

# **US Fish and Wildlife Service 2004 Nutrient Grab Sample Protocol**

**By**

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## **Introduction**

Since 2001, the Arcata Fish and Wildlife Office have spearheaded a collaborative effort with local tribes to monitor water quality of the Klamath River. One of the sections of this program is the collection of water grab samples to identify levels of select nutrients, metals and other constituents. Grab sampling refers to water samples obtained by dipping a collection container into the upper layer of a body of water and collecting a water sample (USGS File Report -00213). Monthly grab samples are normally taken from June to October at established monitoring sites. This document is part of a thorough quality assurance/ quality control (QA/QC) program including specific directions for the proper collection and handling of samples along with collection of duplicate, blank and spike samples during each sampling event. Use of a handheld water quality meter (Hydrolab Quanta) also allows for the collection of general water quality parameters to better relate the results to conditions at the time of sampling.

## **Methods**

### Field Collection

Upon arrival at each site, the sampling churn was rinsed thoroughly three times with deionized (D.I.) water. The goal of rinsing is equipment decontamination, the

removal of substances adhering to equipment from previous exposure to environmental and other media (USGS Open File Report 00213). To do this, take 1/3 of a gallon of deionized (DI) water and pour it in the churn, being careful never to touch any of the inside components of the churn. Shake the churn vigorously so that the small amount of water comes into contact with all of the interior surfaces. Operate the plunger a few times, pour some of the water through the spigot and drain the rest through the lid and repeat two more times with the remaining DI water.

Bring the rinsed churn, Quanta and all other necessary equipment down to the sample site. Set the barometric pressure on the quanta and place it carefully upstream of the collection site. This ensures that when you wade out to collect your sample, you do not disturb sediments that effect its readings. Sampling is generally done by wading from the bank as far as is safe. Be sure to be in water that is representative of the stream and is not affected by eddies or other activity upstream. The churn then is rinsed three times with stream water in the same procedure as before. When rinsing and sampling, be sure to stand downstream of the churn making sure not to stir up the substrate where it would affect the sample. When the sample is to be collected, the churn is fully submerged into the stream then the lid is opened slightly to allow water from under the surface to enter. When it has filled, replace the lid and lift the churn from the water. Completely filling the churn should allow for all samples to be filled from one churn; thereby minimizing differences in water properties and quality between samples. The Quanta should be recorded once it has stabilized and the churn has been collected. Record these values along with any information regarding stream and/ or weather conditions before filling bottles from the churn.

### Churn Operation

Proper use of the churn guarantees the water is well mixed before the sample is collected. The churn should be stirred at a uniform rate by raising or lowering the splitter at approximately 9 inches per second (Bel-Art Products, 1993). This mixing must continue while the bottles are being filled. If filling is stopped for some reason, the stirring rate must be resumed before the next sample is drawn from the churn. As the volume of water in the churn decreases, the round trip frequency increases as the velocity of the churn splitter remains the same. Care must be taken to avoid breaking the surface of the water as the splitter rises toward the top of the water in the churn. The churn does not reliably produce representative samples when it contains less than 4 liters of water, therefore if the water level reaches 4 liters or less, stop filling bottles and refill the churn from the stream.

### Sample Bottles

Sample bottles with and without chemical preservatives were provided by associated laboratories. In the case of bottles that contained chemical preservatives, bottles were not rinsed before sample collection and care was taken to avoid over-spillage that would result in chemical preservative loss. Sample bottles without chemical preservatives were rinsed with stream water from the churn 2-3 times before filling with sample water. Collected samples were placed in coolers on ice for transport to contracted laboratories for analysis. Chain of Custody (COC) forms are filled out by a secondary crew member or after sampling takes place. Be sure to fill in all information completely,

including the time that the churn was collected, who collected the sample and if any changes were made to the analytes collected.

#### QA/QC – Duplicate, Spike and Blank bottle sets

To ensure laboratory and sampling accuracy, one site every sampling period is selected to receive three additional QA/QC bottle sets. These bottle sets incorporate duplicate, blank and spiked water samples. Duplicate samples are collected using the same process as regular samples. These are used to assure the laboratory maintains precision within results.

A limited bottle set containing ‘spiked’ samples is also collected. Spike concentrations are determined based on past findings for each analyte. The spikes should be between 5 and 50 times the minimum detection limit or between 1 and 10 times the ambient level, whichever is greater (Eaton *et. al.*, 1995). Known concentrations of selected analytes are generally added directly to the bottle in place of sample water to provide a sample with known levels of the specified analyte. Some situations have a known quantity and concentration of spike solution added to a known quantity of sample water submitted to the laboratory. The laboratory result then is calculated to find the expected stream concentration and compared to the original and duplicate values. Data forms containing the known spike concentrations are kept to verify that the lab is attaining accurate results.

Blank sample bottles are utilized to assess accuracy of the analysis and verify that the sampling method or equipment does not influence the results. After collection of all other samples at the QA/QC site, the churn is rinsed appropriately three times with D.I.

water before being filled with D.I. water. The blank bottle sets are collected in the same way as other samples, except using D.I. water in place of stream water. Blank samples are collected after all stream water samples are taken and act as a final rinse to decontaminate the churn.

All bottle sets are then placed on ice and are transported to the associated laboratories. This maintains the samples in the dark and as close to 4 °C as possible. All grab samples should be processed within known laboratory holding periods. Any sample that was not processed within known hold times must be reported within the case narrative sent by the analyzing laboratory.

### Turbidity Samples

Turbidity samples are drawn directly from the flowing stream. The appropriately labeled turbidity bottle should be rinsed with stream water three times before taking sample. Once the bottle has been rinsed, it is submerged and allowed to fill to the top, excluding air bubbles. Care should be taken to avoid the collection of surface water in the bottle. Once the bottle is filled, it is capped and placed into a cooler with ice along with the other water samples. Turbidity samples are analyzed at the Arcata Fish and Wildlife Office using a LaMotte 2020 turbidimeter within 24 hours of collection.

### **Bibliography**

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