UNH Nutrient and Light Extinction Monitoring Program
Quality Assurance Project Plan

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A3 – Distribution List

Table 1 presents a list of people who will receive the approved QAPP, the QAPP revisions, and any amendments.

Table 1. QAPP Distribution List

QAPP Recipient Name	Project Role	Organization	Telephone number and Email address
Jonathan Pennock	Project Manager	UNH Marine Program	603-862-2921 jonathan.pennock@unh.edu
Jaimie Wolf	Project QA Officer	UNH JEL	603-862-2175 jswolf@christa.unh.edu
Bill McDowell	Laboratory Director	Water Quality Analysis Lab, Department of Natural Resources, UNH	603-862-2249 bill.mcdowell@unh.edu
Jeff Merriam	Laboratory Manager and QA Officer	Water Quality Analysis Lab, Department of Natural Resources, UNH	603-862-2341 jmerriam@christa.unh.edu or jeff.merriam@unh.edu
Jody Potter	Laboratory Technician	Water Quality Analysis Lab, Department of Natural Resources, UNH	603-862-2341 jody.potter@unh.edu
Jenn Greene	Lead Project Field Technician	UNH	603-862-2175 jenn.greene@unh.edu
Deb Lamson	Senior Field Technician	UNH	603-862-2175 dalamson@cisunix.unh.edu
Holly Abeels	Field Staff	UNH	603-862-2175 holly.abeels@unh.edu
Forbes Horton	Field Staff	UNH	603-862-2175 chorton@cisunix.unh.edu
Phil Trowbridge	NHEP/NHDES Project Officer	NHDES Watershed Management Bureau	603-271-8872 ptrowbridge@des.state.nh.us
Vincent Perelli	NHDES Quality Assurance Manager	NH DES Office of the Commissioner	603-271-8989 vperelli@des.state.nh.us
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Jennifer Hunter	NHEP Director	NH Estuaries Project	603-433-7187 jennifer.hunter@rscs.net

Based on EPA-NE Worksheet #3

A4 – Project/Task Organization

The project will be completed by the UNH Marine Program. Jonathan Pennock will be the Project Manager for UNH. The Project Manager will be responsible for coordinating all program activities. The Project Manager will manage all field staff, be responsible for "stop/go" decisions for daily sampling runs, and will notify the UNH Laboratory Manager when samples will be delivered. The Project Manager will be responsible for resolving any logistical problems and communicating the results to the field staff.

Laboratory operations will be managed by the Laboratory Manager (Jeffrey Merriam). The Laboratory Manager will be responsible for conducting analyses according to the procedures in this QA Project Plan, identifying any non-conformities or analytical problems, and reporting any problems to the Project QA Officer and the Project Manager. The Project Manager will be responsible for resolving any analytical problems and communicating the results to the laboratory staff.

At the end of the project, the Project QA Officer (Jaimie Wolf) will review the results of QA/QC checks and verify that the procedures of this QA Project Plan were completed. The QA Officer will be responsible for a memorandum to the Project Manager summarizing any deviations from the procedures in the QA Project Plan, the results of the QA/QC tests, and whether the reported data meets the data quality objectives of the project.

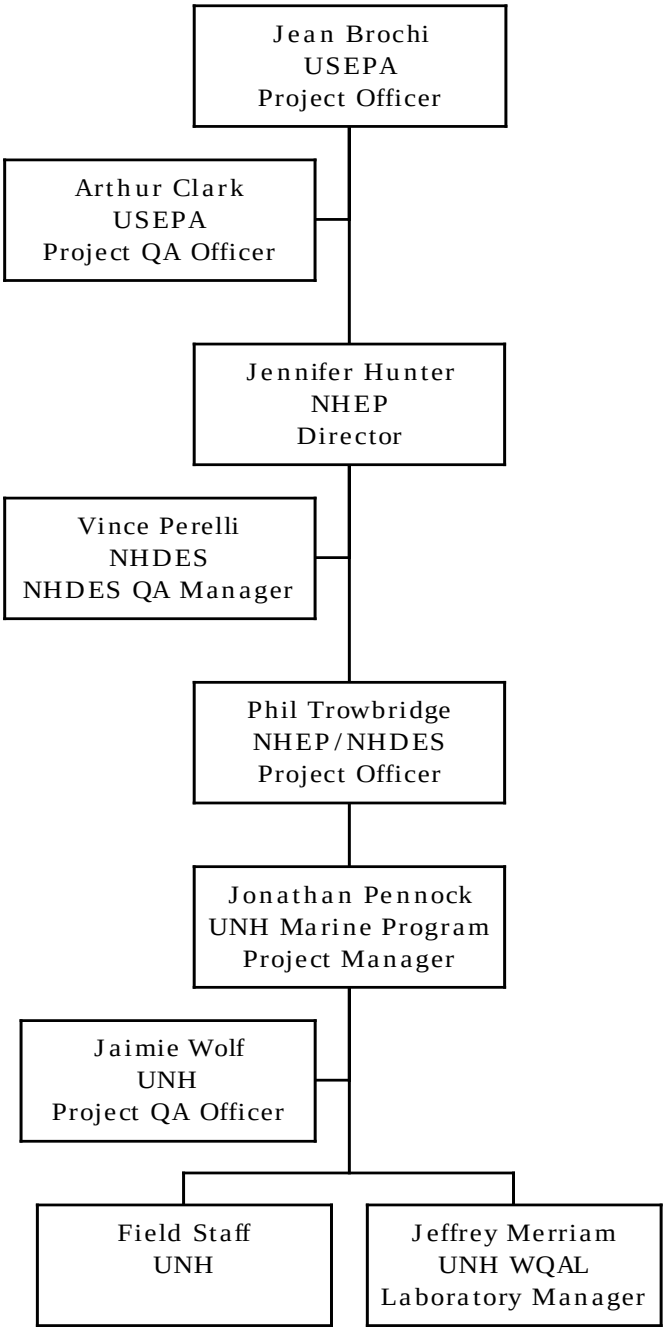
Field staff from the UNH Jackson Estuarine Laboratory include Jenn Greene, Deb Lamson, Holly Abeels, and Forbes Horton. Field staff will report any problems to the Project Manager and the Project QA Officer.

This work is being done under contract for the NH Estuaries Project (NHEP) so the Project Manager will be accountable to the NHEP/NHDES Project Officer (Phil Trowbridge) and the NHEP Director (Jennifer Hunter). Funding for the NHEP is provided by the U.S. Environmental Protection Agency.

The principal users of the data from this project will be the NHEP and NH Department of Environmental Services (NHDES). The Project Manager will submit a report to the NHEP/NHDES Project Officer and the NHEP Director at the end of the project with all the data and the QA Officer's summary report.

Figure 1 shows an organizational chart for this project.

Figure 1. Project organizational chart



A5 – Problem Definition/Background

The NHEP coordinates water quality monitoring in NH's estuarine waters. Historically, water quality stations in the estuaries have been monitored for nutrients, bacteria, eutrophication parameters, and a suite of physicochemical parameters. The specific parameters monitored by existing programs are listed below.

Nutrients	Bacteria	Eutrophication	Physicochemical
Nitrate*	Fecal coliforms	Dissolved oxygen	Temperature
Nitrite*	*E. coli*	Chlorophyll-a	Salinity
Ammonia*	Enterococci	Total suspended solids	pH
Orthophosphate*	*Clostridium perfringens*	Particulate organic matter	

*Dissolved fraction

A major focus of the NHEP is to identify datagaps not being covered by existing programs and to eliminate them. After a review of the monitoring programs, two important parameters were found to be missing from this suite (NHEP, 2002).

First, total nitrogen (TN) cannot be calculated from the nitrogen parameters currently being monitored. The sum of nitrate, nitrite, and ammonia is called "dissolved inorganic nitrogen" (DIN), whereas TN is the sum of DIN, dissolved organic nitrogen (DON), particulate inorganic nitrogen (PIN), and particulate organic nitrogen (PON). TN is expected to be the basis for EPA's nutrient criteria for estuaries so it is important to be able to measure this parameter. Therefore, the program will add DON and PON measurements to the suite of parameters at water quality stations. PIN will not be added because it is considered to be a negligible fraction of TN.

Second, there is no direct measurement of light attenuation. Chlorophyll-a, total suspended solids, and dissolved organic matter affect diffuse light attenuation but measurements of these parameters cannot be used predict the penetration depth of photosynthetically active radiation (PAR) in the water column. Penetration of PAR at depth is important for sustaining seagrasses which are a critical component of the estuarine habitat. Therefore, this project will add measurements of light attenuation using PAR sensors to the suite of parameters measured at the water quality stations.

The stations where these measurements will be taken are described and shown on a map in section B1. Sample collection and analysis will be conducted by UNH under contract with the NHEP.

Measurements of TN in the estuary will be used by the NHEP, NHDES, and other coastal resource managers to compare nitrogen levels in NH's estuaries to EPA's nutrient criteria after these criteria are developed. Light attenuation data will be used to study potential water quality effects of nutrient loading (e.g., loss of water clarity).

A6 – Project/Task Description

This project has four main tasks:

1. Prepare QA Project Plan

A QA Project Plan for both the DON/PON analyses and the PAR measurements in the field will be produced by UNH which must be approved by the NHEP/NHDES Project Officer and EPA Region I before field work on this project begins.

2. Train Project Staff

The Project Manager will organize and implement a training session for field staff. The training session will cover SOPs for field instruments and field data sheets. The training will be based on the QA Project Plan document. Field staff will sign an attendance sheet for the training. The training will be completed before sampling begins.

3. Collect and Analyze Water Quality Samples

UNH will conduct DON and PON analyses on up to 125 samples during 2003. UNH will also measure light attenuation with depth in the field using a PAR sensor. Up to 125 PAR profiles will be measured over the course of the year at the same time that samples are collected for DON/PON analyses.

4. Prepare Final Report

The final work product will be an Excel spreadsheet containing quality assured results of the DON/PON analyses and light extinction coefficients for each station on each date and a final report describing any deviations from the protocols established in the QA Project Plan.

Table 2. Project Schedule Timeline

Activity	Dates (MM/DD/YYYY)		Product	Due Date
	Anticipated Date(s) of Initiation	Anticipated Date(s) of Completion		
QAPP Preparation	2/1/03	8/1/03	QAPP Document	8/1/03
Field Team Training	8/2/03	8/8/03	Training Documentation	8/8/03
Collect and analyze water quality samples	8/9/03	12/15/03	Data	NA
Final project report preparation	12/16/03	12/31/03	Final report	12/31/03

Based on EPA-NE Worksheet #10.

A7 – Quality Objectives and Criteria

Table 3 summarizes the performance criteria for the DON and PON samples that will be collected for this project. More details on each data quality objective are provided in the paragraphs below the table. The data quality objectives for the PAR measurements are discussed at the end of this section.

Data Quality Objectives for DON and PON

Table 3. Measurement Performance Criteria for Nutrient Samples

Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance
Precision-Overall	RPD < 20%	Field Duplicates
Precision-Lab	RPD < 15%	Lab duplicates
Accuracy/Bias	RPD < 15% >85% and <115% recovery	Certified Reference Material Samples Laboratory Fortified Matrix Samples
Comparability	Measurements should follow standard methods that are repeatable	NA
Sensitivity	Not expected to be an issue for this project (see discussion below)	NA
Data Completeness	80 samples meeting data quality objectives	Data Completeness Check

Based on EPA-NE QAPP Workbook for 3/19/02 DES QAPP writing class.

Precision: Relative percent difference (RPD) of duplicate samples is used as one index of precision for nutrient analyses. This is defined as the absolute difference between the duplicates divided by the average of the duplicates. For laboratory duplicates, a difference greater than 10% requires further investigation of the sample run. A difference greater than 15% is failure (unless the average of the two samples is less than 10X the MDL), and results in reanalysis of the entire sample queue, unless there is a reasonable and supported explanation for the inconsistency. For field duplicates, a difference greater than 20% will be flagged as a potential error. Duplicate precision will be analyzed by calculating the RPD using the equation:

$$RPD = \frac{|x_1 - x_2|}{\left(\frac{x_1 + x_2}{2}\right)} \times 100\%$$

where x_1 is the original sample concentration
x_2 is the duplicate sample concentration

Accuracy/Bias. For nutrient analyses, certified reference materials are analyzed periodically (approximately every 20 samples) in each sample queue to assure accuracy. Generally, a RPD from the certified concentration of more than 10% requires further investigation of that run. A difference greater

than 15% is failure (unless the average of the two samples is less than 10X the MDL), and results in reanalysis of the entire sample queue, unless there is a reasonable and supported explanation for the inconsistency. RPDs for certified reference materials will be calculated using the following equation:

$$RPD = \frac{|x_1 - x_2|}{(x_2)} \times 100\%$$

where x_1 is the measured concentration
x_2 is the known concentration for the certified reference material

Laboratory Fortified Matrix samples are also used to assess accuracy of nutrient analyses. The difference of the spiked sample concentration (SA) minus the unspiked sample concentration (SU) divided by the known concentration added (A) (expressed as percent) gives percent recovery (R):

$$R = \frac{(SA - SU)}{A} \times 100\%$$

Generally, a recovery <90% or >110% requires further investigation of the sample run. A recovery greater than or less than <85% or >115% is failure (unless the sample is less than 10X the MDL), and results in reanalysis of the entire sample queue, unless there is a reasonable and supported explanation for the inconsistency

Representativeness: The samples should be taken at the same locations and times as water quality samples for the existing water quality monitoring programs in order to ensure that the data are representative of the same water mass as was monitored for the other parameters.

Comparability: Standardized field and analytical methods should be used. These methods should follow the current industry standard for the types of measurements being taken. Written SOPs should be followed for field and analytical measurements. Standardized field data sheets should be used.

Sensitivity. In northern macrotidal estuaries, studies have shown the total nitrogen concentration to be twice the dissolved inorganic nitrogen concentration (EPA, 2001). In NH's estuaries, the DIN concentration in the middle of the bay is approximately 15 uM (0.2 mg/l), with concentrations increasing in the tidal tributaries. Therefore, the expectation is that DON and PON, which make up the majority of the difference between total nitrogen and DIN, will at least be 0.2 mg/l. Assuming equal apportioning between DON and PON, the methods for each parameter should be able to detect 0.1 mg/l. The analytical method, analytical/achievable method detection limit, and the analytical/achievable laboratory quantitation limits for this project are shown below in Table 4.

Table 4: Surface Water Target Analytes and Reference Limits

Analyte	Analytical method	Project Action Level	Analytical/Achievable Method Detection Limit	Project Quantitation Limit
DON	Appendix B and Appendix C	NA	0.1 mg/L	0.1 mg/L
PON	Appendix D and E	NA	0.1 mg/L	0.1 mg/L

Based on EPA-NE Worksheet #9b and 9c.

Completeness: This study will be deemed successful if data meeting the data quality objectives is obtained for 80 water quality samples (not including field/laboratory duplicates).

Table 5 summarizes the data quality objectives for the in-situ PAR measurements. More details on each data quality objective are provided in the paragraphs below the table.

Data Quality Objectives for PAR measurements

Table 5: Data Quality Objectives for in-situ PAR measurements

Data Quality Indicators	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance
Precision-Overall	SE < 10%	Field Duplicates
Precision-Lab	Not applicable	
Accuracy/Bias	R^2 of correlation >0.95 At least 6 measurements per profile	Regression of ln(PAR) vs. depth. Number of measurements per profile.
Comparability	Measurements should follow standard methods that are repeatable	NA
Sensitivity	Not expected to be an issue for this project (see discussion below)	NA
Data Completeness	80 samples meeting data quality objectives	Data Completeness Check

Based on EPA-NE QAPP Workbook for 3/19/02 DES QAPP writing class.

Precision: Fifteen measurements of PAR (>10% of total) will be replicated three times. The standard error (SE) of the mean light attenuation coefficient value from the three casts will be used to assess the precision of the result. SE values <10% will be acceptable. Casts with SE values >10% will be rejected.

Accuracy/Bias. For PAR measurements, absolute accuracy measurements are not necessary. The light attenuation coefficient is calculated based on the relative change in light with depth. Therefore, the quality of the regressions with depth, not the absolute light intensity, is the measurement of concern. The quality of the regressions will be governed by two criteria. First, each profile will be required to have at least 6 PAR measurements. Second, regressions of light versus depth will be required to have r^2 values of >0.95.

Representativeness: The samples should be taken at the same locations and times as water quality samples for the existing water quality monitoring programs in order to ensure that the data are representative of the same water mass as was monitored for the other parameters.

Comparability: Standardized field and analytical methods should be used. These methods should follow the current industry standard for the types of measurements being taken. Written SOPs should be followed for field and analytical measurements. Standardized field data sheets should be used.

Sensitivity. In general, diffuse light attenuation coefficients for Great Bay should be between –0.02 and –12. Results outside of this range will be flagged for investigation.

Completeness: This study will be deemed successful if data meeting the data quality objectives is obtained for 80 water quality samples (not including field/laboratory duplicates).

A8 – Special Training/Certification

The Project Manager will organize and implement a training session for field staff. The training session will cover SOPs for field instruments and field data sheets. The training will be based on the QA Project Plan document. Field staff will sign an attendance sheet for the training, which will be retained by the Project Manager and included in the final report. The training will be completed before sampling begins.

Table 6. Special Personnel Training Requirements

Project function	Description of Training	Training Provided by	Training Provided to	Location of Training Records
Water quality sampling and field measurements	Field method SOPs and field data sheets. This training will be conducted once at the beginning of the field season.	Project Manager	All field team staff	With Project Manager and included in final report to NHEP.

Based on EPA-NE Worksheet #7.

A9 – Documents and Records

QA Project Plan

The Project Manager will be responsible for maintaining the approved QA Project Plan and for distributing the latest version to all parties on the distribution list in section A3. A copy of the approved plan will be on file with the NHEP/NHDES Project Manager Coastal Scientist at the DES offices, 6 Hazen Drive, Concord NH.

Field Data Sheets

The field data sheets for this project are attached as Appendix G. Field crews fill in these forms during the day and return them to the Project Manager upon completion. The information will be transferred to an Excel Spreadsheet. The original forms will be retained on file by the Project Manager.

Laboratory Data Sheets

Data packages from the laboratory to the Project Manager will be hardcopy laboratory data sheets containing the results of analyses plus the results of QC tests performed. See Appendix A Section VI for details of laboratory electronic and paper records.

Reports to Management

The Project Manager will provide a final report to the NHEP/NHDES Project Officer. The final work product will be an Excel spreadsheet containing quality assured results of the DON/PON analyses and light extinction coefficients for each station on each date and a final report describing any deviations from the protocols established in the QA Project Plan. The final report is due on 12/31/03.

Archiving

The QA Project Plan and final report will be kept on file with the NHEP/NHDES Project Officer at DES in Concord for a minimum of 10 years after the publication date of the final report. The original field and laboratory data sheets will be retained by the Project Manager for a minimum of 10 years.

B1 – Sampling Process Design

This sample design described in this section reflects the original project scope of work in the contract between UNH and NHEP. Sampling began in March 2003 as specified by the contract even though UNH had not prepared a QAPP for the project as required by the contract. This QAPP will cover the samples collected and analyzed starting in August 2003 (the anticipated date of QAPP approval is 8/1/03). UNH understands that the work undertaken prior to QAPP approval is done at risk and could be disqualified. The number of samples listed in Table 7 and Table 8 reflects the total number of samples that UNH will collect for the project in order to be consistent with the contract agreements.

UNH will collect water samples for DON/PON analyses and measure PAR profiles in the field up to 125 times during the 2003 field season. Both the DON/PON analyses and the PAR profiles are critical for the study. The sample breakdown will be:

- Monthly (Mar-Dec) samples at low tide at 9 estuarine stations (all stations except Adams Point).
- Monthly (Mar-Dec) samples at both low and high tide at one estuarine station (Adams Point).
- At least 15 field duplicate samples over the year (every 10^{th} sample).

Table 7 and Table 8 summarize the sampling program. Figure 2 illustrates the locations of the stations.

Table 7: Sampling stations

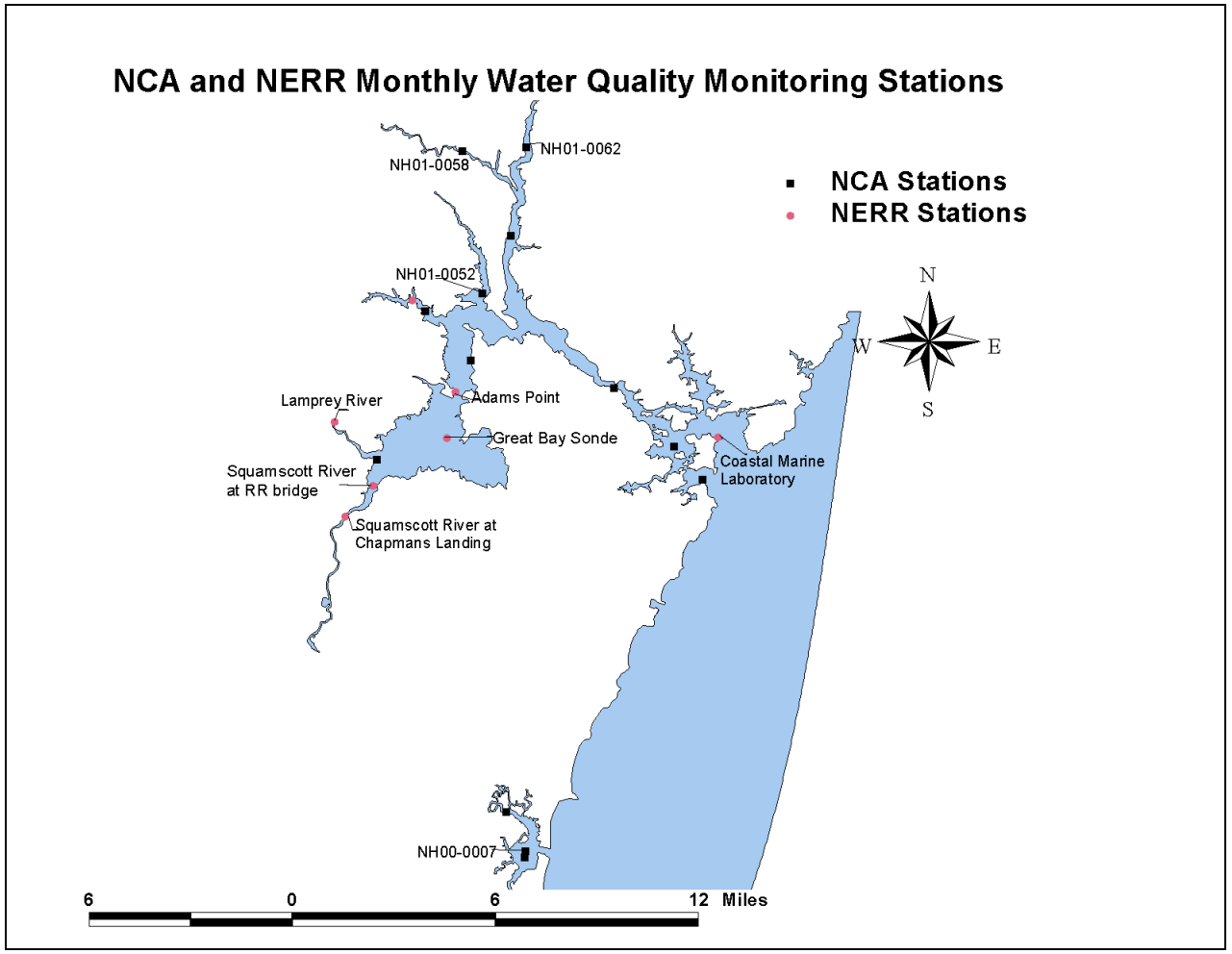
Field Team	Station No.	Latitude	Longitude	Comments
UNH	Adams Point	43° 05' 31"	-70° 51' 51"	High and low tide samples collected at this station
UNH	Great Bay Sonde	43° 04' 20"	-70° 52' 10"	
UNH	Lamprey River	43° 04' 47"	-70° 56' 05"	
UNH	Squamscott River at Chapmans Landing	43° 02' 22"	-70° 55' 44"	
UNH	Squamscott River at RR Bridge	43° 02' 30"	-70° 55' 20"	
UNH	Coastal Marine Laboratory	43° 04' 17"	-70° 42' 40"	Only DON and PON measured at this station
UNH	NH01-0058 Cocheco River	43° 11.71	-70° 51.48	Only DON and PON measured at this station
UNH	NH01-0062 Salmon Falls River	43° 11.81	-70° 49.24	Only DON and PON measured at this station
UNH	NH01-0052 Bellamy River	43° 08.01	-70° 50.82	Only DON and PON measured at this station
UNH	NH00-0007 Hampton Harbor	42° 53.73	-70° 49.71	Only DON and PON measured at this station

Table 8: Sampling design

Parameter	No. of sampling locations	Samples per event per site	Number of sampling events	Number of field duplicates	Number of bottle blanks	Total number to lab
To be analyzed at the UNH lab						
DON and PON	11 (counting Adams Pt twice because it is sampled at high and low tide)	1 sample/ site/event	10 events/yr	>10% (15/yr)	0	125
Measured in the field						
PAR	6 (counting Adams Pt twice because it is sampled at high and low tide)	1sample/ site/event	10 events/yr	>10% (15/yr)	Not applicable	measured *in situ*

Based on EPA-NE Worksheet #9c.

Figure 2: NCA and NERR Monthly Water Quality Monitoring Stations



Note: Only the labeled stations in the figure are part of this monitoring program.

B2 – Sampling Methods

Field samples are collected by boat at all the stations except the Coastal Marine Laboratory. The Coastal Marine Laboratory samples are collected from a fixed pier.

The sample bottle preparation/decontamination and field sampling procedures used for the DON/PON sample collection are listed below.

Sample Bottle Preparation: One-liter Nalgene bottles are prepared before sampling by soaking bottles and caps in a 10% HCl solution for 10 minutes. Bottles and caps are subsequently rinsed with deionized water three times then dried thoroughly before being stored. Before field sampling day, bottles are labeled with appropriate site and placed in a cooler for transfer and storage.
Water Sampling Field Procedures: At each site except the Coastal Marine Lab Pier, one sample bottle is immersed by hand approximately 1 foot below the surface and filled facing the direction of the current (if any current pattern is detected). The bottle is opened individually and rinsed three times with estuarine water before collecting the sample. At the Coastal Marine Lab Pier site, samples are collected by hand using a Wildco alpha bottle (horizontal sampler) lowered to approximately 1 foot below the surface. The Wildo sampler is rinsed multiple times with estuarine water and then the sample is collected. The sample bottle and cap are rinsed three times with sample water from the alpha bottle and then filled from the alpha bottle.
Filtration: Particulate material is separated from dissolved constituents via filtration in the laboratory immediately upon delivery to the laboratory (normally within 5 hours of collection). For dissolved nitrogen species (NO3+NO2, TDN, and Ammonia), a portion of the original sample (at least 180 mL) is filtered through 47mm Whatman GF/C glass fiber filters (nominal pore size of 1.2 µm), collected in a pre-washed HDPE bottle, and then immediately frozen. For particulate nitrogen species (PON), a portion of the original sample (at least 15 mL) is processed using the filtration procedures in Appendix D with a 0.45 µm filter.

The SOP for PAR measurements *in situ* is in Appendix F. No special decontamination procedures are needed for the PAR measurements. Field teams are responsible for reporting sampling method problems to the Project Manager who is reponsible for taking corrective action.

Table 9. Sample Requirements

Analytical parameter	Collection method	Sampling SOP	Sample volume	Container size and type	Preservation requirements	Max. holding time (preparation and analysis)
Nitrate+nitrite (NO_3+NO_2)	Grab	Section B2	60 mL	1000 mL HDPE bottle	Freeze within 8 hours of sample collection	Indefinite once frozen
TDN	Grab	Section B2	60 mL	1000 mL HDPE bottle	Freeze within 8 hours of sample collection	Indefinite once frozen
Ammonia	Grab	Section B2	60 mL	1000 mL HDPE bottle	Freeze within 8 hours of sample collection	Indefinite once frozen
Particulates for PON	Grab	Section B2	60 mL	1000 mL HDPE bottle	Freeze within 8 hours of sample collection	Indefinite once frozen
PAR	measured in-situ	Appendix F	NA	NA	NA	NA

B3 – Sample Handling and Custody

Sample handling and custody procedures for nutrient samples are described in Section III of Appendix A. The Project Manager will be responsible for having the samples delivered to the laboratory within 8 hours of collection so that they can be frozen.

B4 – Analytical Methods

Attachment A is the QA Plan for the UNH Water Quality Analysis Laboratory. This document describes the general SOPs for the laboratory. This QA plan has been included with other QAPPs that have been approved by EPA Region I.

Analytical methods for this study are described in detail in Appendices B, C, D, and E. Appendix B contains the Lachat SOPs for determining nitrate+nitrite concentrations and ammonia concentrations. Appendix C contains the SOP for total dissolved nitrogen concentrations. Dissolved organic nitrogen (DON) concentrations will be calculated from the total dissolved nitrogen (TDN), nitrate+nitrite (NO3/NO2), and ammonia (NH4) measurements as follows:

$$DON = TDN - (NO3 + NO2) - NH4$$

Appendix D contains the protocol for filtering samples to capture particulates. Appendix E contains the protocol for the CHN analysis of the filters (mass of carbon, hydrogen, and nitrogen by mass spectrophotometry) to determine the mass of nitrogen that was retained on the filter. PON will be calculated from these two measurements as follows:

PON = Mass N on filter (mg) / Volume of water filtered (l)

The Laboratory QA Officer is responsible for corrective actions if any problems with the analytical methods arise. Laboratory data reports are expected at least quarterly. All data for the project must be delivered from the laboratory to the Project Manager by March 31, 2004.

Attachment F contains the protocols for calculating light attenuation coefficients from the field measurements of PAR. The field teams are responsible for notifying the Project Manager of any problems with the PAR measurement. The Project Manager is responsible for taking corrective actions to resolve these problems. PAR measurments are made in situ so turn-around times for data are not relevant.

B5 – Quality Control

Section VII of Appendix A describes the quality control measures that will be used for nutrient analyses by the UNH Water Quality Analysis Laboratory. For the PAR monitoring, the field duplicate measurements (every 10^{th} measurement) will serve as the quality control. Section A7 describes how the data quality objectives will be evaluated.

The Project Manager will verify that the field crews are following the protocols correctly during the field sampling audit (see Section C1).

Databases of results will be checked for transcription errors and bad data using two methods. First, the entire data set will be printed and checked against the entries in each field or laboratory data sheet by the Project QA Officer. Second, the Project QA Officer will construct box-plots and other graphical tools (such as residual plots) to determine if there are outliers in the data set. The Project QA Officer will report any outliers to the Project Manager, who will determine whether these data should remain in the dataset.

B6 – Instrument/Equipment Testing, Inspection, Maintenance

Equipment inspections and maintenance schedules for the laboratory are described in Section IX of Appendix A. PAR measurements will be made using a Li-Cor 1400 datalogger and spherical (2-pi) quantum sensors. The instrument will be inspected before each use following the SOP in Appendix F.

B7 – Instrument/Equipment Calibration and Frequency

Equipment calibration procedures for the laboratory are listed in Section V of Appendix A. Calibration runs are stored in the laboratory database along with the run sheets for environmental samples. The PAR sensor will be calibrated every two years per the manufacturers recommendation. However, the calibration is not critical because only the relative light intensities (not their absolute values) are used to determine the light attenuation within the water. Calibration records will be retained by the Project Manager for a minimum of 10 years.

B8 – Inspection/Acceptance Requirements for Supplies and Consumables

Inspection schedules for consumables are listed in Section V of Appendix A. The PAR sensor does not require supplies or consumables.

B9 – Non-direct Measurements

Not applicable. No non-direct measurements will be used for this project.

B10 – Data Management

Field data will be recorded on standard field data sheets (see Appendix G) and transferred to Excel data files. Laboratory data will be transferred from laboratory data sheets to Excel spreadsheets. All data will be stored electronically in Excel spreadsheets which will be transferred to the NHEP/NHDES Project Officer as part of the final report. The NHEP/NHDES Project Officer will be responsible for uploading the data to the NHDES Water Quality Database (STORET compatible). Management of hardcopy data and documents is described in Section A9.

C1 – Assessments and Response Actions

In order to confirm that field sampling, field analysis and laboratory activities are occurring as planned, the Project Manager, field staff, and laboratory personnel shall meet, after the first sampling event, to discuss the methods being employed and to review the quality assurance samples. At this time all concerns regarding the sampling protocols and analysis techniques shall be addressed and any changes deemed necessary shall be made to ensure consistency and quality of subsequent sampling. The Project Manager will have the authority to resolve any problems encountered. Assessment frequencies and responsible personnel are shown in the following table.

Table 10. Project Assessment Table

Assessment Type	Frequency	Person responsible for performing assessment	Person responsible for responding to assessment findings	Person responsible for monitoring effectiveness of corrective actions
Field sampling audit	Once after first sampling day	Project Manager	Project Manager	Project Manager
Field analytical audit	Once after first sampling day	Project Manager	Project Manager	Project Manager
UNH laboratory audit	Quarterly (see Section VIII of Appendix A)	Laboratory Manager	Laboratory Manager	Laboratory Manager

Based on EPA-NE Worksheet #27b.

C2 – Reports to Management

The Project Manager will provide a final report to the NHEP/NHDES Project Officer. The final work product will be an Excel spreadsheet containing quality assured results of the DON/PON analyses and light extinction coefficients for each station on each date and a final report describing any deviations from the protocols established in the QA Project Plan. The final report is due on 12/31/03. Data from the final report will be published biennially in NHEP Environmental Indicator Reports (available on internet).

D1 – Data Review, Verification and Validation

The Project QA Officer will be responsible for a memorandum to the Program Manager summarizing any deviations from the procedures in the QA Project Plan and the results of the QA/QC tests. The Project QA Officer will review all field data sheets and final computer data files for completeness and quality based on the criteria described in Section A7. The Project QA Officer will also *affirmatively* verify that the methods used for the study followed the procedures outlined in this QA Project Plan. If questionable entries or data are encountered during the review process (see methods in Section B5), the Project QA Officer will contact the appropriate personnel to determine their validity.

D2 – Verification and Validation Procedures

The Project Manager will review the memorandum from the QA Officer to see if there have been deviations from the QA Project Plan. Any decisions made regarding the usability of the data will be left to the Project Manager, however the Project Manager may consult with project personnel, the NHEP Project Manager, or with personnel from EPA-NE, if necessary.

D3 – Reconciliation with User Requirements

The Project Manager will be responsible for reconciling the results from this study with the ultimate use of the data. Results that are qualified by the Project QA Officer may still be used if the limitations of the data are clearly reported to decision-makers. Data for this project are being collected as part of a long-term monitoring program. It is not possible to repeat sampling events without disrupting the time series. Therefore, the Project Manager will:

1. Review data with respect to sampling design.

2. Review the Data Verification and Validation reports from the Project QA Officer.

3. If the data quality objectives from Section A7 are met, the user requirements have been met. If the data quality objectives have not been met, corrective action as discussed in D2 will be established by the Project Manager.

4. Draw conclusions from the data.

References

NHEP (2002) Evaluation of Monitoring Programs for the NHEP Monitoring Plan. NH Estuaries Project, Office of State Planning, Portsmouth, NH. September 27, 2002.

EPA (2001) Nutrient Criteria Technical Guidance Manual: Estuarine and Coastal Marine Waters. EPA-822-B-01-003. U.S. Environmental Protection Agency, Office of Water, Washington DC. October 2001.

Appendix A

Quality Assurance Plan
UNH Water Quality Analysis Laboratory

QAPP for the Water Quality Analysis Lab at the University of New Hampshire, Department of Natural Resources, Durham, NH.

I. Laboratory Organization and Responsibility

Dr. William H. McDowell - Director

Jeffrey Merriam – Lab Manager/QA manager. Mr. Merriam supervises all activities in the lab. His responsibilities include data processing and review (QA review), database management, protocol development and upkeep, training of new users, instrument maintenance and repair, and sample analysis.

Jody Potter – Lab Technician. Mr. Potter's responsibilities include sample analysis, logging of incoming samples, sample preparation (filtering when appropriate), daily instrument inspection and minor maintenance.

All analyses are completed by Jody Potter or Jeffrey Merriam, and all data from each sample analysis batch (generally 40-55 samples) is reviewed by Jeffrey Merriam for QC compliance. All users are trained by the lab manager and must demonstrate (through close supervision and inspection) proficiency with the analytical instrumentation used and required laboratory procedures.

II. Standard Operating Procedures

Standard Operating Procedures for all instruments and methods are kept in a 3-ring binder in the laboratory, and are stored electronically on the Lab manager's computer. The electronic versions are password protected. SOPs are reviewed annually, or as changes are required due to new instrumentation or method development.

III. Field Sampling Protocols

Sample collection procedures are generally left up to the sample originators, however we recommend the guidelines described below, and provide our field filtering protocol on request.

All samples are filtered in the field through 0.7 um precombusted (5+ hours at 450 C) glass fiber filters (e.g. Whatman GF/F). Samples are collected in acid-washed 60-mL HDPE bottles. We prefer plastic to glass as our preservative technique is to freeze. Sample containers are rinsed 3 times with filtered sample, and the bottle is filled with filtered sample. Samples are stored in the dark and as cool as possible until they can be frozen. Samples must be frozen within 8 hours of sample collection. Once frozen, samples can be stored indefinitely (Avanzino and Kennedy, 1993), although they are typically analyzed within a few months.

After collection and freezing, samples are either hand delivered to the lab, or are shipped via an over-night carrier. Samples arriving in the lab are inspected for frozen contents, broken caps, cracked bottles, illegible labels, etc. Any pertinent information is entered into a password protected database (MS Access).

We do not require chain of custody paperwork unless a specific project requires it. If a project requires chain of custody, forms are provided by the specific project's manager.

IV. Laboratory Sample Handling Procedures

Samples are given a unique 5-digit code. This code and sample information including name, collection date, time (if applicable), project name, collector, logger, the

date received at the WQAL, sample type (e.g. groundwater, surface water, soil solution) and any other miscellaneous information, are entered into a password protected database. From this point through the completion of all analyses, we use the log number to track samples. Log numbers are used on sample run queues, spreadsheets, and when importing concentrations and run information into the database

After samples are logged into the WQAL, they are stored frozen in dedicated sample freezers located in the laboratory. Samples from different projects are kept separated in cardboard box-tops, or in plastic bags. Samples that may pose a contamination threat (based on the source or presumed concentration range) are further isolated by multiple plastic bags, or isolation in separate freezer space. This is typically not an issue as we primarily deal with uncontaminated samples.

We do not pay special attention to holding time of samples, as frozen samples are stable indefinitely (Avanzino and Kennedy, 1993). However, we do keep track of the date samples arrive at the WQAL, and can report holding times if necessary. After samples are analyzed they are returned to the project's manager for safe keeping or they are held for a period of time at the WQAL to allow necessary review and analysis of the data by the interested parties (not from a laboratory QC sense, but from a project specific viewpoint). Once the data is analyzed by the project's manager(s), the samples are returned or disposed of, based on the preference of the project's manager.

Chain of custody is only implemented when required by a specific project. This is usually only when it's required by the funding agency, or if the samples could be the basis for an enforcement action.

Samples that arrive unfrozen, with cracked bottles/caps, or with loose caps, are noted in the database and are not analyzed. These samples are disposed of to prevent accidental analysis. The sample originator is notified (generally via e-mail) of which samples were removed from the sample analysis stream. Similarly, if while in the possession of the WQAL, a sample bottle is broken or improperly stored (e.g. not frozen), the sample is removed and the sample originator is notified.

V. Calibration procedures for chemistry

Calibration curves are generally linear, and are made up of 4-7 points. A full calibration is performed at the beginning of each run (a run is generally 40-60 samples) with a reduced calibration (3-5 points) performed at the end of the run. Occasionally calibration data is best fit with a quadratic equation, and this is used if it best describes the data within a specific run.

Standards are made from reagent grade chemicals (typically JT Baker) that have been dried and are stored in a dessicator. Working stock solutions are labeled with the content description, concentration, initials of the maker, and the date the stock solution was made. Generally stock solutions are kept less than one week; however some stocks (Br, Na, Cl, C for DOC) can be stored for several months. Standard solutions are kept for less than one week from the date they were made. Stocks and standards are stored tightly covered, in a dark refrigerator.

Control charts are prepared and printed every few months. However data from each run are looked at within days of analyses. Calibration curves, Laboratory Duplicates, Lab Fortified Blanks (LFB), Lab Fortified Sample Matrices (LFM) and Lab

Reagent Blanks (LRB) are reviewed and are checked against known concentrations (where applicable) to ensure QC criteria are met for each run of samples.

VI. Data Reduction, validation, reporting and verification

Data reduction and validation are performed in a spreadsheet (MS Excel). The Raw data page of the spreadsheet lists the date of analysis, user, analysis performed, project, any issues or problems noted with the instrument on that date, and the sample queue and the raw data exported from the instruments. Most raw data is exported as an area or an absorbance value. A second page (typically named "Calculations") is added to the spreadsheet where known concentrations of standards, check standards and reference solutions are added. The calibration curve(s) is calculated and the concentrations are calculated on this page. Calculated concentrations for all standards, LFB, LFM and IPC are compared to the "known" or prepared values. If these are acceptably close (+/- 10% of the "known") no further changes to the calculated concentrations are made. If there is evidence of drift in the response of the instrument during a run, we try to correct for the drift using the responses from the front end calibration curve and the set of standards analyzed at the end of the run. All reference solutions and replicates must meet certain QC criteria (described below) for a run to be accepted.

Data are then exported to the WQAL database. Exported information includes the unique 5-digit code, calculated concentration, the analysis date, the user, the filename the raw data and calculations are saved in, and any notes from the run regarding the specific sample. Data are sent to sample originators upon completion of all requested sample analyses and following review by the WQAL lab manager. Generally the data include

the 5-digit code, the sample name, collection date, and concentrations, in row-column format. Any information entered into the database can be included upon request. Data transfer is typically via e-mail or electronic medium (CD or floppy disk).

All data corrections are handled by the lab manager. Corrections to data already entered into the database are very infrequent. Typically they involve reanalysis of a sample. In this case, the old data is deleted from the database, and the new value is imported, along with a note indicating that it was re-analyzed, the dates of initial and secondary analysis and the reason for the correction.

Hand written or computer printed run sheets are saved for each run and filed, based on the project and the analysis. Spreadsheet files with raw data and calculations are stored electronically by analysis and date. Information in the database allows easy cross-reference and access from individual samples to the raw data and the runsheets. This provides a complete data trail from sample log-in to completion of analysis.

VII. Quality Control

All analyses conducted at the WQAL follow approved or widely accepted methods (Table 1).

Quality Control Samples (QCS) (from Ultra Scientific) are analyzed periodically (approximately every 20 samples) in each sample analysis batch to assure accuracy. The response/unit concentration is also used to monitor day-to-day variation in instrument performance. A difference from the certified concentration of more than 10% requires further investigation of that run. A difference greater than 15% is failure (unless the average of the two samples is less than 10X the MDL), and results in re-analysis of the

entire sample queue, unless there is a very reasonable and supported explanation for the inconsistency. Table 2 lists historical average % recoveries. At least 2 QCS are analyzed on each run.

Standards and reagents are prepared from reagent grade chemicals (typically JT Baker) or from pre-made stock solutions. All glassware is acid washed (10% HCl) and rinsed 6 times with ultra pure-low DOC water (18.2 mega-ohm). All analyses (except CHN) use multi-point calibration curves (4-7) points, which are analyzed at the beginning and the end of each run. A Laboratory Reagent Blank (LRB), Laboratory Fortified Blank (LFB) (a standard run as a sample) and Laboratory Duplicate are analyzed every 10 to 15 samples during each run. At least one Laboratory Fortified Sample Matrix (LFM) is analyzed during each run to insure that sample matrices do not affect method analysis efficiency. Field Duplicates are not required by our lab, and are the responsibility of the specific project's manager.

Laboratory Duplicates must fall within 15% relative percent difference (RPD = abs(dup1-dup2)/average of dup1 and dup 2). A difference greater than 10% requires further investigation of the sample run. A difference greater than 15% is failure (unless the average of the two samples is less than 10X the MDL), and results in re-analysis of the entire sample queue, unless there is a very reasonable and supported explanation for the inconsistency. Long-term averages for relative % difference are included in Table 2.

LFM must show 85% to 115% recovery. A recovery <90% or > 110% requires further investigation of the sample run. A recovery <85% or >115% is failure (unless the sample is less than 10X the MDL), and results in re-analysis of the entire sample queue,

unless there is a very reasonable and supported explanation for the inconsistency. Long-term averages for % recovery are included in Table 2.

Method Detection Limits are calculated at least twice per year, or whenever major changes to instrumentation or methods occur. Table 2 lists most recently measured MDL values.

VIII. Schedule of Internal Audits

Internal audits are not routinely performed, however, review of QC charts, and tables are done at least quarterly by the lab manager.

IX. Preventive maintenance procedures and schedules

The laboratory manager, Jeff Merriam, has 10 years of experience and is highly experienced with all laboratory equipment used within the WQAL. The laboratory manager conducts all maintenance and inspection of equipment based on manufacturer requirements and specifications.

Each day an instrument is used, it receives a general inspection for obvious problems (e.g. worn tubing, syringe plunger tips, leaks). The instruments are used frequently and data is inspected within a few days of sample analysis. This allows instrument (or user) malfunctions to be caught quickly, and corrected as needed.

Each day's run is recorded in the instrument's run log, with the date, the user, the number of injections (standards, samples, and QC samples), the project, and other notes of interests. Maintenance, routine or otherwise, is recorded in the instrument run log, and

includes the date, the person doing the maintenance, what was fixed, and any other notes of interest.

X. Corrective Action Contingencies

Jeffrey Merriam is responsible for all QC checks and performs or supervises all maintenance and troubleshooting. When unacceptable results are obtained (based on within sample analysis batch QC checks) the data from the run are NOT imported into the database. The cause of the problem is determined and corrected, and the samples are re-analyzed. Problems are recorded in the sample queue's data spreadsheet, or on the handwritten runsheet associated with the run. Corrective actions (instrument maintenance and troubleshooting) are documented in each instrument's run log.

XI. Record Keeping Procedures

Protocols, Instrument Logs, QC charts, databases and all raw data files are kept on the lab manager's computer. These are backed up weekly, with the back up stored off site. The computer is password protected, and is only used by the lab manager. Protocols and the sample database are also password protected. Handwritten run sheets are stored in a filing cabinet in the lab. Instrument run and maintenance logs are combined with the QC data to form one large Excel file where instrument performance can easily be compared to instrument repair and the number of analyses, etc. This file is also stored on the lab manager's computer and is password protected.

All information pertinent to a sample is stored in the sample database. From this database we can easily determine the date of analysis and the location of the raw data file

if further review is necessary. The amount of information provided to sample originators is dependent on what is required by the project or funding agencies.

Table 1. List of standard operating procedures and description of analyses done at the Water Quality Analysis Laboratory.

Standard Operating Procedure	Analysis	Instrument Used	Description	Protocol Latest Revision	EPA method or other reference
Ion Chromatography Protocol for Anions and Cations Protocol	Anions and Cations	Ion Chromatograph Ion Chromatograph	Anions via ion chromatography w/ suppressed conductivity. Cations via ion chromatography and conductivity	June 11, 2002	Anions EPA #300.1
Dissolved Organic Carbon Protocol	DOC	Shimadzu TOC 5000 with autosampler	High Temperature Catalytic Oxidation (HTCO)	June 25, 2002	EPA 415.1
Total Dissolved Nitrogen Protocol	TDN	Shimadzu TOC 5000 coupled with an Antek 720 N detector	HTCO with chemiluminescent N detection	June 25, 2002	Merriam et al, 1996
DOC and TDN combined Protocol	DOC and TDN	Shimadzu TOC-V with TNM nitrogen module	HTCO with chemiluminescent N detection	June 25, 2002	EPA 415.1 and Merriam et al, 1996
Lachat QuikChem AE Protocol	Nitrate/Nitrite colorimetric NO_3/NO_2	Lachat QuikChem AE	Automated Cd-Cu reduction	June 25, 2002	EPA 353.2
	Ammonium colorimetric NH_4	Lachat QuikChem AE	Automated Phenate	June 25, 2002	EPA 350.1
	Soluble reactive Phosphorous colorimetric PO_4	Lachat QuikChem AE	Automated Ascorbic acid	June 25, 2002	EPA 365
Acid Washing Protocol	Glass and plastic-ware cleaning		10% HCl rinse and 6 rinses with DDW	June 25, 2002	
Field Filtering Protocol	Sample prep		3-times rinse with filtered sample	June 25, 2002	

Table 2. Detection limits, acceptable ranges, and recent historical averages for QC samples at the Water Quality Analysis Lab.
[1] Detection limit based on user experience and previous analysis (not statistically calculated). [2] Method Detection Limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

Analyte	Units	Typical Range	Regression Type	# of Cal. Points	Detection Limit [1]	MDL [2]	Lab Duplicate % Relative Difference	Limit	LFM % recovery	Limit +/-	IPC % recovery	Limit +/-
SiO_2	mg SiO2/L	0 – 40	Linear	4-7	0.3		3.5	15.0	92.8	15.0		
PO_4	µg P/L	0 – 200	Linear	4-7	2 – 3	1.5	7.8	15.0	95.5	15.0	93.7	15.0
NH_4	µg N/L	0 – 200	Linear	4-7	2 – 3	1.5	7.1	15.0	103.9	15.0	95.0	15.0
NO_3 FIA	mg N/L	0 – 10	Linear	4-7	0.05	0.003	4.6	15.0	100.9	15.0	102.6	15.0
Na^+	mg Na/L	0 – 15	Quadratic	4-7	0.1		0.9	15.0			112.7	
K^+	mg K/L	0 – 7	Quadratic	4-7	0.05		10.4	15.0			97.8	
Mg^{2+}	mg Mg/L	0 – 7	Quadratic	4-7	0.1		4.5	15.0			89.7	
Ca^{2+}	mg Ca/L	0 – 10	Quadratic	4-7	0.1		4.0	15.0			98.2	
Cl^-	mg Cl/L	0 – 15	Quadratic	4-7	0.2	0.02	1.6	15.0			92.7	
NO_3^-	mg N/L	0 – 3	Quadratic	4-7	0.002	0.002	0.3	15.0			96.3	
SO_4^{2-}	mg S/L	0 – 8	Quadratic	4-7	0.1	0.04	2.2	15.0			86.5	
TDN	mg N/L	0 – 10	Linear	4-7	0.1	0.029	7.8	15.0	100.3	15.0	102.1	15.0
DOC	mg C/L	0 – 20	Linear	4-7	0.1	0.048	4.9	15.0	100.5	15.0	97.0	15.0

References

Avanzino R.J. and V.C. Kennedy, 1993. Long-term frozen storage of stream water samples for dissolved orthophosphate, nitrate plus nitrite, and ammonia analysis. *Water Resources Research*, 29(10) 3357-3362.

Merriam, J.L, W.H. McDowell, W.S. Currie, 1996. A high-temperature catalytic oxidation technique for determining total dissolved nitrogen. *Soil Science Society of America Journal*, 60(4) 1050-1055.

Appendix B

Standard Operating Procedures for Lachat Analyses
UNH Water Quality Analysis Laboratory

Lachat Protocol

Instrument overview

All Lachat methods that we use work by measuring a color development brought about by specific reagents. The amount of color is measured via absorbance, which is proportional to the amount of analyte present.

The system is made up of several components. These are described in order from left to right as you look at the instrument.

Autosampler

Holds standards and samples for analysis and has a sampling needle which alternates between being in the sample and being in the rinse vessel. There are 12 lettered vial positions (A – L). These hold the standards in large glass vials. This allows for multiple injections from each standard (typically 2). There are 96 numbered vial positions for samples, blanks, check standards, reference samples, etc.

The sample holders can be removed from autosampler for sample loading. Please do not remove more than three of these holders at one time, as this can cause the autosampler to jam, and requires painful effort to repair.

Pump

A peristaltic pump used to move reagents and sample from their respective reservoirs to the manifold. Pump speed should be 35. Pump tubing should be changed daily to prevent troublesome flow problems. Each pump tube is color coded, indicating its diameter, and thus its flow rate. The manifold diagram found in the specific method you are using will tell you specifically which color to use. The Sample loop, which connects the sampling needle to the manifold should be cut so that 1 inch extends beyond the colored collar on both sides. This ensures accurate timing of sample delivery to the manifold.

Valve

The valve switches sample to and from the sample loop and rinses the loop with carrier. It has six ports. Port 1 and 4 are connected by the sample loop, which supplies a constant volume of sample to be analyzed. The length of the sample loop is specified on the manifold diagram. Port 5 goes to waste from the last channel being used. If you are using two channels, Port 5 on the first channel connects to Port 6 on the second channel. The carrier line connects to Port 2. Port 3 attaches to the manifold.

Manifold

Contains a series of coils, tubes and fittings where the reagents and sample mix and react. There is a heating coil under the manifold that is used when specified in the method. These fittings should be checked periodically to ensure that there are no clogs.

Detector Head

At the end of the manifold is a blue box, which contains the flow cell, the wavelength filter, light source and detector. Be sure that you've installed the correct wavelength filter for the method you are using.

Standard Operating Procedures

1. Select the proper method for the analysis and the sample range you are interested in. Methods are located in two 3-ring binders on the shelf in the lab. The early pages of each method describe the principles of the reaction involved in the analysis, as well as the recipes for the reagents used. There is a manifold diagram (usually toward the end of the method) that describes how the manifold should be set up for each analysis. This description includes the tubing to be used, the length of the sample loop, the wavelength filter, as well as what temperature the heater is set to if needed.

2. Prepare reagents as described in the recipes in the method. Be sure to mix ingredients in the order described as this can affect the outcome of the reagent. Also, pay careful attention to the amounts of acids and bases used in the reagents because many of the color reactions involved in the analyses are pH dependent.

3. Prepare standards as necessary. If you are performing two analyses at once (i.e. running two channels), you should make your standards together (mixed standards). For example, if you were running PO_4 and NH_4, you should have one standard set that contained both NH_4 and PO_4. If you suspect contamination from one standard to the other (i.e. the NH_4Cl used for the NH_4 standard has PO_4 in it), you can make separate standard sets for each analyte. This is normally not an issue.

4. Set up the manifold as specified in the method. Manifolds are found in the cabinet under the lab bench.
   a. Install the correct wavelength filter.
   b. Change pump tubing to appropriate color tubing. Do this daily. Consult the manifold diagram. Be sure the pump tubing is pushed all the way over the nipple on the union before screwing on the fitting. If your fingers don't hurt a little, you probably haven't done it correctly.
   c. Check the manifold for pinched/kinked tubing, plugged unions and T-fittings and unattached tubing. This doesn't have to be done everyday, but taking the 10 minutes required to do this can save you several painful hours troubleshooting later.
   d. Check to make sure the manifold is set up properly by comparing the manifold diagram with the manifold. The sequence that the reagents and samples mix is important and specific for each analysis.

5. If you are generating a hazardous waste, make sure you have enough waste containers on hand before beginning analysis. You will easily go through one or more waste bottles per day. Bottles can be picked up at the Chemistry Stock room in Parsons Hall. See Hazardous Waste protocols for more information on this. The following analyses generate hazardous Waste.

   Ammonium Analysis
   Ortho P and Total P Analysis
   Nitrate/Nitrite Analysis
   Silica

6. Turn the system on by turning the power-strip behind the Lachat on.
7. Stretch pump tubes across the pump cartridge so that the collars are extended beyond the outside edge of the cartridge. Press one side of the cartridge down, and then the other down tightly until you here a light snap, as the cartridge is locked into place. Do this for all the tubes.
8. Turn the pump on, and pump DDW through the manifold and out to waste. Look for any leaks, or pulsing lines. Pulsing indicates some kind of a clog or plug. Fix any flow problems.
9. Check the screen on the Lachat System Unit and note the Diagnostic section on the lower right-hand corner. The instrument reports if there are any problems with the Valves, Detector (alpha only), Reference and Sampler. These should all be "ok" or "OK". Otherwise consult the lab manager.

Computer software use. See attached software flow chart for important components. When you power up the system, the computer should boot into the Lachat software Main Menu. The following describes the different components of the software.

I. **Methods** – Allows you to select a method to load into memory, or to modify or create a method

1. Analysis Select & Download – If the method does not require modification, use this to select and download. You will be put into the **Samples** submenu.
2. Method Definition – Allows you to modify an existing method, or create a new one that meets your analytical needs.
   A. ***File*** – Pick, Save, New, Delete or Quit. Choose the method you want to edit, create, or save, delete a method, or quit from this section.
   B. ***Description*** – Name or Brief notes about the method you selected in the ***File*** menu.
   C. ***Channels*** – Select a channel to modify, add or delete. When you select a channel, you will enter the *Channel Specific Method Definition* (Sheet I.2.C.) screen.
      1. *Info*
         a. *Name of Channel* –
         b. *QuikChem Method Number* – Found on the method sheet used to make reagents.
         c. *Detector Selection* – Ignore. We only have one detector.
      2. *Standards*
         a. *Units* – units of the standards and calculated concentrations.

b. *Format* – format of standards and calculated concentrations.
c. *Concentration* – Enter concentrations of standards for the specific channel. These must be from high to low.

3. *Evaluation*
   a. *Calibration* -- defines the standard curve, and how the software should deal with accepting or rejecting a calibration.
      i. Boundaries – How the standard curve is broken up into high and low ranges, if necessary.
      ii. Strategies – How the software accepts or rejects a calibration. Ignore this unless you're an experienced user.
      iii. Pass/Fail -- Similar to the above. Ignore unless you're an experienced user.
   b. *Signal Processing* -- Ignore
   c. *Auto Dilution Triggers* -- Not Applicable to our system. Ignore.
4. *Presentation* – How the data and peaks are displayed
   a. *Data Window* – Range of the scale and the position of the peak window on the Lachat System display. Ignore unless you're an experienced user.
   b. *Chart Mode* – Ignore.
5. *Timing* – Timing of values and peak detection.
   a. *Periods* – Ignore unless you're an experienced user. You should seek help with this from someone who knows.
   b. *Mode* – Auto or Manual. Ignore this unless you're an experienced user.

D. **Standards** – Allows you to view the current settings and to choose the run protocol for the standards
   1. *Definition* – Read only display of standard concentrations. Ignore.
   2. *Calibration* – Protocols for standard analysis and review.
      a. *Protocol* – How many time each standard gets injected upon doing a calibration (e.g. AA BB CC DD would be standards A through D, each injected twice.).
      b. *Actions* -- Select whether the user must approve all calibrations, or if the software can except it. Chose "User Must Approve" all calibrations.

E. **Timing** – Adjusts system timing. Most timing changes occur in the Channel Specific Method Definition Submenu. Ignore this here unless you're an experienced user.

II. **Samples** – Allows you to start a run, and specify which vials to analyze.
   1. Tray Definition and Submit – Select the appropriate tray to analyze, submit trays, calibrations and kill running trays.
      A. **File** – Select Template Read (public), and pick "temp". This is a sample queue numbered 1 – 96.
      B. **Data Quality Management** – Ignore. We do our own, outside of the Lachat software.

C. **Edit** -- If you have fewer than 96 vials to run, select "Identification", scan down to the last vial in your run, and put a ".." (no quotes) after your last sample. This tells the software to stop sampling.

D. **Submit** – Submit Trays, Calibrations, and kill trays.

1. *Submit Current Tray* – Start a tray. You should answer "yes" when asked if you want to calibrate prior to analysis.
2. *Calibrate Now* -- If you just want to calibrate prior to running for the day to ensure the system is operational, select this.
3. *Kill Running Tray* – Stops the tray currently running.

III. **Results/Approval** – Allows for viewing and downloading of results, and approval of calibrations.

1. View Calibration and Sample Reports

A. **Method Selection** – Select the method used to analyze the data of interest.

B. Tray Selection – Sample tray (*.rps) or Calibration Tray (*.rpc). Runs are saved as the date (YYMMDDrun). For example, 00050801.rps. A sample tray from May 8, 2000, run #01 of the day.

C. Reports – Choose *Report Definition* first and select "runtime" for Sample trays, and "default" for Calibration trays. If you've been shown how to modify a report definition, choose *Define a Report* and make sure you have the correct chord specified. Click *Save Report to Disk*. When the report is prepared, save it to the A: drive.

D. Calibration Graphs and Stats – Allows you to view the calibration graphs in order to approve a calibration. Select the channel of interest. View calibration graph. Further instructions for removing points, etc. are on the screen.

Shut Down

1. Put reagent lines in water. Allow water to pump through manifold for 10 minutes or so. Remember to switch the Cadmium column off line prior to this step if you're doing NO_3/NO_2 analysis.
2. Once water has rinsed all the manifold tubing, pull the reagent lines out of the water so they suck air. Pump until there is little or no water visible in the manifold.
3. Unclip the pump tubing from the pump and release them from the pump cartridges.
4. Clean up your mess, and deal with Hazardous Waste as appropriate.

Notes, things of interest, and specific things

1. NO_3/NO_2 Analysis. The Cadmium column can be ruined easily. To prevent this, keep air OUT of the column. Keep the column out of line, unless you have reagents flowing through the manifold. Remember to switch the column out of line before you flush the manifold with water during shut down.

Quality Assurance and Control

A. Prior to running the Lachat you must log-in on the Log-In Sheet next to the instrument. Please fill-in all designated information. This information will aid in maintenance of the instrument and will be used in conjunction with the Quality Control Table (described later).

B. Every ten to 15 vials (as indicated on the run sheet) will have a blank, a replicate sample, and at least 2 standard replicates.

C. A spiked sample replicate will need to be created for analysis during run as specified on the Lachat run sheet. One spiked sample per run should be generated. The spiked replicate will be made by adding a known volume of 100 mg/L stock solution to 5 mL of sample solution. The weight of each volume should be measured on the balance and recorded. The amount of the spike will depend on the known or anticipated concentration of your samples. 10% to 50% increase in the sample concentration is typically a good rainge.

0.1 mg/L increase = 5 µL 100 mg/L stock in 5 mls
0.5 mg/L increase = 25 µL 100 mg/L stock in 5 mls

If analyzing for multiple constituents (e.g. NH_4 and PO_4 run at the same time) the spiked replicate must contain a known volume of each of these constituents. All volumes should be recorded as weights on the Lachat run sheet.

E. Following completion of your analysis you are responsible for filling out the Quality Control Table located in the main lab filing cabinet. You will need to provide data for your certified reference standards as specified on the Table (see attached Table). This data must be recorded onto the Quality Control Table within one week of sample analysis, failure to do so will result in serious restrictions on laboratory instrument usage. You must also put a copy of your run sheet in the appropriate folder near the Quality Control Tables.

Appendix C

Standard Operating Procedures for Total Dissolved Nitrogen Analyses
UNH Water Quality Analysis Laboratory

TDN using HTCO (Antek 721C)

I.) Preparation of Standard Solutions.

NH_4 is typically used to make the standards. You may change this if you feel your samples may present an unusual situation. We have several N-containing chemicals in the lab that you can use to make check standards, or as an alternative to NH_4.

A. Weigh out 0.3819 g of dried NH_4Cl (should be found in the desicator). Dissolve it in a 100 mL volumetric flask, and fill to volume. This makes a 1000 mg N^{L-1} stock solution (1000 ppm). Quantities for other salts are listed below.

Chemical	grams to add to 100 mL to make 1000 ppm
2Na EDTA $2H_2O$	1.3290
NH_4Cl	0.3819
$NaNO_3$	0.6068
Urea	0.2144
Caffeine	0.3466
Glycine	0.5359

B. If the samples to be analyzed are at the lower end of the concentration range, it may be necessary to make an intermediate standard (100 mg $N\ L^{-1}$).

C. Make working standards by pipetting the appropriate amount of stock (or intermediate standard) into 100 mL volumetric flasks, and bring them to volume. You can put the 100 mL volumetric flasks directly on the analytical balance, allowing you to know exactly how much stock you are adding. This eliminates the necessity of weighing water (to determine the volume dispensed) before using the adjustable pipettes.

D. Store stock solutions and standards in their volumetrics in the refrigerator. Stocks will store for up to a month. Standards should be made weekly, or more frequently if dealing with low concentrations (< 0.3 mg/L). Remember to cover tightly with parafilm. **Please remove you stocks and standards from the fridge when you are done. Dump and rinse glassware, acid wash and return cleaned glassware to the appropriate shelf.** Refer to Acid Washing protocol for details.

II. Sample Preparation

A. Sample vials are prepared by rinsing them at least 2 times with DDW and then combusting them in the muffle furnace at 500°C for 6 hours. It takes the muffle furnace one hour to get up to temperature.
B. If you are not sample limited, you should rinse the vials once with sample before filling. Be consistent within a run. If you are sample limited and can not rinse each vial before filling, then do not rinse the standard vials or blanks either.
C. Put a Blank in the first sample position. This sample will NOT be acquired. It is only there so that the instrument and the software (EZ-Chrom) are in sync.
D. Fill each vial to about one (1) inch from the top, and cover with a 1cm X 1cm piece of parafilm.
E. Put vials in the cardboard tray or other holder (not the sample tray).
F. Carry-over is not typically a problem, unless running a low sample (<0.3 mg/L) immediately following a high sample (10 mg/L). As with any analysis, it's best to analyze similar samples together.
G. Please refer to the **Quality Assurance and Control Section** for information on replicates, certified reference standards and check standards. A copy of the TDN Runsheet is attached.

III.) Plumbing and Gas Connections

A. At the bottom of the combustion tube, turn two-way valve to point toward TDN.
B. Leaks usually have something to do with the membrane dryer. If you suspect a leak, check there first.

IV.) System Inspection

A. Confirm TOC-5000 combustion furnace temperature is at 680 C.
B. Confirm gas pressure on the **ultra zero AIR** cylinder. The cylinder pressure should read at least 400 psi. Carrier gas from the tank should be set to 80 psi. Carrier Flow meter (on TOC-5000) should read 250 mL min^{-1}.
C. Confirm gas pressure on the zero grade Oxygen cylinder. The cylinder pressure should read at least 400 psi. Oxygen from the tank to the flow meter should be set to 40 psi. The flowmeter controlling flow to the Antek should be set to between 50 and 100 cc/sec.
D. Allow Oxygen to flow through the Antek for about 10 minutes prior to turning the Antek ON. This flushes the instrument free of nitrogen. Then turn the Antek **ON** (two switches on back-left side of Antek). The Antek may take up to an hour to warm up and perform correctly, so turn it on early if possible.
E. If you smell ozone after you turn the Antek on, you may need to replace the charcoal in the outlet scrubber or you may have a leak. Check charcoal first.
F. Inspect the syringe. The syringe should be clean and operate smoothly. Confirm syringe movement by entering the **Maintenance** menu, **Mechanical Check** screen. Press 1 to move syringe up, press 2 to move syringe down. If

the syringe leaks around the plunger, the plunger can be replaced. See TOC-5000 operator's manual.

V.) Preparation for Analysis

A. It is necessary to do a "Pre-run" before you analyze your samples. This consists of injections of DDW for an hour or 2 before you begin your run. Fill a large Vial with DDW. Cover with parafilm.
    1. Unscrew the sparge and sample needles from the autosampler arm.
    2. Put both needles into the large vial.
    3. Set Vial on the bench top. **NOT** in the Sample Tray.
    4. Start the instrument as you would a normal run (see below)
    5. Prior to sample analysis, it is recommended that you inject a standard to check for instrument response. Put sample needle into standard solution, and allow the instrument to inject the sample. Using the Preview button in EZ-Chrom, you can watch for a response. If no response, there could be a leak or other problem.

B. Check to see that waste vessel is relatively empty, and that the waste tube is in the waste vessel and has no kinks.

C. The TOC-5000 is normally left ON. If it has been turned OFF, turn on the power switches for the Autosampler and the TOC-5000. If on power-up, the TOC-5000 is in the All Reset mode, all calibration files, date, time, etc., have been erased, and these will have to be entered. I have never seen this in over 4 years. If it does occur, consult the TOC-5000 manual.

D. Press the **NEXT** function key to get to the **Main Menu**.

E. Press **3** and **ENTER**, to get to the **General Conditions** screen.
    1. Confirm that the 250 uL syringe is installed. Check this by physically looking at the syringe, and check that the selected syringe **in General Conditions** agrees with this.
    2. Set the printer to OFF. This must be off, or the acquisition of data will be incorrect.
    3. Confirm that the TC Furnace is ON. This is hardly ever turned OFF. If it is, it will take about an hour for the furnace to reach 680° C.
    4. Exit **General Conditions**

F. Enter the Maintenance Screen by pressing **8** and **ENTER**.

    1. Sroll down to **Ready State Sensor** - Turn this to **INACTIVE** by pressing ENTER while it is highlighted.
    2. Scroll down to the **Mechanical Check** selection and press **ENTER**
    3. Fill the wash-bath reservoir (part of autosampler) with fresh water. You can move the sampling arm by pressing the **ASI** button in **the Mechanical Check** screen. Move sampling arm up, and to the left by pressing the **Arm-Up** button and the **Arm-Left** button.
    4. If proceeding with your actual analysis (not the pre-run), screw the **sampling** needle into the sampling arm (Back hole). Sparge needle

does not need to be installed in to the sampling arm, because no sparging is necessary for TDN analysis.

5. If the sampling needles have been removed and re-installed, you may want to see if they are still in line with the vials. Press the V1 button and the sampling arm moves to vial 1 on the tray. Check to see if the needles line up with the vial by pressing the **Arm-Down** key. Press **the Arm-Down** key again to stop the arm. . The V43 button lines the needle up with vial 43.
6. Exit the **ASI** screen and the **Mechanical Check** screen, and return to the **Main Menu**.

G. Press **9** and **ENTER**, to enter the Autosampler screens.

NOTE: After making any changes, remember to press the ENTER key, or your changes will not be saved.

Sample Measurement Conditions screen. Tells the autosampler what injection volume, number of replicates, etc.

-- measurement group number, up to 15 groups. Each line represents a series of samples that will be subject to the same measurement conditions. TOC analysis requires 2 lines, so the maximum number of groups cannot be used. Typically you'll only have one group, unless you perform TOC analysis.

Type -- type of measurement, Select 4=NPOC (Non-Purgeable Organic C) for TDN.

IS -- Initial Sample, specifies vial position in the sample tray of the first sample in a group.

FS -- Final Sample, specifies vial position in the sample tray for the final sample in a group. Must be the greater than or equal to the value of IS.
NOTE: All spaces between the IS and FS sample positions must be filled, or the autosampler will suck air, and your run could suffer.

C1-C3 -- Ignore these settings. These are used if you plan to create and store a calibration file in the software of the TOC-5000. It is not recommended that you use this feature. For more information on this, consult the TOC-5000 manual.

F1-F3 -- Specifies the calibration file already stored in the instrument. Enter **2** here. This sets the TOC-5000 range to 5, and allows you to inject a low volume.

RG -- Range of the detector. The range set in the calibration curve will be entered automatically. You cannot change this setting unless you use a different calibration file. This should be 5 for TDN analysis.

Vol -- Injection Volume. Entered automatically when a curve is specified, but can be changed. Should be set to 15 uL or 20 uL for TDN analysis.

W -- Washes. Sets the number of times the syringe and the sampling and injection lines are rinsed with sample before sample injection begins. This should typically be set to 3 or 4.

No -- Number of injections for each sample. **Set to 4 to TDN**.

Max -- Maximum number of injections per sample. Set this to **4 for TDN**

SD – Ignore

CV -- Ignore

Dil -- Leave this set to 1. We don't have the dilution option.

SP -- Sparge time. Set this to 0 minutes for TDN

H. Press **NEXT** (**F2** key)

ASI Conditions screen.

RINSE -- Sets whether the surface of the sampling and sparging needles will be rinsed in the needle rinse vessel. If '1' is selected, rinsing will be performed before and after each standard and sample. If '2' is selected, rinsing is not performed. If a wide concentration range does not exist between samples, rinsing is most likely not needed.

NO OF NEEDLE WASHES -- Sets the number of times the flow path from sampling needle to injection needle will be rinsed with water (from the rinse water vessel) after sample injection. Set this to 3 for high salt samples to prevent poor injection performance due to a build up of salts on injection needle.

FLOW LINE WASHES -- Number of times the flow lines will be rinsed with rinse water after all analysis are complete. This is especially important for high salt samples. However, leave this set to 4 as routine, to prevent build up of deposits in the flow lines.

CALIBRATE BEFORE -- Ignore this unless you entered a value under C1, C2, or C3 in the previous screen.

PRINT OUTPUT -- Leave this OFF. We are not concerned with what the TOC-5000 calculates for concentrations. Concentrations will be manually determined in a spreadsheet.

AUTO ADDITION OF ACID -- This may only be specified when a NPOC analysis is being performed. If you manually added acid to the samples, turn this OFF.

ACID VOLUME -- Specifies the volume of acid to be added to each vial. Set to 50 uL if you're using the Auto addition option.

RINSE AFTER ADDITION -- Setting this to 'USED' specifies rinsing of the sampling needle in the rinse water vessel after each addition of acid.

KEY LOCK -- Locks the keys while a run is in progress. Leave this "UNLOCKED".

FINNISH OR RUNNING -- **Finnish**, **running**, or **no change** specified here determines events after the completion of all analyses. When **running** is specified, the following items, TC FURNACE, CARRIER GAS and AUTO START TIME are added to the conditions list, and must be set. **Finnish** shuts down the instrument, except for the cooling fan, which remains on until the instrument may be safely turned off. **No change** specifies that the instrument will remain in the operative state after completion of all the measurements. Set this to RUNNING.

TC FURNACE -- Indicates whether or not the TC furnace will remain on. Leave it ON unless you know that no one will use the machine for a week or so.

CARRIER GAS -- Indicates whether or not the carrier gas will keep flowing after analysis is complete. Set this OFF. No sense wasting gas.

AUTO START TIME -- Specifies the time at which the carrier gas or TC furnace will be turned on again.

I. Press **NEXT**.

VI.) Computer overhead Prior to starting Data Acquisition

A.) Accessing EZ-Chrom.

1.) Double click the EZ-Chrom icon in Windows.

2.) Double click **Instrument 2- Antek-TDN.**

3.) You will be asked to provide a user name and a password. A name and password will be provided by the lab manager. See him or her for this info.

B.) Making a Batch File. The batch file is simply the sample queue, listing the samples in the order that you plan to analyze them.

1.) From the menu bar, select **File, New Batch**, and fill in the appropriate information. Appropriate Information includes:

--Method Path. Name of path to be used to find method files.

--Data Path. Where to store the data files.

--Sample ID. The sample's name. It is usually best just to put in something easy here, and edit the batch file later.

--Method Name. Name of method to use to analyze you run. If you need to create a method for you analysis, see the EZ-Chrom manual for details.

--File Name. As with the sample ID, it's easiest to put something easy here and edit the batch file.

--Sample Amount. The amount of each injection (15 or 20, generally).

--ISTD Amount. Leave blank

--Multiplier. If you've performed a dilution on you samples, include the dilution factor here. If not, set this to 1.

--Number of Runs. The number of samples you will analyze. EZ-Chrom calls a sample one run, and a series of samples a batch.

2.) A spreadsheet will appear containing the number of runs and information that you specified above. Now edit the batch as necessary.

--Run Type. This is UNKNOWN for the TDN analysis, as you will do the regression in a spreadsheet later.

--Sample ID. This is just the sample name, typed as you would like to see it in a spreadsheet or report.

--Method. This should read what ever you selected earlier. If not, change it now for all of the samples in your batch.

--Filename. Needs to be a unique name for each sample. This is what you data will be saved as. It must be less than 9 characters long.

--Level. If you aren't including any calibration data, this can be set to zero. If you are including calibration data, this should be filled in with the appropriate level for each standard. You also must change Run Type to Calibration.

--Sample Amount. Set to the injection volume for each respective sample.

--ISTD Amt. Ignore, unless you are using internal standards.

--Mult. Set to one (1) if you don't have any dilutions. If you do, input the dilution factor here.

--Failure Act. Set this to Continue.

--Description. If you want you can type a detailed description of the sample here.

3.) Go to **File, Save Batch As...** and save it. A suggested name would be today's date (e.g. 980330).

VII.) Data Acquisition

At this point, you should have a Method that you want to use (see manual if you need to create or modify one), a Batch File created, and the Antek and TOC-5000 set up correctly and ready to go.

A.) Start TOC-5000 by pressing the START button.

B.) In EZ-Chrom, click the Run Batch button on the tool bar.

1.) Confirm that the Batch Name listed is the correct name.

2.) Confirm that **Start Run #** and **End Run #** are correct. **First** and **Last** are usually okay.

3.) Click the start button. After a few seconds, it should say "Waiting for Trigger" at the bottom of the screen.

C.) After the first sample (which is a blank) EZ-Chrom will start to acquire data.

IX.) Peak Checking

Peak Checking can be done in the Batch Reprocessing Window of EZ-Chrom, or in the Instrument 2 - Antek window. If you want to begin looking at your data before the entire batch is finished, you must go to the Batch Reprocessing window.

A.) Open the Batch file that you wish to check.

B.) From the menu, select **Batch**, **Reprocess**. A dialog box will appear.

1.) **Reprocessing Mode:** Set this to **Reintegrate**.

2.) **Reprocess From:** Set to **First** to **Last**, or the range you want to reprocess.

3.) Put an X in the box next to **Review Results (Pause After Each Run).**
4.) Leave the box next to **Bracket Calibration** empty.
5.) Click **Start** when you're ready to begin.

C.) If your batch contains Calibration files, a dialog box **entitled Calibration Options** will appear. Put an X in the box next to **Clear Response Factors At Batch Start**. Then click **Ok**.

D.) At this point, the chromatograms should be displayed on the screen. **Cntl - Z** normalizes the chromatogram to its highest peak. The baseline can be expanded by clicking and dragging the mouse over the baseline you'd like a better look at. You can return to the normalized view of the chromatogram by using **Cntl - Z**. You can look at the previous image (section of expanded baseline) you viewed by double clicking on the chromatogram.

E.) Select **Method**, **Graphical Events Programming**, **Integration Tools** (or **Cntl-T**). This displays the different Integration tools for changing peak starts and ends and baseline in a button format. These can also be accessed by selecting **Method**, **Graphical Events Programming**, and selecting one from the menu.

The EZ-Chrom manual explains what each of the Integration Tools does. This protocol will give details of the most commonly used tools.

1.) **Manual Baseline (Man BL)**. Draws a new baseline for a peak. Click and drag an new baseline from the start of the peak to the end. This tool allows you to correct the start and end of the peak at the same time. After making this change, a dialog box appears, showing the start and stop time of the change you just made. You may have it reintegrate the chromatogram by clicking that button, or if you have several other changes to make, click **Ok**, which will allow you to make other changes before integrating. It also asks if you want to save changes to the **Manual Integration Fixes Table**, or to the **Integration Table**. Saving to the **Manual Integration Fixes Table** just makes the changes to the current chromatogram. Saving changes to the **Integration Table**, changes the method, and will be applied to each subsequent chromatogram. Generally you'll want to save changes to the **Manual Integration Fixes Table**.

2.) **Start** and **Stop.** Each of these is pretty self explanatory. Changes the start and stop of the peak.

3.) **Integration Off**. Turns off the integration for the selected time range. Can be used when a peak has been detected and identified as one of the peaks of interest, but it isn't the correct peak. Also can be used if there is a two humped peak, and the peak has been split into two peaks by EZ-Chrom. **Integration Off** for one of the peaks, and then use **Manual Baseline** to put in a peak covering both humps.

F.) After you have made all necessary changes to the chromatogram, go to the next chromatogram by typing the down arrow (⇩). If you need to correct a previously viewed chromatogram, you can get back to it by pressing the up arrow (⇧).

G.) After you have checked each of the runs in a batch, you need to make a summary file that you can use in a spreadsheet to calculate concentrations and get you data into a usable form.

1.) Select **Batch**, **Summary** from the menu.

2.) A dialog box will appear. Check the following.

--**Enable Summary for Channel:** A

--**Peak Parameters:** Select those that you are interested in.

--**Group Parameters:** Leave these unchecked.

--**Summary Report Path:** Type in the complete path name where you would like your file saved. EZ-Chrom calls the file Batch.sum, where Batch is the name of you batch file.

--Click OK.

3.) Next, Select **Batch**, **Reprocess** from the menu. A dialog box will appear.

--**Reprocessing Mode:** Set this to **Reintegrate**.

--**Reprocess From:** Set to **First** to **Last**, or the range you want to reprocess.

--make sure **Review Results (Pause After Each Run)** is NOT checked**.**

--Leave the box next to **Bracket Calibration** empty.

--Click **Start** when you're ready to begin.

4.) Open this file in Excel, make changes to it as needed, and save the file. If you want to do your calculations in Quatro-Pro, open it in Excel, and save it as a *.DIF file. Then open this file in Quatro-Pro.

X.) Data Reduction and Number Crunching

This is easily done in the spreadsheet of your choice. The parameters for data reduction presented here are typical, and should be used as a guideline.

A.) Calculate the mean, and %CV of the three peaks for each sample. If the CV is greater than 5 %, use the two peaks closest in area counts to calculate the mean area for that sample.

B.) Because the Blank is generally zero (or very close to zero), use the blank in the calculation of the regression.

X1.) Quality Assurance and Control

A. LOG-IN

Prior to running the ANTEK you must log-in on the Log-In Sheet next to the instrument. Please fill-in all designated information. This information will

aid in maintenance of the instrument and will be used in conjunction with the Quality Control Table to be described later.

B. PRE-RUN

A pre-run should be done prior to analysis. Set the instrument up to inject aliquots of DDW, tap water, or samples from the previous day's TDN run. This should run for several hours prior to your analysis. This is primarily to condition the catalyst.

C. BLANK STABLIZATION

Three blanks will be at the start of your run. The first acts as the "timing blank" (no data is collected for this), and the second is to get a blank value. A third blank is recommended, especially if you're analyzing low TDN (<1 or 2 mg N/L).

D. Standard Replicates, Sample Replicates, Certified Reference Standards

A blank, a sample replicate, and two or three standard replicates will be run every 12 samples as specified on the TDN run sheet. The TDN standard curve frequently drifts during the run, and multiple and frequent replicate standards will allow you to correct for this drift.

1. Sample replicates will consist of one spiked replicate as noted on the TOC run sheet. This replicate is required to check for potential problems due to matrix interference. The spiked replicate will be made by adding a known volume of 1000 mg/L stock solution to 5 mls of a given sample (to be determined by user). If your standard curve ranges from 0 to 10 mg/L or less then you must use a 1 mg/L spike. If your standard curve exceeds 10 mg/L at the high end then you should use a 5 mg/L spike. For calculation of an increase of 1 mg/L above the sample concentration add 5 µL of 1000 mg/L stock to 5 mls of sample.

1 mg/L increase = 5 µL 1000 mg/L stock in 5 mls
5 mg/L increase = 25 µL 1000 mg/L stock in 5 mls

All volumes should be recorded as weights on the TDN run sheet.

2. Two vials will be filled with a certified reference solution (Ultra Scientific, or otherwise) in your run. This should be treated like a sample and will be placed in the positions specified on the TDN run sheet. The Lot # for the certified reference standard should be written on each run sheet. This will allow you to track the run to run variability of your analysis, as well as confirm the accuracy of your standard solutions.

3. At the end of your run, a standard curve consisting of four standards and a blank will be run. This will help to detect and account for any drift in the calibration during the run.

E. QUALITY CONTROL TABLE

Following completion of your analysis you are responsible for filling out the Quality Control Table located in the main lab filing cabinet. You will need to provide data for your EPA certified reference standards as specified on the Table (see attached Table). This data must be recorded onto the Quality Control Table within one week of sample analysis. Failure to do so will result in serious restrictions on laboratory instrument usage. You must also put a copy of your runsheet in the appropriate folder near the Quality Control Tables.

Appendix D

Standard Operating Procedures for Particulate Filtration
UNH Water Quality Analysis Laboratory

Particulate Organic C and N Filtration

The filters we are using are prepared and pre-weighed for suspended sediment analysis. They are in numbered pans. **It is very important that the filter be kept in its respective pan to assure correct identification.**

Sample Filtration

1. Filters are listed on the data sheets in order from the bottom of the stack to the top. Take the pan and filter from the **bottom** of the stack.
2. Check to see that the filter weight (written on the side of the pan) is recorded correctly on the data sheet. These weights have been entered previously, but check to see that it is correct.
3. Using forceps, place the filter on the base of the filter tower.
4. Gently place the top of the filter tower on the base, and secure as necessary.
5. Record the sample name and collection date (if available) of the sample on the data sheet.
6. Shake the sample bottle several times to produce a homogenous solution.
7. Measure 15 mL of sample in a graduated cylinder.
8. Pour into filter tower, and apply a vacuum.
9. Continue to add 15 mL aliquots (or larger if the samples is relatively clear) until the filter is nearly plugged.
10. Record how many mL of sample you filtered on the data sheet.
11. Put you initials next to the volume filtered.
12. Rinse the graduated cylinder 3 times with DDW, pouring the rinse water into the filter tower each time.
13. Rinse the sides of the filter tower with DDW until there is no visible sediment on the sides.
14. Continue to suck the filter dry for about 3 minutes after the filtration is complete.
15. Remove the top of the filter tower.
16. Carefully remove the filter using forceps, and place it in its identified pan. Be sure to get every piece of the filter.
17. Place pan/filters in the drying oven at 105 C for at least 6 hours.
18. Record the date and time you put the samples in the oven on the data sheet.
19. Record any notes, problems, observations, difficulties, etc. on the data sheet.
20. Perform a replicate filtration every 10-15 samples.

Balance Procedures

1. Be sure the balance is level by verifying that the bubble is in the center of the circle.
2. Be sure the balance is isolated from wind and vibrations.
3. Don’t lean on the bench when you are weighing things.
4. Brush out any debris that might be on or near the balance pan.
5. Calibrate the balance daily, both before and after you weigh filters.
    a) Zero the balance.

b) Place the smaller of two calibration weights on the balance (use 2 weights that are appropriate for the masses you'll be measuring).
c) Record the mass on the log sheet, including you initials, and the date.
d) Remove the weight from the balance.
e) Zero the balance.
f) Place the larger of the two calibration weights on the balance.
g) Record the mass on the log sheet, including you initials, and the date.

Weighing Filters

1. Remove the pan/filters from the drying oven and cool in a desiccator to balance temperature.
2. Record the time and date on the data sheet.
3. Try to limit the time the filters are out of the desiccator prior to weighing as they will have absorb moisture from the air.
4. Calibrate the balance and record the weights.
5. Zero the balance.
6. Using forceps, place the filter on the balance. Weigh only the filter! Not the tin!
7. Allow the balance to stabilize and record the weight on the data sheet.
8. Put you initials next to the Notes: column on the data sheet.
9. Record any notes, problems, or observations on the data sheet.
10. Put the weighed filter back into its respective pan, and put the pan/filter back into a desiccator until the filter can be prepared for CHN Analysis.

Appendix E

Standard Operating Procedures for CHN Analyses
UNH Water Quality Analysis Laboratory

CHN Sample Preparation Protocol

This assumes you have a homogenous sample that has been ground or sieved.
Always have the pan arrests in the raised position when placing or removing items from the weighing tray.
Always close the balance door when not placing or removing items.
Be very careful placing and removing samples from the weighing pans. Cases of the "shakes" are not allowed.

Calibrating the Micro-Balance

1. Remove all samples and weights from the sample and reference trays.
2. Be sure that the trays are free of debris (there are small brushes in the drawer beneath the balance if you need to sweep small particles away).
3. Lower the pan arrests.
4. Press the AUTOTARE button and wait until integration ("Int") is complete.
5. Press the RANGE button until "200 mg" appears on the left display.
6. Raise the pan arrests and place a 100 mg calibration weight on the sample pan (the pan on the right). The calibration weight is in a box labeled "AD6 Kit", located in the drawer to the left of the balance. DO NOT touch the weight with your fingers. Use the forceps.
7. Lower the pan arrests, and enter 100 on the numeric key pad.
8. Press the CALIB button. Calibration is complete when the "INT" stops flashing.

Using the Micro-Balance

1. Press the RANGE button until it reads 20 mg. This is normally the most appropriate range, although for ultra-low, super-critical weighing, you can use the 2 mg range.
2. Place the tare weight on the LEFT tray. This is a small piece of copper wire that weighs approximately as much as a sample tin and tin holder
3. Place a sample tin into a black tin holder (found in the drawer below the micro-balance) and carefully place the combination on the right tray.
4. Lower the pan arrests.
5. Press the AUTOTARE button and wait until integration ("Int") is complete.
6. Raise the pan arrests and remove the tared sample tin and holder.
7. Add 2 mg (+/- 0.5 mg) of you sample to the tin. Be careful not to get any sample material on the outside of the tin. NOTE: For mineral soils, you may add 10 to 20 mg of sample to the tin in order for there to be enough C and N to measure accurately.
8. Place the sample, sample tin and holder on the right weighing tray.
9. Lower the pan arrests and wait for the weight to stabilize.
10. Record weight.
11. Raise pan arrests and remove sample from Micro-Balance.
12. On a clean surface. Fold the top of the tin over to seal it and flatten the bottom of the tin with the butt end of the forceps. Then fold again so it is in thirds.

13. Place the tin so that the area where most of the sample is, is facing up. Fold into thirds again so the sample is surrounded by an equal amount of tin. Page 4-61 in the CHN manual shows a modified version of this procedure.
14. Record your sample name and weight on the forms provide and store in a labeled sample tray.

RUNNING THE INSTRUMENT

Gas Flow

Stable and precise gas flow to the CHN analyzer is critical for successful analysis. Prior to starting the CHN analysis, check that all three gas tanks have an **internal pressure of at least 500 psi** (typically the dial on the left). Please inform the lab manager if any of the tanks are below 500 psi, or are close that limit.

Check the regulators (dial on the right) to see that each is set to deliver the appropriate pressure to the instrument.

Helium (He) – The "carrier", 20 psi.
Air – Runs the pneumatics in the instrument (valves, etc), 60 psi.
Oxygen (O2) – Allows for oxidation of the sample, 16 psi.

Check the Run Counters

1. Press the **PARAMETERS** button.
2. Press **4** and **ENTER**.
    i. You should see RUN COUNTERS
REDUCTION ###
    ii. If this number is less than the number of samples you're planning to analyze, and is less than 30, you need to fill and install a new reduction tube (see maintenance section). If the number is more than 30, you should run up to that many samples, and plan on changing the reduction tube after that.

3. Press the **ENTER** key.
    i. You should see RUN COUNTERS
COMBUSTION ###
    ii. If this number is less than the number of samples you're planning to analyze, and is less than 30, you need to fill and install a new combustion tube (see maintenance section). If the number is more than 30, you should run up to that many samples, and plan on changing the combustion tube after that.

4. Press the **ENTER** key.
    i. You should see RUN COUNTERS
VRCPT ###
    ii. If this number is less than the number of samples you're planning to analyze, and is less than 30, you need to replace the vial receptacle (see maintenance section). If the number is

more than 30, you should run up to that many samples, and plan on replacing the vial receptacle after that.

5. Press the **ENTER** key again, and you should be back to the **PARAMETERS** prompt.
6. Press PARAMETERS button and you should return to **STANDBY**.

System Purge

1. Press the **PURGE GAS** button.
2. You should see PURGE GAS
HELIUM Y/N
3. Press the **YES** button.
4. You should see PURGE GAS
ENTER TIME
5. Enter the time you want to purge in seconds. Typically 180 is sufficient for Helium.
6. Press **ENTER**.
7. You should see PURGE GAS
OXYGEN Y/N
8. Press the **YES** button.
9. You should see PURGE GAS
ENTER TIME
10. Enter the time you want to purge in seconds. Typically 120 is sufficient for Oxygen
11. After the gasses have finished purging, you should be back in **STANDBY**.

Tray Set Up

Your first sample on you tray should be a series of Blanks, Conditioners, and K-factors, in the following order;

1. Blank
2. Blank
3. Blank
4. Conditioner
5. Blank
6. Conditioner
7. Blank
8. K-Factor
9. K-Factor
10. K-Factor

Blanks are sample tins with nothing in them. Conditioners have some type of sample in them, usually standard material. K-Factors have a precisely measured amount of standard material in them. The standard is usually Acetanilide, although there are other standard materials in the dessicator in room 228.

These initial samples only need to be run at the beginning of a tray, and will allow you to assess how the machine is running. For the blanks and the K-factors, consistency is as important as the actual value. Blanks will likely start off higher and decrease slightly. They should be consistent by the last blank. If not, run additional blanks. K-factors should also be consistent. Typical values are:

Blank
C = 20 H = 120 N = 30

K-Factor
C = 12.700 H = 32.5 N = 4.485

Your samples follow these initial samples. Please run a Blank, K-factor, a replicate of one of your samples and a reference sample every 12-15 samples. There are several reference samples near the instrument (2 mineral soils, ground Ivy, or you can also run some standard material and call it a sample).

Setting up a New Run Sequence

1. Press the **AUTORUN** key
2. You should see
   AUTO RUN NO. XX
   1B 2K 3S 4RP
3. If the number is not 1, press **4** to reset the starting number.
4. You should see
   1 RESET 2 PRINT INFO
   3 PRINT RESULTS
5. Press **1**.
6. You should see
   RESET ALL? Y/N
7. Press **YES**.
8. You should then see
   AUTO RUN NO. 1
   1B 2K 3S 4RP
9. Enter the appropriate number that describes the sample for the specified position on the sample tray; 1 for Blank, 2 for K-factor, 3 for Sample, or 4 to reset or print.
10. If you enter 1 (blank), you will immediately go to the next sample.
11. If you enter 2 (K-factor), you will see
    THEORY STANDARDS
    S1 S2 S3 S4
    i. Enter 1 (S1 = Acetanilide), and then the weight of the standard, and press **ENTER**
12. If you enter 3 (Sample), you will see
    ID _
13. You must put some number or letter combination here. It does not need to be unique, or relevant to your sample, but the machine requires a value.

14. The instrument will automatically prompt you for information about the next sample.
15. When you are done entering information for all your samples, press **AUTORUN**, which will put you back at **STANDBY**.
16. With the sample carousel removed from the instrument, turn it so position 60 is over the hole in the bottom of the carousel.
17. Fill your tray with your blanks, standards and samples up to position 59 (leave 60 empty for now).
18. Put the sample carousel on the instrument so that position 60 is lined up with the arrow on the front of the machine, and tighten the knurled nut.
19. Turn the carousel on click clockwise (to the left) so that position 1 is lined up with the arrow.
20. Put sample 60 in position 60.
21. Press the **START** button.

Adding to a Run Sequence

See manual, page 5-117, and is also attached to the protocol in the lab Protocol binder.

Modifying Run Parameters

See manual, page 5-119, and is also attached to the protocol in the lab Protocol binder.

Data Retrieval and Processing

Data from each of your runs is printed out at the completion of each sample. You will need to manually enter this information into a spreadsheet. Your spreadsheet should have at least the following columns and the appropriate information for each sample in your run. Occasionally, the raw signals are useful to correct for a bad blank, or other problem with the run. Don't through your print out away until you're sure your run is perfect, or you've entered all of the data (including the raw signal values), as there is no other way to retrieve your data once the paper copy is gone.

Tray position	Sample	Weight (mg)	C (mass, blank count, or K-factor)	H (mass, blank count, or K-factor)	N (mass, blank count, or K-factor)

Once you have the mass for C, H, and N, you can easily calculate %C, %N, C:N, etc. for your sample.

Maintenance

Routine maintenance can be quite extensive and time consuming because it involves letting the furnace cool, repacking tubes, and heating the furnace again. Cooling and heating can take nearly a full day. Do not underestimate this when planning your analyses.

Filling the Combustion Tube

The combustion tube should be replaced after 1000 samples. Before doing any maintenance, be sure to turn the furnace off, and let the furnace cool to room temperature.

1. Press **PARAMETERS**.
2. Enter **12**, and press **ENTER**
3. Press **2** to turn furnace OFF
4. Press **PARAMETERS** button again to return to **STANDBY**
5. Press **MONITOR** button.
6. When asked if you want to PRINT LIST, press **NO**.
7. Press **1** and **ENTER**, to monitor the temperature of the combustion furnace.

Detailed instructions for packing and installing a new combustion tube are found in the manual, starting on page 4-6.

Filling the Reduction Tube

The combustion tube should be checked after 250 samples. If the copper grains in the tube are still orange, you can still use it. If more than 5/6 of the copper is grey or black, replace the reduction tube. Before doing any maintenance, be sure to turn the furnace off, and let the furnace cool to room temperature.

1. Press **PARAMETERS**.
2. Enter **12**, and press **ENTER**
3. Press **2** to turn furnace OFF
4. Press **PARAMETERS** button again to return to **STANDBY**
5. Press **MONITOR** button.
6. When asked if you want to PRINT LIST, press **NO**.
7. Press **2** and **ENTER**, to monitor the temperature of the reduction tube.

Detailed instructions for packing and installing a new reduction tube are found in the manual, starting on page 4-10.

Cleaning the Vial Receptacle

The vial receptacle should be checked anytime the furnace is cooled and the tubes are exposed. It is located in the top of the combustion tube (the one on the left). It should definitely be cleaned/replaced after 600 samples. Before doing any maintenance, be sure to turn the furnace off, and let the furnace cool to room temperature.

1. Press **PARAMETERS**.
2. Enter **12**, and press **ENTER**
3. Press **2** to turn furnace OFF
4. Press **PARAMETERS** button again to return to **STANDBY**
5. Press **MONITOR** button.
6. When asked if you want to PRINT LIST, press **NO**.
7. Press **1** and **ENTER**, to monitor the temperature of the combustion tube.

8. The vial receptacle can be cleaned and reused. Over time it can become brittle and may be damaged in the process of removing it. If it is damaged, replace it with a new one.
9. Specific directions can be found in the Combustion Tube filling instructions.

Appendix F

Standard Operating Procedures for PAR Field Measurements
UNH Marine Program

LI-1400 DataLogger PAR Measurements

Standard Operating Procedures

Overview

The LI-1400 DataLogger and calibrated quantum sensors provide the capability to quantify Photosynthetically Active Radiation (PAR) both above and below the water surface. The 'air' sensor remains above the water and quantifies downwelling radiance from the sun each time a discrete measurement is taken; this is most often used to normalize readings taken over several minutes to a constant downwelling value. The 'underwater' sensor is deployed on a frame that is lowered into the water. This sensor is generally used to measure a profile of in-water irradiance versus depth so as to estimate the diffuse attenuation coefficient (K_d), a measure of the rate at which photosynthetically active radiation is attenuated as it passes down through the water-column.

In general, the attenuation of light is exponential versus depth. To obtain K_d from a series of light readings with depth, a series of measurements at at least 8 depths is desired (in shallow waters this may not be possible). One obtains K_d by making a linear regression of sample depth versus ln (PAR) and calculating 1/(slope of the regression). Additional information of interest includes the percent of surface radiation reaching the bottom.

Estimates of K_d are considered robust if the r^2 of the regression is >0.95 (generally >0.98). The precision of the method is estimated by taking 3 complete profiles sequentially and calculating the standard error (SE) of the measurement. The SE should be less than 10%.

Before First Sampling of the Day

1. Insure that the sensors are securely attached to their frames and confirm that the calibrations factors stored in the DataLogger are correct for the sensors in use.
2. Hook up the Underwater BNC connector to Channel I1 labeled "underwater".
3. Hook up the Air BNC connector to Channel I2 labeled "air".
4. Turn the DataLogger 'ON'.
5. Under View, press 'ENTER' to view new data.
6. The first view should say "I1I" which corresponds to the underwater connector. "I2I" corresponds to the air connector.
7. Switch to view I2I, take the cap off of the air sensor, check the reading then cover the sensor with your hand to confirm the reading changes (the reading should decrease with a decrease in light).
8. Switch to view I1I, take the protective covering off of the underwater sensor, check the reading and then cover the sensor with your hand to confirm the reading changes (again the reading should decrease).

At Each Station

1. Turn on the DataLogger.
2. Take out the respective data sheet for the site. Record the time when the underwater sensor is put in the water.
3. Lower the sensor to 10cm. Allow the reading to stabilize (1-2 seconds) and then press 'ENTER'. This logs the data into the DataLogger. Cross off 10cm (and each subsequent depth for which you log data into the DataLogger) on the data sheet.
4. Lower the sensor to the next depth. In shallow areas, record measurements every 25cm as marked on the cable. In deep and/or clearer water areas the sensor can be lowered every 50cm. At least 6-8 depths should be recorded in the DataLogger for each station.
5. When (If) the sensor reaches bottom, write the bottom depth (approximate using the depth markers) on the datasheet and press 'ENTER' to log data into the DataLogger. You do not need to go to the bottom if you have >10 good readings; if the DataLogger is showing light readings less than 0.5 or if the sensor begins to stream out in strong currents.
6. Raise the underwater sensor out of the water and put the protective cover on. Put the cap on the air sensor also. Turn the DataLogger 'OFF' until reaching the next station.

At End of Sampling

1. Unplug the BNC connectors from the LI-1400.
2. Rinse underwater sensor, frame and cable with freshwater and let dry before storage.

Download Data to Excel in the Laboratory

1. After returning to the lab the data should be retrieved from the DataLogger.
2. Attach the DataLogger to the computer using the serial cable.
3. Open the LI-1400 program and then turn the DataLogger on.
4. Under the remote menu click on 'CONNECT'. Under the connect window, type '2' next to com port number and click 'CONNECT'.
5. Under the remote menu click 'RECEIVE DATA'. Save the data on the computer.
6. Open Microsoft Excel and then open the file you just saved. The file is a delimited file and click 'FINISH'.
7. Download LiCor Data into a new Excel file and **Save As** GBSWMP Raw Light Profile (MMDDYY) where the MMDDYY represents the sampling date.
8. Once you are certain that you have successfully downloaded and saved the data the data in the DataLogger should be cleared from memory. This can be done 2 ways:
    a. On the DataLogger, press the 'FCT' key. Arrow to the right twice till clear memory is in the window. Arrow down to clear all, down to date, down to time, and down to clear memory yes/no. Confirm that "clear memory yes" is in the window and then press 'ENTER'. (This may not clear the memory).
    b. In the LI-1400 program, under the remote menu click 'CLEAR DATABASE'. In the clear database window confirm that all is chosen then click 'OK'.

9. Under the remote menu click 'DISCONNECT'. Unplug the DataLogger from the computer, turn it off, make sure that there is no dirt or salt on it and put it away.

Data Processing

1. **Open** *GBSWMP Light Profile Master* Excel File and **Save As** *GBSWMP Light Attenuation (MMDDYY)*

2. Cut and paste the raw light data into the appropriate rows/columns in the *GBSWMP Light Attenuation (MMDDYY)* file making sure to separate each station as indicated by the station labels in column A.

3. Edit the depths Column E so that they reflect the correct depths at which each of the readings at a particular station was taken.

4. Run individual regression analyses for each of the light profiles as follows (using APL as an example):

    a. Click on **Tools**, **Data Analysis**, **Regression**
    b. Select 'Input Y Range' as the range of measured Depth [m] in Column E
    c. Select 'Input X Range' as the range of calculated Quantum [LN] in Column H
    d. Select 'Output Range' as the Yellow Shaded Block in Column J
    e. Select 'OK'; this should insert the regression statistics to the right of the data [Note: Do not include data for which the Quantum [Raw Water] data is < 0.1]
    f. Save File (intermediate save so as to not lose data)

5. At this point, the Diffuse Attenuation Coefficients ($\mathbf{K_d}$) should have been calculated for each station at which you did the regression. $\mathbf{K_d}$ = 1/x-coefficient from the regression. For Great Bay, values of $\mathbf{K_d}$ should range from -0.5 (clear) to -6.0 (very turbid).

6. QA/QC: Examine the regression output data:

    a. Acceptable regressions must have an $R^2 > 0.95$ and, for stations with an optimal number of sample depths (>8) should have an $R^2 > 0.98$.
    b. Examine the Quantum [Raw Water] data. These data should show a continuous decrease with depth except for the odd cases where the Quantum [Air] data increased significantly from the preceding reading (e.g. the passing of a cloud). Highlight any questionable reading by applying an 'Orange' fill to the cells in question.

7. In the event that the $R^2 < 0.95$ and there is a data point at the top or bottom of the profile that is clearly bad (these are the most likely places for this to occur because of surface reflection or sediment resuspension) you may choose to run the regression again omitting the suspect data. In such cases it is imperative that you make a notation in Column I, just below the $\mathbf{K_d}$ calculation block.

Appendix G

Field Data Sheets

GREAT BAY SWMP STATION LOG SHEET

Station: _____ ***Lat Deg:Lat Min***: _____:_____

Date: _____ ***Lon Deg:Lon Min***: _____:_____

Time: _____

Tide: ***Low*** ***High*** ***(Circle One)***

CTD / YSI

	Depth	*Temp*	*Salinity*	*DO*	*%SAT*
Surface					
Bottom *(If Necessary)*					

Bottle Cast Data

	Nutrient/TSS Bottle ID	*DON/PON Bottle ID*	*(Additional Sample)*	*(Additional Sample)*
Surface				
Bottom *(If Necessary)*				

LiCor PAR Measurements *(Cross off Depths Where Measurements are Taken/Stored; Make a Minimum of 6 Measurements)*

Time: _____

Depth (cm):

10	25	50	75	100	125	150	175	200
250	300	350	400	450	500	550	600	650
700	750	800	850	900	950	1000	1050	1100
1200	1300	1400	1500					

Bottom Depth: _____

Comments