DEPARTMENT OF
ECOLOGY
State of Washington

Quality Assurance Project Plan

Puget Sound Total Alkalinity and Dissolved Inorganic Carbon Pilot Project

September 2014

Publication No. 14-03-116

Publication Information

Each study conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance Project Plan. The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, Ecology will post the final report of the study to the Internet.

This Quality Assurance Project Plan is available on Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1403116.html

Ecology's Activity Tracker Code for this study is 01-800.

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Puget Sound
Total Alkalinity and
Dissolved Inorganic Carbon
Pilot Project

September 2014

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EAP: Environmental Assessment Program

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Abstract

The Washington State Department of Ecology (Ecology) is conducting a pilot study to test whether total alkalinity (TA) and dissolved inorganic carbon (DIC) water sampling can be incorporated into the existing Marine Flight Program. Another goal of this study is to provide high-quality pH data to better understand the dynamic conditions that affect pH measurements in Puget Sound, using potentiometric electrodes for in situ pH sensor measurements. Ecology will accomplish this with these steps:

- measure TA and DIC to quantify pH with greater accuracy
- calculate the saturation state of aragonite (a form of calcium carbonate created through shell biomineralization)
- assess the side-by-side comparisons of the potentiometric pH measurement routinely collected on the marine flight oceanographic conductivity-temperature-density (CTD) sensor package
- calculate improved pH values using TA and DIC

The focus of this study will be to determine the range of TA, DIC, and pH at select sites and assess their seasonal variability in the upper water column of Puget Sound.

Background

When carbon dioxide (CO_2) is absorbed by seawater, it forms a weak acid that reduces the pH of seawater and changes the carbonate ion concentration and saturation states of biologically important calcium carbonate minerals. Alkalinity plays an important part in how well seawater can buffer increases in CO_2. Chemical reactions that shift the carbonate system to produce lower pH are termed *ocean acidification* or *OA*. Calcium carbonate minerals are the building blocks for the skeletons and shells of many marine organisms. In areas where most life now congregates in the ocean, the seawater is supersaturated with respect to calcium carbonate minerals. This means there are abundant building blocks for calcifying organisms to build their skeletons and shells. However, continued ocean acidification or a reduced capacity of the water to buffer the dissolution of CO_2 into a weak acid is causing marine water to become undersaturated with calcium carbonate minerals of a specific form (aragonite). This is likely to affect the ability of some organisms to produce and maintain their shells.

Using a potentiometric electrode for measuring pH is a standard operating procedure for nation-wide water quality monitoring programs. However, this procedure is known to yield potentially large and variable biases from true pH values because most electrodes do not take changing buffering capacities of the water into account. Stated accuracy of many potentiometric pH sensors is on the order of 0.1–0.2 pH units, but under typical use, pH measurement biases may be higher. This is especially problematic when there are significant differences between the ionic strength and composition of the seawater being measured as compared to the internal reference solution of the pH electrode. Thus, in estuarine systems with strong salinity gradients and multiple sources of freshwater with varying composition among them, such as Puget Sound, it is challenging to ascertain the magnitude, direction, and variability of the bias in pH, as measured with potentiometric pH sensors.

The marine inorganic carbon system has four measurable parameters: TA, DIC, the partial pressure of carbon dioxide (pCO_2), and pH. From any pair of measurements, all other inorganic carbon parameters can be calculated. These parameters include saturation states for the aragonite and calcite forms of the biomineral calcium carbonate and concentrations of carbonate and biocarbonate ions. These parameters are calculated by a program such as CO2SYS (Lewis et al., 1998). When using the selected pair of measured carbon parameters, we also need measurements of temperature, salinity, nutrient concentrations (phosphate and silicate specifically), and the appropriate dissociation constants for the salinity range of the ecosystem (Dickson et al., 2007). We will use Lueker et al. (2000) constants if all the data have salinity > 16 psu, and we will use Millero et al. (2006) if any of the data have salinity < 16 psu.

For decades, Ecology's ongoing Long-term Marine Flight Program has been providing monthly full depth profiles of estuarine state variables: temperature, salinity, pressure, and conventional electrode-based pH. Water samples for TA and DIC will provide an independent and refined assessment of the magnitude, direction, and variability of pH biases between potentiometric electrode measurements and the more accurate pH values inferred by using multiple variables relevant for the carbonate system. We plan to add measurements of TA and DIC in the near-surface waters, which are naturally very variable due to the influence of less dense freshwater and biological activity. This will allow us to leverage resources and provide the context of a large pre-existing data set to help delineate spatial and temporal information on pH in sensitive areas. Determining a systematic bias between potentiometric and the calculated pH would allow constraining and leveraging Ecology's large historical data set. It may also be possible to develop regional relationships between alkalinity and salinity, which could provide insight into TA and DIC outside of each point measurement collected by the Marine Flight Program.

Project Description

Ecology will collect approximately 173 samples from 6 stations in the Central Sound, Whidbey Basin, Hood Canal, and Totten Inlet from June 2014 to May 2015. Sampling procedures will be incorporated into the routine marine flight field work (Bos et al., 2014). TA and DIC samples will be taken at each station as well as a nutrient sample. Due to the current limitation of our CTD package, we will be using a seawater collection method that allows an alternative water collection method. This method uses a peristaltic pump and extended holding time of samples suited for external batch analysis (Bockmon and Dickson, 2014). The Pacific Marine Environmental Laboratory (PMEL) Carbon Group will analyze the samples, using standard operating procedures used in the marine carbon community (Dickson et al., 2007).

Ecology's Marine Monitoring Unit will use the data to focus on TA and DIC in a spatial and temporal context and support efforts to develop specific relationships between alkalinity and salinity within Puget Sound and its regions.

This project has three goals:

- Test the feasibility of collecting these samples from a floatplane, as part of the already established Marine Flight Program.
- Provide context for a very large existing pH sensor data set and delineate spatial and temporal variability of pH sensitive areas.
- Assess the bias introduced in potentiometric pH measurements in historic data collected by Ecology in the absence of TA and DIC data.

Based on the results, we will evaluate the need and feasibility of continuing alkalinity monitoring as an integral part of our marine monitoring operations and determine regions and depth ranges where additional TA and DIC samples will be required.

Study Area

Six stations were chosen from the 2014 Marine flight stations. Both data needs and logistics were considered. Two stations are in Whidbey Basin: Saratoga Passage (SAR003) and Possession Sound (PSS019). Contrasting stations will provide information on the difference between the rain-fed Skagit River (SAR003) and the snowmelt-fed Snohomish River (PSS019) and the expected ranges. The Admiralty Inlet station (ADM001) will provide data on water entering from the north. The northern-most Hood Canal station was chosen (HCB010) because of its close proximity to commercial oyster beds. Oakland Bay (OAK004) and Totten Inlet (TOT002) in the South Sound were chosen because of their highly productive commercial harvested shellfish beds. For a more detailed description of the marine flight study area and complete station list, see Bos et al. (2014).

Table 1. Description of Ecology’s Long-term Ecology Marine Waters Monitoring stations to be sampled for this study.

Station	Short Description	Latitude	Longitude	Basin	WRIA	WA Level-8 HUC Boundaries
ADM001	Admiralty Inlet – Bush Pt.	48° 01.789' N	122° 37.076' W	Admiralty Inlet	06	17110019
SAR003	Saratoga Passage – East Point	48° 06.4557' N	122° 29.4925' W	Whidbey Basin	06	17110019
PSS019	Possession Sound – Gedney Island	48° 00.6556' N	122° 18.0750' W	Whidbey Basin	07	17110019
HCB010	Hood Canal – Send Creek, Bangor	47° 40.2000' N	122° 49.2000' W	Hood Canal Basin	15	17110018
OAK004	Oakland Bay – Near Eagle Point	47° 12.806' N	123° 4.659' W	South Basin	14	17110019
TOT002	Inner Totten Inlet	47° 07.2890' N	123° 01.2754' W	South Basin	13	17110019

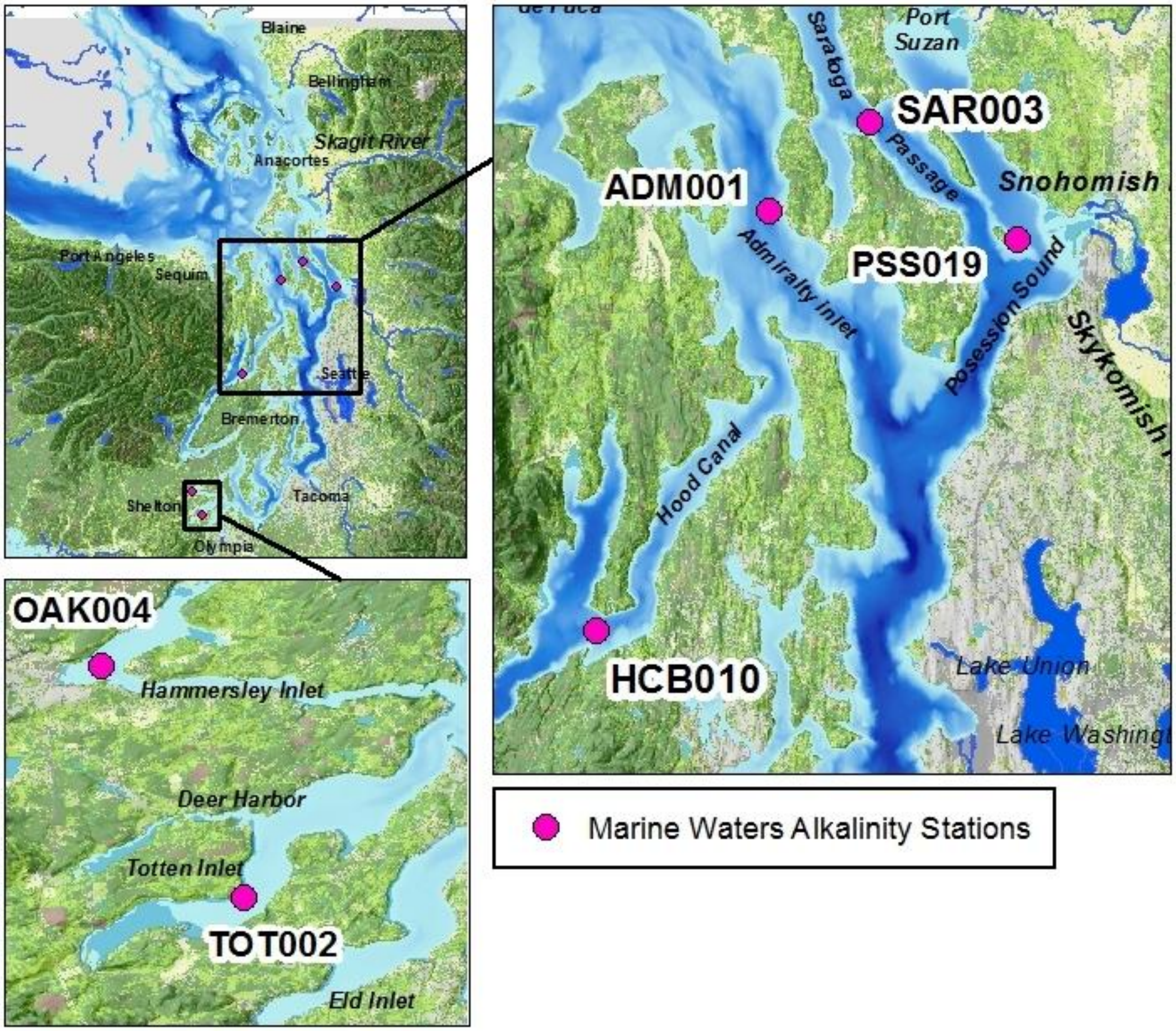

Figure 1. Ecology's TA and DIC pilot sampling locations.

Organization and Schedule

Table 2 lists staff involved in this project. All are employees of the Washington State Department of Ecology. Table 3 presents the proposed schedule for this project.

Table 2. Organization of project staff and responsibilities.

Staff	Title	Responsibilities
Mya Keyzers Marine Monitoring Unit Western Operations Section Phone: 360-407-6395	Project Manager	Writes the QAPP. Oversees field sampling and transportation of samples to the laboratory. Conducts QA review of data, and analyzes and interprets data. Writes the draft report and final report.
Christopher Krembs Marine Monitoring Unit Western Operations Section Phone: 360-407-6675	Principal Investigator	Creates the project scope and budget, tracks progress, and reviews the draft QAPP. Analyzes and interprets data. Reviews final report.
Laura Hermanson Marine Monitoring Unit Western Operations Section Phone: 360-407-0273	Field Technician	Helps collect samples and records field information.
Carol Maloy Marine Monitoring Unit Western Operations Section Phone: 360-407-6742	Unit Supervisor for the Project Manager	Provides internal review of the QAPP, approves the budget, and approves the final QAPP.
Julia Bos Marine Monitoring Unit Western Operations Section Phone: 360-407-6674	Data Management & analysis	Enters data into EIM/data management system. Supports data analysis, coding, and reporting.
Robert F. Cusimano Western Operations Section Phone: 360-407-6596	Section Manager for the Project Manager	Reviews the project scope and budget, tracks progress, reviews the draft QAPP, and approves the final QAPP.
William R. Kammin Phone: 360-407-6964	Ecology Quality Assurance Officer	Reviews and approves the draft QAPP and the final QAPP.

QAPP: Quality Assurance Project Plan

Table 3. Proposed schedule for completing field and laboratory work, data entry into EIM, and reports.

Field and laboratory work	Due date	Lead staff
Field work completed	May 2015	Mya Keyzers
Laboratory analyses completed	August 2015	
Final report		
Author lead / Support staff	Mya Keyzers/Christopher Krembs	
Schedule		
Draft due to supervisor	September 2015	
Draft due to client/peer reviewer	September 2015	
Draft due to external reviewer(s)	October 2015	
Final (all reviews done) due to publications coordinator	October 2015	
Final report due on web	November 2015	

Quality Objectives

Water samples collected in the field and analyzed in a laboratory have an inherent amount of error. Concerns about precision and consistency in sample collection will be minimized by following the appropriate protocols and SOPs.

Bias is defined as the difference between the population mean and the true value of the parameter being measured (Lombard and Kirchmer, 2004). It is a challenge to address precision and bias in pumped samples because they represent spatially averaged samples that are affected by patterns of natural variability and plane drift. We recognize that taking the samples from deeper in the water column would reduce horizontal variability, yet safety aspects of the peristaltic pump tubing limits sampling to 5-7 meters. We will be taking triplicate samples, but they will be less of a control on bias and more of a check on station representativeness.

If the data quality objectives are not met, the data are qualified. UW's Marine Chemistry Laboratory performs replicate sample analyses using sample splits within laboratory batches for quality control purposes. The difference between field and laboratory variability is a measure of the sample field variability.

Table 4. Measurement quality objectives for TA/DIC laboratory analyses.

Laboratory Measurement	Precision (relative standard deviation, RSD)	Accuracy (% difference from true value)	Lowest Value (reporting Limit)
TA/DIC	<0.5%	<0.25%	NA*

* Not yet defined by laboratory.

The PMEL lab uses certified reference materials (CRM) with known DIC and TA produced by Dr. Andrew Dickson at Scripps Institution of Oceanography, University of California, San Diego. These are included with every sample batch and used to correct field sample TA/DIC measurements for small offsets between measured and certified values of the CRM. Recovery percentage is calculated from these results and therefore can be used as a measure of analytical accuracy and bias. If the results fall outside of established limits, data associated with the batch are flagged by the reviewer. Any measurement problem that cannot be resolved is given a data quality flag

Sampling Process Design (Experimental Design)

TA, DIC, and nutrient water samples will be collected in addition to the routine marine flight sampling described in Bos et al. (2014). The six stations in this pilot project will be visited every month for a year, with sample collection from May 2014 through May 2015.

Ecology will collect samples from 6 different areas of the Puget Sound (Figure 1). Each station will be visited every month during the sampling year. Field staff will collect one water sample, to be analyzed for both TA and DIC, from 5-m depth at each station once a month for a total of about 173 samples.

TA and DIC will be analyzed from the same water sample. The TA, DIC, and nutrient samples will be collected using a peristaltic pump during the routine CTD vertical profile cast. Once the TA and DIC samples have been collected, the glass lids will be greased and then capped with a rubber band and clamp. The samples will be stored in the dark until they are transported back to the lab, where mercuric chloride will be added. After the nutrient samples have been collected, they will be stored in a cooler with ice. Once back at the lab, they will be frozen until delivery to the UW Marine Chemistry Lab.

The marine flight CTD is equipped with temperature, conductivity, pH, dissolved oxygen, pressure, photosynthetically active radiation (PAR), light transmission, in situ chlorophyll fluorescence, and turbidity. The CTD is deployed through a hatch in the belly of the plane, descends to the bottom of the water column, and then comes back up to the surface, collecting data at eight scans per second. The CTD's measurements of conductivity and temperature will be used in the data analysis.

Alkalinity and nutrient samples will be delivered at the end of each month to Pacific Marine Environmental Laboratory (PMEL) and UW Marine Chemistry Lab, respectively.

Characteristics of Existing Data

Alkalinity data was collected in the 1970s by Eugene Collias (Collias, 1970). Ecology collected TA and DIC on the 2008 University of Washington collaboration cruise on the R/V Bold. Ecology's South Puget Sound Dissolved Oxygen Study (Roberts et al., 2008) collected TA data. NOAA collected TA and DIC data from the various locations in the Puget Sound in 2008 (Feely et al., 2010). Although the studies have different precision and accuracy and provide large scale, seasonal and spatial ranges, they establish expected ranges and variabilities in the environment.

Expected range of results

Table 5. TA/DIC results from previous studies in the target area of our study.
All results are from surface data.

Study	Location	TA		DIC	
		Minimum	Maximum	Minimum	Maximum
		µM/kg		*µM/kg*	
SPSDO - Roberts et al., 2008					
	Oakland Bay-surface	1860	2130	NA*	NA
	Totten Inlet-surface	1460	2080	NA	NA
	Main Basin-surface	2020	2150	NA	NA
Feely et al., 2010					
	Admiralty Inlet-surface	2107	2129	2032	2053
	Hood Canal-surface	2018	2032	1881	1981
	Main Basin-surface	2041	2052	1884	1998

*SPSDO did not collect DIC.

Sampling Procedures

Ecology field staff method has been informed by Bockmon and Dickson (2014). To analyze DIC and TA, a minimum of 2.5 L of water is needed for one 500 mL water sample. The method uses a peristaltic pump and a filter system to remove particulates (0.45 µm Durapore membrane filters). We will be using a Geofilter 350 RPMs peristaltic pump with silicone tubing. The method allows transportation of the samples to the lab before adding mercuric chloride. This is a critical requirement for in-flight operations and safety on board the plane. We will be collecting the samples aboard a small and unstable seaplane, so post-sampling fixation is a critical step. Based on Leinweber and Gruber (2013), a delayed addition of mercuric chloride will not compromise the integrity of the sample. After preservation, the samples will be stored in a dark walk-in cooler at 35-39°F until delivery at the end of each month.

The marine flight CTD, including the potentiometric electrode for *in situ* SeaBird SBE 18 pH sensor will be deployed at each station. The pH sensor is stored with a pH 4 buffer, per the manufacturer’s recommendations. On the morning of a flight, the buffer is changed to pH 8, priming it for the expected conditions in the Puget Sound waters. The pH 8 buffer is taken off and replaced before and after each cast. This extends the life of the electrode. A CTD cast is conducted at each monitoring station. Each time the CTD is turned on, data are recorded internally (minimum sampling rate is eight scans per second), and this cast is assigned a cast number and time. For each cast, the station name, cast number, and cast start time are recorded in the field log.

The sensors on the CTD are equilibrated at in situ conditions for a minute. The CTD is turned on, lowered into the water until the entire unit is submerged, and held stationary. This time has been empirically established over the years to stably equilibrate the sensor to the environment, although the sensor response is generally within seconds of turning the instrument on. The CTD is then lowered to 1-2 meters above the bottom, at a rate no faster than 0.5 m/sec., held near the bottom for 2 minutes, raised to the surface, and turned off.

Measurement Procedures

Because this is a pilot project, several external SOPs were used to develop the method testing for float plane feasibility. Generated data are under an experimental SOP for which a method does not currently exist. These data will only be used in the development of a SOP for the sampling procedure. If this method proves to be successful, an SOP will be written and the data can then be used.

Field procedures will follow:

- EAP025- Standard Operating Procedure for Seawater Sampling.
- Bockmon, E. E., and A.G. Dickson, A seawater filtration method suitable for total dissolved inorganic carbon and pH analyses. *Limnology and Oceanography*: Methods. 2014.
- Dickson, A.G., C.L. Sabine, and J.R. Christian, editors. 2007. *Guide to Best Practices for Ocean CO_2 Measurements*. PICES Special Publication 3, 191 pp.

Quality Control Procedures

Field

Replicate Sample Collection

We will collect triplicate samples at each station, to determine the representativeness of each station rather than to use as a control on bias.

Laboratory

Laboratory Control Samples

Table 6. The laboratory budget for the projected field samples to be collected for the study.

Sample Type	# of stations	Field Replicates	# of samples	$/sample	Months of sampling	Subtotal
TA/DIC	6	3	18	$125	11	$24,750
Nutrients	6	0	6	17.65	11	$1,165
					Total	$25,915
					Total Budget	$28,000

For testing laboratory performance and analyst proficiency, check standards or laboratory control samples of known concentrations are included with every sample batch. Recovery percentage is calculated from these results and therefore can be used as a measure of analytical accuracy and bias. If the results fall outside of established limits, data associated with the batch are flagged by the reviewer. Any measurement problem that cannot be resolved is given a data quality flag.

Non-detects for analytes of DIC, TA, and pH are not expected for Puget Sound samples and do not require a separate data analysis.

TA/DIC analyses will be conducted by PMEL. PMEL uses Certified Reference Material for oceanic CO_2 measurements produced by Dr. Andrew Dickson at Scripps Institution of Oceanography, University of California, San Diego.

Data Management Procedures

Field measurements will be collected in a field notebook. We will enter data into Microsoft Excel® spreadsheets as soon as possible after we return from the field.

Generated data are experimental because a method does not currently exist. These data will only be used in the development of a SOP for the sampling procedure. If this method proves to be successful, an SOP will be written and the data will be used and stored as described below.

No data generated before the approval and publication of a final SOP will be entered into Ecology's Environmental Information Management (EIM) database or published in any other data system.

Spreadsheet files, paper field notes, laboratory results, and reports will be kept together with the project data files. All digital files are stored on a secure, shared network server.

Audits and Reports

The report will describe the outcome of this pilot project and include recommendation on the feasibility of continuing this type of sampling as part of the marine flight program. Reports on the findings of this study will be completed at the end of the project in October 2015.

Data Verification and Validation

Data Verification

While on station, field staff will check all field logs for missing data before departing for the next station. All laboratory data will be labeled as "Draft" until data verification is complete. The appropriate QA and QC codes will be applied to the data.

Data verification and review are conducted by the Marine Waters Monitoring group by examining all field and laboratory-generated data to ensure:

- Specified methods and protocols were followed.
- Data are consistent, correct, and complete, with no errors or omissions.
- Data specified in the *Sampling Process Design* section were obtained.
- Established criteria for QC results were met.
- Data qualifiers (QC codes) are properly assigned.

Data Validation

Once the sampling is complete, all data are finalized, and all QA, QC and validation are complete, the data will then be vetted by the Puget Sound Ecosystem Monitoring Program (PSEMP) Marine Waters work group. This group includes scientists specialized in ocean acidity.

Data Quality (Usability) Assessment

Sample results from laboratory analyses will be examined for completeness (all samples, all analyses). Processing logs and laboratory reports will be scrutinized for adherence to specified methods and QA/QC requirements.

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Appendix. Glossary, Acronyms, and Abbreviations

Glossary

Alkalinity: measures the ability of a solution to neutralize acids to the equivalence point of carbonate or bicarbonate. The alkalinity is equal to the stoichiometric sum of the bases in solution.

Ambient: Background or away from point sources of contamination.

Conductivity: A measure of water's ability to conduct an electrical current. Conductivity is related to the concentration and charge of dissolved ions in water.

CTD: A set of sensors (conductivity-temperature-depth) combined into an instrument package used for collecting continuous water column profile data. The CTD is equipped with sensors to measure additional variables and a pump to draw water through the sensors. Profiles at each station are collected from the sea surface (top bin = 0.5 m) to the sea bottom. The CTD and sensors are operated and maintained according to manufacturer's recommended protocols, with factory calibration occurring annually.

Dissolved Inorganic Carbon (DIC): The sum of inorganic carbon species in a solution. The inorganic carbon species include carbon dioxide (CO_2), carbonic acid (H_2CO_3), bicarbonate anion (HCO_3^-), and carbonate (CO_3^{2-}).

Nutrient: Substance such as carbon, nitrogen, and phosphorus used by organisms to live and grow. Too many nutrients in the water can promote algal blooms and rob the water of oxygen vital to aquatic organisms.

Ocean Acidification: When carbon dioxide (CO_2) is absorbed by seawater, chemical reactions occur that reduce seawater pH, carbonate ion concentration, and saturation states of biologically important calcium carbonate minerals. These chemical reactions are termed *ocean acidification*. Calcium carbonate minerals are the building blocks for the skeletons and shells of many marine organisms. In areas where most life now congregates in the ocean, the seawater is supersaturated with respect to calcium carbonate minerals. This means there are abundant building blocks for calcifying organisms to build their skeletons and shells. However, continued ocean acidification is causing many parts of the ocean to become undersaturated with these minerals, which is likely to affect the ability of some organisms to produce and maintain their shells.

Parameter: A physical chemical or biological property whose values determine environmental characteristics or behavior.

Peristaltic Pump: is a type of positive displacement pump used for pumping a variety of fluids. The fluid is contained within a flexible tube fitted inside a circular pump casing.

pH: pH is the negative logarithm of the activity of the (solvated) hydronium ion, more often expressed as the measure of the hydronium ion concentration. pH is a measure of the acidity and basicity of water. A low pH value (0 to 7) indicates that an acidic condition is present, while a high pH (7 to 14) indicates a basic or alkaline condition. A pH of 7 is considered to be neutral. Since the pH scale is logarithmic, a water sample with a pH of 8 has ten times less hydronium ion activity than one with a pH of 7.

Potentiometric electrodes: Potentiometry passively measures the potential of a solution between two electrodes, affecting the solution very little in the process. The potential is then related to the concentration of one or more analytes.

Acronyms and Abbreviations

PMEL	Pacific Marine Environmental Laboratory
TA	Total Alkalinity
DIC	Dissolved inorganic carbon
CO_2	Carbon dioxide
pCO_2	Partial pressure of carbon Dioxide, the amount of CO2 in the atmosphere
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management
et al.	And others
GPS	Global Positioning System
MQO	Measurement quality objective
QA	Quality assurance
QC	Quality control
RPD	Relative percent difference
RSD	Relative standard deviation
SOP	Standard operating procedures
ORNL	Oak Ridge National Laboratory
CDIAC	Carbon Dioxide Information Analysis Center
DAAC	Distributed Active Archive Center
PSEMP	Puget Sound Ecosystem Monitoring Program

Units of Measurement

m	meter
mL	milliliters
L	liters
µm	micromolar
µm/kg	micromolar per kilogram