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State of Washington

Standard Operating Procedure EAP088, Version 1.1

Marine Waters Data Quality Assurance and Quality Control

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Purpose of this Document

The Washington State Department of Ecology develops Standard Operating Procedures (SOPs) to document agency practices related to sampling, field and laboratory analysis, and other aspects of the agency's technical operations.

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Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by Ecology.

Although Ecology follows the SOP in most instances, there may be instances in which Ecology uses an alternative methodology, procedure, or process.

SOP Revision History

Revision Date	Revision History	Summary of Changes	Sections	Reviser(s)
05/13/2019	1.0	Draft to recertification	All	Arati Kaza
09/12/2022	1.1	Recertification. Reformatted tables. Added alternative text to figures and tables. Minor edits.	All	Julia Bos, Suzan Pool

1.0 Purpose and Scope

1.1 This document is the Environmental Assessment Program (EAP) Standard Operating Procedure (SOP) for a system of quality assessment (QA) and quality control (QC) procedures conducted for marine water quality data collected under the long-term Marine Waters Monitoring (MWM) Program.

1.2 Marine waters monitoring is driven by key questions about the long-term conditions of Washington's marine waters. Part of answering these questions is determining the data to collect and setting quality objectives to ensure the data can fulfill the needs. Good QA procedures are used to determine if data collected meets the quality objectives. High data quality is mandatory for Ecology's Long-Term Monitoring Program and ensure that trends accurately reflect true environmental change. We have implemented an overall data quality assessment (QA) system which includes routine data quality control (QC) procedures during all phases of the data life cycle including internal peer group reviews to ensure that our data meet highest quality standards. Data quality codes are applied to datasets allowing users to decide the appropriate level of quality for their analyses.

1.3 This document describes test procedures for QC of measurements and analyses performed on marine waters data that are part of the overall QA system. Observations covered by these procedures are collected as a measure of water quality in Washington state marine water bodies, some in real-time or near-real-time settings. Many of these procedures were established in 1991 for the Puget Sound Water Quality Authority, after the Marine Waters Monitoring Program was formalized and mandated by the state legislature (PSEP, 1991; PSEP 1997). Many of these procedures have been updated and improved over the years, as methods and technology has evolved.

1.4 Post-processing and post-deployment data treatment and adjustment issues are not part of the scope of this document.

2.0 Applicability

2.1 This SOP represents a set of tests and procedures for a variety of data types. The goal is to improve QA/QC through documented, reproducible standard processes. Although certain tests are recommended, thresholds for tests may vary among and within the Marine Waters Monitoring programs and projects, depending upon technology, location seasonality and type of deployment and sampling. For example, the upper limit for DO observations for an instrument moored in deeper coastal waters might not be suitable for use in a shallow nutrient-rich bay.

3.0 Definitions

The following list of definitions includes terms relevant to the Marine Waters Monitoring Program. There may be undefined terms used in this document and it is assumed the user can infer the meaning of these terms. An extensive list of definitions specific to QA and QC can be found on Ecology's QA website.

3.1 **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.

3.2 **Blank:** A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process. (Jones, 1999)

3.3 **Calibration:** The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured.

3.4 **Check Standard:** A substance or reference material obtained from a source independent from the calibration standard source; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards, but should be referred to by their actual designator. (i.e., CRM, LCS, etc.) (Lombard and Kirchmer, 2004; Kammin, 2010)

3.5 **Chlorophyll *a*:** Chlorophyll is the pigment that allows plants, including algae, to convert sunlight into organic compounds in the process of photosynthesis. Chlorophyll *a* is the predominant type of this pigment found in algae and phytoplankton, and its abundance can be used as an indicator of the amount of algae present in seawater.

3.6 **Clarity:** A qualitative measurement of the ability of water to transmit light. Clarity can be assessed using transmissometer and turbidity sensors (see 3.45).

3.7 **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.

3.8 **Continuing Calibration Verification Standard (CCV):** A quality control sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run. (Kammin, 2010)

3.9 **Control Chart:** A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system. (Lombard and Kirchmer, 2004; Kammin, 2010)

3.10 **Control Limits:** Statistical warning and action limits calculated based on control charts. Warning limits are generally set at +/- 2 standard deviations from the mean, action limits at +/- 3 standard deviations from the mean. (Kammin, 2010)

3.11 **CTD (Conductivity-Temperature-Depth):** A set of sensors that is combined into a submersible instrument package used for collecting continuous data of conductivity, temperature, and depth in the water. The CTD can be equipped with auxiliary sensors to measure additional variables and a pump to draw water through or pass by the sensors. The CTD and auxiliary sensors are operated and maintained according to manufacturer's recommended protocols, with factory calibration occurring annually. It is commonly used in both marine and freshwater applications.

3.12 **Derived Data:** Derived data are defined or calculated using other data, called base or primary (raw) data. An example of a derived data variable is the density of seawater, calculated using salinity, temperature, and pressure, based on the equation of state for seawater.

3.13 **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-}).

3.14 **Dissolved Oxygen (DO):** The amount of gaseous oxygen (O_2) dissolved in water. Oxygen gets into water by diffusion from the surrounding air, by aeration (rapid movement), and as a product of photosynthesis. It is consumed by respiration and decay processes, as well as in some chemical reactions. Dissolved oxygen levels are used as an indicator of water quality.

3.15 **Fluorometer:** An instrument that provides an indication of the concentration of a given material by measuring the amount of fluorescence attributed to the material. For example, a fluorometer provides an excitation beam at a wavelength that is known to cause fluorescent emission from chlorophyll and measures light at a wavelength that matches the chlorophyll emission. As a result, the amount of chlorophyll-containing algal biomass can be estimated through in situ fluorescence.

3.16 **Initial Calibration Verification Standard (ICV):** A quality control sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples. (Kammin, 2010)

3.17 **Instrument Detection Limit (IDL):** The minimum quantity of analyte or the concentration equivalent which gives an analyte signal equal to three times the standard deviation of the background signal at the selected wavelength, mass, retention time, absorbance line, etc.

3.18 **Interquartile Range:** In descriptive statistics, the interquartile range (IQR) is a measure of variability. Quartiles divide a rank-ordered data set into four equal parts. The IQR is equal to the difference between the upper and lower quartiles, such that 25% of the results are above and below those values, respectively. IQR = Q3 − Q1. It is the most significant basic robust measure of scale.

3.19 **Laboratory Control Sample (LCS):** A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples. (U.S. EPA, 2016)

3.20 **Material Safety Data Sheet (MSDS):** MSDSs provide both field staff and emergency personnel with proper procedures for handling or working with a particular substance. MSDSs include information such as physical data (e.g., melting point, boiling point, flash point, etc.), toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill/leak clean up procedures.

3.21 **Measurement Quality Objectives (MQOs):** Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness. (U.S. EPA, 2006)

3.22 **Method Blank:** A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples. (Lombard and Kirchmer, 2004; Kammin, 2010)

3.23 **Method Detection Limit (MDL):** MDL is defined there as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero. (Code of Federal Regulations, 2012)

3.24 **Niskin Bottle:** Water sampling bottle used to collect sub-surface water for subsequent measurements. Niskin bottles are plastic tubes (PVC) with spring-loaded end caps, an air-vent valve at one end and a dispensing stopcock at the other.

3.25 **Nutrient:** A substance such as nitrate, nitrite, silicate, ammonium and phosphate. These compounds are used by organisms to live and grow. Nutrient measurements are used as an indicator of water quality.

3.26 **Parameter:** A distinguishing physical, chemical or biological property whose values determine environmental characteristics or behavior.

3.27 **Particulate Organic Carbon (POC):** Particulate matter is defined as suspended particles in seawater having a size greater than 0.45 uM. The particulate organic carbon fraction of total organic carbon is defined as organic matter that is larger than 0.45 uM. POC inputs to the sea are divided into two categories: allochthonous inputs from land and atmosphere and autochthonous (internal) inputs from biogenic material formed from in situ photosynthesis or decomposition of organic matter or organisms.

3.28 **Particulate Organic Nitrogen (PON):** The fraction of particulate nitrogen that is from biogenic material, such as material formed from in situ photosynthesis or decomposition of organic matter or organisms

3.29 **Percentile:** An estimated portion of a sample population based on a statistical determination of distribution characteristics. For example, the 90th percentile value is a statistically derived estimate of the division between 90% of samples, which should be less than the value, and 10% of samples, which are expected to exceed the value.

3.30 **Percent Relative Standard Deviation (%RSD):** A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$\%RSD = 100 * (s/x)$$

where s = sample standard deviation, and x = sample mean (Kammin, 2010)

3.31 **pH:** A measure of the acidity or alkalinity 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 is ten times more basic than one with a pH of 7.

3.32 **Photosynthetically Active Radiation (PAR):** Wavelengths, roughly 400 - 700 nanometers, of incoming sunlight that can be absorbed by plants for photosynthesis.

3.33 **Phytoplankton:** Free-floating aquatic flora that convert inorganic compounds into complex organic compounds using light. This process of primary productivity supports the pelagic food-chain. Phytoplankton vary in size from less than 1 to several hundred µm.

3.34 **Quality Assurance (QA):** A set of activities designed to establish and document the reliability and usability of measurement data. (Kammin, 2010)

3.35 **Relative Percent Difference (RPD):** RPD is commonly used to evaluate precision. The following formula is used:

Abs|a-b|/((a+b)/2) * 100

Where a and b are 2 sample results, and abs() indicates absolute value

RPD can be used only with 2 values. If there are more than two values, use %RSD. (Lombard and Kirchmer, 2004)

3.36 **Replicate Samples:** Two or more samples taken from the environment at the same time and place, using the same protocols followed for regular samples. Replicates are used to estimate the random variability of the material sampled. (Jones, 1999)

3.37 **Salinity:** Salinity is the total amount of dissolved material in grams in one kilogram of sea water.

3.38 **Secchi Disk:** Measures transparency of the water using an 8-inch diameter white disk attached to a rope. The rope is marked at 0.5 meter intervals for easy determination of depth. This also specifies the depth resolution of the measurement.

3.39 **Secchi Depth:** Depth in the water at which a deployed secchi disk is no longer visible. It is usually the average between the depth at which the disk is no longer visible when it is lowered into the water and the depth at which it is again visible as the disk is raised. The secchi depth can be used to estimate the amount of colored substances (i.e., phytoplankton, algae, and detritus) in the water. Changes can be caused by sediment runoff from land or increased phytoplankton populations. Changes in secchi depth over time are used as an indicator of water quality.

3.40 **Secondary Data:** Data from sources other than the Marine Waters Monitoring Program, used for advanced analyses or contextual interpretation of monitoring data. An example of a secondary data source is stream flow data from the USGS.

3.41 **Sediment:** Soil and organic matter that is covered with water (for example, river or estuary bottom).

3.42 **Spiked Blank:** A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method. (U.S. EPA, 2016)

3.43 **Total Nitrogen (TN):** Total nitrogen is the amount of nitrogen found in water and consists of dissolved nitrogen (DN) and particulate nitrogen (PN) of either organic or inorganic sources.

3.44 **Total Organic Carbon (TOC):** Total organic carbon is the amount of carbon found in an organic compound and is often used as a non-specific indicator of water quality. Total organic carbon consists of dissolved (DOC) and particulate organic carbon (POC) and is therefore affected by pronounced fluctuations in suspended solids in riverine systems. Sources of organic carbon in fresh and marine waters include living material and waste materials and effluents. Organic matter from living material may arise directly from plant photosynthesis or indirectly from terrestrial organic matter.

3.45 **Transmissivity (light transmission):** A measure of light scattering and absorption through a defined distance of the water, reported as a percent or ratio of light received relative to light that was originally transmitted. Light transmission is used as an indicator of water quality, providing information about water clarity, light absorption and light scattering (beam attenuation)

3.46 **Turbidity:** A measure of water clarity at a specified wavelength of light. High levels of turbidity can have a negative impact on aquatic life.

3.47 **303(d) list:** Section 303(d) of the federal Clean Water Act, requiring Washington State to periodically prepare a list of all surface waters in the state for which beneficial uses of the water – such as for drinking, recreation, aquatic habitat, and industrial use – are impaired by pollutants. These are water quality-limited estuaries, lakes, and streams that fall short of state surface water quality standards and are not expected to improve within the next two years.

4.0 Personnel Qualifications/Responsibilities

4.1 Experience with oceanographic or marine waters data collection, analysis and interpretation.

4.2 Training and experience working with basic statistical and graphical analysis.

4.3 Training and experience with software programs, e.g., Excel, PowerPoint, and, if possible, some MATLAB, R, or other statistical software and tool development skills.

4.4 Typical job class performing SOP: Natural Resource Scientist 1/2/3/4, Environmental Engineer 1/2/3/4/5, Environmental Specialist 1/2/3/4/5.

5.0 Equipment, Reagents, and Supplies

5.1 Equipment consists of computer hardware and appropriate software with connection to data files and databases stored on shared Ecology network servers.

6.0 Summary of Procedure

6.1 QA system description.

6.1.1 The ongoing effort to provide high quality data occurs in many steps before, during and after data collection. Figure 1 provides a high-level summary of our QA system and QC steps.

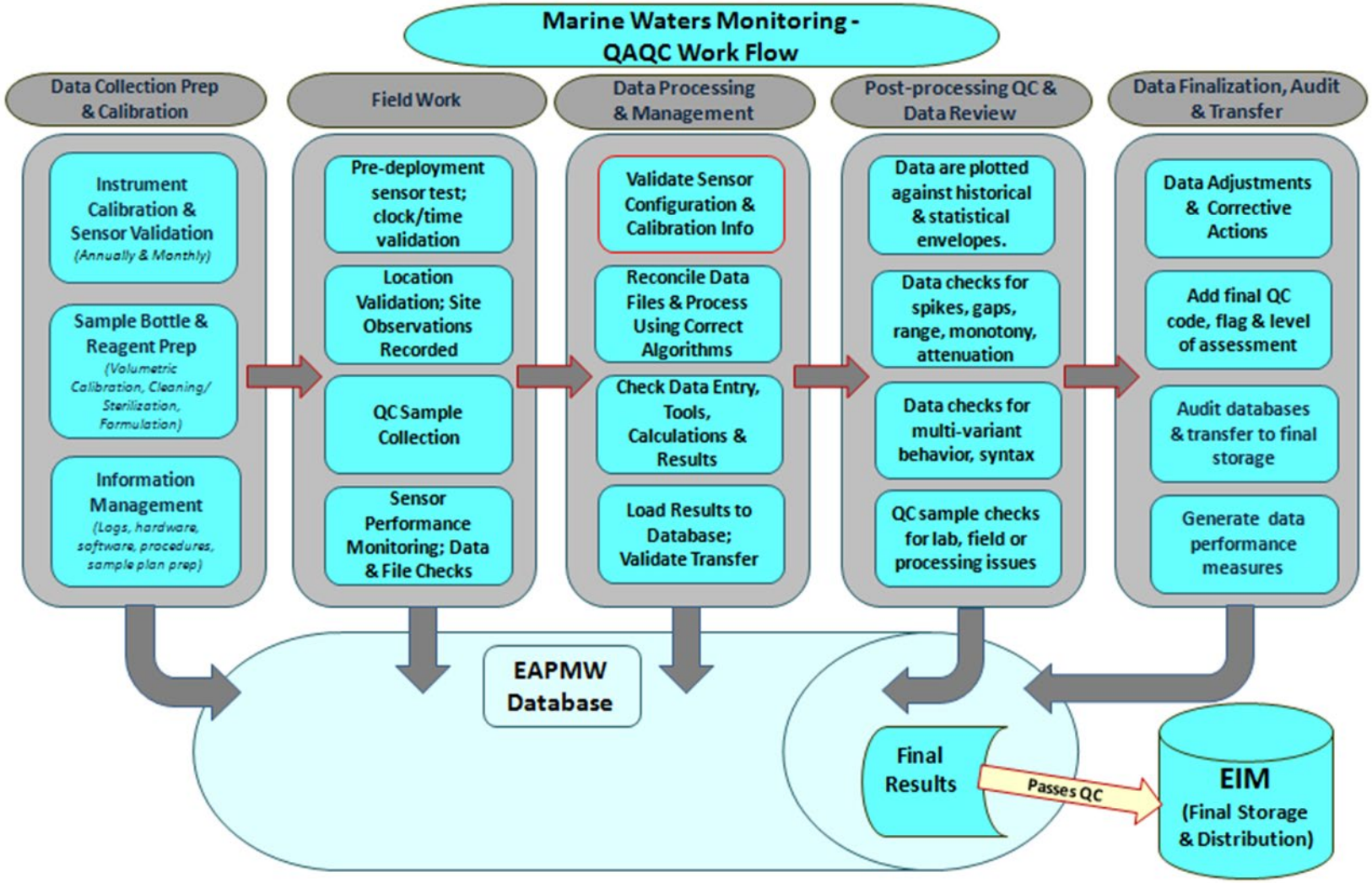


Figure 1. Marine Waters Monitoring Program's data QA system with QC steps.

6.2 Essentially, all QA and QC activities for the Marine Waters Monitoring Program are categorized by steps in our workflow that occur at routine intervals in the data life cycle:

6.2.1 Preliminary QC – data collection prep and calibration

6.2.2 Data Collection QC – activities during field sampling & related work

6.2.3 Post-Data Collection & Analysis QC – activities after field work such as lab sample analysis QC procedures & sensor checks

6.2.4 Data Processing & Management QC – activities during data entry, calculations, processing and management

6.2.5 Post-Processing QC & Data Review – analytical tests and activities such as statistical analyses, sensor signal checks, and contextual checks

6.2.6 Final Data QA & Audits – activities such as database, web and product audits, to determine if data quality objectives have been met

6.3 Our QA system includes multiple actions to ensure all data collection, reporting and analyses are of high quality and appropriate for assessing marine water quality. We emphasize using standard, validated and scientifically recommended procedures which are thoroughly documented and independently reviewed for appropriate and correct application. Our QA system includes the following key elements incorporated into the data life cycle.

6.3.1 Meeting QA/QC objectives.

6.3.2 Training and performance checks of personnel.

6.3.3 Calibrating/validating equipment and proper maintenance.

6.3.4 Performing proper sample custody.

6.3.5 Performing proper data and information management.

6.3.6 Conducting repetitive sensor performance assessment or verification.

6.3.7 Field measurement and analytical laboratory QC procedures.

6.3.8 Data verification and validation through routine data review.

6.3.9 Periodic data usability (method) assessment.

6.3.10 Conducting audits.

6.3.11 Performance measure evaluation.

6.4 The first five activities are discussed at length in Quality Assurance Monitoring Plans (QAMPs) with specific application to the different Marine Waters Monitoring programs. Sensor performance assessment procedures to validate sensors are described in QAMPs, and relevant SOPs while treatment of the assessment results are discussed briefly in this SOP. The last five elements on the list - analytical lab and field QC procedures, data verification and validation through data review and data usability assessments are described in this SOP. Conducting audits and performance measure evaluation are described in program QAMPs, and briefly in this document.

6.5 These procedures are conducted using any current and available oceanographic data QA/QC standards. Yet, current practices and technologies for oceanographic sampling and marine monitoring continue to evolve. Different types of data (sensor, discrete laboratory sample analyses, field observations) require unique data QC techniques. As technology evolves, steps in the QC process change also. Therefore, the current routines used for QA/QC activities for data review and assessment are published and updated every 3 years in this SOP.

6.6 Field measurement (CTD or Sensor) QC procedures – before and during data collection.

6.6.1 A major prerequisite for establishing QC standards for field sensor data collection is a strong QA program. A national consensus amongst a broad group of oceanographers and marine scientists is that good QC requires good QA, and good QA requires dedicated, good scientists, engineers, and technicians. An effective QA effort continuously strives to ensure that end data products are of high value and to prove they are free of error. (U.S. IOOS, 2012)

6.6.2 For this reason, the Marine Waters Monitoring Program has implemented multiple levels of QA to test performance and operation of sensors before, during and after deployment and engage in routine, frequent assessment to determine if measurement procedures are functioning as expected and generating high quality data. Technicians routinely collect a variety of quality control samples and conduct evaluations to test whether quality objectives are being met, in the field and in the lab. After data collection and processing, data is subjected to several QC tests, including coordinated statistical and graphical review by multiple staff members. Each datum is given an overall “pass” or “fail” QC code, any qualifying QC flags, and a code for level of assessment. Tables 1 to 3 list the system of current QC codes used by the Marine Waters Monitoring Program.

Table 1. QC code definitions of the data quality values applied to Marine Waters Monitoring Program's data. The data quality value represents the first character of the 3-character QC codes.

Data Quality Value	Definition	Description
0	None	Data quality not yet determined
1	Fail	Data fails QC, unacceptable
2	Pass	Data passes QC, acceptable

Table 2. QC code definitions of the data quality flags applied to Marine Waters Monitoring Program's data. The data quality flag represents the second character of the 3-character QC codes.

Data Quality Flag	Specific to Laboratory Data	Definition	Description
0	No	No Specification	No specific reason given for pass or fail.
1	No	Sensor or equipment performance	Inconsistent instrument performance.
2	No	Procedure modification	Data collection method modified from standard procedures.
3	No	Method limitation	Method limitation.
4	No	Outlier	Discontinuous or unexpected single result.
5	No	Data behavior	Unexpected or unlikely continuous data pattern.
6	No	Out of range	Data exceeds engineering range specified for instrument, valid range for datatype, range based on climatology or range that calculation should allow.
7	No	Estimate or missing information	Result is an estimate or is missing underlying source or related information needed for validation.
8	No	Non-survey	Result, such as sensor equilibration data, collected during operations but not considered to be an ambient measurement.
9	No	Calculated	Data generated by calculation from other measurements.
JB	Yes	Blank contamination	Analyte found in blank.
JE	Yes	Exceedance of calibration	Reported result is an estimate because it exceeds the calibration range.
JH	Yes	Holding time exceedance	Analyzed past recommended holding time; recommended holding conditions not met.
J	Yes	Estimate	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
JM	Yes	Method modification	Analysis or data collection method modified from routine practices.

Data Quality Flag	Specific to Laboratory Data	Definition	Description
M	Yes	Missing result	Sample collected but lost in transit or lab; result not returned by lab.
NAF	Yes	Not analyzed for	Not analyzed for.
NC	Yes	Not calculated	Not calculated.
R	Yes	Rejected	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
U	Yes	Undetected	The analyte was not detected at or above the reported sample quantitation limit.
UJ	Yes	Undetected, but limits insufficient to generate accurate results	The analyte was not detected at or above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of the of quantitation necessary to accurately measure the analyte in the sample.

Table 3. QC code definitions of the data quality assessment levels applied to Marine Waters Monitoring Program's data. The assessment levels represent the level of data processing and quality control to which the data have been subjected. The data quality flag represents the third character of the 3-character QC codes.

Data Quality Assessment	Definition	Description
0	None	No quality control done
1	Preliminary	Automated processing done and initial value generated
2	Reviewed	Manually reviewed; data flags applied
3	Final	Review is final

6.7 Table 4 lists criteria for quality objectives specified for marine water column variables, including precision, accuracy, measurement ranges and reporting limits. Table 5 lists basic analytical procedures used to test that these objectives are met. Since the tests performed for these assessments may change with advancing technology in sensor or laboratory methods, this SOP will be updated every 3 years. The overall QA/QC objectives may change depending on the monitoring plan, study design or with advancing technology in sensor or laboratory methods. Any changes are noted in annual updates to be published as an addendum to the monitoring QAMPs.

Table 4. A summary of quality control objectives, measurement ranges, and reporting limits for field sensor measurements.

Field Measurement	Precision (relative standard deviation, RSD)	Bias (% deviation from true value)	Manufacturer (Model Number)	Manufacturer Reported Range	Manufacturer Reported Accuracy	Lowest Value
Chlorophyll Fluorescence	10%	5%	WET Labs, Inc. (ECOFL-NTU)	0 to 50 µg/l	0.025 µg/l, reported as "sensitivity"	0.1 µg/l
Conductivity	10%	5%	Sea-Bird Electronics (SBE 4)	0.0 to 7.0 Siemens/meter (S/m)	0.0003 S/m	1 uS/cm
Density	10%	5%	Sea-Bird Electronics	dependent on temperature and conductivity	dependent on temperature and conductivity	0.1 sigma-t
Dissolved Oxygen	5%	5%	Sea-Bird Electronics (SBE 43)	0 to 120% of saturation	2% of saturation	0.05 mg/L
Light Transmission	10%	5%	WET Labs, Inc. (C-Star)	0 to 100%	99% R^2, reported as "linearity"	0.01%
Nitrate	2.4 uM	10%	Sea-Bird Electronics (SUNA Vx)	0 to 200 uM, reported as "best"	2 uM	0.3 uM
pH	0.1 pH	N/A	Sea-Bird Electronics (SBE 18)	0 to 14 pH	0.1 pH	0.1 pH

Field Measurement	Precision (relative standard deviation, RSD)	Bias (% deviation from true value)	Manufacturer (Model Number)	Manufacturer Reported Range	Manufacturer Reported Accuracy	Lowest Value
Pressure	5%	1%	Sea-Bird Electronics (SBE 29)	0 to 500 m	0.1% of full scale range	0.1 db
Secchi Depth	0.5 m	N/A	N/A	N/A	N/A	N/A
Temperature	0.025 °C	0.05 °C	Sea-Bird Electronics (SBE 3)	Negative 5.0 to positive 35 °C	0.001 °C	0.01 °C
Turbidity	10%	5%	WET Labs, Inc. (ECOFL-NTU)	0 to 25 NTU	0.01 NTU	0.1 NTU

Table 5. A summary of quality control objectives and QC procedures for field sensor measurements. (Y = yes, N = no)

Field Measurement	Precision (relative standard deviation, %RSD)	Accuracy (% from true value)	Manufacturer Calibration Report Reviewed	Pre-deployment Performance Assessment via Lab Seawater Bath	In Field Performance Assessment	Preliminary Processing and Flagging of Raw Data	Graphical and Statistical Data Review and Flagging	Adjustment Based Performance Assessments	Performance Assessment Based on Independent Discrete Water Samples	Annual Review and Final Data Assessments
Conductivity	10%	5%	Y	Y	Y	Y	Y	N	N	Y
Density	10%	5%	Y	Y	Y	Y	Y	N	N	Y
Dissolved Oxygen	5%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Fluorescence	10%	5%	Y	N	Y	Y	Y	Y	Y	Y
Light Transmission	10%	5%	Y	Y	Y	Y	Y	N	N	Y
Nitrate	10%	10%	Y	Y	Y	Y	Y	N	Y	Y

Field Measurement	Precision (relative standard deviation, %RSD)	Accuracy (% from true value)	Manufacturer Calibration Report Reviewed	Pre-deployment Performance Assessment via Lab Seawater Bath	In Field Performance Assessment	Preliminary Processing and Flagging of Raw Data	Graphical and Statistical Data Review and Flagging	Adjustment Based Performance Assessments	Performance Assessment Based on Independent Discrete Water Samples	Annual Review and Final Data Assessments
pH	10%	10%	Y	Y	Y	Y	Y	N	N	Y
Pressure	5%	1%	Y	Y	Y	Y	Y	N	N	Y
Salinity	10%	5%	Y	Y	Y	Y	Y	N	Y	Y
Temperature	1%	1%	Y	Y	Y	Y	Y	N	N	Y
Turbidity	10%	5%	Y	N	Y	Y	N	N	N	Y

6.8 QC procedures start prior to sensor deployment with industry-standard, well-controlled sensor calibration by a manufacturer at the factory. The primary instrument used for Marine Waters Monitoring is a Sea-Bird Electronics, Inc. (SBE) CTD package. The CTD is a system composed of multiple specialized sensors that will give accurate and precise results when properly calibrated and maintained. High quality, controlled manufacturer calibrations help assure that quality objectives can be met. Maintenance and calibration procedures are fully described in various operating manuals and application notes for the specific sensors used. A full list of sensor models is included in Table 5. References for specific manuals and application notes for each sensor can be found at various manufacturer websites, including Sea-Bird Electronics, Inc., and related companies, WET Labs and Satlantic. Calibrations are performed at the factory for all sensors on an annual or bi-annual basis, with servicing and repairs occurring as needed. With each calibration, the manufacturer generates a new set of calibration coefficients. In addition to providing a new set of calibration coefficients, the manufacturer also reports on drift and loss of sensitivity relative to the previous calibration. The most recent calibration coefficients are applied to the data during processing prior to storage in the database.

6.9 A schedule listing the frequency of factory calibrations is shown in Table 6. The calibration and maintenance schedule tracks age and behavior of sensors over each instrument's operational lifetime. Sensors returning from annual calibrations are tested prior to deployment, using a seawater bath as well as standards and other reasonable tests to determine proper and correct operation. If performance checks and data review indicate that instrument performance may be compromised from the original factory state, the problem is investigated and resolved, and instruments are returned to the manufacturer for diagnostics and repair, as needed. Sensor calibration histories are maintained to track sensor behavior, characterize reasonable operation and correct measurement by each sensor. One current SOP, EAP086, provides information on sensor assessment via controlled seawater baths (Friedenberg et al., 2016).

Table 6. CTD calibration and maintenance schedule. (Y = yes, N = no)

Sensor	Monthly In-House Calibration Performance Assessment	Annual Factory Calibrations
Conductivity[1]	Y	Y
Temperature	N	Y
Pressure	N	Y
Dissolved Oxygen[1,2]	Y	Y
pH[3,4]	Y	Y
Transmissometer[3]	Y	Y

Sensor	Monthly In-House Calibration Performance Assessment	Annual Factory Calibrations
Fluorescence[1]	Y	Y
Turbidity	N	Y
Nitrate[1,3]	Y	Y

[1] Performance checks using in-situ samples.
[2] Monthly performance check via lab bath.
[3] Bi-monthly calibration.
[4] During factory calibrations, pH sensor is checked for internal electrolyte and electrical connections. Probe to be replaced every 6 months.

All calibration/validation data are recorded in appropriate separate sensor forms and archived in the data management file system. Calibration and sensor performance verification results are maintained in the database. Sensor behavior and aging are tracked via control charts or other appropriate analytical tools.

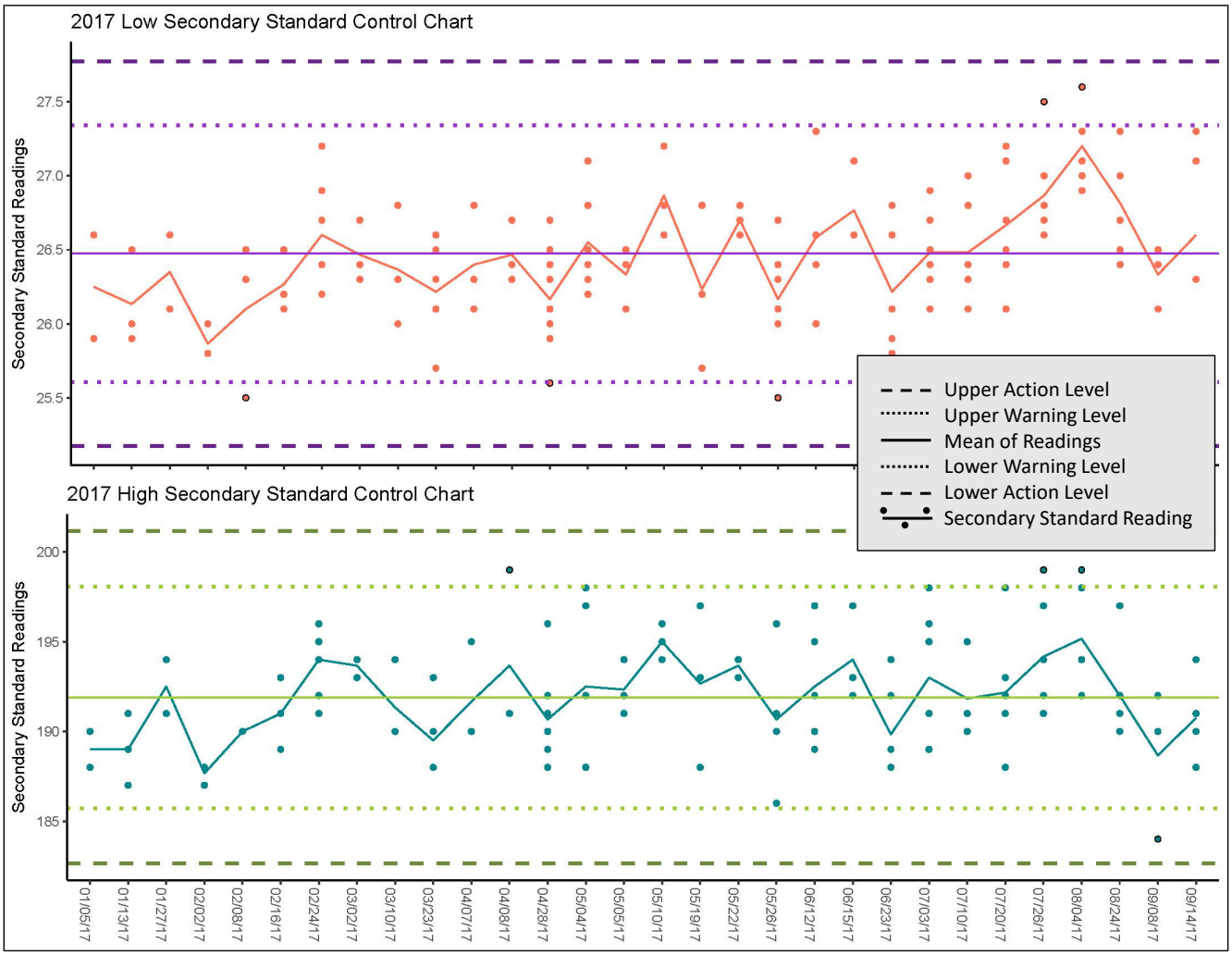


Figure 2. Example of an instrument control chart used to assess and track performance of a Turner Designs fluorometer. Standard deviations are used to determine warning (2x the standard deviation) and action limits (3x the standard deviation) for managing instrument operation.

6.10 Pre-survey performance tests of instruments are conducted and compared to expected value ranges determined by sensor-specific performance testing and to specifications determined during factory calibration. Technicians test instrument packages under controlled conditions to ensure proper operations prior to any field survey. Table 4 lists MQOs for CTD sensor performance testing in the lab.

6.11 During sensor deployments, several test readings are taken, using standards or other available tests to ensure proper configuration and operation. Technicians take voltage and frequency readings during field surveys, before and after deployments to ensure correct operation of all sensors. These tests help identify sensor issues or failures during sampling. Examples of readings from vertical profiles are shown in Table 7. These measurements are compared to ranges (minimum and maximum) of all good test results for each respective sensor using plotting tools. If a problem is detected and confirmed in the field using plotting tools, then data collection is suspended. Data collection starts again once the problem is resolved and the sensor repaired or replaced.

Table 7. Example of raw CTD voltage readings from one month used for pre-survey validation in the field prior to CTD casts. The pH sensor is soaked in pH 8 buffer for reading. No other sensors are controlled using standard reference materials for this test.

Voltage Channel	Voltage Description	Sensor SN	Month Year	Count	Average Voltage	Minimum Voltage	Maximum Voltage
1	CTD Alkaline Batteries	2538854-0381	March 2013	772	2.406	2.166	2.649
2	CTD Lithium Battery	2538854-0381	March 2013	772	1.317	1.278	1.364
3	Pressure	290559	March 2013	772	4.408	4.399	4.422
4	Temperature	34501	March 2013	772	1.452	1.269	1.567
5	Dissolved Oxygen	430049	March 2013	772	2.981	2.267	3.295
6	pH	180530	March 2013	772	2.909	2.128	2.987
7	Transmission	CST-850PR	March 2013	772	2.948	0.337	4.598
8	Fluorescence	FLNTURT-299	March 2013	772	0.091	0.055	0.18

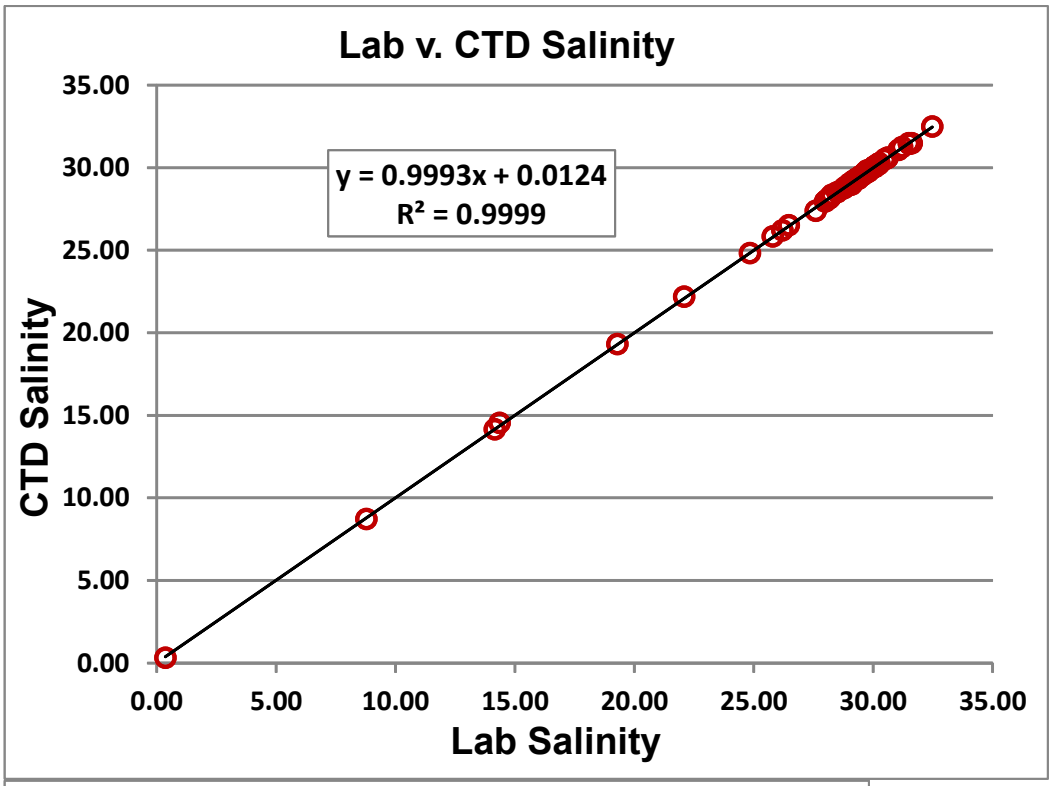
Voltage Channel	Voltage Description	Sensor SN	Month Year	Count	Average Voltage	Minimum Voltage	Maximum Voltage
9	Turbidity	FLNTURT-299	March 2013	722	0.375	0.243	0.603

6.12 CTD QC Sample Collection.

6.12.1 During field deployments, independent QC samples are collected to validate measurements for salinity, dissolved oxygen (DO), chlorophyll *a* fluorescence, and nitrate. Independent QC verification samples provide information about sensor behavior during field deployments between scheduled lab or field bath assessments. These QC samples also provide a way to determine if sensors have drifted, are damaged, or have failed during deployment. Verification samples for salinity measurements, and reference samples for chlorophyll *a* fluorescence, and nitrate are collected during each daily survey to compare with sensor values and verify CTD sensor performance. These reference samples are used to adjust data as appropriate.

6.12.2 Water samples are collected at stations with little to no vessel drift to minimize effects of rapidly changing horizontal water masses. Chlorophyll *a* and nitrate samples are collected from 0, 10 and 30 meters to capture a variety of levels observed in the upper water column. Salinity samples are collected at a few various locations throughout the day to cover a range of expected salinities.

6.12.3 Should the CTD values differ substantially from the analyzed water samples, CTD data are "flagged" until differences are resolved. At the end of a sampling year, as part of data finalization, independent verification sample results are analyzed and used to determine if there were any substantial anomalies in quantitative sensor measurements. If anomalies are found, lab and sensor data are checked for any QC flags such as outlier, gap, or contextual issues which may explain the difference. If the lab sample is good, depending on the severity of the difference (e.g., <0.1 PSU), a “pass” QC flag is applied to the sensor data if no issues are found. Figure 3 shows a typical result for analyses of independent (salinity) lab samples against sensor data, confirming validity of the sensor data.



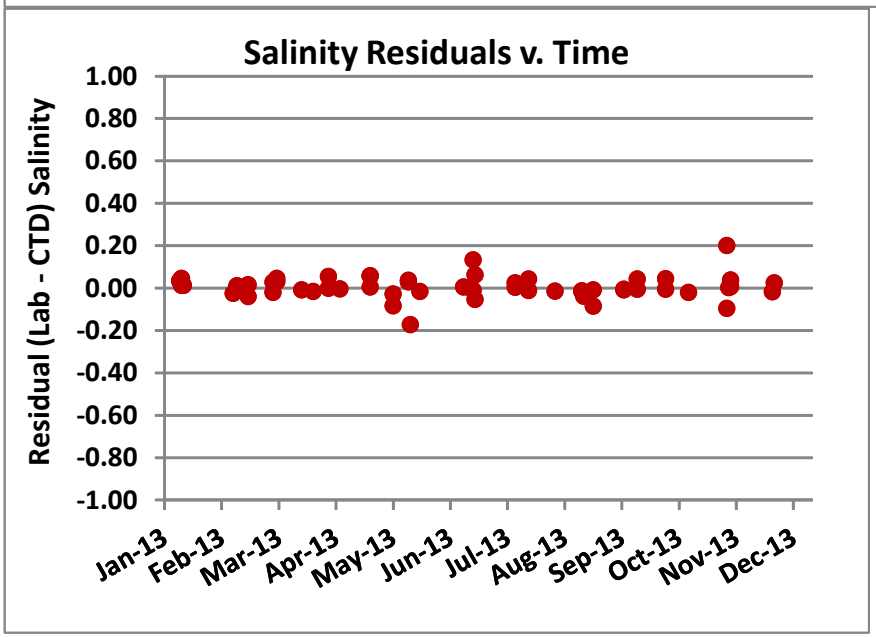


Figure 3. Example plots of sensor validation using independent lab samples.

6.13 Due to the nature of marine water column sampling via a Lagrangian approach, that is drifting with a water parcel and currents rather than holding one static position, replicate CTD casts in the field do not provide a good test of precision. At some sites, currents and winds cause the vessel to drift a significant amount, and along with rapidly changing water conditions, replicate casts collected one after another provide a measure of field variability in space and time rather than a test of CTD precision and accuracy. For this reason, the MWM group uses independent, in situ QC sample collection and lab testing under controlled conditions (e.g., a bath) to perform QA of CTD performance.

6.14 Laboratory-based CTD QC procedures via seawater bath assessment of CTDs.

6.14.1 A seawater bath is set up and maintained at Ecology's Marine Laboratory. This bath is used to assess clean, recently factory-calibrated sensors prior to deployment, and monthly to track sensor performance during the course of a sampling year. More information on this procedure can be found in the SOP EAP086 Version 2 (Friedenberg et al., 2016).

6.14.2 For the laboratory bath procedure, a reference CTD (model SBE 37-SMP-ODO) is used to evaluate the performance of the dissolved oxygen sensors on the field CTDs. The lab reference and field sensors are run side-by-side in a semi-controlled seawater bath where environmental effects are minimized. A side-by-side (paired sample) approach generates a data volume adequate for a statistically robust comparison. For dissolved oxygen, this type of sampling is referred to as "reference sampling" (Sea-Bird Electronics Application Note No. 64-2, June 2012).

6.14.3 Each month during the laboratory bath procedure, the calibration of the reference instrument is checked against laboratory methods to ensure highest data quality. To minimize air exposure and dissolved oxygen bias in Winkler samples, the lab bath is maintained near 100% dissolved oxygen saturation. Winkler samples are collected from the lab bath to coincide with reference CTDs. Dissolved oxygen measurements between the field CTDs, the reference instrument, and the Winkler samples are quantitatively compared to assess both field and reference sensor performance (stability, slope and offset) and whether measurement quality objectives for accuracy and precision are met.

6.14.4 For dissolved oxygen, a sensor passes the instrument-based performance check if values fall within 2% of the reference instrument measurements (i.e., the paired bath measurement values are within 98 to 102% of each other). Any instrument that does not pass performance checks is not deployed and is removed from the instrument pool for additional diagnostics. The reference instrument- is confirmed monthly by laboratory analysis (Winkler DO replicates). The instrument should fall within 5% of the established Winkler to DO sensor ratio, based on ongoing sensor control methods. The Carpenter method for DO titrations is used to determine the dissolved oxygen concentration in collected reference samples (Bos, 2015). Verification DO samples are analyzed by staff in the Ecology's Marine Laboratory.

6.14.5 For pressure, performance is verified in the bath by confirming whether values are near expected pressure values, given the depth of the bath water, and whether there are continuous, stable measurements and general agreement with the reference instrument held at the same depth within the bath.

6.14.6 For salinity, which is derived from the CTD's conductivity measurements, performance is verified based on agreement (difference <0.2 PSU) between the reference CTD and the assessed CTD. In general, sensors are expected to hold their calibration well within measured quality objectives (McPhaden et al., 1990). Verification salinity samples are sent to the UW's Marine Chemistry Laboratory for analysis.

6.14.7 For temperature, sensor performance is based on agreement (difference <0.2 °C) between the reference CTD and the assessed CTD.

6.15 Analytical Laboratory (Discrete Water) Sample QC Procedures – pre- and during sample collection.

6.15.1 QC procedures for discrete water sample results via laboratory analyses start prior to sample collection with several pre-collection activities. These include verification that:

6.15.1.1 Lab instrument calibrations are current and instrument meets control criteria based on standards analysis.

6.15.1.2 All methods and standards are up-to-date.

6.15.1.3 Chemicals and reagents are not expired.

6.15.1.4 All equipment and sample bottles are properly cleaned and prepped, certified or calibrated as required by methods used.

6.15.2 In addition to QC activities that occur before and during sample collection, analytical laboratories perform additional QC procedures throughout sample analyses and result calculations. These procedures are not covered in this SOP and are reported in method procedures or reports generated by each lab.

6.15.3 Prior to sample collection, all information necessary for sample management and analysis is defined and appropriately documented. During collection, information is recorded and verified by a second staff member for correctness and completeness.

6.15.4 Along with regular environmental samples, QC samples are collected or generated at the lab to accompany each batch of samples. These include:

- Blanks, both lab and field
- Replicate samples
- "Standards" or Standard Reference Materials (SRM)
- Lab Control Samples (LCS)
- "Blind" SRMs submitted to the laboratory

6.15.5 The Definitions section of this SOP contains descriptions of various types of QC samples. QC samples have MQOs (evaluation criteria) associated with them and are described in Tables 8 and 9. Specified criteria must be met to obtain fully usable data.

6.16 **Replicate Sample Collection.** Replicate samples for dissolved oxygen, nutrients, and chlorophyll *a* are collected during every long-term monitoring survey to determine field and sample variability. Ten percent of sites are sampled to conduct a quantitative determination of homogeneity of conditions, along with precision and bias of sampling methods.

6.17 **Analytical Replicates.** Total variation in lab samples is assessed by collecting replicates from the same Niskin sampling bottle for all parameters at 5% to 10% of sites. These replicates are used to assess whether the data quality objectives for precision are met. If the objectives are not met, the data are qualified. In addition, Ecology's Manchester Environmental Laboratory, UW's Marine Chemistry Laboratory, and Ecology's Marine Laboratory all routinely perform replicate sample analyses using sample splits within laboratory batches for quality control purposes. The difference between analytical field replicates and laboratory replicate results is a measure of the field sample variability.

6.18 **Laboratory Performance Samples.** 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 is flagged by the reviewer. Any measurement problem that cannot be resolved is given a data quality flag.

6.19 **Blanks.** Blanks are prepared and analyzed in each laboratory to determine if samples were contaminated during processing and analysis. Blanks are run before and after each batch of samples and compared to established acceptance limits. Blank results are reported by each lab and are included with each dataset. Blank results are evaluated by the MWM group and receive final approval from the monitoring coordinator or senior oceanographer.

A positive blank can indicate laboratory contamination. Blanks are important to measure to determine the accuracy of low level samples near the detection limits. Blank responses are used to determine method detection limits (MDLs) and in some cases, to apply data quality flags to sample batches. Table 8 lists the QA/QC samples used to perform quality assessment of laboratory procedures and data results.

Table 8. A summary of quality control steps for analytical laboratory samples. (Y = yes, N = no)

Laboratory Measurement	Precision (relative standard deviation , %RSD)	Accuracy (% from true value)	Instrument Control Check using Blanks	Laboratory Standards Check	Laboratory Control Samples	Replicate Analysis	Method Detection Limits Check	Preliminary Review and Flagging of Raw Data	Graphical and Statistical Data Review and Flagging	Annual Review Assessments
Chlorophyll *a*	10%	NA	Y	Y	N	Y	Y	Y	Y	Y
Dissolved Inorganic Carbon	<0.5%	<0.25%	Y	Y	N	Y	Y	Y	Y	Y
Dissolved Oxygen	5%	NA	Y	Y	N	Y	Y	Y	Y	Y
Nitrate	10%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Nitrite	10%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Ammonium	10%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Orthophosphate	10%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Particulate Nitrogen (PN)	≤20%	5%	Y	Y	N	Y	Y	Y	Y	Y
Particulate Organic Carbon (POC)	≤20%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Silicate	5%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Salinity	10%	5%	Y	Y	N	Y	Y	Y	N	Y
Total Alkalinity	<0.5%	<0.25%	Y	Y	N	Y	Y	Y	Y	Y
Total Organic Carbon (TOC)	≤20%	5%	Y	Y	Y	Y	Y	Y	Y	Y
Total Nitrogen (TN)	≤20%	5%	Y	Y	Y	Y	Y	Y	Y	Y

Table 9. Quality assurance/quality control procedures for water sample analysis and sensor performance testing in the laboratory. This table shows the laboratory samples part of the procedures. Nutrients, dissolved oxygen and chlorophyll *a* laboratory samples are replicated in the field.

Analytical parameter	Calibration and standardization	Lab control (check) samples or standards (30 or less samples)	Replicates (30 or less samples)	Blanks per Batch
Ammonia (NH_4)	5 point standardization	2 to 3	2	2
Nitrite (NO_2)	5 point standardization	2 to 3	2	2
Orthophosphate (PO_4)	5 point standardization	2 to 3	2	2
Silicate (SiO_4)	5 point standardization	2 to 3	2	2
Chlorophyll and phaeopigments	Calibration 1 time per year	4 total using 2 high and 2 low	3	2 for method and 2 for reagent
Dissolved oxygen	3 point standardization	3	3	2
Salinity	1 per batch	1	1	2
Total alkalinity	5 point standardization	1	2	N/A
Dissolved inorganic carbon	2 point calibration (high and low)	1	2	N/A
Particulate organic carbon	5 point standardization	1	2	1
Particulate nitrogen	5 point standardization	1	2	1
Total organic carbon	5 point standardization	1	2	1
Total nitrogen	5 point standardization	1	2	1

Table 10. Quality assurance/quality control procedures for water sample analysis and sensor performance testing in the laboratory. This table shows the sensor part of the procedures.

Analytical parameter	Calibration and standardization	Lab control (check) samples or standards (30 or less samples)	Replicates (30 or less samples)	Blanks per Batch
pH (electrode sensor)	5 point calibration	NA	NA	NA
Light transmission	2 point calibration (high and low)	NA	NA	NA
Dissolved oxygen (Clark cell with membrane)	Standardization with full saturation	NA	NA	NA

6.20 Data Processing QC Procedures

6.20.1 Quality control for data processing consists of a few basic activities, best performed prior to processing to reduce the need for more extensive work later such as tracking down errors and redoing work, and to avoid propagating errors.

6.20.2 Processing and data adjustment activities often are not given adequate attention. This is unfortunate because errors can still occur after data have been collected. Just as field, instrument or technician performance could introduce measurement error, data processing staff may potentially introduce processing error, sometimes systematically. Often a few errors are responsible for the majority of QC issues. To reduce effort, and possibly minimize error, checks are performed throughout the field collection period and data processing rather than waiting until the end of data collection. The burden of QC programming and checking should not be underestimated.

6.20.3 The QC activities during processing are:

- Verifying all source information and files.
- Checking source data and data files for correctness.
- Checking source data and files for completeness (e.g., if 20 samples were collected, there should be 20 sample results).
- Checking data processing tools and software for correct operation, formatting, calculation, and references.
- Documenting any necessary data processing results or confirmations, especially any issues or exceptions that occur during processing and would be informative for further data analyses and finalization.

6.20.4 These activities apply to all types of data collected for processing – sensor measurements, analytical lab samples, and field observations – and to any secondary data used for more advanced analyses or contextual assessment.

6.21 Data verification and validation through routine data review. (Post data processing QC)

6.21.1 One of the most critical phases of quality control occurs during post-processing of all data, prior to comprehensive data analysis. At this step, multiple types of tests and analyses are performed, including statistical and graphical exploration of lab and sensor data.

6.21.1.1 **Post processing QC for laboratory data**

6.21.1.1.1 **QC tests.** All lab data results are subjected to the following tests:

- **Range check.** Do data fall within the expected ranges?
- **Gap or missing value check.** Are any expected results missing?
- **Spurious results check.** Are any values negative or of an unreasonable magnitude?
- **Outlier check.** Do any results fall outside the expected data pattern, either being too high or too low?
- **Climatology check.** Do results seem reasonable compared to historical results – range and pattern?
- **Neighbor check.** Do results seem reasonable compared to results from the same site, same day or similar depths?
- **Seasonality check.** Do results reflect seasonal processes or effects or are they extraordinarily different?
- **Logical relationships check.** Do result fractions from related variables (e.g., total nitrogen and dissolved nitrogen) make sense?

These tests use statistical and graphical analyses, and a suite of numerical and auditing/reconciliation procedures. Figures 4 and 5 show examples of graphs used to determine spurious results, outliers, climatology, seasonality and neighbor checks.

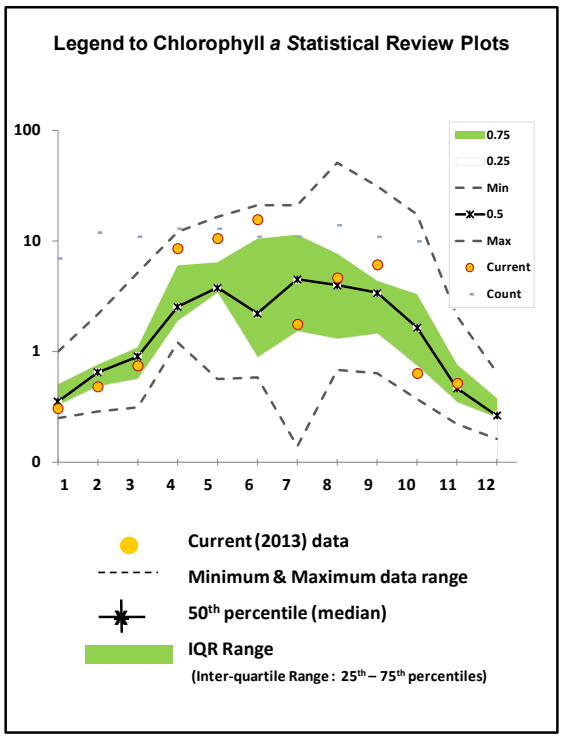


Figure 4. Example of statistical graph for chlorophyll *a* used to apply QC tests to lab sample results.

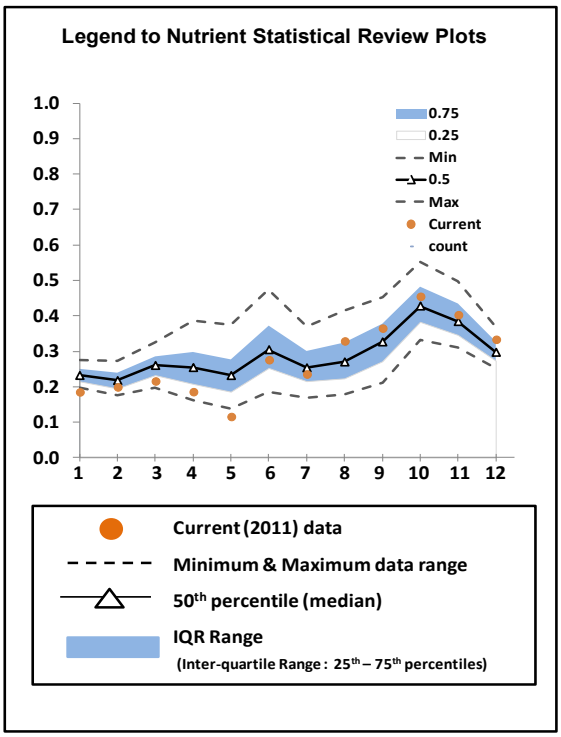


Figure 5. Example of statistical graph for nutrient used to apply QC tests to lab sample results.

6.21.1.1.2 **Analysis of replicates.** All replicate samples are treated as follows:

- **Step 1.** Replicate lab samples are paired with the nearest 0.5 m sampling depth recorded by the Automatic Firing Module (AFM) during vertical profiles.
- **Step 2.** Depths or times between field replicates are compared (e.g., samples collected out of different Niskin bottles at identical depth or differing times). At a vertical difference > 0.25m depth, or a significant time difference (>5 min.) for baths, samples are "disqualified" as replicates and treated as unique samples. We do not deploy moorings at this time, however, this step can be applied to samples collected during prior mooring deployments or field baths.
- **Step 3.** If field replicates meet sampling depth or time criteria, an average, standard deviation and coefficient of variance (relative standard deviation) are calculated. The same metrics are also calculated for lab replicates. This requires that at least 3 replicates be collected for every event.
- **Note:** Variance results outside of MQOs are evaluated to determine if field or lab procedures have created a systematic bias in the results and if samples need to be rejected. Typically, higher variance is associated with samples of very low concentrations. In these cases, small relative differences are checked against proportion of concentration and if the concentration is very low, high variance may be considered acceptable.

6.21.1.1.3 **Analyses of blanks.** For Marine Waters Monitoring lab sample collection, the type of blank used depends on the parameter, and thus the type of analysis for blank results varies by parameter. For each parameter listed in Table 11 below, the type of blank is denoted, along with the test procedure and criteria for passing or failing the test.

Table 11. Laboratory blanks included in Marine Waters quality control analytical procedures.

Analytical Parameter	Type of Blank	Analysis Test Method	Method Criteria	Comment
Dissolved oxygen (mg/L)	Spiked blank	Recovery efficiency	2 blanks run and must be within plus or minus 0.001 uL of each other	Deionized water spiked with surrogate analyte, KIO_3, equivalent to 0.001 normality.
Chlorophyll *a* (µg/L)	DI water; named laboratory reagent blanks (LRB)	Threshold exceedance	< 3 times MDL (based on acetone blanks)	A secondary test determines if blank exceeds 3% of lowest sample concentration.
Chlorophyll *a* (µg/L)	Method blank; named filtration reagent blanks (FRB)	Threshold exceedance	< 3 times LRB	A secondary test determines if blank exceeds 5% of lowest sample concentration.
Nutrient (µM) sample analysis for ortho-phosphate (PO_4), silicic acid (aka silicate; SiO_4), nitrate (NO_3), nitrite (NO_2), and ammonium (NH_4)	Method blank	Threshold exceedance	< 3 times reported blank concentration	Method blank based on seawater matrix with known low level concentrations of analyte. Blank to test reagent contamination.
Particulate sample analysis for particulate organic carbon and particulate nitrogen	Method blank	Threshold exceedance	< 10% analyte level reported for sample	Blank matrix, either pre-combusted filter or sediment capsule.

Analytical Parameter	Type of Blank	Analysis Test Method	Method Criteria	Comment
Total nutrient sample analysis for total organic carbon	Method blank	Threshold exceedance	< 5% to 10% analyte level reported for sample	Reagent water.
Total nutrient sample analysis for total nitrogen	Method blank	Threshold exceedance	< 3 times MDL	Reagent water.
Salinity (PSU)	Method blank	Threshold exceedance	< 3 times MDL	Deionized water.
Dissolved inorganic carbon (µmol/kg)	NA	NA	NA	Blanks not applicable.
Total alkalinity (µmol/kg)	NA	NA	NA	Blanks not applicable.

6.21.1.1.4 **Analyses of Standards.** As for laboratory blanks, standards are analyzed with every batch of lab samples. Depending on the parameter and the type of analyses different types of standards are used. Standards can consist of certified reference materials (CRMs), laboratory control standards (LCSs) and calibration verification standards (CVSs). These standards are used to test for bias in a measurement system. For each parameter listed in Table 12 below, the type of standard is denoted, along with the test procedure and criteria for passing or failing the test.

Table 12. Laboratory standards included in Marine Waters quality control analytical procedures.

Analytical Parameter	Type of Standard	Analysis Test Method	Method Criteria	Comment
Dissolved oxygen (mg/L)	ICV	Recovery efficiency	3 standards run. Must be within plus or minus 0.001 uL of each other.	Deionized water spiked with surrogate analyte, KIO_3, equivalent to 0.01 normality.
Chlorophyll *a* (µg/L)	CRMs, primary standards	Calibration	Establish measurement relationship.	Calibration performed annually.

Analytical Parameter	Type of Standard	Analysis Test Method	Method Criteria	Comment
Chlorophyll *a* (µg/L)	CRMs, secondary standards	Control limits	Results within plus or minus 2 standard deviations of the mean, consistently.	Results within plus or minus 3 standard deviations of the mean, result in corrective action.
Nutrient (µM) samples for five analytes: ortho-phosphate (PO_4), silicic acid (aka silicate; SiO_4), nitrate (NO_3), nitrite (NO_2), ammonium (NH_4).	ICV	Calibration verification	Establish measurement relationship.	Calibration performed before and after every sample run.
Nutrient (µM) samples for five analytes: ortho-phosphate (PO_4), silicic acid (aka silicate; SiO_4), nitrate (NO_3), nitrite (NO_2), ammonium (NH_4).	LCS	Recovery efficiency	Plus or minus 5% of known concentration	"Blind" control samples created with CRMs.
Nutrient (µM) samples for five analytes: ortho-phosphate (PO_4), silicic acid (aka silicate; SiO_4), nitrate (NO_3), nitrite (NO_2), ammonium (NH_4).	CCV	Recovery efficiency	Plus or minus 5% of known concentration	Secondary test to monitor system bias during analytical runs.
Particulate organic carbon	ICV	Calibration verification	Plus or minus 30% of known concentration	Calibration performed before and after every sample run.
Particulate organic carbon	LCS	Recovery efficiency	Plus or minus 10% of known concentration	"Blind" control samples created with CRMs.
Particulate organic carbon	CCV	Recovery efficiency	N/A	Secondary test to monitor system bias during analytical runs.

Analytical Parameter	Type of Standard	Analysis Test Method	Method Criteria	Comment
Particulate nitrogen	ICV	Calibration verification	Establish measurement relationship.	Calibration performed before and after every sample run.
Particulate nitrogen	LCS	Recovery efficiency	Plus or minus 10% of known concentration	"Blind" control samples created with CRMs.
Particulate nitrogen	CCV	Recovery efficiency	N/A	Secondary test to monitor system bias during analytical runs.
Total organic carbon	ICV	Calibration verification	Establish measurement relationship.	Calibration performed before and after every sample run.
Total organic carbon	LCS	Recovery efficiency	Plus or minus 20% of known concentration	"Blind" control samples created with CRMs.
Total organic carbon	CCV	Recovery efficiency	Plus or minus 25% of known concentration	Secondary test to monitor system bias during analytical runs.
Total nitrogen	ICV	Calibration verification	Establish measurement relationship.	Calibration performed before and after every sample run.
Total nitrogen	CCV	Recovery efficiency	Plus or minus 25% of known concentration	Secondary test to monitor system bias during analytical runs.

Analytical Parameter	Type of Standard	Analysis Test Method	Method Criteria	Comment
Salinity (PSU)	CRM, standards	Calibration	Establish measurement relationship.	Calibration performed before and after every sample run.
Dissolved inorganic carbon (µmol/kg)	CRMs, primary standards	Calibration	Establish measurement relationship.	Calibration performed before and after every sample run.
Total alkalinity (µmol/kg)	CRMs, primary standards	Calibration	Establish measurement relationship.	Calibration performed before and after every sample run.

6.21.1.1.5 **Detection Limits.** For each type of analyses, various detection limits are established as a measurement quality objective. For analyses at Ecology's Marine Lab (ML), we establish an Instrument Detection Limit (IDL) based on the analyses of method blanks. For EPA method 445.0 the background is a solution of 90% acetone. For dissolved oxygen analyses, the determination of instrument detection limit is by replication of spiked or fortified blanks within a recovery efficiency range equivalent to +/- 0.001 µL sodium thiosulphate. Any sample batches with blanks that exceed expected IDLs are flagged as an "estimate" due to potential contamination revealed by analyses of blanks.

Method detection limits are established for each analytical lab parameter by analyses of multiple (at least 7) replicates of seawater containing the analyte at 5 times the concentration of the estimated detection limit. Table 13 includes the MDLs for all analytical lab parameters. If any reported sample results fall below the MDL, that sample result is flagged as an "estimate".

Table 13. Method detection limits for Marine Waters laboratory samples.

Laboratory Analyte	Laboratory	Analytical Method	Expected Range of Results	Reporting Limit
Alkalinity	PMEL	Dickson et al., 2007 (SOP3b)	1100 to 2300 µmol/kg	1 µM/kg
Dissolved inorganic carbon	PMEL	Dickson et al., 2007 (SOP2)	1050 to 2300 µmol/kg	1 µM/kg

Laboratory Analyte	Laboratory	Analytical Method	Expected Range of Results	Reporting Limit
Dissolved oxygen	ML	Carpenter, 1966	0.00 to 15.00 mg/L	0.01 mg/L
Nitrate	MCL	Armstrong et al., 1967	0.00 to 40.00 µM	0.15 µM
Nitrite	MCL	Armstrong et al., 1967	0.00 to 2.00 µM	0.01 µM
Ammonium	MCL	Slawyk and MacIsaac, 1972	0.00 to 10.00 µM	0.05 µM
Ortho-phosphate	MCL	Bernhardt and Wilhelms, 1967	0.00 to 4.00 µM	0.02 µM
Silicate	MCL	Armstrong et al., 1967	0.00 to 200.00 µM	0.21 µM
Chlorophyll *a*	ML	Arar and Collins, 1997	0.00 to 60.00 µg/L	0.02 mg/L
Salinity	MCL	Grasshoff et al., 1999	0.00 to 36.00 PSU	0.01 PSU
Particulate Nitrogen (PN)	MEL	Zimmerman et al., 1997	140 to 380 µg/L	1 µg/L
Particulate Organic Carbon (POC)	MEL	Zimmerman et al., 1997	0 to 3000 µg/L	1 µg/L
Total Organic Carbon (TOC)	MEL	Standard Methods, 2022 (method 5310 B)	0 to 3000 µg/L	500 µg/L
Total Nitrogen (TN)	MEL	Standard Methods, 2021 (method 4500-n B)	15 to 50 µM	0.01 µM

6.21.1.2 **Post processing QC for CTD sensor data**

6.21.1.2.1 **QC Tests.** All measurements generated by sensors are subjected to the following tests:

- **Range check.** Do data fall within the expected ranges?
- **Syntax check.** Are sensor outputs reasonable – of proper format and magnitude?
- **Gap or missing value check.** Are any expected results missing?
- **Flat line check**. Are data results abnormally uniform given environmental condition or context?

- **Attenuated signal check.** Are sensor outputs the correct length or number or lines or characters?
- **Rate of change check.** Does the sensor signal exhibit the proper rate of change given environmental conditions or context, or is it too fast or slow?
- **Spurious results check.** Are any values negative or of unreasonable magnitude?
- **Outlier (spike) check.** Do any results fall outside the expected data pattern, either being too high or too low?
- **Climatology check.** Do results seem reasonable compared to historical results – range and pattern?
- **Multi-variant check.** Do sensor results exhibit coherence with related parameters collected or measured at the same time or depth?
- **Neighbor check.** Do results seem reasonable compared to proximal results from the same site, day and adjacent depths?
- **Seasonality check.** Do results reflect seasonal processes or effects or are they extraordinarily different?
- **Logical relationships check.** Do result fractions from related variables (e.g., total nitrogen and dissolved nitrogen) make sense?

6.21.1.2.2 **Statistical Analyses.** These tests are conducted using statistical and graphical analyses, as well as a suite of numerical and auditing/reconciliation procedures. Site-specific statistical evaluation of water column data is conducted every month by the Marine Waters Monitoring group. The interquartile ranges of historical results for each station and each depth are calculated and compared to the current monthly data. An example of this type of plot is shown for station PSB003 in Figure 6. These graphs are used to visually determine gaps, spurious results, outliers/spikes, flat line or unexpected data patterns, climatology, seasonality, multi-variant and neighbor checks. Data that are significantly different than the historical ranges are automatically flagged and reviewed. Any results failing the QC tests are flagged with a QC code of "fail" and are eliminated from further analyses or external data distribution.

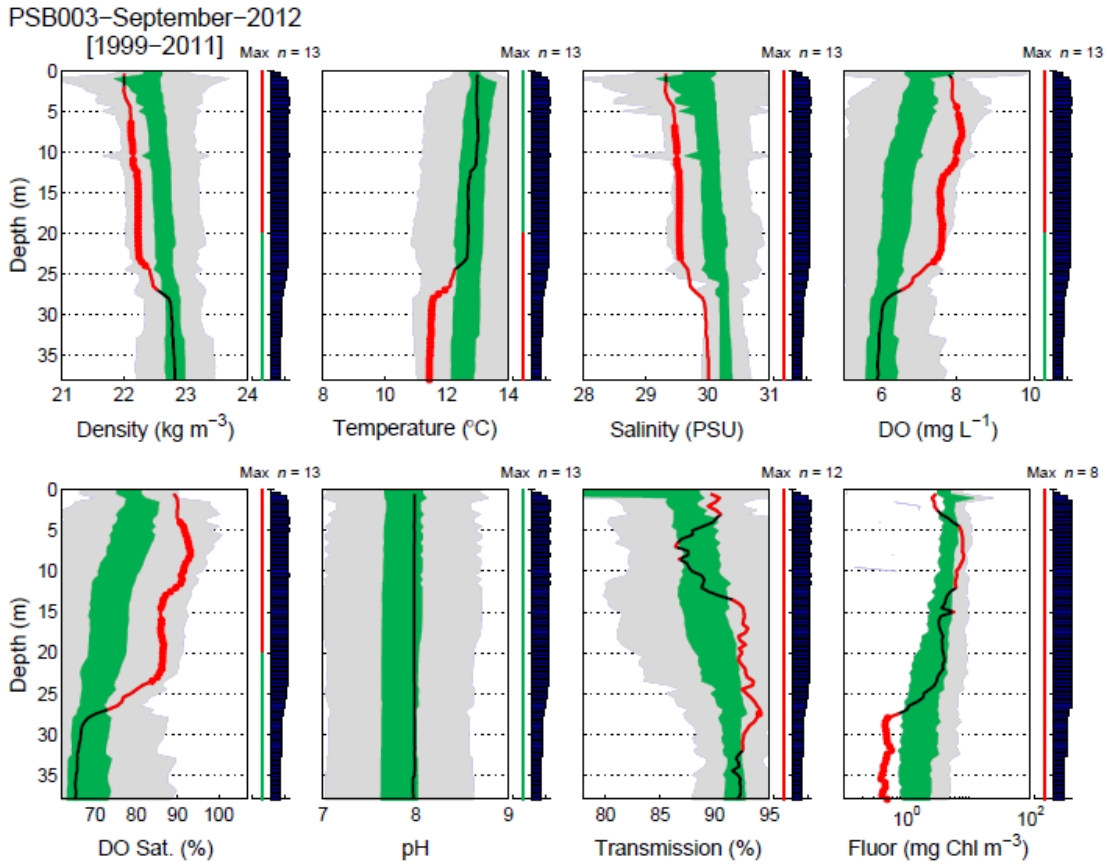


Figure 6. Vertical sensor profile data plotted in context of interquartile ranges based on historical results specific to a station and sampling time of the year. Graphs are used to visually inspect the temporal context of measurements and used to apply QC tests to sensor measurement results.

6.21.1.2.3 **Additional QC actions.** Other conditions warrant further review/research and follow up actions to correct or understand whether data passes or fails quality objectives. These include:

- Missing data.

- Values that exceed detection limits (data at, below or above detection limits).
- Weather or environmental events that cause anomalous values.
- Laboratory method changes.
- Field data collection method changes.
- Personnel changes.
- Equipment malfunctions.

Samplers try to avoid or mitigate these circumstances through good planning, preparation and by using standardized protocols and methods and good communication. When any of these things do affect data, every effort is made to determine if data can be used or re-generated. Even so, the data may be still be flagged and commented as "estimates" to alert users to potential analytical effects. If data can't be used it is flagged as "fail" and eliminated from analysis and distribution.

6.21.1.2.4 **Corrective action processes.** QC results may indicate data problems. Staff and external lab analysts will follow prescribed procedures to resolve the problems. Options for corrective action may include:

- Retrieving missing information.
- Re-calibrating analytical instruments or sensors.
- Re-analyzing samples (must be done within holding time requirements).
- Modifying the analytical procedures.
- Collecting additional samples or taking additional field measurements.
- Qualifying results using QC codes.

6.21.1.2.5 **QC Codes.** Following quality assessment, all data is given a quality description (QC code) and released for public use or removed from the dataset. A quality flag is given to each data point to communicate any specific reason for the QC code. Also, quality assessment allows the marine waters group to describe and quantify the accuracy and expected error associated with all marine data generated. At various stages of assessment, a code specifying the QA level is used to denote the status of data in the QC and review process. Once all QC procedures have been applied and quality objectives passed, data are finalized. Prior to finalization, all data in the process of review are considered provisional and may be subject to change. Status of the data is clearly communicated to data users. Descriptions of all QC codes, flags and level of assessment can be found in Tables 1 to 3.

6.21.1.2.6 **Secondary Data.** Secondary data from external sources are used for several purposes. We use publicly available data collected by programs or agencies that follow documented procedures. Typically, the external party provides data in a provisional state and subsequently finalizes them. We rely on the external party to generate and publish data and related QA/QC information. We also review the data to assure they make spatial and temporal sense. For our final reports and products, all secondary data will be thoroughly reviewed and only data collected under formal QA/QC procedures will be published. Any developmental products, such as the hypoxic intrusion index, will be identified as such.

6.21.1.2.7 **QA/QC of Analytical and Descriptive Products.** As part of our final assessment and reporting on marine water quality conditions, all analytical and descriptive products are reviewed by internal colleagues to catch errors or potential mistakes.

- Analytical (quantitative) products are calculated or computed results, intended to provide exact determinations or assessments based on data. These products are given a comprehensive review, with secondary checks of calculations and computations, validation of source data, equations and methods used to determine analytical results.
- All data inputs and calculated output for analytical products are preserved and reviewed to ensure consistency with previously reported or published products, assure no loss or inclusion of erroneous data and validate calculation and reporting methods.
- Descriptive products which are intended to provide graphical or illustrative information undergo a “basic” check for overall correctness, completeness and reasonableness within context of expected or related information.
- Current marine water column monitoring products are defined as analytical or descriptive and are listed in Table 14.

Table 14. Marine Waters Monitoring analytical and descriptive products and type of quality assurance and quality control required.

Analytical Product	Type of Product	Level of QC Required
Marine Water Condition Index (MWCI) through annual plots and heat maps	Quantitative	Comprehensive Review
Annual anomalies in the dissolved oxygen deficit, light transmission, salinity (0 to 50 m heat maps)	Quantitative	Comprehensive Review
Monthly condition summaries through heat maps and text	Qualitative (Descriptive)	Comprehensive Review
Monthly or annual weather and river summaries based on 5 long term stations	Qualitative (Descriptive)	Basic Review
Monthly anomalies in Pacific Decadal Oscillation Index (PDO)	Qualitative (Descriptive)	Basic Review
Monthly anomalies in the Pacific Fisheries Environmental Laboratory Upwelling Index (PFEL)	Qualitative (Descriptive)	Basic Review
Monthly anomalies in North Pacific Gyre Oscillation Index (NPGO)	Qualitative (Descriptive)	Basic Review
Monthly anomalies in Hypoxic Intrusion Index (HI)	Quantitative	Comprehensive Review
Annual Long Term Water Column Monitoring Condition Summary	Qualitative (Descriptive)	Comprehensive Review
Trends and Correlation in Long Term Water Column Monitoring Annual Data Results	Quantitative	Comprehensive Review
Annual Watermass Summaries through plots and text	Quantitative	Comprehensive Review
Annual Long Term Water Column Monitoring QA/QC Summary	Quantitative	Comprehensive Review

6.21.1.2.8 **Periodic data usability (method) assessment.** Upon completion of the QA/QC, data review and the data verification process, data quality (Usability) assessment (Lombard and Kirchmer, 2004) is conducted by senior oceanographers in the Marine Waters Monitoring Program.

- Data from laboratory QC procedures, as well as results from field replicates, laboratory duplicates, check samples and sensor performance tests provide information to determine if MQOs have been met. The usability assessment includes review of laboratory and sensor precision, accuracy and the success of meeting control limits. Sample results from laboratory analyses and sensor deployments are examined for completeness (all samples, all analyses). Processing logs and laboratory reports are scrutinized for adherence to specified methods and QA/QC requirements.
- A review of sample results is performed following each sampling year to determine need for modifications to the sampling or analysis program. Laboratory and quality assurance experts who are familiar with assessment of data quality are consulted if guidance is needed for assessment. Annual summaries include data quality and whether project objectives are being met. If limitations in the data are identified, they are noted.
- If MQOs are met, the quality of the data is considered usable for meeting project objectives. If MQOs have not been met, MWM staff members examine the data to determine whether they are still usable and whether the quantity is sufficient to meet project objectives.

6.21.1.2.9 **Conducting audits.** Audits are conducted every month, on incoming data once it's been processed and uploaded to the EAPMW database. Annual audits are conducted for every sampling year, once data has been completely reviewed and quality control and assessment activities are completed. These audits occur 4-6 months after the sampling year is completed.

- MWM technicians track and reconcile the status of samples being analyzed by the laboratories, focusing on QC problems as they arise. The monitoring coordinator periodically performs QA/QC of files including raw data field sheets, calibration records, laboratory QA/QC, and other program related materials. Summaries (statistical evaluations and plots) of all QC information collected during a sampling year are generated and reviewed routinely by the MWM group.
- All laboratories participate in routine performance and system audits of various analytical procedures. Audit results are available upon request. The Laboratory Accreditation Unit of Ecology's EAP accredits all contract laboratories that conduct environmental analyses for the agency, and the accreditation process includes performance testing and periodic lab assessments. No additional audits are envisioned.

- To assure accurate entry of data into the database, the monitoring coordinator or data manager checks 10% of all values against the source data. If errors are found, an additional 10% of values are checked and the process will continue in this way until no errors are found or all values have been verified or corrected.
- The senior oceanographer, monitoring coordinator or data manager checks 10% of the annual, finalized data in Ecology databases and available via the internet against the source data. If errors are found, an additional 10% of values are checked and the process will continue in this way until no errors are found or all values have been verified, corrected, or flagged.
- The results of QA/QC and audits including performance assessment of all measurement systems, significant QA problems, and recommended solutions are available upon data finalization following the completion of a sampling year.

6.21.1.2.10 **Performance measure evaluation.** Once a year, in the month (July) following the end of the state fiscal year, we report the attainment of our monitoring performance measure to the Washington State's Office of Financial Management. Our performance measure is an accounting of the percentage of data collected that met MQOs. Table 15 shows performance measure attainment for recent years.

Table 15. Example performance measures for Marine Waters Monitoring data of vertical water profiles. This example shows the performance measures for the last five years of the monitoring program. Event, site, and weather observations are not included in the performance measures.

Data Type	Year 2017	Year 2018	Year 2019	Year 2020	Year 2021
Total number of discrete results collected	8,449	14,386	10,119	3,175	9,119
Total number of discrete results that meet DQOs	8,437	14,249	9,864	3,142	9,099
Percent of discrete results that meet DQOs	99.9%	99.0%	97.5%	99.0%	99.8%
Total number of continuous vertical profile results collected	686,936	716,356	706,834	269,630	735,499
Total number of continuous vertical profile results that meet DQOs	643,592	684,201	690,895	266,832	717,701
Percent of continuous vertical profile results that meet DQOs	93.7%	95.5%	97.7%	99.0%	97.6%

Data Type	Year 2017	Year 2018	Year 2019	Year 2020	Year 2021
Total number of sampling sites	39	39	39	39	39

7.0 Records Management

7.1 All data reviews, QC analyses, and related activities are performed using standardized data templates, software routines and documentation. These tools are stored on a secure network drive, in appropriately organized and designated folders along with the original field, lab and instrument files and data. All decisions and QC activities are documented using independent records, so that any unusual results or procedures can be verified after the review or process is completed.

8.0 Quality Control and Quality Assurance

8.1 This section is redundant to the overall procedure documented in Section 6.

9.0 Safety

9.1 There are no specific safety requirements for this work beyond the stated considerations in the agency guidance.

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