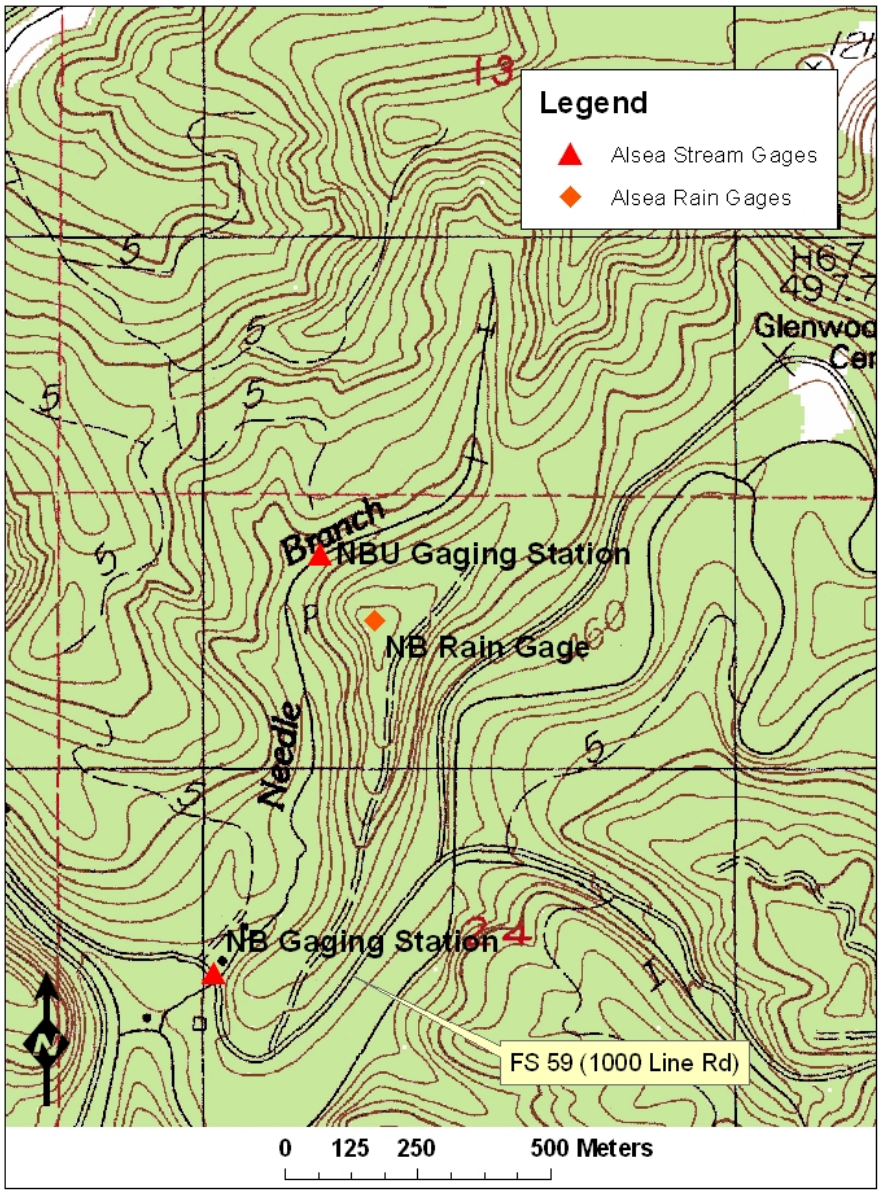


Figure 3. Map of Needle Branch stream and rain gage locations.

Servicing and Retrieving Data from Stream Gages

Servicing intervals for the stream gages vary by season and by current climate patterns. It is recommended that the gages be serviced at least weekly during the wet season. Attempts should be made to service the gages immediately prior to an expected storm system whenever possible. This allows for correction of any fouling that may occur during a recession period (especially

common early in the wet season with leaf fall) and ensures the highest quality data. Gages should also be serviced as soon as possible following a storm. Batteries should be changed on a monthly basis, or when voltage nears 12.0 volts.

Field personnel should plan to visit the gaging stations during several of the storm events throughout the year to observe and document conditions and collect a depth integrated sample using a DH-48 integrated sampler. Flow measurements should also be made at this time. This is accomplished by:

- Stretching a tape across the channel (use rebar and heavy duty clips to secure tape above water's surface)
- Use a Global Water flow probe FP101 (Global Water Instrumentation, Inc., Gold River, CA) or comparable device to make flow velocity measurements at equal intervals across the channel (every foot or half foot depending on channel width and flow characteristics)
- Measure and log depth and distance from left or right edge of water at the same intervals

Gages can be serviced on a bi-weekly basis during the dry season. At that time, turbidity probes and ISCOs will be removed from the system and brought to the laboratory for any necessary maintenance and storage. The turbidity probe's calibration should be checked with a calibration standard prior to storage. This check should be conducted again prior to deployment, and the probe calibrated if necessary (see OBS-3 Instruction Manual and TTS Manual).

The following outline provides a step-by-step guide for field personnel servicing the stream gages (*italics provide an explanation for each step)*:

1) Visually assess weir and TTS boom for any obvious interference and note in field book and on field forms. Typical interferences include, but are not limited to, debris caught in the weir or on the instrumentation, sediment covering the stilling well intake, and/or beaver activity causing backwater effect in the vicinity of the gage (*quality control step to ensure data accuracy*).
    a. If there is an obvious problem, continue to step 2 without fixing it and then repeat steps 3 and 4 after fixing it, specifically noting changes in stage and/or turbidity that occurred as a result of the maintenance.
    b. If there is no problem, note and continue to step 2.

2) Enter the gage house and connect laptop to the Campbell Scientific datalogger via the 9-pin connector cable (*provides opportunity to immediately assess datalogger real-time display for potential problems and determine the next "awakening" time for measurement; awakenings occur on ten minute intervals and are indicated by the illumination of the OBS button in the "Ports and Flags" window*)
    a. Open LoggerNet software
    b. Connect to the correct datalogger (from list)
    c. Open "Numeric Window" and "Ports and Flags" window

3) Log the following information in field notebook and on gage house data sheet (*data collection*)
    a. Field visit records
        i. Date and time

ii. Field personnel present
iii. Current weather conditions

b. Data retrieval and manual verification measurements (*steps i-ix provide data necessary for quality control used when compiling, correcting, and analyzing data at a later date*)
   i. Stage
      1. TTS (from numeric window)
      2. Reference stage (*verifies accuracy of instrumentation*) – Measure the outside reference "tape-down" with the staff plate located in each gaging house. Tape-down is located:
         a. Flynn Creek–on the upstream side of the weir, right of the notch, top of bolt; subtract measured value from 3.27 to get reference stage in feet
         b. Needle Branch–on the left bank, weir approach wall, top of lowest bolt; subtract measured value from 2.89 to get reference stage in feet
         c. Deer Creek–on the upstream side of the weir, right of the notch, top of bolt; subtract measured value from 2.78 to get reference stage in feet
      3. Stevens stage measurement (*back up for datalogger*)–at time of reading, mark on scroll: current position (by rotating the pulley gently), date, time, initials, and reference stage
         a. Complete section of field form associated with Stevens recorder
         b. Reset time or stage, if necessary
         c. Wind clock, if necessary
   ii. TTS system battery voltage (new and old battery in the case of a battery change)
   iii. Median turbidity
   iv. Dump #
   v. Next Bottle #
   vi. If probe was wiped or boom was cleared
   vii. If fouling was evident
   viii. Samples attempted and retrieved (from ISCO)
      1. note if correct volume was collected
      2. note that ISCO was reset after collection, if applicable
   ix. Other information pertinent to specific visit

4) If TTS samples have been collected since the previous visit, perform a data dump.
   a. Fill out bottle labels for each sample collected. Provide:
      i. Dump #
      ii. Location ID
      iii. Bottle number (from ISCO carousel)
      iv. Date of retrieval
      v. Total number of bottles retrieved for this visit
      vi. Initials

   see TTS Field Manual for explicit instructions for the following steps:
   b. If collecting a depth integrated sample (*collected intermittently during wet season, especially during storm events, to verify the performance of the auto-sampler against a USGS accepted method for suspended sediment concentration sample acquisition*)
      i. Click and highlight the "DI" button in the "Ports and Flags" window
      ii. Wait for ISCO sample to be triggered at next "awakening" (awakening is when OBS-3 turbidity probe and other sensors are activated to take a measurement, every 10

minutes under current settings and indicated by the illumination of the OBS button in the "Ports and Flags" window)

iii. Collect depth integrated sample with DH-48
    1. Place bottle in sampler
    2. Beginning on one side of the flow, submerge sampler slowly through the water column and move to the opposite bank perpendicular to flow while continuing to lift and submerge the sampler
    3. The goal is to completely fill the bottle with one pass across the channel

c. Immediately following an "awakening", click and highlight the "Dump" button in the "Ports and Flags" window (performed to download data from datalogger and collect samples from ISCO)
d. In LoggerNet window, choose "Custom Collection"
e. Depending on data needs, choose "Collect All" (this datalogger with current settings only holds approximately one month's worth of data, so don't expect to access the entire record with this setting) or "Collect All Since Last Collection". Name the file with station initials and dump date (i.e. FC_01_01_06) and save in "newdata" folder.
f. While file is downloading, stop ISCO program (press stop), access and cap bottles, remove and label in sequential order
g. Replace retrieved bottles with clean ones and replace cover
h. Reset ISCO by pressing "Start" then "Enter"
i. Check downloaded data by using the TTS RawPlot software
    i. Launch program
    ii. Select file number to view
    iii. Plot Stage-Turbidity and look for any obvious problems that may need to be addressed in the field
j. If battery change is required, after dump (*batteries should be changed when voltage reaches 12 volts or lower*):
    i. Power off ISCO and disconnect computer from datalogger
    ii. Quickly switch battery leads
    iii. Power on ISCO and reconnect to computer
    iv. Reset any settings in the numeric window, if necessary (minimum stage, turbidity offset, stage offset, etc.)
    v. Check to make sure "Dump" number and "Next bottle" have been changed to appropriate values (usually current dump number plus one for "Dump" and one for "Next bottle"); manually change if necessary
k. Upon return to office, immediately create back up file to be saved on another server

Downloading Rain Gage Data

Rain gages should be downloaded bi-weekly during the wet season. Dataloggers have the capacity to collect data over longer time periods, but this interval will allow field personnel to detect any problems with the instrumentation (electrical or mechanical) without losing a critical amount of data. The following steps should be taken upon arrival at the rain gage:

1) Note rain gage name, date, time of visit, and initials in field book.
2) Note any necessary observations that may affect rain gage performance (clogging or evidence of tampering, mainly)

3) Remove cover by twisting clockwise
4) Use Hobo Shuttle to download data
    a. Connect to data logger
    b. Press button on shuttle
    c. Toggle through the options (see Shuttle directions)
    d. Make sure "Relaunch" is successful
5) Replace cover
6) Upon return to the office, immediately download Shuttle data to laptop computer and create a backup file on another server

Collecting Water Quality Samples

Water quality samples are to be collected at each gaging location (Figures 1 and 2) on a monthly interval. Physical samples will be accompanied by *in situ* water quality measurements made with a Hydrolab Quanta (Hach Company, Loveland, CO). Dissolved oxygen (in milligrams per liter and percent saturation), temperature (C˚), pH (standard units), and specific conductivity (µS/cm) will be recorded. Turbidity will be measured using a Hach 2100P turbidimeter either in the field or in the laboratory using a re-suspended sub-sample of the nutrient sample collected in the field. A field equipment checklist for water quality sampling is included herein. Laboratory analytical methods and Quality Assurance/Quality Control procedures are outlined in the QAPP.

The following steps should be followed when collecting samples:

- Prior to leaving for the field
    - Make arrangements with NCASI laboratory for sample delivery date and request bottles and labels
    - Calibrate HydroLab according to its instruction manual
    - Calibrate turbidimeter according to its instruction manual
    - Pick up bottles and labels from NCASI laboratory
    - Buy ice
- Upon arriving at sample location
    - Submerge Hydrolab Quanta in channel thalweg and allow to equilibrate
    - Measure reference stage as described in Servicing Stream Gages outline (3.iv.2) and note in field book
    - Put on nitrile gloves (new pair at each site)
    - Apply label to bottle and fill in necessary information, including site ID, date, time, and initials
    - Collect sample from mid-depth in channel thalweg
    - Record site ID, date, time, dissolved oxygen (in milligrams per liter and percent saturation), temperature (C˚), pH (standard units), and specific conductivity (µS/cm) in field book
    - Either
        - Measure and record turbidity in the field using cuvette, or
        - Sub-sample each sample in the laboratory using nitrile gloves and the cuvette in the turbidimeter case to measure turbidity
    - Fill out chain of custody form
    - Deliver samples to NCASI laboratory within 24 hours of collection:

        720 SW 4th Street
        Corvallis, Oregon 97333

Safety Plan

Safety is the first priority when working in the field. The following list is not intended to be exhaustive but should be used as starting point for conducting safe field research:

- A contact person should always know your intended destinations and return time
- Follow all field safety requirements stipulated by the landowner
- All safety gear listed in the field checklist should be carried
- Wear clothing and footwear adequately suited to the climate and field conditions
- Always drive with lights on
- Tune CB radio to 17 when traveling FS 31 and 4 when traveling FS 59 and announce mile number, direction, and vehicle type at every posted mile
- Have safety numbers easily accessible
  - Samaritan Toledo Health Clinic–541-336-5181
  - George Ice/NCASI–541-752-8801
  - Jeff Light/ Plum Creek–541-336-6227
- Report any unsafe conditions to NCASI and/or supervisor immediately upon return

Directions to Samaritan Toledo Health Clinic, Figure 4 (541-336-5181):

- From Alsea research watersheds, take FS 59 (1000 Line Rd) north towards Toledo
- Dead end into South Bay Rd, take right
- Veer left onto Elk City Rd (turns into Butler Bridge Rd)
- Take slight right onto S Main St
- Take left onto US Hwy 20 Business
- Go to 1744 NW Hwy 20 Business, Toledo, OR 97391

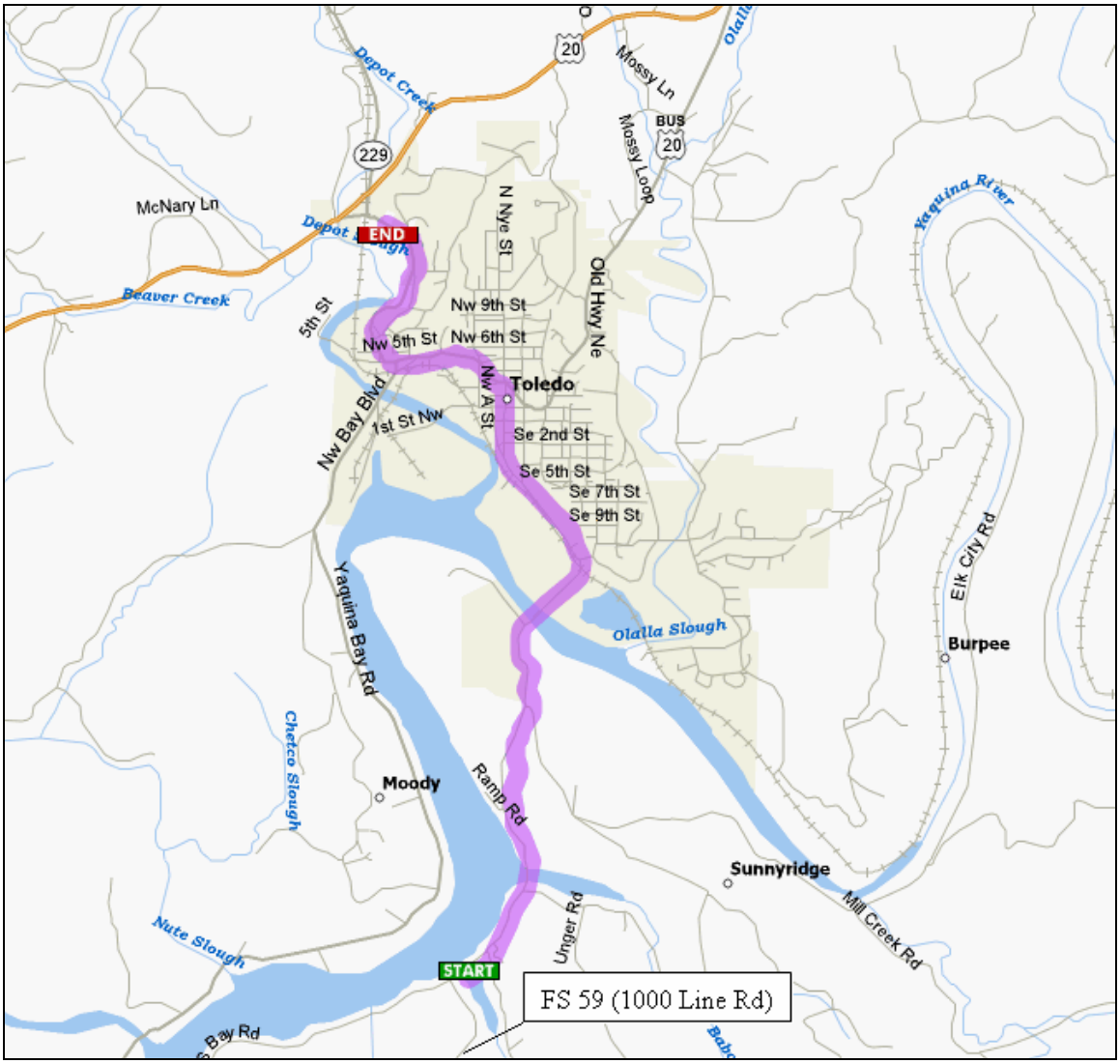


Figure 4. Route from FS 59 (1000 Line Rd) to Samaritan Health Clinic

Field Equipment Checklist for Servicing Gaging Stations and Rain Gages			
First aid kit*		Laptop computer	
CB radio*		Campbell Scientific interrogation cable (9-pin connector)	
Hardhat*		Toolbox	
Hand saw*		Bottle labels (SSC)	
Pulaski*		24 ISCO bottles per station	
Waders/high boots*		Bottle caps	
Orange field vest*		Rain gage interrogation cable	
Safety numbers*		Hobo Shuttle for rain gage	
Whistle*		5, 3v lithium battery (CR2032) for rain gages (if needed)	
Let contact person know plans*		Charged 12v batteries (if needed)	
Field book and field forms		DH-48 integrated sampler	
Write in the Rain pen, pencils, and sharpie		DH-48 bottles (1 per station)	
Backpack		Instruction manual (ISCO)	
Camera		TTS field manual	

* indicates necessary field safety equipment

Field Equipment Checklist for Water Quality Sampling			
First aid kit*		Laptop computer	
CB radio*		Campbell Scientific interrogation cable (9-pin connector)	
Hardhat*		Toolbox	
Hand saw*		Bottle labels (nutrients)	
Pulaski*		Sample bottles	
Waders/high boots*		Nitrile gloves	
Orange field vest*		Quanta Hydrolab (calibrated)	
Safety numbers*		Hach 2100P Turbidimeter (calibrated)	
Whistle*		Flagging	
Let contact person know plans*		Cooler	
Field book and field forms		Ice	
Write in the Rain pen, pencils, and sharpie		Machete	
Backpack		Miscellaneous	
Camera			

*indicates necessary field safety equipment

ATTACHMENT B

OREGON STATE UNIVERSITY STANDARD OPERATING PROCEDURE FOR DETERMINATION OF SUSPENDED SEDIMENT CONCENTRATIONS USING VACUUM FILTRATION

Received via personal communication (email) from Joanna Warren, Faculty Research Assistant, Oregon State University Forest Engineering Department, March 18, 2005

DETERMINATION OF SUSPENDED SEDIMENT CONCENTRATION USING VACUUM FILTRATION

Materials:

Wash bottles with DI (deionized) water
Vacuum filtration manifold with Buchner funnels
Vacuum pressure hose/rubber stopper/glass tube assembly for carboy and vacuum line
Large carboy for filtrate
Desiccator cabinet and jar
Forceps/tweezers
1.5 µm glass fiber filter paper (Whatman 934-AH) sized to fit Buchner funnels – **ALWAYS** HANDLE GLASS FIBER FILTER PAPERS WITH FORCEPS/TWEEZERS
Oven at 105^{o} C
Numbered aluminum weighing tins that will fit in appropriate analytical balance – **ALWAYS** HANDLE ALUMINUM WEIGHING TINS WITH FORCEPS/TWEEZERS
Analytical balance accurate to 0.0001 gram
Analytical balance able to handle load of one sample bottle filled with sediment and water

Procedure: (**ALWAYS** HANDLE PAPERS AND TINS WITH FORCEPS/TWEEZERS)

1. Inspect the filters carefully when removing them from the box and separate them if they are stuck together. Hold each filter up to the light to verify there are no holes present. Discard the defective filters.

2. Write filter number on one side of the filter paper with an ultra-fine Sharpie. Underline the numbers so that they will not be confused when read upside down. Record filter ID number on initial tare weight form. Place many (25-50) glass fiber filters in oven at 105^{o} C for 24 hours.

3. Remove filters from oven and place in desiccant cabinet to cool for at least 15 minutes before weighing. Do not remove filters from desiccant cabinet until you are ready to weigh them since they will absorb moisture from the air. Weigh oven-dried filter paper for each sample. (Take out only a few filters at a time.) Do not stack or overlap the filters.

4. Be sure at least two (2) lines of the manifold are open/on, then turn on the vacuum line.

5. Be sure to keep track of filter paper and its corresponding sample number. Place a pre-tarred filter, numbered side down, on vacuum manifold and wet it down with DI water. This will create a seal and prevent floating of the filter paper when a during sample filtration. Use the other manifold line to throttle the flow of air through the funnel being used for filtration.

6. Record sample bottle number.

7. Weigh sample bottle and sample (without cap) and record.

8. Shake the bottle with fluid in it. Pour the fluid into the funnel **slowly**, taking care that suction is continuously maintained – not too much; not too little. If the water has too much sediment, just filter what you can on the first filter and filter the remainder with another filter and combine the weights. Watch the carboy to make sure it isn't too full to pull water through the lines; empty the carboy as needed.

9. Wash all sediment out of empty sample bottle onto filter paper with DI water. Weigh empty/washed sample bottle.

10. When the sample has been completely filtered and the paper appears to no longer be saturated, release the vacuum suction from the funnel, and remove the filter paper with sediment from the funnel using blunt tweezers. Dry at 105^{o}C for 24 hours.

11. After 24 hours, remove the filter from the oven and place in the desiccator cabinet or jar immediately and allow to cool to room temperature. Then weigh filter and record.

12. At least one (1) blank filter paper per every ten (10) water samples should be run using only DI water. Note: blanks will typically lose weight; this represents loss of filter fibers during filtration. The mean fiber loss should be added back into sample weights.

13. Clean funnels with DI water and Kimwipes after use.

14. Calculation: suspended sediment concentration, SSC (mg/L) =
    a. Mass of water and sediment (g) - mass of sediment (g) = mass of water x 1 cc/g = ml (to convert from grams to cubic centimeters and then to ml).
    b. Mass of sediment /particle density (2.65 g) (or grams of soil x 1cc/2.65 g) = cc = ml
    c. Then add together ml of water and ml of soil to get actual volume of sample.
    d. (mass of sediment * 1000000)/ actual volume of sample.

***Note**: Total suspended solids (TSS) involves using an aliquot of a sample.
Suspended sediment concentration (SSC) involves using the entire sample.
(Sources: http://www.epa.gov/etv/sitedocs/meetings/wqp/sum_111301.pdf and http://water.usgs.gov/osw/pubs/ASCEGlysson.pdf)

Adapted from:

Method 2540D in: Clesceri, L.S., A.E. Greenberg and A.D. Eaton, eds. 1998. Standard Methods for the Examination of Water and Wastewater. 20th ed. American Public Health Association, Washington, DC.

USFS Redwood Sciences Lab Sediment Lab Manual. Laboratory Procedures for Determining Suspended Sediment Concentration. 13p. Arcata, California. http://www.fs.fed.us/psw/topics/water/tts/manuals/sedlab_manual.doc

Figure 1. Vacuum filtration manifold with 8-55mm Buchner funnels.

Figure 2. Vacuum filtration manifold with 4-110mm Buchner funnels.

ATTACHMENT C

NATIONAL COUNCIL FOR AIR AND STREAM IMPROVEMENT STANDARD OPERATING PROCEDURES FOR NUTRIENTS USING AN ALPKEM FLOW INJECTION ANALYZER

Provided by NCASI's West Coast Regional Center Laboratory, Corvallis, OR

NATIONAL COUNCIL FOR AIR AND STREAM IMPROVEMENT STANDARD OPERATING PROCEDURES FOR NUTRIENTS USING AN ALPKEM FLOW INJECTION ANALYZER

The Table C1 lists the NCASI Standard Operating Procedures (SOPs) being used for the analysis of nutrient parameters using an Alpkem flow injection analyzer (FIA) and the most recent revision date. These SOPs are currently undergoing revision and will be incorporated in an update to the QAPP when they are complete. An example SOP for the nitrate-nitrite analyses is included herein.

Table C1. NCASI Standard Operating Procedures for Nutrient Analysis.

NCASI Method ID	Method Title	Revision Date
CH18A	Nutrient Content by Flow Injection Analysis, Nitrate/Nitrite Analysis	10/05
CH18B	Nutrient Content by Flow Injection Analysis, Orthophosphate Analysis	10/05
CH18C	Nutrient Content by Flow Injection Analysis, Ammonia Analysis	12/05
CH18D/F	Nutrient Content by Flow Injection Analysis, Persulfate Digestion and Total Phosphate Analysis	10/05
CH18G/F	Nutrient Content by Flow Injection Analysis, Kjeldahl Digestion and Total Phosphate Analysis	10/05
CH18G/H	Nutrient Content by Flow Injection Analysis, Kjeldahl Digestion and Total Nitrogen Analysis	10/05

NCASI STANDARD OPERATING PROCEDURES

CHAPTER 18: DETERMINATION OF NUTRIENT CONTENT BY FLOW INJECTION ANALYZER

SECTION A: NITRATE AND NITRITE ANALYSIS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the guidelines to be followed when performing nitrate and nitrite analyses. The method is also used to analyze persulfate digestates for total nitrogen. This SOP is based on the Flow Solutions 3000 total phosphorus procedure, which complies with the requirements of EPA Method 363.2. The applicable range of EPA 363.2 is 0.05 to 10.0 mg/L of N as NO_3^-. The MDL determined in the NCASI WCRC laboratory is limited by the blank level (refer to current control charts).

Nitrate and nitrite are the total oxidized inorganic nitrogen forms found in natural waters. Typically, concentrations range from less than 0.01 mg/L in surface and ground waters to more than 30 mg/L in some wastewaters. Nitrate is the predominate form of nitrogen and usually makes up the majority of the combined nitrate/nitrite concentration. Nitrate is an essential plant nutrient and in some cases has been identified as the growth-limiting nutrient.

Using this method, the unfiltered/undigested or persulfate digested sample (for total persulfate nitrogen) is injected into the instrument and flows through an open tubular cadmium reactor (OTCR) that reduces all forms of nitrate to nitrite.

Refer to NCASI SOP Chapter 18, Section D: Persulfate Digestion for the Determination of Total Nitrogen and Total Phosphorus, for the digestion procedures.

2.0 GLASSWARE AND EQUIPMENT

2.1 Prepare an HCl bath by adding approximately 3 L of concentrated HCl to approximately 4 gallons of deionized (DI) water. Soak all glassware in the HCl bath for a minimum of 12 hours.

2.1.1 Glassware may only be added to the acid bath after being thoroughly rinsed with hot tap water and then DI water.

2.2 Rinse the glassware copiously with DI water and allow to air dry before use. **ALL** nutrient glassware **MUST** be segregated from other glassware and should **NEVER** be washed using any detergents.

2.2.1 Reagent storage bottles

2.2.1.1 three 500 mL amber bottles

2.2.1.2 1 L amber bottle

2.2.1.3 four 100 mL amber bottles

2.2.2 Reagent preparation glassware

2.2.2.1 4 L vacuum flask and aspirator system

2.2.2.2 1 L beaker

2.2.2.3 two 600 mL beakers

2.2.2.4 two 100 mL beakers

2.2.2.5 50 mL beaker

2.2.2.6 200 mL Erlenmeyer flask

2.2.2.7 1 L volumetric flask

2.2.2.8 500 mL volumetric flask

2.2.2.9 ten 100 mL volumetric flasks

2.2.2.10 two 25 mL volumetric flasks

2.2.2.11 100 mL graduated cylinder

2.2.2.12 50 mL graduated cylinder

2.2.2.13 petrie dish with cover

2.2.3 Sample preparation glassware

2.2.3.1 disposable culture tubes (VWR Catalog No. 60825-618)

2.3 All Teflon™-lined screw caps, reagent line caps, and scoopulas need not be acid washed and may be thoroughly rinsed with hot tap water and DI water.

3.0 PREPARATION OF STANDARDS AND REAGENTS

3.1 Prepare the following standards and reagents ***in advance***:

3.1.1 Ammonium chloride-ethylenediaminetetraacetic acid, disodium salt dihydrate buffer (EDTA)–Dissolve 85 g of ammonium chloride (NH_4Cl) and 0.1 g of EDTA ($C_{10}H_{14}N_2Na_2O_8 \bullet 2H_2O$) in approximately 900 mL of Barnstead water using a 1 L beaker. Adjust the pH to 8.5 using ammonium hydroxide (NH_4OH) and dilute the solution to 1L with Barnstead water using a volumetric flask. Transfer the solution to a 1 L amber bottle and store the solution in the refrigerator. The reagent is stable for several weeks.

3.1.2 Color reagent–Slowly add 50 mL of phosphoric acid (H_3PO_4) to approximately 400 mL of Barnstead water in a 600 mL beaker equipped with a stir bar and stir plate. Add 20 g of sulfanilamide ($C_6H_8N_2O_2S$) and 1g of N-1-napthylethylenediamine dihydrochloride (NED) ($C_{12}H_{14}N_2 \bullet 2HCl$) and stir until the solids are dissolved. Transfer the solution to a 500 mL volumetric flask with rinses of Barnstead water. Fill the flask to volume with additional Barnstead water and transfer the solution to a 500 mL amber bottle. The reagent is stable for a month and should be stored in the refrigerator. Filter the solution in-line by placing a 25 mm, 0.45 μm cellulose acetate syringe filter (Value Prep, catalog number 28145-472) on the top of the reagent bottle during the analysis.

3.1.3 2% copper sulfate (coil activation)–Place 2 g of copper sulfate, pentahydrate ($CuSO_4 \bullet 5H_2O$) in a 100 mL amber bottle. Using a graduated cylinder, add 100 mL Barnstead water to the reagent bottle. If desired, a larger quantity of solution may be prepared.

3.1.4 0.5 N HCl (coil activation)–Dilute 4.15 mL of concentrated hydrochloric acid (HCl) to 100 mL with Barnstead water using a Finn pipette and volumetric flask. Transfer the solution to a 100 mL amber bottle. If desired, a larger quantity of solution may be prepared.

3.1.5 1000 mg/L nitrate stock solution–Dry approximately 1 g of potassium nitrate (KNO_3) in a 105°C oven for a minimum of 2 hours using a petrie dish and cover. Allow the solid to cool in a dessicator for approximately 15 minutes. Weigh 0.7218 g of the dried potassium nitrate and dissolve the solid in 100 mL of Barnstead water using a 100 mL volumetric flask. The standard stock may be used for several months and must be stored in the refrigerator.

3.1.6 5 mg/L nitrite stock solution (coil efficiency test)–Dilute 2 mL of 250 mg/L nitrite stock solution (Hach Catalog No. 23402-49) to 100 mL with degassed Barnstead water using a volumetric flask.

3.1.7 1 mg/L nitrate standard (ICV)–Transfer a small amount of nitrate standard from a different source than the standard utilized for the calibration curve to a 16 X 100 mm disposable culture tube (VWR Catalog No. 60825-618) just before analysis.

3.1.8 Calibration curve–Add approximately 90 mL of Barnstead water to each volumetric flask. Add the appropriate levels (Table 1) of 100 mg/L nitrate stock solution (Section 3.1.6) with an automatic pipette or gas tight syringe. Dilute each flask to 100 mL with additional Barnstead water using a volumetric flask.

Table 1. Nitrate Standard Stock Levels

Standard Level (mg/L)	100 mg/L Nitrate Stock Solution
0.02	20 µL
0.05	50 µL
0.10	100 µL
0.50	500 µL
1.00	1.0 mL
5.00	5.0 mL
Blank	0 mL

3.2 Prepare the following standards and reagents ***on the day of analysis*** and dispose of after use:

3.2.1 Degassed Barnstead water–Add approximately 3 L of Barnstead water to a 4 L vacuum flask. Attach the vacuum flask to a smaller flask to catch any backflow. Attach the smaller vacuum flask to the laboratory sink using an aspirator setup. Place the 4 L flask on a heating stir plate and add a stir bar. Turn the tap water to full flow, turn on the stir plate, and turn the heat on to the lowest setting. The water should be degassed for a minimum of 1 hour just prior to use. After removing portions of the degassed water, continue to degas the remaining volume of Barnstead water.

3.2.2 Brij water–Add approximately 0.5 mL of Brij surfactant (P/NA21-0110-33) to a 500 mL reagent bottle. Fill the reagent bottle with degassed Barnstead water.

3.2.3 Working buffer–Add approximately 1 mL of Brij surfactant to a 500 mL reagent bottle. Fill the reagent bottle with ammonium chloride-EDTA buffer (Section 3.1.1). Filter the solution in-line during analysis using a syringe filter (Section 3.1.2).

3.2.3.1 The surfactant degrades rapidly and should only be added to the aliquot of working buffer that will be used for the day.

3.2.3.2 A newly opened bottle of Brij-35 surfactant should only be used for 5 months, after which a new bottle must be used. The older bottle

of surfactant may be used in other analyses so it is not necessary to dispose of it.

4.0 COIL ACTIVATION

4.1 Using a 10 mL syringe with a threaded tip, flush the OTCR with the following solutions in the order listed. Avoid introducing air into the coil by assuring that no bubbles are injected into the coil along with the reagent.

4.1.1 DI water–one or two flushes with 10 mL

4.1.2 0.5 N HCl (Section 3.1.4)–one flush with 10 mL
Do not let the HCl sit in the OTCR; proceed directly to the next solution.

4.1.3 DI water–one or two flushes with 10 mL

4.1.4 2% $CuSO_4$ (Section 3.1.3)–Fill syringe with approximately 10 mL of solution. Flush half of syringe through the coil and allow the solution to sit in the coil for one minute. Slowly flush the remainder of the solution through the coil, as plugging will occur if the coil is flushed too quickly.

4.1.5 DI water–Forcefully flush with water until precipitate is no longer extracted from the coil. This usually requires two rinses of approximately 15 mL each.

4.1.6 After analysis, fill the coil with DI water for storage, taking care to keep oxygen out of the coil.

4.2 The coil efficiency must be tested prior to nitrate and nitrite analyses. Because the coil converts all nitrate to nitrite, the peak height of the 5 mg/L nitrate standard (Section 3.1.8) from the curve is compared to the peak height of the 5 mg/L nitrite stock (Section 3.1.6). The efficiency is calculated as follows:

4.2.1 (5 mg/L (NO_3^-) peak height / 5 mg/L (NO_2^-) peak height) X 100

4.2.2 The coil efficiency must be greater than 80%. If the coil efficiency is NOT greater than 80%, the activation process must be repeated until the coil efficiency criterion is achieved (Section 4.0).

5.0 QA/QC SAMPLES

5.1 Prepare the following samples to verify instrument and operator performance during the analysis.

5.1.1 Sync peak (SYNC)–The SYNC is an initial run of the second highest point on the calibration curve. This sample *does not* need to be individually prepared and can be drawn from a point on the calibration curve (Section 3.1.8). Because the instrument does not calculate concentrations

for these peaks, the two sync peak heights should be compared to one another. The two sync peaks should be strong and approximately the same height.

5.1.2 Continuing calibration verification (CCV)–The CCV is a point from the calibration curve that is analyzed repeatedly throughout the run. This sample *does not* need to be individually prepared and can be drawn from a point on the calibration curve (Section 3.1.8). Recoveries should be between 80 and 120% or within the warning limits of the current control charts. The RSD between the multiple recoveries should be less than 25%.

5.1.3 Independent calibration verification (ICV)–The ICV is a known standard prepared from a source other than the source the curve was prepared from. This solution is ordered in the appropriate concentration (Section 3.1.7). Recovery should be between 80 and 120% or within the warning limits of the current control charts.

5.1.4 Matrix spike and matrix spike duplicate–If persulfate digested samples are being analyzed they should have been spiked with both nitrate and phosphorus standards prior to digestion (Section 3.1.6). If undigested samples are being analyzed the standard can be added to the tube prior to analysis at a concentration that is one to five times the anticipated native level. The recoveries should be between 80 and 120% or within the warning limits of the current control charts and the RPD between the two recoveries should be less than 25%.

5.1.4.1 The sample chosen for the matrix spike and spike duplicate should be different for each analysis.

6.0 INSTRUMENT SETUP

6.1 Turn on the power for each component beginning with the autosampler, analyzer, and computer**. It is important that the power of each component be turned on in this order**.

6.2 From the "Method Editor" prompt, load the "no2no3" (nitrate/nitrite) method. Page through the method to verify that the parameters are the same as indicated in the Alpkem Operators Manual (Section 6, pages 17 and 18) and Attachment 1.

6.2.1 If necessary, change the calibration curve table to reflect the actual curve that was prepared. The table in the method *must be identical* (names, levels, concentrations) to the sample table that is prepared (example in Attachment 2). In addition, this table from the method should include the CCV (Section 5.1.2) and its minimum allowable recovery.

6.3 From the "Sample Table" prompt, load an old nitrate/nitrite sample table. Save the table as the analysis name followed by the current date and prepare a new sequence table that includes the following information. If conducting analyses of persulfate

digested samples include the digested calibration curve solutions as samples analyzed in triplicate. Refer to Section 5, page 11, Table 5.1, in the Alpkem Flow Solution™ 3000 operation manual for sample type abbreviations, names, and functions.

6.4 Insert the nitrate/nitrite cartridge into the instrument.

6.5 Attach the appropriate tubing to the nitrate/nitrite cartridge. The tubing is located in a resealable bag in each cartridge box.

6.5.1 Inspect each tube for wear and replace tubes when cracks, deformity, or excessive wear become apparent.

6.6 Insert the 540 nm filter into the detector. Either Channel 1 or Channel 2 may be used, as long as the same channel is consistently designated throughout the method and instrument setup.

6.6.1 Change the filters by pulling out the detector cartridge. Remove the plastic casing that holds the filter by pulling straight up. Remove the filter by inserting a small allen wrench into the bottom of the plastic casing, loosening the allen screw, and catching the filter on a lint-free cloth. Insert the desired filter into the plastic casing, center the filter, and tighten the allen screw. Replace the plastic casing containing the filter in the detector cartridge *with the mirrored side of the filter facing out*. Replace the detector cartridge in the instrument.

6.6.2 Filters that are not being used should be stored in a sealed container with desiccant.

6.7 Connect all reagent lines to one platen and all waste lines to an additional platen (if possible). Place the platens on the instrument, add a thin bead of silicone oil to one of the rollers, close the platen levers, and tighten until the last thread disappears on the tightening screws.

6.7.1 Do not over-tighten screws, as this can result in baseline instability.

6.8 Connect all reagent lines to DI water and all waste lines to the waste container (Attachment 3).

6.9 Start the pump using the “pump” pull-down from the menu at the top of the screen. Select the appropriate channel from the list. Watch for leaks and flow problems. Begin monitoring the baseline by clicking the “Collect Data” prompt. When prompted, select the current sample table and method. After the blank chromatogram comes up, click the “play” button (the red arrow located on the left of the screen). To bring the baseline back to zero, click the “ZERO” button at the top left corner of the chromatogram. A stable baseline should occur once all bubbles have been removed from the system. Bubbles are removed by kinking the

"debubbler waste" line for several seconds and releasing. The pressure build-up will flush the bubbles out of the system. Repeatedly kink the line, as necessary.

6.9.1 *Do not* connect the OTCR until the lines have been connected to the reagents and a stable baseline with the reagents has been established (Section 6.14).

6.10 Connect all reagent lines to Kleenflow Acid (P/N A001251) and run the acid through the system for 10 minutes.

6.11 Connect all reagent lines to degassed Barnstead water and run through the system for a minimum of 15 minutes. A stable baseline should occur in approximately 5 minutes or less and once all bubbles have been removed from the system. Zero the baseline as necessary.

6.12 Connect the buffer reagent line to the Brij water solution for 10 to 15 minutes. The surfactant should help eliminate any remaining bubbles from the system and a stable baseline should be established in approximately 5 minutes or less. If a stable baseline cannot be established, repeat the process (Sections 6.10 through 6.12).

6.13 Once a stable baseline has been established with degassed Barnstead water and Brij water, connect all reagent lines to the appropriate reagents (Attachment 3). Once a stable baseline has been established connect the nitrogen line. This analysis is a segmented flow analysis; therefore nitrogen bubbles will be observed from this point on during the run.

6.13.1 Carrier–degassed Barnstead water

6.13.2 Wash–degassed Barnstead water

6.14 Once a stable baseline has been established with the reagents and the nitrogen flow is established connect the OTCR to the instrument without introducing air into the coil. This can be achieved by connecting one end of the OTCR to port 8 so that the reagents are flowing through the system and into and out of the coil. Next, connect the other end of the coil to port 4. Verify that there are no leaks.

6.14.1 Either end of the coil may be connected to port 8 or port 4.

6.14.2 Air is destructive to the OTCR and must not be introduced into the coil.

6.15 Establish a stable baseline with the coil connected. The baseline can usually be established in 5 minutes or less.

6.16 Transfer the samples to the autosampler tray as indicated on the sample table.

6.16.1 If the samples are acid preserved, neutralize them with NaOH prior to analysis.

6.17 Monitor the stable baseline for 10 to 15 minutes and begin the coil test run (Section 4.2).

6.17.1 The pump should be set on low speed.

6.17.2 Prepare an additional table that contains only the coil test samples. This table should be prepared and run to verify coil efficiency before the entire sample table is run. Include the coil test in both sample tables to first check the coil efficiency and then verify the coil efficiency immediately before the samples are run.

6.17.3 If the coil efficiency is calculated to be greater than 80%, run the sample table. If the coil efficiency is calculated to be less than 80%, repeat the coil activation (Section 4.0) and re-run the coil test after a stable baseline has been established (Sections 6.13 through 6.16).

6.18 Run the sample table.

6.18.1 It is preferable that analyses be carried out on fresh, unpreserved samples. If analysis cannot be completed within 48 hours from the day of collection, analysis can be performed on samples preserved at pH 2 with H_2SO_4. The holding time for preserved samples is 28 days and they must be neutralized with NaOH prior to analysis.

6.18.2 Monitor the beginning of the run to verify that the sync peaks elute and are of comparable height to one another.

7.0 DATA WORK UP

7.1 Audit the chromatogram for correct peak selection and move any peaks that are incorrectly selected by clicking on the "peak editor" button (peak with a plus sign) that is located to the left of the screen. Place the cursor on the desired peak, click and hold the left mouse button. While holding down the left mouse button, drag the peak label number to the new desired location. Release the left mouse button when the peak label is located at the apex of the correct peak.

7.1.1 Peaks cannot be dragged past other peaks.

7.1.2 The "recalculate" button must be clicked to update any changes made to the chromatogram.

7.1.3 Any changes must be saved under a new file name.

7.2 Audit the calibration curve for linearity. The calibration curve correlation coefficient must be 0.99 or greater. If the correlation coefficient criterion is not met, the calibration curve must be reprepared and the QC samples reanalyzed.

7.2.1 Each calibration curve point is run in triplicate. Therefore, if one or two of the three peaks appear to be outliers, they can be deleted to improve the linearity of the curve. Delete a peak by left-double clicking on the undesired point of the calibration curve.

7.2.2 The "recalculate" button must be clicked to update any changes made to the calibration curve.

7.2.3 Any changes must be saved under a new file name.

7.3 Transfer the data from the results page to the Excel file for nitrate/nitrite analysis. The files are located on the network in the DC_Com drive in the Nutrients folder. As required for total nitrogen only recalculate sample concentration based on the digested calibration curve.

8.0 INSTRUMENT SHUTDOWN

8.1 After the last sample has run and the software has finished collecting data, remove the coil from the instrument and flush with DI water to remove reagents. Do not introduce air to the column

8.1.1 The coil and connecting tubing should be filled with DI water for storage.

8.2 Switch all reagent lines to water and pump for at least 15 minutes.

8.3 Stop the pump, loosen the platen levers, disconnect the tubing from the platens, and disconnect the tubing from the DI water, waste container, and instrument.

8.3.1 The tubing may remain connected to the instrument and to the DI water and waste container overnight, but the tubes *must* be removed from the platens in an effort to prolong the life of the tubes.

8.4 Turn off the power to the autosampler and analyzer and close out of the EnviroFlow software.

8.4.1 Shutdown can be done in any order.

9.0 WASTE DISPOSAL

9.1 All reagent and sample wastes must be pH adjusted until neutral and disposed of down the drain with copious amounts of water.

ATTACHMENT 1

Method Edit – NO2NO3.mth (Timed Events)

File Name: C:\FLOW_4\NUTRIE~1\METHODS\NO2NO3.MTH
Date: October 27, 2005

Device	Name	State	Time	T
Cycle Start		On	2:00:00	R
Autozero		On	1:59:00	R
Cycle Duration		On	80.00	I
Sampler		Sample	0.00	I
Sampler		Wash	40.00	I
Ch1 Valve1		Load	0.00	I
Ch1 Valve1		Inject	45.00	I

ATTACHMENT 2

Method Edit – NO2NO3.mth (Calibrants Table)

File Name: C:\FLOW_4\NUTRIE~1\METHODS\NO2NO3.MTH
Date: October 27, 2005

Name	T	nitrate + nitrite	%
Cal 0.02	C	0.020000	
Cal 0.05	C	0.050000	
Cal 0.10	C	0.100000	
Cal 0.50	C	0.500000	
Cal 1.00	C	1.000000	
Cal 5.00	C	5.000000	15
CCV 0.50	CCV	0.500000	25

ATTACHMENT 3