Standard Operating Procedure

Water Quality Sampling and Laboratory Processing

Synopsis: A standardized method for collecting water quality data according to Great Lakes Coastal Wetland Monitoring Program protocols for monitoring Great Lakes coastal wetlands

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Contents:

1.0 PURPOSE

The following sections describe protocols to be used for field water quality measurements, water sampling, and water sample processing. Standard Operating Procedures (SOPs) are based on the protocols developed previously by NRRI-UMD for the National Park Service's Great Lakes Monitoring Network (Elias et al. 2008) and the Great Lakes Coastal Wetlands Consortium. Relevant sections were extracted from these documents for use by the Great Lakes Coastal Wetland Monitoring Program.

2.0 RESPONSIBILITIES

Scientists and field teams sampling wetlands as part of a monitoring program following Great Lakes Coastal Wetland Monitoring Program protocols should ensure that all work performed satisfies the specific tasks and requirements outlined in this SOP and the project's quality assurance project plan (QAPP).

3.0 SAMPLE COLLECTION LOCATIONS

Chemical/physical measurements will be made in each vegetation zone where fish and macroinvertebrate data are collected. Fish and macroinvertebrates are collected by vegetation zone; water quality should be collected in association with fyke nets, one water quality sample per vegetation zone. In the event that fyke nets are not set due to very shallow depth, but invertebrates are collected, then a water quality sample should also be collected from that zone. These samples are required.

Crews have the option of taking water quality meter samples at each individual fyke net location (or invertebrate dip net replicate point if fyke nets are not set in that zone). This additional sampling effort is recommended if vegetation patches forming the zone are separated rather than contiguous. Water quality data collection is critical at each wetland, but parameters will be classified as critical, recommended, or supplementary on an individual parameter-by-parameter basis in this section. Defining parameters as "critical" does not mean that biological samples should not be taken at a site if water quality parameters cannot be taken because, for example, the DO sensor on the meter is malfunctioning. Every attempt should be made to obtain critical measurements, including borrowing equipment from a nearby field crew and obtaining a replacement meter as soon as possible. We also define water quality parameters in terms of 1) field measurements using instruments with sensors used at the site, 2) parameters requiring analysis of a water sample either the evening or the day after the sample was collected, or 3) parameters measured at one of our project water quality laboratories.

Critical:

- Field: temperature, dissolved oxygen, pH, specific conductivity
- Lab: alkalinity, turbidity, soluble reactive phosphorus (SRP), [nitrate+nitrite]-nitrogen, ammonium-nitrogen, chlorophyll-*a*

Recommended:

- Field: transparency (or secchi) tube clarity
- Lab: total nitrogen (TN), total phosphorus (TP), chloride, color

Supplementary:

- Field: oxidation-reduction potential (redox), in situ chlorophyll fluorescence
- Lab: Sediment percent organic matter

In general, two or three vegetation zones will be sampled by fish and macroinvertebrate crews in each wetland, although one or four zones are possible. The basic water quality sampling design is based on the placement of three fyke nets within each vegetation zone at a site. If fyke nets cannot be set in a zone, then macroinvertebrate sampling points will be used instead as water quality sampling locations. Water quality data will be collected from fyke net or macroinvertebrate locations as follows:

- Field: critical, recommended, and supplementary measurements will be made at the first net set location within each vegetation zone. It is recommended that water quality measurements be made at each net set within the zone, but only one location is required.

- Lab: water will be collected from each of the fyke net locations within a vegetation zone and combined to form a single composite sample, which will be analyzed for critical and recommended water quality parameters.

4.0 SAMPLING

Field measurements should represent, as closely as possible, the natural condition of the surface water at the time of sampling. Experience with and knowledge of the sampling equipment and the collection, storage, and processing of water samples for subsequent laboratory analyses are critical for collecting data of high quality. To ensure consistent, high-quality data, always:

- Make field measurements only with calibrated instruments that have been error-checked.
- Maintain a permanent log book for each field instrument for recording calibrations and repairs. Review the log book before leaving for the field. This book should also be used for recording results of pre-and post-calibration checks.
- Test each instrument (meters and sensors) before leaving for the field. Become familiar with new instruments and new measurement techniques before collecting data.
- Have backup instruments readily available and in good working condition, whenever possible.
- Follow quality assurance/quality control procedures. Such protocols are mandatory for every data collection effort, and include practicing good field procedures and implementing quality control checks. Make field measurements in a manner to minimize artifacts that can bias the result.
- Refer to the checklist of supplies and equipment needed for field sampling (Table 1) prior to each sampling trip. Keep on hand all necessary forms, calibration logbooks, field

logbooks, field data sheets, procedural manuals, and equipment instructional manuals.

Table 1. Field supplies and equipment checklist

√	**Equipment and Supplies**
	Field notebook, pencils and pen (waterproof ink)
	New field data sheets on waterproof paper
	Up-to-date field folders containing recent maps and field notes
	Multiparameter instrument (calibrated), calibration standards, data logger
	Calibration logbook for each instrument
	All maintenance parts and calibration standards for field instruments
	Backup instruments in case of electronic failure of multiprobe (for example: YSI 85 [T- DO-EC25] or equivalent, YSI 200 [T-DO], Hannah Dist3 [EC25], armored NIST certified thermometer [°C], portable pH meter)
	Secchi tube
	Composting jug and other bottles
	Sonde with barometric pressure sensor, weather radio barometer, or other way to obtain barometric pressure
	Extra batteries for all field equipment (multiparameter probe, calculator, GPS, etc.)
	Rain gear
	First aid kit
	Personal flotation device(s)
	Field trip itinerary
	Cell phone
	Digital camera with sufficient memory and battery
	Global positioning system (GPS) with extra batteries
	Deionized or distilled water for field rinsing
	Relevant sections of this manual copied onto waterproof paper

4.1 Sequence of Activities during Field Workday

1. Review field gear checklist.
2. Prepare datasheets for all anticipated sites and zones, printed on waterproof paper.
3. Prepare sample bottles and labels in advance and place in a cooler.
4. Conduct daily calibration of appropriate meters and probes.
5. Inspect vehicles at the beginning of every field day, including all safety and directional lights, oil, gasoline, tire air pressure levels.
6. Inspect boat; ensure all safety gear is on board.
7. Drive to boat landing or portage boat into lake to be sampled. Load boat with sampling gear, launch boat, and navigate to monitoring site. Set up a clean work space on the boat for sampling.
8. Refer to description of wetland, polygon maps, and comments from field crews who have visited previously to orient to site and vegetation zones (see macroinvertebrate and fish SOPs).

9. Measure field water quality variables per Sections 4.5-4.9 and collect samples per 5.0.
10. Be sure that all samples are correctly labeled and preserved on ice.
11. Verify that data sheets are completely filled out, and sign form.
12. If sampling from more than one site in a day, follow procedures for decontamination of equipment between sites.
13. Upon return to shore, inspect boat, trailer, and all equipment that has come into contact with the water for invasive species.
14. Return to office or field station after all sampling is complete.
15. Process samples according to appropriate protocol. Refrigerate or freeze samples, as required and package samples for sending to analytical laboratory.
16. Rinse sensors with deionized water and perform calibration re-checks.
17. As soon as possible after returning from the field, review both hardcopy and sonde data; offload sonde data; review laboratory data as it is received.

4.2 Recording Field Information Upon Arrival at Monitoring Site

Consistent methods are important to long-term data quality. In actuality, the ideal conditions are not always met in the field or in the lab and changes in staff occur. Therefore, documentation of procedures, site conditions, laboratory analysis, and reasons for deviations of any kind is important. Personnel are encouraged to write down more than they feel may be necessary in the moment, as the future interpretation of their data will depend on the written record and not the memory of an individual. Waterproof data sheets should be prepared ahead of time, labeled with the project and wetland ID. Field data sheets are used to record the physical and chemical water quality variables measured at the time of sample collection. In addition to recording the field variables, any samples collected for laboratory analyses must be indicated. Documentation should include calibration date for each instrument, field conditions at the time of sample collection, visual observations, and other information that might prove useful in interpreting these data in the future. Datasheets include the required fields for staff to fill in. These sheets should always be used when collecting project data.

4.3 Decontamination Procedures

Field technicians should be aware of and record potential sources of contamination at each field site. Decontaminate field sampling equipment for minimizing the risk of spreading invasive species. Clean field and laboratory equipment to avoid contamination of analytes to be measured. Do not allow sample water to touch hands; do not touch insides of sampling equipment, containers, or laboratory bottles. The GLCWMP Fish SOP, Section 9 includes procedures for decontaminating field equipment to reduce the spread of invasive species.

4.4 Bottle Preparation – Types and Sizes of Bottles

For each monitoring station, select the bottles appropriate for each analyte and label them with, at a minimum, wetland ID, vegetation zone type, sample date, and analyte code according to the requirements of the analytical laboratory, and sampling crew code. Store pre-labeled bottles in a dry box or in separate bags for each station.

4.5 Field Measurement Procedures

For all sampling, it is critical to avoid sampling water showing evidence of oil, gasoline or anything else from the boat motor. Turn off the boat motor and do not set an anchor if the boat position can be held without it. If there is any evidence of sediment disruption, reposition the boat to get into clear, undisturbed conditions. Avoid surfactants, floating debris, and turbid aeration during sample collection. Discard rinse water or excess sampling water on the opposite side of the boat from where samples are being collected. If sampling by wading, extra care must be taken to ensure that sample water does not contain sediment or debris disturbed by the person's movement in the wetland.

4.6 Stabilization of Sensor Probe Readings

Before making field measurements, properly-calibrated sensors must be allowed to equilibrate to the condition of the water being monitored. Sensors have equilibrated adequately when instrument readings have stabilized, that is, when the variability among measurements does not exceed an established criterion.

Table 2. Recommended instrument stabilization criteria for recording field measurements.

Standard Direct Field Measurement	Stabilization Criteria (O'Ney 2005)	Stabilization Criteria *In situ* Multisensors (WOW 2005)
Temperature:		
Thermistor thermometer	± 0.2 °C	± 0.2 °C
Liquid-in-glass thermometer	± 0.5 °C	(5%)
Specific conductivity (EC25)		
When ≤ 100 µS/cm	± 5 %	< 5 uS/cm
When > 100 µS/cm	± 3 %	(10%)
pH: Meter displays to 0.01	± 0.1 unit	± 0.2 uni (10%)
Dissolved oxygen:		
Amperometric (same as polarigraphic) method	± 0.3 mg/L ± 2%	± 0.5 mg/L (10%)

4.7 Guidelines for Taking Measurements

Acquiring high quality results requires the use of consistent measurement methods. Adhere to the following guidelines:

1. Submerse sonde to mid-water column depth.
2. Wait for the DO value to stabilize first, record the value, then read the other parameters. Because DO takes the longest to stabilize this assures all parameters have equilibrated. Stabilization of the DO value will typically take anywhere from 30 seconds to several minutes, depending on the gradient from the previous sample and the age and type of oxygen probe. Extra time should be allowed for equilibration when values are below approximately 3 mg/L. If the sonde does not have a stirring mechanism, gently move the cable vertically a few centimeters once per second.

3. Enter all data on field forms.

Instrument problems or failure

- If water quality sensor measurements are not representative of field conditions based on previous data or limnological knowledge, re-calibrate and try again. If readings seem reasonable, proceed. If not, first check the troubleshooting guide in the instrument manual. If problem persists, collect as much data as possible using back-up handheld instruments, if available.
- In the case of measuring DO, move the probe vertically or horizontally without causing bubbles to form. If DO is very low, move the probe for ca. 2 min for values to stabilize.

4.8 Detailed Description and Troubleshooting Hints for Field Variables

Because temperature, DO, and other water quality variables are important determinants of biotic habitats, it is important that observers write down values on field forms and think about their ecological meaning, even if a data logger is recording the measurements. The hard copy also serves as backup in case there is an electronic failure.

4.9 Temperature

Temperature (T) is measured in units of degrees Celsius (°C) and recorded to the nearest tenth of a degree as warranted by instrument. The upper few centimeters of soft sediments are often several tenths of a degree warmer than the overlying water. The rise in temperature can be an indication that the probe is submersed into the sediments. If this happens, be sure to vigorously rinse the instrument in shallow water to clean it before re-taking measurements.

4.10 Specific Conductivity

Specific conductivity (SpC) is the ability of water to conduct an electrical current for a unit length and unit cross-section at a certain temperature, measured in units of microsiemens per centimeter (µS/cm), and recorded to the nearest µS/cm. Commonly used in water quality monitoring, SpC is a general measure of the concentration of ions dissolved in the water. It is important to be aware of the difference between EC (conductivity at the ambient temperature of the sensor) and SpC (an abbreviation for conductivity that is temperature compensated to 25°C). The difference between EC and SpC can confound analyses of seasonal patterns of dissolved ions since water temperatures vary throughout the year. SpC can be used to monitor seasonal

changes in total dissolved salts (TDS) such as a spring flush of road salt, which is why the temperature compensation is important. Many instruments will display EC in addition to SpC. In the event that an uncompensated sensor must be used, the value of SpC can be calculated from EC and temperature values.

A common physical problem in using a specific conductance probe (or meter) is entrapment of air in the conductivity probe chambers. Its presence is indicated by unstable specific conductance values fluctuating up to 100 µS/cm. This problem can be minimized by slowly and carefully placing the probe vertically into the water and when completely submerged, quickly moving it back and forth through the water to release any air bubbles. An SpC probe with an open flow design does not trap air.

Having specific conductance standards in the field can help verify values that fall outside the expected range. For example, the expected specific conductivity is around 200 and the reading is 1500. A known standard can be put in the instrument storage cup to determine if the instrument is reading correctly or is out of calibration.

4.11 Hydrogen Ion Activity (pH)

Commonly used in water quality monitoring, pH is a measure of the acidity of water, measured in standard pH units (SU), and recorded to the nearest 0.1 pH unit. The pH scale is from 1 to 14: neutral water is pH 7, acidic waters have pH <7 and alkaline waters have pH >7.

- Is the value real or is the instrument out of calibration? Having pH standards in the field can help verify values that fall outside the expected range. For example, the expected pH is around 7.0 and the reading is 9.5. A known standard can be put in the instrument storage cup to determine if the instrument is reading correctly or out of calibration.
- As with dissolved oxygen, a pH probe can take longer to equilibrate when the gradient from the previous measurement is large (>1.0 pH SU).
- Low ionic strength waters with SpC < 50 µS/cm can cause pH measurement stability problems with some probes, necessitating use of low ionic strength probes. Probes will often calibrate fine in strong ionic strength buffers, but will not read accurately in lower ionic strength surface waters. If you suspect this is the case, use a sensor that is designed for low ionic strength waters.

4.12 Dissolved Oxygen

Dissolved oxygen (DO): units of mg/L record value to nearest 0.1 mg/L unless otherwise justified; percent saturation (% DO) record value to nearest %; also temperature compensated.

- Avoid touching the bottom, as the membrane may become fouled.
- Equilibration time is critical; the steeper the DO gradient, the longer the equilibration time. It may take >5 minutes when DO drops abruptly to near zero.
- The DO probes with membranes (Clark cell) actually consume oxygen in the immediate vicinity around the membrane as they work; therefore, measurements require moving water using either a built-in stirrer (typical in multiparameter sondes and BOD probes) or moving the cable up and down (e.g., 6” each side of the desired depth) during the

measurement. Optical sensors do not consume oxygen and hence do not require moving water.

- Accuracy of an optical DO probe can be compromised if it is covered with a residue that inhibits or increases oxygen reaching the sensor surface. Biofilm on the sensor surface may increase DO measurements, while oils or sediments may lower them. If measurements seem suspect, or if the instrument was used in contaminated water, the sensing surface should be cleaned with a soft brush and a mild detergent following manufacturer's instructions.

4.13 Clarity: Secchi Tube

Procedure:

- Remove sunglasses. If you wear prescription glasses that darken to protect eyes from sun, wear a billed cap to shade the glasses. Turn your back to the sun and position the tube to avoid direct sun the full length of the tube.
- Gently pull up the inside string to remove the black and white Secchi disk from the tube.
- Fill the tube to the top with water from the sampling bottle or carboy. Let the water drain out of the string guide hole to the zero mark on the tape measure attached to the side of the tube.
- While looking down into your tube from the top, slowly lower the Secchi disk down into the tube until the disk disappears from sight. When it disappears, stop lowering.
- While continuing to look down the top of the tube, slowly pull the string to raise the disk until it reappears. Lower and raise the disk until you have found the midpoint between disappearance and reappearance of the disk.
- Pinch the string against the top rim of the tube to hold the disk at this measured depth of disappearance/reappearance. Look at the side of the tube, across the top of the disk, to see the closest centimeter mark on the tape.
- Write down this depth, to the nearest centimeter, on your stream data sheet under "Secchi tube depth." If the disk does not disappear, and you see it clearly sitting on the bottom of the tube, record "greater than 100".

Maintenance notes and other precautions:

- Clean the secchi tube periodically with mild dish soap and a soft cloth.
- Although water from the tube may be saved for turbidity and TSS measurements do not save it for nutrient or other pollutant analyses because the tubes are not cleaned according to certified protocols.
- Dissolved color due to organic matter (humic and fulvic acids, usually from bogs and conifer needles) can confound comparisons of secchi tube data between lakes. Also, wetlands with similar concentrations of suspended sediments can have different transparency because smaller particles scatter more light.

5.0 WATER SAMPLE COLLECTION

Before its first use, the sampler must be cleaned thoroughly by rinsing three times with tap or

deionized water, rinsing three times with 0.1 N HCl, and finally, rinsing three times with deionized water.

5.1 Integrated Sampling

- Rinse compositing jug (or carboy) 3 times before sampling. Increase surface water flushing to 6 rinses if the compositing jug previously contained water that was contaminated with sediment or water deemed to have much higher nutrient levels.
- Ensure the collection bottle on the telescoping sampling pole is securely attached
- Extend the telescoping rod to the length necessary to reach sampling location.
- Dip the rod into the water and rinse the bottle three times.
- Invert the bottle and fill from just below the water surface.
- Fill the carboy sufficiently to ensure adequate water for all analyses.
- Always ensure composite water is well mixed prior to dispensing it to any other containers.
- Once water is collected in compositing jug, conduct secchi tube reading. Next, dispense into clean “cubitainer” or similar container.
- Keep cubitainer cold and dark until processing in the home lab, campsite, or motel later in the day.
- Rinse bottles and caps for chlorophyll-a, nutrients, etc. with sample water prior to filling.
- Fill nutrient bottle to the bottom of the neck of the bottle – this will prevent the bottle from breaking when the water expands as it freezes.
- Store water samples in cooler with ice until return to home base for further processing.

5.2 Quality Assurance of Field Duplicates

Collect field duplicates approximately every 10 samples. The duplicate should be collected in the same spot as the primary sample. Label the duplicate analysis bottles with the code appropriate for the duplicate. Indicate on the data field form which site and zone contain the duplicate. Treat the duplicate as a regular sample for all phases of collection, processing, and analysis.

6.0 DEPARTURE FROM MONITORING SITE

Before leaving the monitoring site, all data sheets and sample labels must be reviewed for legibility, accuracy, and completeness. Any changes in procedure due to field condition must be explained in the comments section. Make sure the information is complete on all data sheets. After reviewing each sheet, the crew leader must sign on the appropriate lines to certify that sampling has been conducted in accordance with all SOPs.

7.0 QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)

QA/QC includes all aspects of a project intended to ensure measurements are accurate, reproducible (precise; consistent), and include accurate estimates of uncertainty. It specifically involves following established protocols in the field and lab to assure everyone that the sample is representative of the site, free from outside contamination by the sample collector, and that it

has been analyzed following standard QA/QC methods.

7.1 Calibration of Field Instrument Sensors

Calibration schedules overlap but differ from sampling schedules, so calibration methods are listed here as a separate procedural step. Instrument calibration is an essential part of quality assurance. Table 4 summarizes the ideal calibration frequency and minimum acceptance criteria for sensors (i.e., probes). Logistical constraints at remote sites may preclude calibration and checks of calibration at the ideal frequency. This SOP provides only general guidelines for equipment use and maintenance. A wide variety of field instruments is available; such instruments are continuously being updated or replaced using newer technology. Keep equipment manufacturers' maintenance and calibration instructions for all instruments for reference purposes. Field personnel must be familiar with the instructions provided by manufacturers. Contact manufacturers for answers to technical questions.

7.2 Instrument Calibration and Maintenance Logbooks

Calibration and maintenance logs for multi-parameter sondes and all back-up sensor probes should be maintained and will document the frequency of calibration, maintenance, and calibration checks. Keep calibration logs with each instrument during the sampling season. Logs should later be archived. Each instrument should have a logbook for recording all maintenance and calibration information, including:

- Serial number, date received, manufacturer’s contact information, especially technical service representatives
- Service records, dates of probe replacements
- Maintenance records, for example, whenever the following general maintenance occurs: DO membrane replacement, pH reference probe junction and filling solution, probe cleanings, sonde (the sensor housing) replacement, impellor replacement or cleaning, etc.
- Calibration dates and calibration data
- Any problems with sensors
- Pre-mobilization, post-calibration checks performed on individual sensor probes

Table 4. Ideal calibration frequency and acceptance criteria.

Parameter	USEPA Method	Minimum Calibration Frequency and QC checks	Acceptance Criteria	Corrective Actions
Temperature	170.1	Annually, 2-point calibration with NIST thermometer	±1.0 ºC	Re-test with a different thermometer; repeat measurement
Specific Conductance (EC25)	120.1	Daily, prior to field mobilization; calibration check prior to each round of sampling; 10% of the readings taken each day must be duplicated or a minimum of 1 reading if fewer than 10 samples are read.	±5% RPD 10%	Re-test; check low battery indicator; use a different meter; use different standards; repeat measurement
		Daily, prior to field mobilization (2 buffers should be selected that bracket the anticipated pH of the water body to be sampled:	±0.05 pH unit;	
pH	150.1	Calibration check w/ third buffer prior to each round of sampling; check with low ionic strength buffer in addition, if conductivity is <50 µS/cm	±0.1 pH unit RPD 10%	Re-test; check low battery indicator; use different standards; repeat measurement; don't move cords or cause friction/static
		10% of the readings taken each day must be duplicated or a minimum of 1 reading if fewer than 10 samples are read.		
Dissolved Oxygen	360.1	Daily, prior to field mobilization; check at the field site if elevation or barometric pressure changed since calibration	0.2 mg/L concentration or ±10% saturation	Re-enter altitude; re-test; check low battery indicator; check membrane for wrinkles, tears or air bubbles; replace membrane; use a different meter; repeat measurement
Depth	--	Daily, prior to field mobilization, check at the field site. Check annually against commercially purchased brass sash chain labeled every 0.5 m to ensure that it reads zero at the surface and varies <0.3 m for depths <10 m and no more than 2% for greater depths.	±0.1 m	Retest, check low battery indicator; repeat measurement; use with accurately calibrated line
Secchi/Transparency tube			--	Transparency tubes have a 100 cm scale; ensure tube is clean.

7.3 Handling of Calibration Standard Solutions

Store all calibration standards in a temperature-controlled environment. Standards should be dated upon receipt and upon opening. Commercially-purchased calibration standards come with an expiration date that must be observed. Ensure that calibration standards are not used beyond expiration dates.

Properly dispose of all waste materials. Used calibration solutions, in general, may be rinsed down a sink with water after consideration of the wastewater treatment system available to that sink. Material safety data sheets (MSDS) that are sent with manufacturer purchased calibration solutions should be kept on file. These documents describe the flammability, toxicity, and other safety hazards of reagents. Some reagents may include constituents toxic to aquatic life. These should not be rinsed down a sink in any large quantities in primitive areas where the ultimate destination of wastewater is the aquatic environment. Instead, these reagents should be collected in a properly-marked leak-proof container for disposal in an adequate treatment system.

7.4 Temperature Calibration

Temperature is typically not adjustable on an electronic sensor but should be cross-compared to a National Institute of Standards and Technology (NIST) traceable thermometer at the beginning of each field season, as follows:

- Compare against a NIST-certified or NIST-traceable thermometer at a broad range of temperatures, for example 0 to 40°C;
- The sensor should read within ± 1.0°C of the NIST thermometer;
- Typically you cannot adjust the instrument to calibrate it, but check the manual. It is a good idea to check the instrument at 0°C in slurry of ice-water if a calibrated (NIST) thermometer is not available since electronic and non-electronic temperature sensors are typically linear over the likely range of field temperatures.

7.5 Specific Conductivity Calibration

Specific conductivity (SpC) should be calibrated using a KCl solution as specified by the instrument manual. Stock calibration solutions can be purchased commercially, prepared by a water quality contract lab, or made in an academic or agency lab. Set the instrument to record temperature-compensated SpC. Because the typical modern SpC sensor is linear to <3% over the range from approximately 20 to 10,000 µS/cm, a single point calibration is typically sufficient, though a 2-point calibration is preferred. A typical standard is 500 or 1000 µS/cm. Pre-mobilization error checks of this sensor using 100, 500 or 1000 uS/cm standards may be used to establish sensor error over the range of most freshwater work.

7.6 pH Calibration

The pH is calibrated using the standard two or three buffer technique, using either pH 4, pH 7, or pH 10, depending on the expected field values. Calibrate the probe according to the

manufacturer's recommendations. If a water body is classified as low acid neutralizing capacity (ANC; ANC approximately 100 µeq/L or lower), a low ionic strength pH combination electrode may be necessary to acquire adequate sensitivity if stabilized pH measurements are not achieved with standard pH sensors. Prior to each round of sampling, check the calibration with a third standard having a pH value similar to what is expected in the field.

7.7 Dissolved Oxygen Calibration

Two types of dissolved oxygen (DO) sensors are typically utilized: Clark cell and optical. A Clark cell (membrane) sensor is air-calibrated, while an optical DO sensor is calibrated in 100% saturated tap water. For a Clark cell, it is assumed that the dry sensor will read 100% saturation in an enclosed airspace with enough water in the bottom of the container to saturate the air with water vapor. For optical DO sensors, the sensor window must be submerged in 100% DO saturated water, which is obtained by agitating tap water in a container for one minute and then decanting it into the calibration chamber until the DO sensor is completely covered with water. A Clark cell should be checked for bubbles under the surface of the membrane. If bubbles are observed, change the electrolyte solution and replace the membrane. An optical DO sensor's surface should be observed to make certain that no air bubbles are clinging to it. The calibration chamber can be tapped gently to dislodge any bubbles.

Temperature affects DO saturation, as does the air pressure, which varies with elevation and ambient weather. Both sensors require a known barometric pressure (BP), obtained by the multiprobe itself (if equipped with a BP sensor) or from another source. Acceptable sources for BP are from another instrument, such as a calibrated barometer, or a third-party source, such as the local weather bureau or airport.

7.8 Post-Field Calibration Checks

Post-field calibration checks should be performed after each use of the instrument and before any instrument maintenance. The sooner this procedure is performed, the more representative the results will be for assessing performance during the preceding field measurements. Calibration and post-calibration should be no more than 24 hours apart if possible. When sampling daily, the second day's calibration can serve as the first day's post-field calibration check. Take the same care used in performing the initial calibration by rinsing the sensors and waiting for sensors to stabilize.
Record post-field calibration check values in the calibration logbook (generally on the same page with the initial calibration for that sampling trip).

The purpose of the post-calibration is to determine if the instrument has held calibration during the day of sampling. Compare the post-calibration values to the expected values for the standards. This will ensure that the field measurements for the day can be reported with confidence. The difference between the post-calibration value and expected standard value can be used to indicate both calibration precision and instrument performance. If post-calibration values (Table 5) fall outside the error limits for DO, pH, and specific conductance, data collected do not meet quality assurance (QA) standards and should be flagged appropriately.

Measurements may be repeated with a different or back-up instrument. If post-calibration measurements do not consistently fall within the error limits after in-house trouble shooting, the instrument should be returned to the manufacturer for maintenance

Table 5. Post-calibration check error limits.

Parameter	Value
Temperature	± 1 °C, annual calibration check
Specific Conductance	± 5%
pH	± 0.1 standard units
Dissolved Oxygen	± 0.2 mg/L, ± 10% saturation

7.9 Sensor Maintenance and Storage

Most multi-parameter sondes should be stored with a small amount of water in the storage cup. Refer to the manufacturer's manual for tips on cleaning the probes and housing, routine maintenance procedures, and proper storage procedures.

7.10 Sampling Duplicates

The purpose of a duplicate sample is to estimate the inherent variability of a procedure, technique, or characteristic. Duplicate samples are collected and duplicate analyses may be made in the field: 1) as a form of field quality control; 2) to measure or quantify the homogeneity of the sample, the stability and representativeness of a sample site, the sample collection method(s) and/or the technician's technique. Duplicates are analyzed in the laboratory for the same parameters as the monitoring sample to which they apply. Duplicate samples also document the technique and ability of the technician and analyst to produce representative data.

Duplicate field samples should be collected every sampling trip for each type of sample collected and the results must have a Relative Percent Difference (RPD) less than or equal to the guidelines stated in the project QAPP. Required field parameter measurements can be duplicated to estimate the precision of the equipment. Every tenth measurement should be duplicated, and the results of both measurements recorded and evaluated as RPD. The result can be compared with the stated precision of the instrument.

7.11 Summary of Quality Assurance/Quality Control Field Procedures

Quality assurance protocols are means to ensure data collected are as representative of the natural environment as possible. Quality assurance procedures are required in all data collection efforts as part of this monitoring protocol. Elements of quality assurance/control include (in summary):

- Field staff must be trained by personnel experienced in the protocol.
- Use calibrated instruments for all field measurements. Test and/or calibrate the instruments before leaving for the field. Each field instrument must have a permanent log book for recording calibrations and repairs. Review the log book before leaving for the field. All manually recorded field measurement data will be collected on field forms; data

that are automatically recorded will be captured electronically and the equipment used will be documented on field forms. Hard and electronic copies will be made as soon as possible after surveys and kept at a separate location as backup.

- Complete records will be maintained for each sampling station and all supporting metadata will be recorded appropriately (field forms or electronically).
- Make field measurements in a manner that minimizes bias of results.
- Check field-measurement precision and accuracy.
- Collect 10% duplicate water samples; conduct duplicate measurements of field parameters at approximately 10%.

8.0 PROCESSING WATER SAMPLES AND ANALYTICAL LABORATORY REQUIREMENTS

This standard operating procedure (SOP) is designed to provide detailed instructions on the handling and processing of water samples prior to analysis by an analytical laboratory.

Water chemistry will be performed by one or more analytical laboratories that have demonstrated the ability to measure analytes at detection levels adequate to meet the needs of the GLCWMP. Preferably, the laboratories will be state- or federally-certified for performing the above water chemistry analyses in natural waters, or an academic research laboratory that can demonstrate quality assurance and quality control (QA/QC) procedures consistent with current EPA procedures that are used as the basis for state certification of commercial environmental laboratories.

8.1 Sample Handling and Processing Procedures

The following general techniques will be observed throughout the procedures detailed below.

- Keep all water samples cool and dark until processing is complete and samples are shipped to the analytical laboratory.

- Use only new, clean sample bottles supplied by the analytical laboratory or purchased pre cleaned from a supplier.

- Rinse filtration equipment with deionized water (DIW) three times between samples.

- Avoid touching the inside of sample bottles and filtering apparatus, tips of forceps, and filters to prevent contamination of the samples.

- When filtering samples in the field, use an enclosed filtering apparatus to minimize contamination from airborne sources.

- Wear disposable, powderless gloves when working with acids and other preservatives.

- Rinse the apparatus three times with 0.1N HCl, followed by three times with DI water before processing the next sample. Alternatively, if the nutrient concentrations are not

expected to be dramatically higher between one sample and the next (e.g., different zones within the same site or similar sites within a region), the filtering apparatus may be rinsed sufficiently with DI water. However, acid-rinsing should be conducted in most cases.

- Prepare QA/QC samples in the same manner as regular samples, using water from the same sample collection container.

- Ensure all sample bottles are labeled correctly, completely, and legibly.

- Check laboratory equipment and supplies list (Table 6) and ensure equipment is clean and ready for use and supplies are adequate prior to beginning sample processing.

The following sections detail the procedures to be followed when processing water samples for particular analyses.

8.2 Total Chlorophyll-*a*

1. Fit rinsed filtering device with a Whatman GF/C glass fiber filter using forceps, smooth side down (curl is up). Do not touch the filter with your fingers.
2. Agitate water sample (always shake well to minimize subsampling error for solids).
3. Set pump vacuum to < 0.5 atmospheres (7.5 PSI or 380 mm Hg). If using a hand pump, maintain pressure at or below 10 PSI.
4. Use a glass or plastic graduated cylinder to measure 100 - 1000 ml of water sample. Filter sample. If water is very turbid, filter small aliquots (100 ml) to avoid clogging the filter. Sufficient volume has been filtered when a green, brown, or tan color is clearly visible on the filter and the flow decreases to a few drops/second.
5. Rinse graduated cylinder and filtering apparatus with DIW and pass through filter to include any algae that may have adhered to the sides of the cylinder.
6. Record volume filtered on data sheet (excluding DIW rinse).
7. Use forceps to fold filter into quarters with sample on the inside; do not touch filter with fingers.
8. Wrap filter in foil; label foil with site number, zone, date sample was collected, and volume filtered. Place foil in small, sealable baggie with standard laboratory label.
9. Refrigerate immediately and freeze as soon as possible. Place small baggies with foils together in a large, sealable freezer bag. A third watertight container may be used for shipping to ensure that melt water in transport will not corrupt the samples.

8.3 Unfiltered (Raw) Samples

1. Rinse sample bottle provided by analytical laboratory 1x with sample water.
2. Fill sample bottle with sample water (allowing for expansion if sample will be frozen).
3. Refrigerate or freeze, as per laboratory instructions, until packaging for transport to analytical laboratory.

8.4 Filtered Samples

1. Using clean forceps, place a 0.45µm Millipore cellulose membrane filter (or equivalent) in

the filtration apparatus. Rinse with 10 ml DIW into a cleaned (0.1N HCl and DIW rinsed as per sample bottle cleaning protocol) filtering flask (glass or plastic). Rinse flask with filtrate and discard rinse water.
2. Filter a small amount (~50 ml) of sample water; rinse filtering flask with filtrate and discard filtrate.
3. Filter enough of the sample to produce the required amount of filtrate to be tested.
4. Dispense the filtrate into separate bottles provided by the analytical laboratory.

8.5 Sample Delivery to Laboratory

1. If samples are to be shipped call FedEx or other courier service ahead of time to arrange pick-up.
2. Make large quantities of ice cubes and ice blocks (or buy ice) ahead of time.
3. Line cooler with large plastic garbage bag.
4. Place all total chlorophyll-*a* baggies containing aluminum foil wrappers in one large sealable plastic bag. Place this baggie between 2 ice packs or bags of ice. It is critical that melt water does not soak the filters, so you may want to place large sealed bag of foil wrappers in a sealed plastic jar before surrounding with ice.
5. Use ice cubes, doubly bagged in plastic bags, to pack around samples; use other ice blocks (water bottles, soda bottles, etc.) as they will fit.
6. Include a temperature check bottle with the sample bottles in each cooler, if used by the contract analytical laboratory.
7. Complete a chain of custody (COC) form, keeping the 'client copy' for the project files. Seal the laboratory's copies in a one-gallon plastic sealable bag and tape to the inside cover of the cooler. Prepare separate COC forms for each cooler.
8. If the refrigerated samples are sent in the same insulated cooler with the frozen samples, protect them from freezing by wrapping them in newspaper, bubble wrap, etc.
9. Ship samples overnight so they are received the following day during a work-week, whenever possible. Contact the laboratory about Saturday shipment receipt availability before shipping samples on a Friday. Many laboratories do not have sample receipt staff on Saturday or charge extra for staff time.
10. Alert the contract laboratory when samples have been shipped via phone or e-mail. Be sure to get acknowledgement from the lab that they know the samples are en route. Ask the lab to confirm when the samples are received.

Acknowledgements

Combinations of existing protocols were used to develop this standard operating procedure. We consulted protocols written for the EPA (Baker et al. 1997), USGS (Hoffman et al. 2005, USGS 2005), and NPS (Elias, et al., 2008, Magdalene et al. 2007, O'Ney 2005).

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CWMP: Coastal Monitoring 2017

water quality field "cheatsheet"

Water Quality Component Update: 5/15/2017

Contents:

1. Draft water processing SOPs.
2. Draft notes for maintaining clean labware in the field during sample processing.
3. Water sampling bottles to be prepared for field crews by labs.
4. Draft supplies and field instruments lists.

Label protocol:	
Site ID	Replicate #
Plant zone	Vol (vol filtered)
Sample type	Crew code
Date	Crew chief name

Collecting water for lab/hotel processing

- Goal: collect 6 L surface water in a clean carboy (10 L) from EACH fish/bug vegetation zone (carboy is acid-washed polypropylene)
  - Grab samples are collected with a 1 L acid-washed polyethylene bottle attached to a 1 m pole from the surface water.
  - Grab samples are made from the boat or just upon entry to the zone, before ANY disturbance to the zone by sample crew members.
  - First, collect 1 L water to rinse collection bottle and carboy thoroughly. REPEAT 3 times.
  - Then collect 2 each 1 L grab samples from each fyke net (or bug D-net) location in the zone.
  - Water is poured into the carboy through an acid-washed polyethylene funnel to which a 500 um screen is attached to remove any detritus.
  - No sediment should be allowed into sampling containers. If sediment is disturbed, all containers with sediment should be emptied and thoroughly rinsed 3 times before starting over.
- Mix water in carboy thoroughly and dispense into an acid-washed 4L polyethylene cubitainer. Store in cooler on ice.
- Use remaining 2 L to determine turbidity tube water clarity (remix water before adding to T-tube.

Water quality sampling "cubitainer" protocol

Collect 1 cubitainer (4L) composite per vegetation zone (1 to 4 samples per wetland site)

Within 24 hrs- hotel/tent procedures: MIX VIGOROUSLY!

1. chlorophyll-*a*: GF/C filter (42.5 or 47mm) 300 - 1000 mL into a clean* filter flask; fold/freeze
2. dissolved nutrients: refilter ~110 mL of the GF/C filtrate through a 0.45um Millipore membrane into the 125 mL poly bottle using an acid-clean * * 60 mL polypropylene syringe filtrator; chill/freeze
3. TN+TP(raw) : fill to the neck, 2 x 250 mL poly bottles; chill/freeze (one will be archived)
4. Turbidity (raw): fill 1 x 60 mL brown poly bottle; chill (DO NOT FREEZE)
5. Color and chloride: fill 1x 60 mL brown poly bottle w/ GF/C filtrate from #1; chill (DO NOT FREEZE)
6. Alkalinity/pH: use 25-50 mL of GF/C filtrate leftover from #1; titrate that evening, but can delay w/chilled filtrate for 72 hrs if needed)

Chlorophyll-*a*: fold filter to ¼ size, foil wrap, freeze 

Process *cubitainer* water

Titrate alkalinity

125 ml poly
filtered (0.45 um)
frozen

[NO2 + NO3]-N
NH4-N
SRP

250 ml poly (n=2)
raw
frozen

TN + TP
Archived

60 ml brown poly
raw chilled

turbidity

60 ml brown poly
filtered (GF/C)
chilled

Color
chloride

Notes

- Fill bottles only as far as the neck of the bottle, especially when freezing.
- Chlorophyll filters should be folded into quarters using forceps, wrapped in foil, labeled with lab tape (don't forget volume filtered), and frozen. Store in zip-lock bag. Be aware that acids will degrade chlorophyll so don't handle with fingers and be sure to extra rinse.

FILTER CLEANING PROCEDURES (assume pre-cleaned w/acid and rinsed with DIW prior to start):

- Large filter receiving flasks: rinse with DIW 3x, and shake out well before filtering water for chlorophyll-*a* through GF/C glass fiber filters. Pre-rinse the GF/Cs with ~20 mL of DIW and then discard this filtrate before filtering a measured amount of well-shaken sample water.
- Syringe filter apparatus + 0.45 um millipore filter for dissolved nutrients:
  - Only "push" water through filter – if you suck water, the filter may tear and you won't know it;
  - First push a few mLs of DIW through the filter.
  - Remove syringe again to draw in GF/C filtrate, then pressure push it directly through filter apparatus and into 125 mL pre-cleaned bottle.
  - Change filters and clean the apparatus between samples from different vegetation zones.
- Labs should prepare sets of pre-cleaned bottles in zip-lock bag packets: 2 x 250Ml, 1x 125mL, and 2 x 60mL brown plastic bottles.
- If necessary, clean new ones by rinsing with 0.1N HCl (~1% of full strength) followed by at least 3 DIW rinses.

Lab bottle preps for field crews

- Each wetland site: 1-4 vegetation zones, each generating a single carboy of water in the field, that will be used to:
    - perform a field measurement (1 rep per wetland) of transparency tube clarity (or secchi tube)
    - fill a 4 L cubitainer that will be split back at the field “lab” after hours

- Field crew needs: water sample containers, prepped and bagged; 1-4 per site; plus they will have spares. Ice coolers must be large enough to hold at least 4 full 4-liter cubitainers + at least 4 *gel packs* or ~ 5 -10 lbs of ice

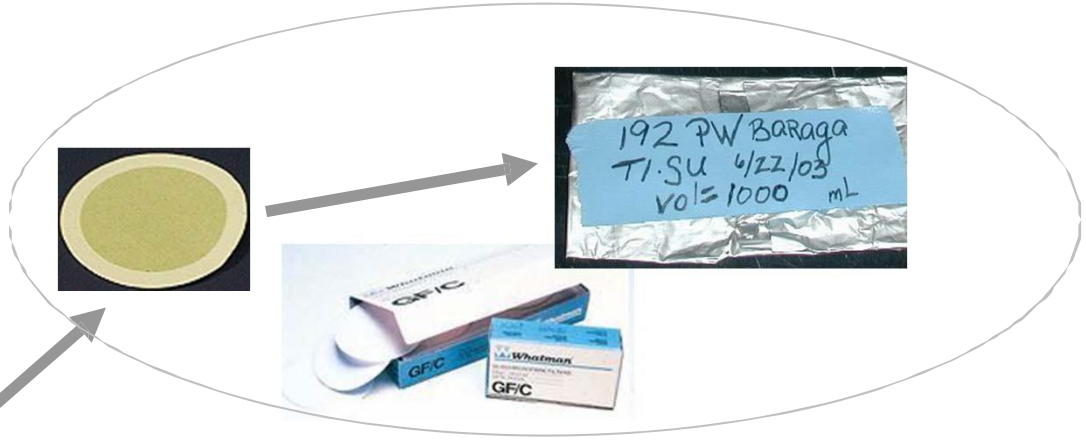
WQ supplies - 1

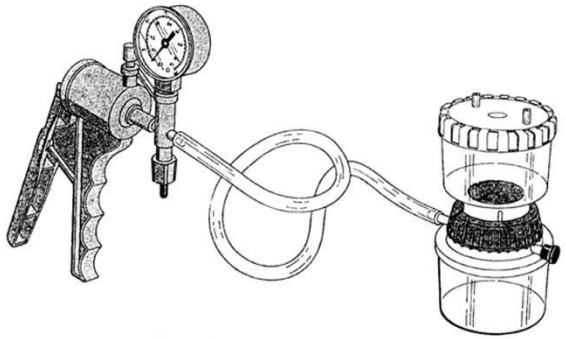
Chlorophyll-a \ and nutrient filtration

250 mL plastic

graduated cylinder

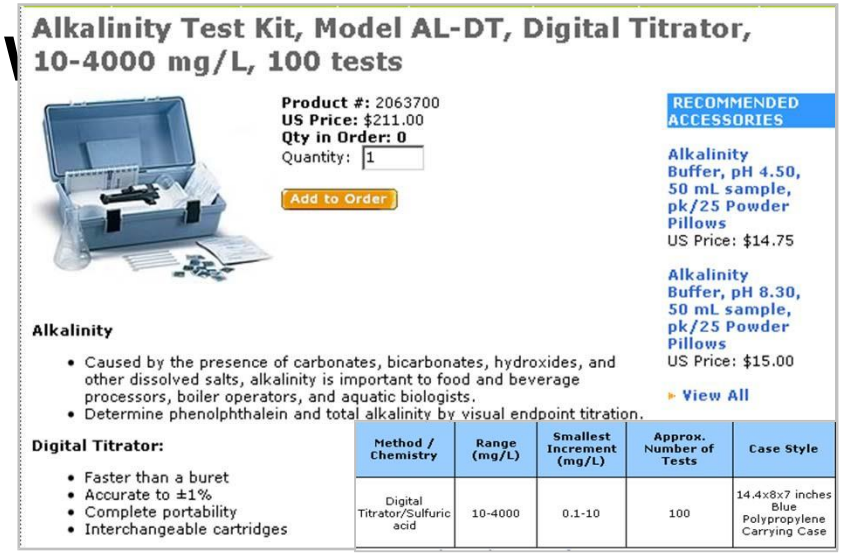
GF/C glass filters for chlor-*a*; filtrate for alkalinity, color, Cl^-





millipore 0.45 um membrane filters – refilter GF/C filtrate for dissolved nutrients using 60 mL syringe + luer-lok filtrator

OR



Alkalinity Test Kit, Model AL-DT, Digital Titrator, 10-4000 mg/L, 100 tests

Product #: 2063700
US Price: $211.00
Qty in Order: 0
Quantity: 1
Add to Order

RECOMMENDED ACCESSORIES

Alkalinity Buffer, pH 4.50, 50 mL sample, pk/25 Powder Pillows
US Price: $14.75

Alkalinity Buffer, pH 8.30, 50 mL sample, pk/25 Powder Pillows
US Price: $15.00

View All

Alkalinity

- Caused by the presence of carbonates, bicarbonates, hydroxides, and other dissolved salts, alkalinity is important to food and beverage processors, boiler operators, and aquatic biologists.
- Determine phenolphthalein and total alkalinity by visual endpoint titration.

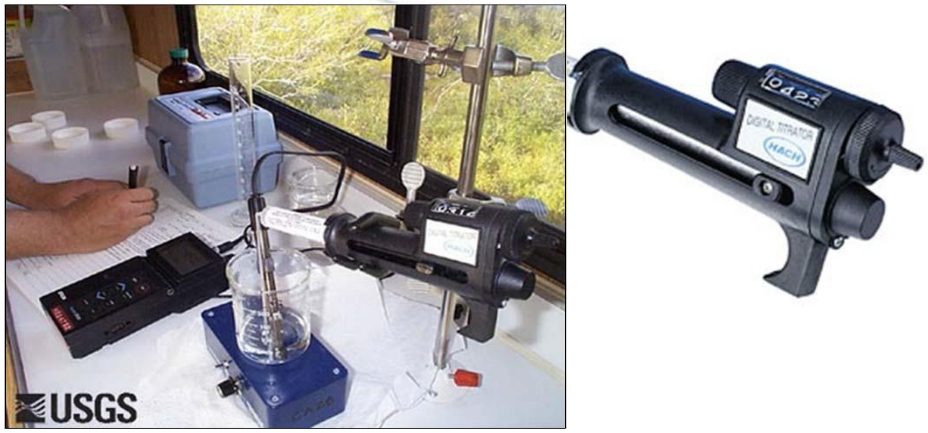
Digital Titrator:

- Faster than a buret
- Accurate to ±1%
- Complete portability
- Interchangeable cartridges

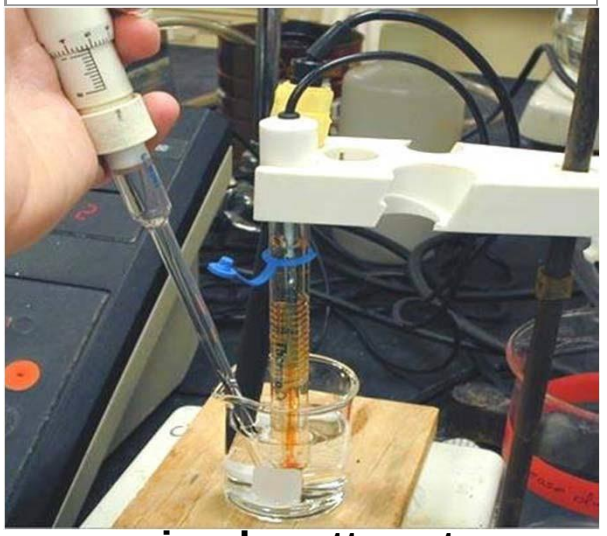
Method / Chemistry	Range (mg/L)	Smallest Increment (mg/L)	Approx. Number of Tests	Case Style
Digital Titrator/Sulfuric acid	10-4000	0.1-10	100	14.4x8x7 inches Blue Polypropylene Carrying Case

If someone is starting from scratch, I suggest the Hach kit, but get extra reagents; still need a stir plate and pH meter.

Hach #: 2063700 $211.00
GO TO www.hach.com and search "alkalinity test kit"



Alkalinity titration



microburette set-up

conventional set-up

WQ supplies - 3

www.Coleparmer.com

1. **Gilmont Micrometer Burette – <u>but cheaper to buy Hach titrator on previous page ($211 w/reagents)</u>**

- EW-07846-00 Gilmont® micrometer burette, 2 mL $243.00
- EW-07847-00 Gilmont® micrometer burette spare tip, 2 mL, $153.00

2. **Hand-operated vacuum/pressure pump, 35 mL/stroke, PVC body**
   - EW-79301-21 Qty: 2 (a spare) @ $96.00 each
- Tygon tubing to fit

3. **Plastic filtrators, syringes, etc**
   - EW-06137-58 Economical graduated plastic cylinder, 250 mL @ $19.00 each
   - EW-06623-32 Polypropylene filter holder for 25-mm membranes $108.00 / pkg of 6
   - 2-3 pairs of flat filter forceps
   - boxes of Whatman GF/C glass fiber filters (42.5mm diECs) – start with ~ 500
   - boxes of 25 mm millipore 0.45 micron “HAWP” membrane filters ~ 500
   - 2 or 3 squirt bottles for DIW + a bunch of 1 gallon plastic bottles for DIW (can use supermarket <u>deionized</u> water jugs (not distilled) because their easy to pour; check for screwcaps; or use cubitainers)
   - 1 or 2 squirt bottles for 0.1 N HCl + a couple gallons of 0.1N HCl (supermarket DIW jugs or cubitaniers)

4. EW-62502-00 **Nalgene *compositing* carboy with spigot, 9 L @ $153 each**

Nalgene funnel ~150mm diam with cemented disc of 500 um Nitex netting <u>Need spare + Goop</u>

- EW-06123-40 polypropylene powder funnel, 700 mL; $24.00 / package of 4:
- NOTE- Water for nutrients will be poured through the screen and so will contact the screen and the cement (Goop or other silicone underwater glue). So after sealing the screen in place taking care to make it “leak-proof,” soak it in 1N HCl overnight, then in tap water for a few days, then in DI water to leach excess cement residues.

WQ supplies - 4

www.Coleparmer.com

5. **Stir plate for alkalinity titrations** – Cole-Parmer has AC and battery powered models ~$180-300. No need for heat, so get what you prefer depending on field or motel use. Need a couple of small plastic or teflon coated stir bars. If heated, bring a piece of ¼ inch plywood.

- a couple of 50-100 mL plastic beakers for titrations
- 35, 50, or 100 mL plastic graduated cylinders for measuring out sample for titrations (smallest needed)

6. **pH meter for titrations in lab**, but also could be used in field for oRP if desirable.

- Matt Cooper suggests **Denver Instruments Waterproof UltraBasic model UP-5 using a high-performance glass combination electrode**. It's battery-powered and he's used it for a few years *However*, Cole-Parmer shows:

6-1. It costs $427 for the portable waterproof meter at Cole-Parmer: EW-59505-00.

- Also need EW-59505-55 ATC probe (autoTempCompensation) @ $168 and an EW-59505-60 Power adapter, 115 VAC @ $35.00 /EA
- Also need a EW-59505-55 ATC probe (autoTempCompensation) @ $168 and an EW-59505-60 Power adapter, 115 VAC @ $35.00 /EA
- Would also need a probe holder; CHECK ALL THIS OUT BEFORE BUYING.

6-2. Or their 115VAC Ub-5 bench model EW-59503-00 @ $472.75 that includes: combination pH electrode with built-in ATC, base, electrode arm, power supply, and manual. Or UB-10 Bio-Kit for $791 includes: TRIS-compatible, combination pH electrode with built-in ATC, base, electrode arm, power supply, and manual- This meter provides a mV output to use for ORP, but probe is not included. NOTE- this suggests it is also battery powered but not in a waterproof case. CHECK COMBINATION PROBE w/Matt

- **Beckman pHI® 410, pH/mV meter, waterproof, handheld (meter only)** part No.A58734 is another one with a good reputation and similar price

WQ Supplies - 5

www.Fodriest.com (or elsewhere) field WQ meters

7. NRRI recommends **YSI Model 85 temperature, dissolved oxygen (both mg/L and % saturation) and conductivity (with/without temp compensation)**

- Fondreist # 85-10 with 10' cable $1,224.00
- Buy extra DO membrane kit; NRRI will suggest an EC25 spec. conductivity standard for field calibration.

8. For those so inclined: NRRI recommends a **Hach 2100Q Portable Turbidity Meter**

- Fondreist # 2100Q 01 2100Q portable turbidimeter, meets EPA Method 180.1 $930.00
- Turbidity standards kit needed also

9. Water sampling pole –NAECO Telescoping Swing Sampler 6' to 12' @ $140 Fondriest #W1310

- Or duct tape a 1 liter wide mouth Nalgene or HDPE bottle onto a 2 m pole or PVC pipe.