

Community Clean Water Institute

Volunteer Water Quality Monitoring Program

Sampling and Analysis Methods

Sampling

Samples are collected by direct insert of a probe, collection of surface water by a container on a pole or by a bucket attached to a rope. Field analysis is performed on the samples collected on site. For samples returning to CCWI the sampling containers for nutrient testing are sterilized Whirl Pak bags. Containers for bacteria testing are sterilized wide mouth bottles with attached lid. Samples containers are marked with Site ID, time, and date either with label stickers or by writing with a Sharpie pen. Samples are placed in a cooler with ice or blue ice for transport to CCWI. CCWI staff keep samples refrigerated until analyzed. Operators are instructed to refrigerate samples at home if they are not to be returned to CCWI right away. Chain of Custody is recorded on the field datasheet.

For details on sampling collection and instrument operation, visit http://www.ccw.org/resources/water_tests.html. Though not available at our website, the instructions for calibration of the polarographic dissolved oxygen meter as well as operation of the recently acquired (10/2006) luminescence dissolved oxygen meter are sent into the field along with field datasheets.

Data collection

The Stream Quality Field Data Form captures the site location & conditions, instrument ID, Field results with sampling method and serves as a chain of custody. (See attached form page 6)

Each of the instruments has a data collection form that organizes the sample identifiers, the observed data and calculation of the result. (See attached forms page 7)

Instrument maintenance is documented in the Equipment Maintenance Log (See attached form page 8). All reagents, equipment and standards are logged, given an ID and all accompanying documentation is filed. Maintenance and narrative about the functioning of the ion chromatograph is described in a bench laboratory log.

Calibration and accuracy checks are recorded in the Calibration Record (See attached form page 9).

Results and metadata from these forms are posted to an Excel spreadsheet (the Ultimate) as a centralized data repository. The database is reviewed against original data sheets for validation, and outliers investigated and qualified.

Methods:

	METHODNAME	METHODDESCR	Res	Reporting Limit	Units
Field	EPA170.1B	Temperature by Bulb	0.3	NA	Deg C
Field	EPA170.1T	Temperature by Thermocouple	0.1	NA	Deg C
Field/LAB	EPA150.1	pH	0.1	NA	pH Units
Field/LAB	EPA120.1	Specific Conductance	--	10	micro Siemens
Field/LAB	EPA180.1	Hach 2100P Turbidimeter	--	0.01	NTU
Field	ICM-DO	Dissolved Oxygen (polarographic)	0.1	NA	mg/L
Field	Hach10360	Dissolved Oxygen (luminescent)	0.1	NA	mg/L
LAB	IDEXX9223	Bacteria	--	1	MPN
LAB	IDEXX9223	<i>E. coli</i> Bacteria	--	1	MPN
LAB	HachNI-14	NO ₂ +NO ₃ -N (color wheel)	--	0.02	mg/L
LAB	LaMotte3649-SC	NO ₂ +NO ₃ -N (colorimeter)	--	0.02	mg/L
LAB	EPA300.0M	NO ₃ -N (Ion Chromatography)	--	0.02	mg/L
LAB	HachPO-24	PO ₄ -P (color wheel)	--	0.03	mg/L
LAB	LaMotte3653-SC	PO ₄ -P (colorimeter)	--	0.03	mg/L
LAB	EPA300.0M	PO ₄ -P (Ion Chromatography)	--	0.03	mg/L

Method Names: Is a condensation of source+identification. Example: HachNI-14 refers to the Hach kit number NI-14.

RS = resolution (size of the smallest observed interval)

NA = Not Applicable

MPN = Most Probable Number

NTU = Nephelometric Turbidity Unit

Field Testing

Temperature

Alcohol filled bulb thermometer and or a thermocouple in the Dissolved Oxygen Meter. The range of the Bulb Thermometer is from -5 to 50 deg C in 0.5 divisions. The thermocouple is a digital output to the tenths of a degree Celsius.

pH

Instrument is a pHTester model 10 from Eutech instruments. The method is electrometric with temperature compensation, with a two point calibration (7 & 10). Results are recorded as displayed to the tenths place.

Specific Conductance

Instruments are the ECTester from Eutech. One instrument the ECTester Low operates in the 0 to 2000 micro Siemens range with a resolution of 10 micro Siemens. The ECTester High operates in the 0 to 19900 micro Siemens Range with a resolution of 100 micro Siemens. The method is direct conductivity measurement with temperature compensation, with a single point calibration. Results are recorded as displayed.

Turbidity

Instrument is a Hach Model 2100P Portable Turbidimeter Catalog number 46500-88, range 0-1000 NTU. Instrument calibration is performed with Hach StablCal Calibration set Catalog number 26594-05 as per manufacturer's instructions on a quarterly or as needed basis. Secondary Gelex Standards are used between calibration to check accuracy to within 5%, and accompany the meter to the field for use as an accuracy check should meter malfunction be suspected. Meter set to autorange and signal average. Data recorded as displayed in digital format. No rounding rules applied.

Dissolved Oxygen

1. Polarographic Electrode method using an ICM Model 31050 Oxygen Meter. Instrument calibrated to moist air at the lab or in the field prior to use per day. Reported values are as observed on the meter with out compensations for altitude, barometric pressure or salinity.
2. Hach Luminescent Dissolved Oxygen (LDO) Model HQ10, method 10360. Calibrations are not normally required for this probe. The calibration is verified by sampling an air saturated water. Reported values are as observed on the meter with out compensation for salinity. This instrument compensates for barometric pressure based on an internal pressure sensor. EPA approved the validation study for this method in July of 2006.

Note: This LDO meter model has been replaced with HQ d series which feature an improved cable connection to the meter.

Lab Testing

Bacteria:

Total Coliform Bacteria by multiple well Quani-Tray Method: 9223 (IDEXX Corp.)

The lowest Most Probable Number is 1 when a single small well is positive out of the 48 small and 49 large wells.

E. coli Bacteria by multiple well Quanti-Tray Method: 9223 (IDEXX Corp.)

The lowest Most Probable Number is 1 when a single small well is positive out of the 48 small and 49 large wells.

Individually wrapped sterile pipettes are used for dilutions.

pH

Instrument is a pHTester model 10 from Eutech instruments. The method is electrometric with temperature compensation, with a two point calibration (7 & 10). Results are recorded as displayed to the tenths place. The probe is calibrated to 7.0 and 10.0 buffers by CCWI staff before each deployment.

Specific Conductance

Instruments are the ECTester from Eutech. One instrument the ECTester Low operates in the 0 to 2000 micro Siemens range with a resolution of 10 micro Siemens. The ECTester High operates in the 0 to 19900 micro Siemens Range with a resolution of 100 micro Siemens. The method is direct conductivity measurement with temperature compensation, with a single point calibration. Results

are recorded as displayed. The probe is checked for accuracy and calibrated as needed before each deployment.

Turbidity

Instrument is a Hach Model 2100P Portable Turbidimeter Catalog number 46500-88. Instrument calibration is performed with Hach StablCal Calibration set Catalog number 26594-05 as per manufacturer's instructions on a quarterly or as needed basis. Secondary Gelex Standards are used between calibration to check accuracy to within 5%, and accompany the meter to the field for use as an accuracy check should meter malfunction be suspected. Meter set to auto range and signal average. Data recorded as displayed in digital format. No rounding rules applied.

Nitrate+Nitrite-Nitrogen

1. Color wheel

By Hach Low Range Nitrate Test Kit Model NI-14 Catalog number 14161-00 using the Nitrate Nitrogen (0-1 mg/L) procedure. A separate procedure to assess the Nitrite Nitrogen is not performed. The mg/L Nitrate+Nitrite Nitrogen is read directly from the color wheel. Color Wheel Part number 14171 is from zero to 1.0 mg/L Nitrate Nitrogen with divisions at 0.02 mg/L. The lowest non zero division for this wheel is 0.02 mg/L Nitrate+Nitrite Nitrogen. The reporting limit for this method is 0.02 mg/L Nitrate+Nitrite Nitrogen.

2. Colorimetric

By LaMotte's Smart 2 Colorimeter with the reagent kit code: 3649-SC. This is a low range cadmium reduction method. The colorimeter measures the amount of color in the sample. This sample absorbance is compared to a calibration curve stored within the meter to return a concentration of Nitrate-Nitrogen (NO₃-N) in mg/L. The calibration curve is set by LaMotte. Nitrite present in the sample is included in the result. Results are mg/L Nitrate+Nitrite-Nitrogen with a reporting limit of 0.02 mg/L.

Nitrate-Nitrogen

Ion Chromatography

Dionex Model 4000i with Anion Separator 14S column running a Carbonate / bicarbonate eluant (0.005 Molar Sodium Carbonate / 0.0007 Molar Sodium bicarbonate). A six point calibration curve is prepared following each eluant prep. The calibration is verified by performance on an externally prepared reference solution which also serves as the continuing calibration verification.

This method measures Nitrite separately from Nitrate. The results are Nitrate-Nitrogen (NO₃-N) with a reporting limit of 0.02 mg/L.

Phosphate-Phosphorus

1. Color wheel

By Hach Total Phosphate Test Kit Model PO-24 Catalog 2250-01 using the Low Range procedure. The observed value from the disk is divided by 50 to obtain the mg/L phosphate then divided again by 3 to represent the mg/L PO₄-P.

The color wheel part number 24898-00 is from 0 to 50 mg/L in unit divisions. For the Low Range method this represents a 0.02 to 1 mg/L phosphate range or 0.007 to 0.33 mg/L PO₄-P. The reporting limit for this method is the first non zero division at 0.007 mg/L PO₄-P. For the medium range method the reporting limit is five times the Low Range method at 0.03 mg/L PO₄-P.

2. Colorimetric

By LaMotte's Smart 2 Colorimeter with the reagent kit code: 3653-SC. This is an Ascorbic Acid reduction method. The colorimeter measures the amount of color in the sample. This sample absorbance is compared to a calibration curve stored within the meter to return a concentration of phosphate in mg/L. The calibration curve is set by LaMotte. The phosphate result provided by the instrument is divided by three to present the phosphate-phosphorus value (PO₄-P). The reporting limit for this method will be the same as the color wheel at 0.03 mg/L PO₄-P.

3. Ion chromatography

Dionex Model 4000i with Anion Separator 14S column running a Carbonate / bicarbonate eluant (0.005 Molar Sodium Carbonate / 0.0007 Molar Sodium bicarbonate). A six point calibration curve is prepared following each eluant prep. The calibration is verified by performance on an externally prepared reference solution which also serves as the continuing calibration verification. A blank begins each run, and reference solution is run at the beginning and end of each run. The results are Phosphate-Phosphorus (PO₄-P) with a reporting limit of 0.03 mg/L.

Laboratory methods: all glassware is cleaned with alconox detergent and rinsed twice with deionized water before use and in between samples. All reagents and standards are tracked for expiration date.

-end of section

STREAM QUALITY FIELD DATA FORM

Community Clean Water Institute Citizen Monitoring Program

Weather in past 48 hours:

COLD SUNNY

Weather now:

COLD overcast

(TERMS: Storm, Rain, Intermittent Showers, Overcast, Partially Cloudy, Sunny)

Stream name: Dutch Bull Creek
Watershed name: Dutch Bull Creek
Citizen Monitors: Tom Austin
Date: 12/20/06

QC check 1/24/07 dd

Instrument ID	TB-CCWI-14	PHEL-CCWI-6	TB-CCWI-15	DOL-CCWI-01	DOL-CCWI-01	TUN-CCWI-01	EC-CCWI-1	Please fill in one number	Stream Reach Type
Site ID	Air Temperature (°C)	pH	Water Temperature (°C)	Water Temperature (°C)	Dissolved Oxygen (mg/L)	Turbidity (NTU)	Electrical Conductivity (us or ms)	Observed Flow:	1.) Pool 2.) Run 3.) Riffle
D3C000	3:00 10	8.0	5.5	6.5	10.6	2.48	330	1.) Dry 2.) Isolated pool 3.) <0.25 gal/sec 4.) <5 gal/sec 5.) >5 gal/sec 6.) Full waterway, no observed flow	21
D3C050	3:30 6.5	7.8	6.2	7.0	11.3	3.21	300	1.) pole 2.) bucket 3.) grab	1
LAW010	3:45 6	8.2	4.8	5.6	11.8	4.33	220		2
D3C030	4:00 7	8.0	4.5	5.5	11.8	2.67	240		2
D3C020	4:15 6.5	7.9	4.9	5.6	11.8	2.06	230		2
D3C010	4:45 5.5	8.0	5.0	5.9	11.9	2.10	240		2

Chain of Custody

Relinquished By:	Received By:	Date:	Time:
<u>Tom Austin</u>	<u>Sarah Shaffer</u>	<u>12/21/06</u>	<u>9:30am</u>

Dissolved Oxygen Meter Calibration:

Date: _____ Time: _____
Pressure (assumed): _____ mmHg
DO Temp: _____ °C
Pre-Calibration reading: _____ mg/L
Post-Calibration reading: _____ mg/L

Write any Comments on Back of Page

Creek Name	Date & Time Collected
Santa Rosa	9/29/05 11:45a

Date Tested in Lab:

9/30/05

Phosphate and Nitrate Tests

- ☒ All lab ware washed in Alconox
- ☒ All lab ware rinsed in De-ionized water
- ☒ Standard procedure followed

Test For Nitrate

Kit ID#: NO3C-CCWI-7g
NO3P-CCWI-7g

Site Name	Color Wheel (Nitrate-N)	NO3 (X4.4)	Time Tested	Signature
SRCD40	0.08		5:30p	R
BLANK				

Comments:

Test for Phosphate

Kit ID#: PO4A-CCWI-15 7g

Site Name	Color Wheel	PO4 ($\div 50$ Lo or 10mid)	Range (Lo/Mid)	P (PO4 value $\div 3$)	Time Tested	Signature
SRCD40	A	0.4	MID	0.133	4:40p	R
BLANK						

Comments:

Revised 8/10/05

0 x 001

QC check
2-4-07

Equipment Maintenance Log

Date	Parameter	Description of Action and Instrument/Standard ID
1-3-06	DO-ccwl-01	allowed meter to dry for 2 weeks. when turned on today, calibrated fine & appears to be in working order.
1-20-06	DO-ccwl-01	added solution during calibration
4-24-06	TUN-ccwl-01	changed all 4 Batteries
5-1-06	DO-ccwl-01	changed membrane + added solution
5-1-06	DO-ccwl-01	meter unable to calibrate; O ₂ reading unstable; Temp reading stable. Buffed all 8 Batteries, meter returned to normal; calibrated.
5-2-06	DO-ccwl-01	meter calibrated in office, but upon delivery to site, O ₂ read 0.00 w/ no response to anything, Temp reading stable.
5-5-06	DO-ccwl-01	meter still reading 0.00 for O ₂ .
4-28-06	phel-ccwl-05	unable to calibrate; E1, E2; ordered replacement meter from forestry suppliers
5-10-06	DO-ccwl-01	newly ordered new probe 3511-10 FF. Rcvd 5-10-06. meter calibrated fine w/ new probe attached. changed membrane solution.
5-6-06	phel-ccwl-05	rec'd new ph test R 10 (new model). Attempts to cal w/ existing Hach Powder Buffers (7.0; 10.0) did not work. Readings standard values @ 6.9 & 9.2; not 7.0 & 10.0. changed standard solutions; still nothing.
5-10-06	phel-ccwl-05	attempts to calibrate again; still not reading values correctly.
5-11-06	phel-ccwl-05	changed setting on meter from NIST standards to USA stand. meter calibrated correctly @ both 7.0 & 10.0. (✓)
5-11-06	DO-ccwl-01	changed membrane solution; meter was reading low @ 6.0mg
6-7-06	ec-ccwl-01 low	changed all 4 Batteries
6-7-06	DO-ccwl-01	changed all 8 Batteries
6-7-06	DO-ccwl-01	added membrane solution
6-12-06	DO-ccwl-01	meter doesn't change from blw 0.2-0.5 on O ₂ setting. tried changing solution; temp setting works fine. called ICM - mailing unit in for repair. (R)

CALIBRATION RECORD

CCW 2006

Date & Time	Parameter and Instrument ID	Operator Name	Purpose: calibration, accuracy, drift	Standard value and ID	C°	Reading pre-calibration	Action
1-12-06 2:15p	EC-CCW-01	JS	cal	ECST-CCW-1L 440-450us		450	none
1-12-06 2:30p	DOE-CCW-01	JS	cal	wet paper towel = 742mmHg	22.6	10.9	cal to 8.6
1-12-06 2:22p	TUN-CCW-01 TUST-CCW-5	JS	accuracy - check	select secondary standard 5.53		5.15	none
1-12-06 2:21p	TUN-CCW-01 TUST-CCW-6	JS	accuracy - check	select secondary standard 53.8		53.7	none
1-14-06 2:40p	EC-CCW-01	JS	cal	ECST-CCW-1L 440-450us		440	none
1-14-06 2:48p	PHEL-CCW-06	JS	cal	PHB7-CCW-1C PHB10-CCW-1B		7.3/10.0	cal to 7.0/10.0
1-14-06 2:48p	DOE-CCW-01	JS	cal	wet paper towel 760mmHg	20.8	8.5	cal to 9.0
1-20-06 9:35a	EC-CCW-01	JS	cal	ECST-CCW-1L 440-450us		450	none
1-20-06 9:35a	PHEL-CCW-06	JS	cal	PHB7-CCW-1C PHB10-CCW-1B		7.4/9.9	cal to 7.0/10.0
1-20-06 9:30a	DOE-CCW-01	JS	cal	wet paper towel 760mmHg	20.5	10.0	cal to 9.0
1-24-06 10:20a	PHB-CCW-01	JS	cal	wet paper towel 760mmHg	20.5	8.3	cal to 8.6
1-24-06 10:20a	EC-CCW-01	JS	cal	ECST-CCW-1L 440-450us		460	cal to 450
1-24-06 10:15a	PHB-CCW-06	JS	cal	PHB7-CCW-1C PHB10-CCW-1B		7.4/9.9	cal to 7.0/10.0
1-23-06 9:15a	DOE-CCW-01	Tom Austin (DRC)	cal	PHB wet paper towel air humidity 760	22.7	9.4	8.7

