

Water Sampling and Testing for Formaldehyde at Northwest Fish Hatcheries

Report Finalized August 2017

Prepared for the EPA Region 10 Office of Water and Watersheds



WATER SAMPLING AND TESTING FOR FORMALDEHYDE AT NORTHWEST FISH HATCHERIES

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Cover Photo: Little White Salmon River, Washington

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EXECUTIVE SUMMARY

In 2016, the U.S. Environmental Protection Agency (EPA) Region 10's Office of Environmental Review and Assessment (OERA) and the Washington State Department of Ecology (Ecology) conducted water sampling and field analysis at 10 federal and state fish hatcheries in Washington and in Idaho. The objective was to provide data on the concentrations of formaldehyde being discharged from hatcheries after applications of formalin, which is used by the hatcheries to control external parasites on hatchery fish and their eggs.

Formalin is a generic term that describes a solution of 37% formaldehyde gas dissolved in water. The Food and Drug Administration (FDA) requires a 10-fold dilution of finfish treatment water and a 100-fold dilution of finfish egg treatment water, which should lead to a discharge concentration of no more than 25 parts per million (ppm), equivalent to 25 µL/L formalin, or 10 ppm formaldehyde (active ingredient). According to the FDA, additional instream dilution, infrequent use, and rapid degradation should render the discharged formalin below a level that causes significant environmental effects on aquatic animals.¹

In a recent risk assessment of hatcheries covered by the EPA National Pollutant Discharge Elimination System (NPDES) General Permit for Federal Aquaculture Facilities and Aquaculture Facilities in Indian Country within Washington State, EPA Region 10 concluded that formalin use at hatcheries covered that NPDES General Permit is not likely to affect salmonids listed under the Endangered Species Act (ESA) in concentrations below 10 ppm formaldehyde in the receiving water.² Therefore, the EPA has used 10 ppm formaldehyde as the level of concern for this study, against which formaldehyde concentrations detected in samples were compared.

The EPA and Ecology worked with staff and managers at U.S. Fish and Wildlife Service and Washington State Department of Fish and Wildlife to identify which hatcheries in the Northwest use the most formalin, and to ensure that sampling included a range of formalin use scenarios (i.e., egg, juvenile, and adult treatments). By sampling formaldehyde in the effluent at facilities that use the most formalin across Washington and Idaho, and by sampling during peak formalin use, this study intended to capture a reasonable worst-case scenario for estimating formaldehyde concentrations in Pacific Northwest hatchery effluent.

Sampling was performed in accordance with an approved quality assurance project plan (QAPP) at a minimum of three locations per fish hatchery: 1) the influent raw water; 2) the effluent water; and 3) the receiving water. Samples from facility outfalls where treated effluent would be discharged were collected as both grab samples collected by hand and discrete interval samples collected with an automated sampler. Grab samples were collected after at a period when it was believed that the plume with the highest likely concentration of formaldehyde was being discharged through the outfall. The analytical parameters for influent raw water and effluent, are applicable field measurements (temperature, total chlorine, ammonia, dissolved oxygen, conductivity, turbidity, pH, and formaldehyde screening) and laboratory analysis for formaldehyde. The EPA Region 10 Laboratory performed all of the analytical chemistry required for this study.

In all of the hatcheries sampled for this study, the maximum concentration of formaldehyde measured did not exceed the FDA and EPA Region 10 level of concern of 10 ppm.

1 http://water.epa.gov/scitech/wastetech/guide/aquaculture/upload/2005_09_01_guide_aquaculture_EEBA_EEBA-Chapter-7.pdf accessed 12/5/2014

2 https://www3.epa.gov/region10/pdf/permits/npdes/wa/WA_Hatchery_GP_WAG130000_BE.pdf

Based on data collected at the hatcheries that participated in this study, as well as the available toxicological data for threatened and endangered salmonids and an EPA risk assessment for formalin in Washington hatcheries, the EPA believes that current levels of formalin use are generally protective of aquatic life and ESA listed salmonids in Pacific Northwest waters.

INTRODUCTION

In 2016, the U.S. Environmental Protection Agency (EPA) Region 10's Office of Environmental Review and Assessment (OERA) and the Washington State Department of Ecology (Ecology) conducted water sampling and field analysis at 10 federal and state fish hatcheries in Washington and in Idaho. The objective was to provide data on the concentrations of formaldehyde being discharged from hatcheries after applications of formalin used by the hatcheries to control disease among hatchery fish. The data resulting from this study will be used by the EPA Region 10 Office of Water and Watersheds (OWW) to inform the development of aquaculture permits issued under the National Pollution Discharge and Elimination System (NPDES). This report details the sample collection techniques used by EPA and summarizes the results of analysis for samples collected by EPA and Ecology.

BACKGROUND

NPDES Aquaculture Permits

The EPA and Ecology issue permits under the National Pollutant Discharge Elimination System (NPDES) to establish conditions for the discharge of pollutants in wastewaters to waters of the United States. In order to ensure protection of water quality and human health, the NPDES permits place limits on the types and amounts of pollutants that can be discharged and places other conditions on such activity, such as monitoring and best management practices.

At 40 CFR §122.24, the EPA defines concentrated aquatic animal production (CAAP) facilities as point sources subject to the NPDES permit program. In Idaho, EPA Region 10 is the NPDES permitting authority for CAAP facilities.³ In Washington, EPA is the NPDES permitting authority for federal aquaculture facilities (i.e., National Fish Hatcheries), and for aquaculture facilities in Indian Country within the State of Washington.⁴ Ecology is the NPDES permitting authority for all other hatchery and aquaculture facilities.⁵

Formalin

Formalin is a generic term that describes a solution of 37% formaldehyde gas dissolved in water. The Parasite-S formulation is administered in a bath treatment to control external protozoa (*Chilodonella* spp., *Costia* spp., *Epistylis* spp., *Ichthyophthirius* spp., *Scyphidia* spp. and *Trichodina* spp.), and the monogenetic trematode parasites (*Cleidodiscus* spp., *Dactylogyrus* spp., and *Gyrodactylus* spp.) on all finfish. It is also used for the control of fungi of the family Saprolegniaceae on all finfish eggs (Western Chemical Label, no date).

3 <https://yosemite.epa.gov/r10/water.nsf/npdes+permits/general+npdes+permits#Aquaculture>

4 <https://yosemite.epa.gov/r10/water.nsf/npdes+permits/general+npdes+permits#fedaqua>

5 http://www.ecy.wa.gov/Programs/wq/permits/fin_fish/index.html

Formalin is administered to salmon and trout as a bath treatment for prolonged or short periods of time. The standard dosage recommended in the INAD #9013 Protocol to prevent or control fungus on fish and eggs is to administer formalin as a static-bath or flow-through treatment at 15 - 2000 $\mu\text{L/L}$ (ppm) active drug. Eggs are treated daily or every other day until hatch. Fish are treated every other day to weekly for 30 to 60 minutes, and then transferred to clean water. The formalin concentration is water temperature dependent and 50°F is the cutoff for the two treatment concentrations. Salmon and trout are treated up to 170 $\mu\text{L/L}$ at water temperatures above 50°F and 250 $\mu\text{L/L}$ at temperatures below 50°F. All other finfish are treated up to 250 $\mu\text{L/L}$ regardless of temperature. Treatment is not recommended to exceed 1.0 hour.

In the finding of no significant impact for Parasite-S, the Food and Drug Administration (FDA) requires a 10-fold dilution of finfish treatment water and a 100-fold dilution of finfish egg treatment water, which should lead to a discharge concentration of no more than 25 ppm (equivalent to 25 $\mu\text{L/L}$ or 10 ppm formaldehyde).⁶ The FDA contended that additional in-stream dilution, infrequent use, and rapid degradation would render the discharged formalin below a level that causes significant environmental effects on aquatic animals (formaldehyde, the active ingredient in formalin, is oxidized in the aquatic environment into formic acid and ultimately into carbon dioxide and water; the estimated half-life of formaldehyde in water is approximately 36 hours). Directions for dilution of treatment water and additional environmental precautions are described on the labeling of the product. An example of the Parasite-S labeling is displayed in Appendix 1.

As part of its Biological Evaluation for EPA's NPDES General Permit for federal aquaculture facilities and aquaculture facilities in Indian Country within Washington State, and in compliance with the Endangered Species Act (ESA)⁷, the EPA performed risk assessments to determine whether formalin use at EPA-permitted aquaculture facilities in Washington and Idaho have the potential to affect threatened or listed species or their critical habitat. These risk assessments used the best available science and toxicological information. The EPA's risk assessments likely resulted in unrealistically conservative assumptions, and did not account for in-stream dilution. Based on the available toxicological data for threatened and endangered salmonids, the EPA believes that the FDA's dilution requirement (10 ppm formaldehyde) will be protective of aquatic life in Pacific Northwest waters. Therefore, the EPA has used 10 ppm formaldehyde as the level of concern for this field study, against which formaldehyde samples were compared.

There have been very few studies examining the amounts of formaldehyde discharged from fish hatcheries after formalin treatments. Some of the hatcheries participating in this study provided anecdotal information on unpublished or informal studies. One published study which did focus on measuring the formaldehyde concentration in effluent outfalls at freshwater aquaculture facilities in Nova Scotia was done by Environment Canada in 2011. Formaldehyde was applied at an effective rate of 250 $\mu\text{L/L}$ to a 10-meter diameter pond with a water temperature of 50° Fahrenheit (F). Samples were collected at the effluent outfall and at another location 100 meters downstream of the effluent outfall. Samples were collected with the aid of automated ISCO® samplers set to obtain 100 milliliters (ml) of water every 5 minutes over a 3-hour period. The ISCO® samples were composited in a glass container through Teflon tubing. Additional grab samples were also collected at three other aquaculture facilities at different times, with one effluent sample collected from an outfall 72 hours after treatment. The results of the study revealed the formaldehyde concentrations in samples

6 http://water.epa.gov/scitech/wastetech/guide/aquaculture/upload/2005_09_01_guide_aquaculture_EEBA_EEBA-Chapter-7.pdf accessed 12/5/2014

7 https://www3.epa.gov/region10/pdf/permits/npdes/wa/WA_Hatchery_GP_WAG130000_BE.pdf

collected at the outfall location during treatments ranged from 1.8 to 7.1 milligrams per liter (mg/L). The analysis of the sample collected 72 hours after treatment revealed 0.2 mg/L formaldehyde. The analysis of samples collected at the downstream locations varied in formaldehyde concentration of 0.9 to 1.9 mg/L. The study concluded that formaldehyde concentration downstream of land based aquaculture facilities could cause an adverse chronic impact to aquatic organisms.⁸

PROJECT DESCRIPTION

Selection of Sampling Sites

In order to select hatcheries at which to sample, the EPA and Ecology worked with staff and managers at U.S. Fish and Wildlife Service (U.S. FWS), Idaho Department of Fish and Game (IDFG), and Washington State Department of Fish and Wildlife (WDFW) to identify hatcheries that use the most formalin, and to ensure that sampling included a range of formalin use scenarios (i.e., egg, juvenile, and adult treatments). By sampling formaldehyde in the effluent at facilities that use the most formalin, and by sampling during peak formalin use, this study intended to capture a reasonable worst-case scenario for estimations of formaldehyde concentrations in Pacific Northwest hatchery effluent. The hatcheries at which sampling was conducted are covered under NPDES aquaculture permits by the EPA or Ecology. A list of all the hatcheries participating in this study is included in Table 1. The locations are displayed on the map in Figure 1.

Table 1: Hatcheries Sampled for this Study.

Hatchery Name	Ownership	Formalin Consumption		Sampling Date	Growth Stage Treated
		Gallons Per Year	Sampled By		
Carson National Fish Hatchery	U.S. FWS ^a	516	EPA/OERA	7/13/2016	Adults
Clearwater Fish Hatchery	IDFG ^b	1,650	EPA/OERA	7/20/2016	Adults
Cowlitz Salmon Hatchery	WDFW ^c	6,000	Ecology	9/30/2016	Adults
Dworshak National Fish Hatchery	U.S. FWS	2,680	EPA/OERA	7/18/2016	Adults
Hoodspoint Fish Hatchery	WDFW	1,155	Ecology	12/7/2016	Eggs
Kalama Falls Fish Hatchery	WDFW	1,554	Ecology	9/12/2016	Adults & Eggs
Leavenworth National Fish Hatchery	U.S. FWS	4,400 ^d	EPA/OERA	8/17/2016	Adults
Little White Salmon National Fish Hatchery	U.S. FWS	370	EPA/OERA	7/11/2016	Adults
Priest River Fish Hatchery	WDFW	2,310	Ecology	11/4/2016	Adults
				11/30/2016	Eggs
Wallace River Fish Hatchery	WDFW	2,575	Ecology	9/19/2016	Adults & Eggs

^a United States Department of Fish and Wildlife Service

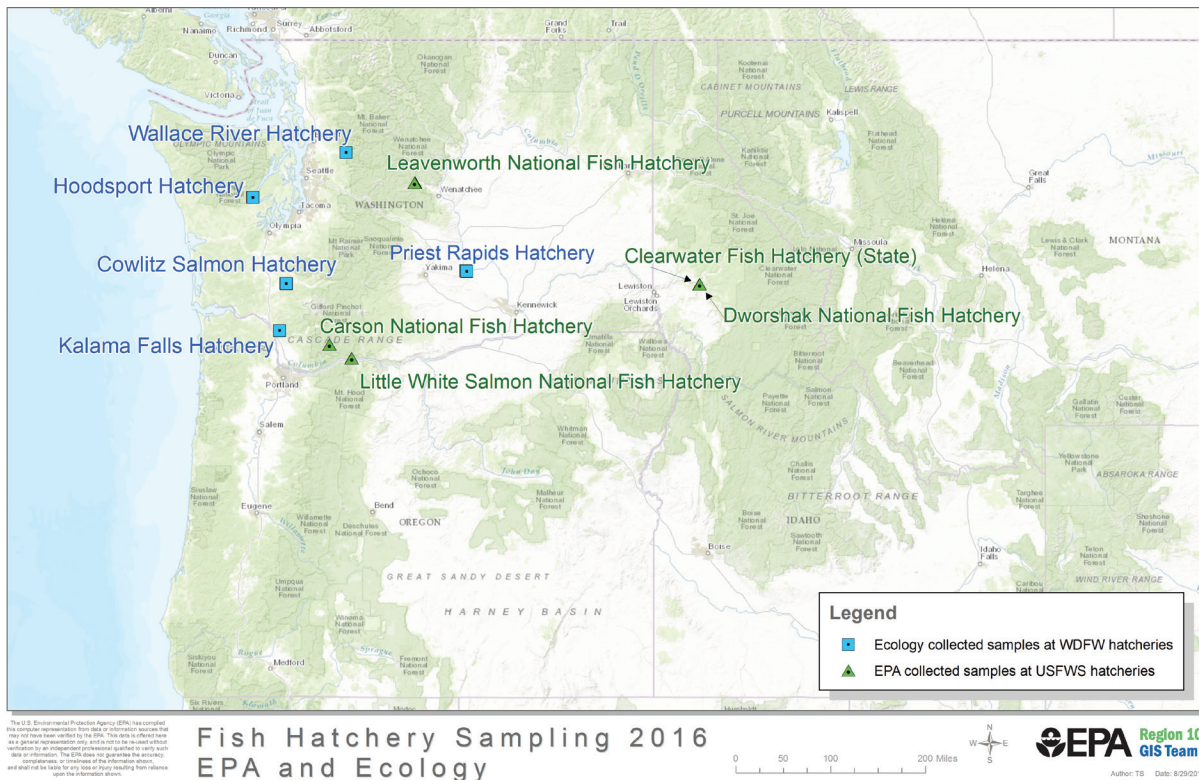
^b Idaho Department of Fish and Game

^c Washington State Department of Fish and Wildlife

^d Not including treatment of egg, juvenile, and adult Coho Salmon done by the Yakama Nation

8 Lalonde BA, Earnest W, Garron C, (2015) *Formaldehyde Concentration in Discharge from Land Based Aquaculture Facilities in Atlantic Canada*. Bulletin of Environmental Contamination and Toxicology 94:444-447

Figure 1: Locations of fish hatcheries participating in the EPA sampling events



EPA Sampling of Hatcheries

Between July 11, 2016 and August 17, 2016, OERA conducted site visits at five hatcheries to obtain samples for analysis and associated supporting data to determine the concentrations of formaldehyde being discharged through hatchery outfalls following formalin treatments. Prior to visiting the sites, the managers of the hatcheries were interviewed by phone to discuss the logistics of the planned site visits. Appendix 2 includes contact information and aerial photographs showing the sampling locations at each of the hatcheries visited by OERA. Appendix 3 includes a collection of digital images showing examples of the sampling and field measurements taken during the site visits.

Sampling was performed in accordance with an approved quality assurance project plan (QAPP) at a minimum of three locations per fish hatchery: 1) the influent raw water; 2) the effluent water; and 3) the receiving water. A copy of the QAPP is included in Appendix 4. Samples from facility outfalls where treated effluent would be discharged were collected as both grab samples collected by hand and discrete interval samples collected with an automated sampler. The grab samples were collected after at a period when it was believed that the plume with the highest likely concentration of formaldehyde was being discharged through the outfall. The analytical parameters for influent raw water and effluent, are applicable field measurements (temperature, total chlorine, ammonia, dissolved oxygen, conductivity, turbidity, pH, and formaldehyde screening) and laboratory analysis for formaldehyde. Ammonia reacts with formaldehyde thus the presence of ammonia will result in a low bias for formaldehyde. Chlorine reacts with ammonia to create chloramines and will slowly oxidize the formaldehyde to formic acid.

While significant concentrations of formaldehyde in the influent water were not expected, it was possible that at certain locations, formaldehyde occurs naturally or that other contaminants from upstream (including but not limited to pesticides and fertilizers used in agricultural or forestry operations) may interfere with formaldehyde analysis. The composite downstream sample was collected to reveal the concentration of formaldehyde downstream of the facilities after mixing the effluent with the receiving water.

Ecology Sampling of WDFW Hatcheries

Between September 12, 2016, and December 7, 2016, Ecology collected samples during six events from five fish hatcheries managed by the WDFW (Table 1). Appendix 5 includes contact information and aerial photographs showing the sampling locations at each of the hatcheries visited by OERA. At the Priest Rapids Hatchery, sampling was conducted once during adult treatment, and on a separate date during egg treatment in order to capture any differences in formaldehyde concentrations during the two treatment types. At the other four hatcheries, one site visit was performed, in which adults and eggs were concurrently being treated, only adults were being treated, or only eggs were being treated. Ecology's sampling was performed in accordance with Ecology's QAPP for this project based on the original EPA QAPP. Differences between Ecology and EPA sampling included minor variations in the equipment used for sampling and field screening. Copies of Ecology's QAPP, field notes, and results of analysis have already been provided to the NPDES Permits Unit. Ecology's QAPP is included in Appendix 6.

EPA staff participated in one of Ecology's field sampling events, assisting with sampling performed at the Kalama Falls Hatchery. EPA also provided supplies including sample containers and field test kits. All of the samples collected by Ecology were analyzed for formaldehyde by the EPA Region 10 Laboratory.

METHODS AND MATERIALS

1. Field Measurements

Chlorine measurements were performed on site using a glass sample cell designed for use with a HACH® Chlorine Colorimeter with the quality control verification performed with the aid of a HACH® Chlorine DPD Secondary Standard Kit. Measurements of pH, temperature, turbidity, conductivity, and dissolved oxygen were made in-situ with a Horiba® model U53 water quality meter during collection of the physical samples. Ammonia screening was performed with the aid of a HACH® ammonia test strip kit and the formaldehyde screening was performed with the aid of a Quantofix® formaldehyde test kit.

2. Water Sampling

Water samples were collected with portable Sigma® model 900 composite water samplers. The samplers were deployed to three locations at each hatchery: the influent point, the effluent point, and downstream. The samplers were programmed to obtain a timed composite sample from the influent and downstream locations and set for timed discrete samples from the effluent location. The samples were collected over a facility specific sampling period that bracketed the application of formalin. The sample collection containers were iced during sample collection and preserved, stored, and shipped with wet ice as the coolant. The temperature of each cooler containing samples were checked with an infrared thermometer upon arrival at the laboratory. The formaldehyde sample containers were QC-Class 125 milliliter (ml) glass jars with Teflon lids.

EPA Health and Safety Considerations

OERA fieldwork performed during this project was done according to health and safety guidelines approved by OERA Management. There were no adverse health and safety incidents identified by the sampling team during sample collection or field analysis. Special care was taken to disinfect equipment between facilities in an effort to avoid potential spread of contamination or pathogens from one facility to another.

3. Analytical Methods

The water samples were analyzed for formaldehyde at the U.S. EPA Region 10 Laboratory using method EPA 1667a titled Formaldehyde, Isobutyraldehyde, and Furfural by Derivatization Followed by High Performance Liquid Chromatography. This method is for surveying and monitoring under the Clean Water Act. Method sensitivity can be varied depending upon the expected concentration. At the start of this project it was decided to pursue the lowest range of sensitivity to capture concentrations well below the target maximum of 10 ppm. Doing so required that most of the positive samples were analyzed at a 10:1 dilution. A short holding time and only a single container of each sample precluded re-extraction or duplicate analysis for any sample except the field duplicates.

4. Quality Assurance and Quality Control

One field blank and one field duplicate sample was collected during most of the sampling events. The field blank sample consisted of laboratory-grade deionized (DI) water that was prepared for formaldehyde analysis by running approximately five gallons of DI water through the Sigma® automatic composite samplers prior to initial use and before moving to the next sample location. Samples were shipped to the laboratory under chain of custody with coolers sealed shut with application of custody seals. Signed copies of the chain of custody documentation showing the condition of samples upon receipt at the laboratory are included in Appendix 7. Copies of Ecology chain of custody documentation is included in Appendix 8.

RESULTS OF ANALYSIS

EPA Sampling

The results of the formaldehyde analysis of effluent samples collected from four of the facilities are summarized in Figure 2. The graph displays the results for samples collected at ten minute intervals at the Little White Salmon National Fish Hatchery, the Carson National Fish Hatchery, the Dworshak National Fish Hatchery, and the Clearwater Fish Hatchery. ***In all cases the maximum concentration of formaldehyde measured did not exceed the FDA Acceptable Formaldehyde Discharge Concentration of 10 parts per million (ppm), which is equivalent to 10 mg/L.*** Effluent samples collected from the Leavenworth National Fish Hatchery were collected at 20 minute intervals. The results of the formaldehyde analysis of effluent samples collected from the Leavenworth National Fish Hatchery are summarized in Figure 3. The results reported are expressed in micrograms per liter (µg/L).

The results of the OERA field measurements taken during sampling events at the hatcheries are summarized in Table 2. The water quality measurements, including the results of ammonia and chlorine analysis, did not impact the analysis of water samples tested for formaldehyde in the laboratory.

Water Sampling and Testing for Formaldehyde at Northwest Fish Hatcheries

Table 2.

Summary of OERA Field Measurement Data

Hatchery Name	Measurement Location	Temp. °C	Temp. °F	pH	Turbidity NTU	Conductivity Ms	Dissolved O ₂ ppm	Ammonia test strip ppm	Chlorine ppm	Formaldehyde test strip ppm
Little White Salmon NFH	Influent	7.91	46.24	7.08	0.82	0.043	12.83	ND	0.03	ND
	Effluent	7.73	45.91	6.48	1.85	0.043	11.05	ND	0.01	10
	Receiving Water	8.27	46.89	6.99	0.85	0.043	11.24	ND	0.05	ND
Carson NFH	Influent	9.36	48.85	5.79	0.18	0.049	12.79	ND	0.08	ND
	Effluent	9.23	48.61	5.93	2.04	0.049	11.27	0.25	0.01	10
	Receiving Water	11.58	52.84	5.81	0.76	0.058	8.96	0.25	0.01	10
Dworshak NFH	Influent	7.34	45.21	6.76	0.7	0.032	14.73	ND	0.05	ND
	Effluent	7.59	45.66	6.27	0.76	0.032	16.62	ND	0.09	10
	Receiving Water	7.54	45.57	6.72	0.8	0.032	16.62	ND	0.09	ND
Clearwater FH	Influent	10.73	51.31	6.34	0.75	0.032	14.73	ND	0.07	ND
	Effluent	12.58	54.64	6.64	1.15	0.035	14.82	0.25	0.11	10
	Receiving Water	7.36	45.25	6.29	1.04	0.033	13.77	ND	0.06	ND
Leavenworth NFH	Influent	14.62	58.32	7.26	No Meas	0.035	11.94	ND	ND	ND
	Effluent	14.73	58.51	7.36	2.04	0.076	8.05	0.25	ND	ND
	Receiving Water	15.58	60.04	7.53	0.19	0.041	15.39	0.25	ND	ND

Analysis of the influent water at all of the hatcheries resulted in no formaldehyde detected. In addition, there were no detectable interferences noted, these would be seen as absorbing in the near-ultraviolet and interfering with the quantitation of the derivatized formaldehyde. The results of analysis of the receiving waters collected as composite samples revealed varying concentrations of formaldehyde ranging from an estimated 96 µg/L downstream from the Clearwater Hatchery to 1000 µg/L downstream from the Carson Hatchery. Analysis of the grab samples collected at four of the fish hatcheries revealed concentrations of formaldehyde ranging from none detected (<20 µg/L) at the Clearwater Fish Hatchery to 2000 µg/L at the Little White Salmon National Fish Hatchery.

Analysis of the field blanks collected at the hatcheries revealed no formaldehyde detections. Also, the results of analysis for field duplicates showed variable reproducibility. The field duplicate results are summarized in Table 3. For the most part, there was pretty good agreement between field duplicates with the relative percent difference ranging between 0 and 32.3%. The one exception was for the field duplicate collected at the Clearwater fish hatchery where the initial sample had 32 µg/L and the duplicate was < 20 µg/L.

Water Sampling and Testing for Formaldehyde at Northwest Fish Hatcheries

Figure 2: Concentrations of formaldehyde for hatchery effluent sampled at 10-minute intervals.

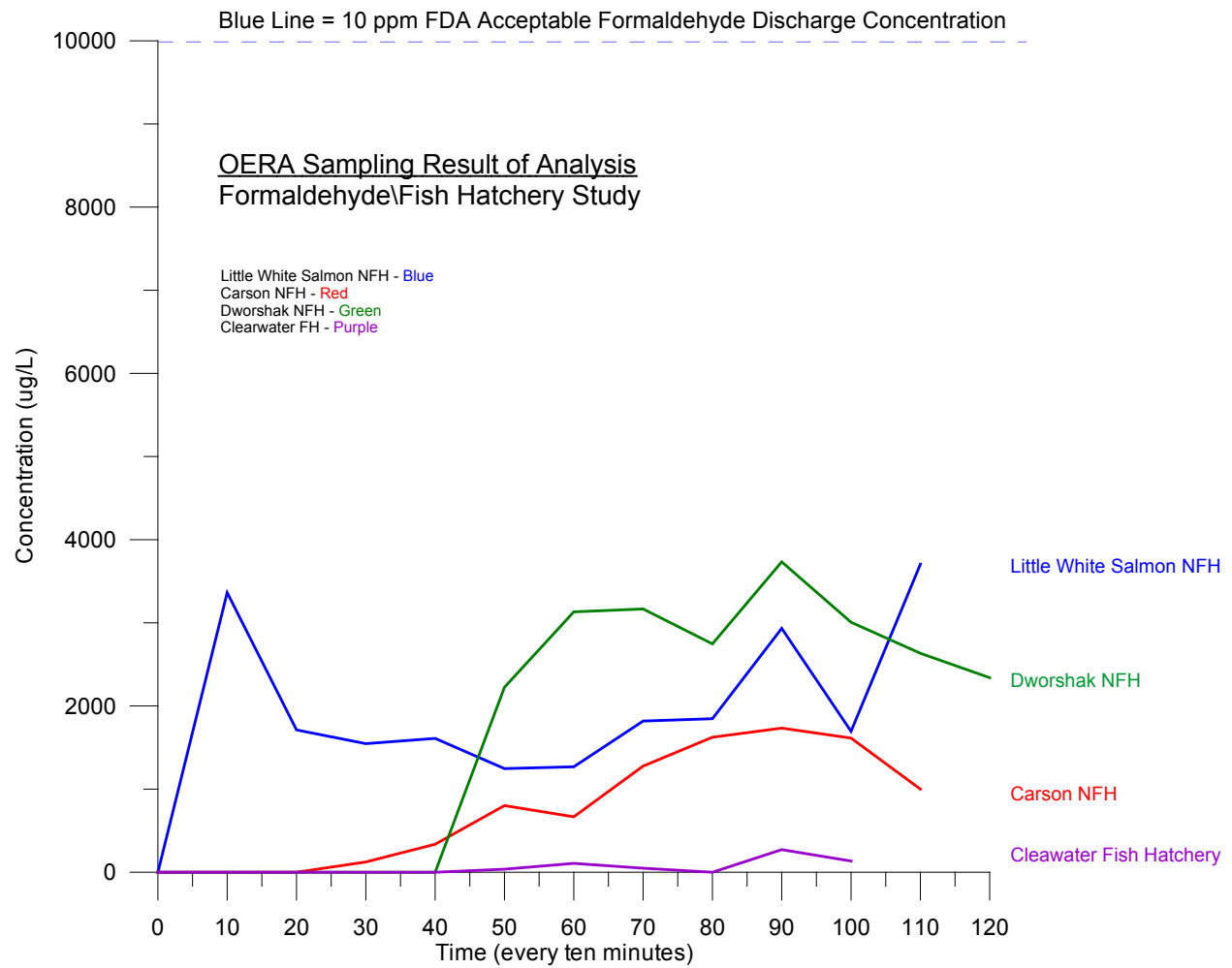


Figure 3: Concentrations of formaldehyde for hatchery effluent sampled at 20-minute intervals.

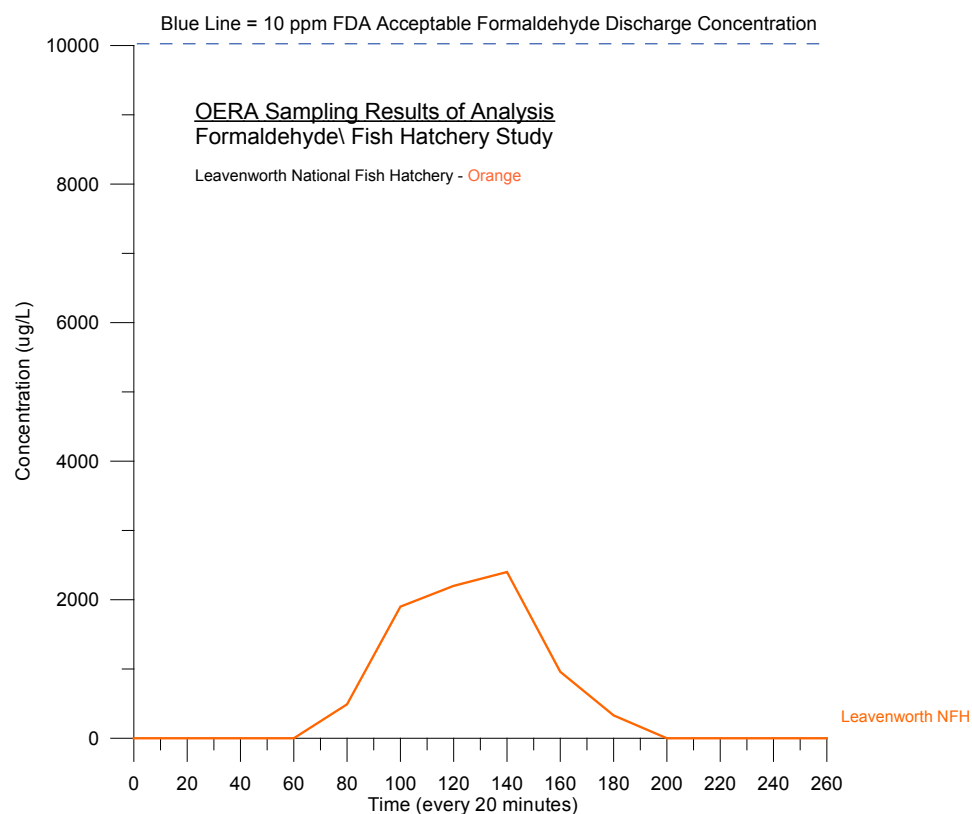


Table 3.

Summary of Field Duplicate Measurements

Hatchery Name	Measurement Location	Sample Number	Result $\mu\text{g/L}$	Duplicate Number	Result $\mu\text{g/L}$	RPD
Little White Salmon NFH	Effluent	16284107	1300	16284116	1800	32.3
Carson NFH	Effluent	16284124	120	16284135	140	15.4
Clearwater FH	Effluent	16294128	32	16294136	<20	50 -200
Leavenworth NFH	Effluent	16334105	490	16334118	490	0

The relative percent difference (RPD) for the Clearwater fish hatchery duplicate samples is expressed as a range given the detection limit of 20 $\mu\text{g/L}$.

Additional Information Collected

During the site visits, the field team collected additional information on the species of fish being treated with formalin, the type of formalin being applied, and the conditions under which it was being applied. The aquatic species being treated during all of the site visits was

adult spring Chinook salmon (*Oncorhynchus tshawytscha*). The formulation of formalin used at all of the federal hatcheries was Parasite-S, New Animal Drug Application (NADA) number 140-989, manufactured by Western Chemical, Inc. In the case of the Clearwater fish hatchery, the fish were being treated with a generic version of Parasite-S containing 37% formaldehyde called Formacide-B, Abbreviated New Animal Drug Application (ANADA) number 200-414, distributed by Georgia Pacific.

During the field visits, the sampling team noted there was variation in the layout of the facilities and the methods used to measure water flow and administer formalin. All the facilities were using a flow-through method of treatment which typically lasted for 60 minutes. The target concentration of formalin applied ranged between 150-200 ppm. In each case, it appears the facilities were making a good effort to follow the formalin label instructions. One minor discrepancy noted was at the Leavenworth National Fish Hatchery. The treatment was applied at a rate of 200 ppm ($\mu\text{g/L}$), but the water temperature measured by EPA was 14.62°C (58.32°F) for the influent water and 14.73°C (58.51°F) for the effluent water. The formalin label states that in water greater than 50°F (10°C) the concentration of formalin should be up to 170 $\mu\text{g/L}$. It would appear the formalin is being over applied for the temperature conditions at this facility.

One observation made at the Little White Salmon National Fish Hatchery was that the outfall used for routine monitoring under the NPDES permit appears to be different from the water channel used to discharge formalin treated water into the Little White Salmon River. Another complication encountered at this facility was an apparent power outage during sampling that caused the pump administering formalin to stop during treatment. This resulted in an incomplete and relatively distorted data displayed as the blue plot in Figure 2. As the sampling was terminated at 110 minutes, we do not know if the concentration of formaldehyde exceeded the highest level measured which was 3700 $\mu\text{g/L}$ for sample 16284112.

Ecology Sampling

Results of analysis for the Ecology samples were similar to the results of analysis from the samples collected by EPA, and the concentration of formaldehyde did not exceed the 10 ppm acceptable threshold set by FDA.

Data resulting from analysis of the Ecology samples are displayed in Figures 4, 5, and 6. The only potential discrepancy noted is shown in Figure 10 representing the data from analysis of samples from the Cowlitz Hatchery. There is a steep decline (from 2100 $\mu\text{g/L}$) in the curve to none detected ($<20 \mu\text{g/L}$) at 180 minutes and then a steep incline (to 1700 $\mu\text{g/L}$) afterward. This means that formaldehyde, if any, was below the detection limit for sample 16395258, a discrete sample collected at 1:00 PM. It is likely that this was an error caused by a mislabeled container.

The results of the Ecology field measurements taken during sampling events at the hatcheries are summarized in Table 4. The water quality measurements including the results of ammonia and chlorine analysis, did not impact the analysis of water samples tested for formaldehyde in the laboratory.

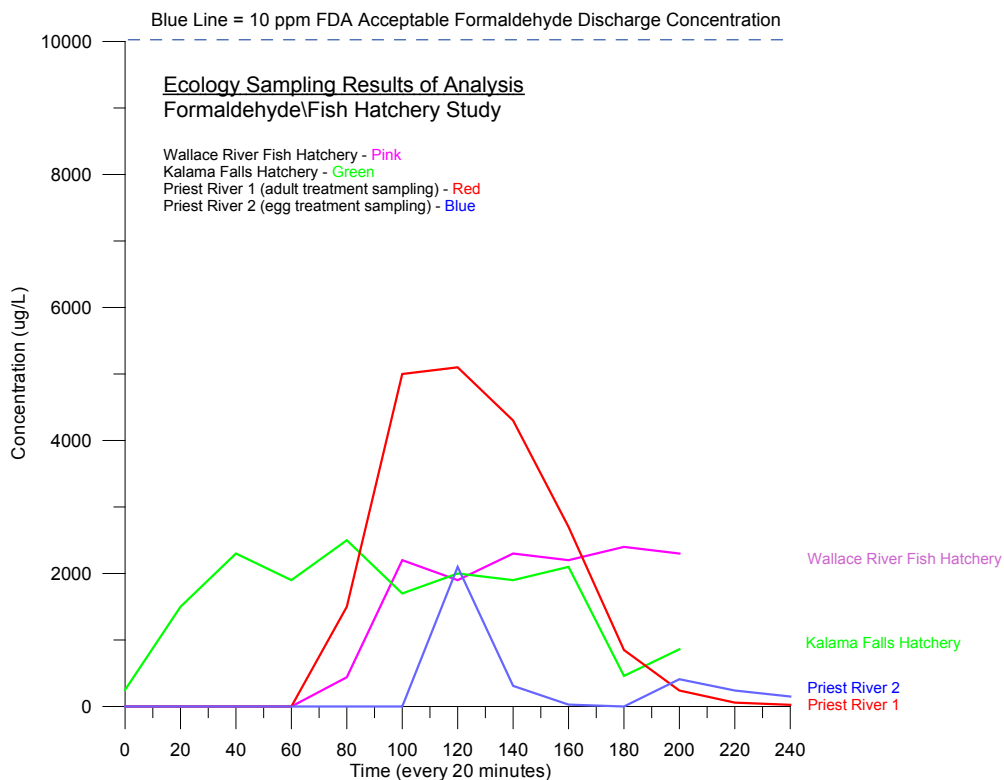
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Table 4.

Summary of Ecology's Field Measurement Data

Hatchery Name	Measurement Location	Temp (deg C)	Temp (deg F)	pH	Specific Conductance (u S/cm)	Dissolved Oxygen (ppm)	Ammonia Test Strip (ppm)	Chlorine (ppm)	Formaldehyde Test Strip (ppm)
Cowlitz Salmon Hatchery	Influent	12.29	54.122	7.62	49.1	10.51	ND	ND	ND
	Effluent	12.17	53.906	7.55	49.6	11.04	ND	ND	ND
	Receiving Water	11.94	53.492	7.5	49.2	10.49	ND	0.07	ND
Hoodsport Hatchery	Influent	6.94	44.492	7.6	72.2	12.13	ND	0.03	ND
	Effluent	6.83	44.294	7.43	72.2	11.52	ND	0.015	10
	Receiving Water	6.81	44.258	7.62	83.5	12.18	ND	0.02	0-10
Kalama Falls Hatchery	Influent	11.94	53.492	7.53	60.5	11.1	ND	0.01	ND
	Effluent	12.38	54.284	7.76	61.4	10.42	ND	ND	ND
	Receiving Water	12.4	54.32	7.61	60.7	11.06	ND	0.09	ND
Priest Rapids Hatchery (Visit 1)	Influent	15.85	60.53	7.78	227.4	9.72	ND	ND	ND
	Effluent	14.5	58.1	7.87	142.8	10.11	ND	0.02	10
	Receiving Water	14.68	58.424	8	141.4	10.34	ND	ND	10
Priest Rapids Hatchery (Visit 2)	Influent	13.31	55.958	7.9	135.1	10.56	ND	0.07	0-10
	Effluent	10.88	51.584	8	138.4	11.01	ND	0.06	0-10
	Receiving Water	10.94	51.692	8.01	137.9	11.32	ND	0.04	ND
Wallace River Hatchery	Influent	13.2	55.76	7.3	30.1	NA	ND	ND	ND
	Effluent	13.16	55.688	7	33.1	NA	0.25	ND	10
	Receiving Water	12.89	55.202	7.19	27.3	NA	ND	0.06	ND

Figure 4: Concentrations of formaldehyde for hatchery effluents sampled at 20-minute intervals (Ecology).



Water Sampling and Testing for Formaldehyde at Northwest Fish Hatcheries

Figure 5: Concentrations of formaldehyde for hatchery effluent sampled at 30-minute intervals (Ecology).

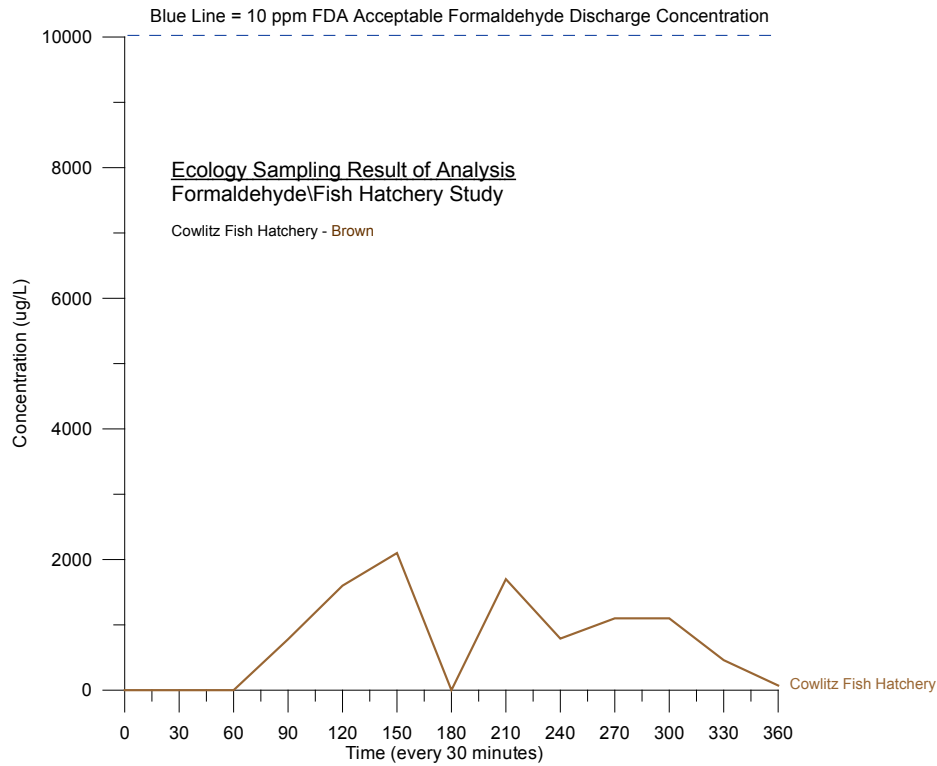
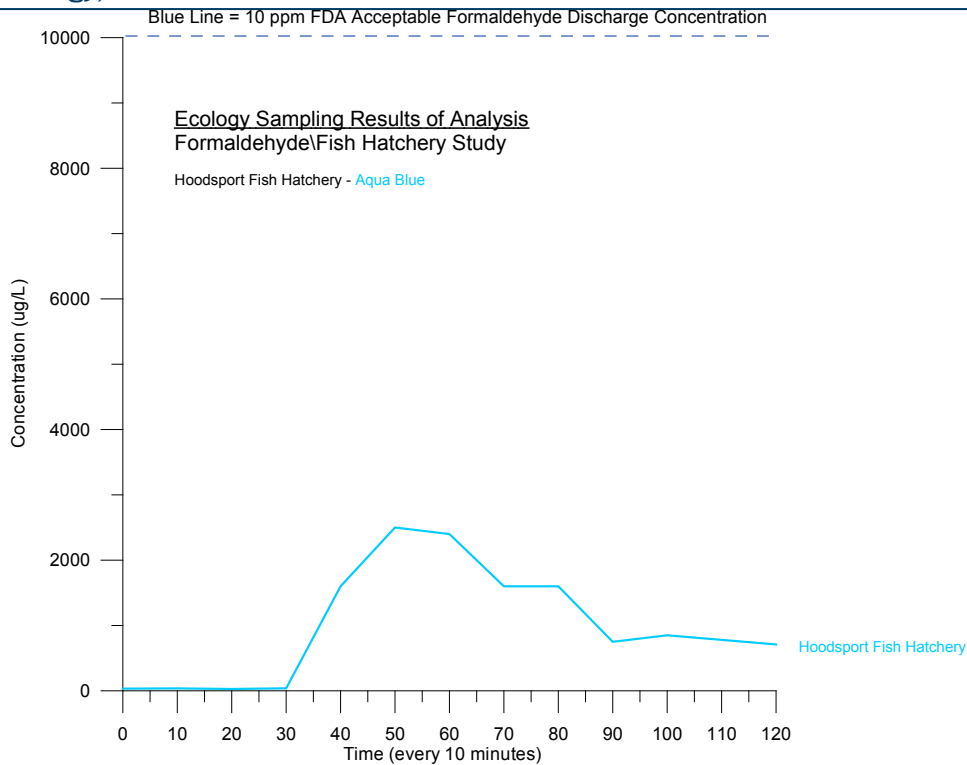


Figure 6: Concentrations of formaldehyde for hatchery effluent sampled at 10-minute intervals (Ecology).



Additional Information Collected

At the five WDFW hatcheries sampled, both adults and eggs were treated using Parasite-S from Western Chemical. Among the hatcheries, adult treatments lasted 1–2 hours, while egg treatments lasted 15 minutes. Differences in treatment types among the hatcheries (adults, eggs, or adults & eggs) during the time of sampling were noted. At the Kalama Falls and Wallace River Hatcheries, both adults and eggs were on station and treated during sampling. At the Cowlitz Hatchery, only adults were being treated during sampling. During the first Priest Rapids Hatchery sampling event, both adults and eggs were on station, but only adults were treated during the time of sampling. During the second Priest Rapids Hatchery sampling event, only eggs were on station and treated. The Hoodspport Hatchery typically only treats eggs, unless adult treatments become necessary. During the sampling the typical formalin concentration used for adults was 1:6000 (~166 ppm), while the concentration used for eggs was 1:600 (~1666 ppm).

The physical layout of each facility was unique to each hatchery. At the Wallace River Hatchery, adult and egg treatment effluents had separate discharge points. At the Cowlitz Hatchery, adult and egg treatment effluents had a single common discharge point. At the Priest Rapids Hatchery, hatchery effluent was mixed with river water in a long concrete canal structure before being discharged into Jackson Creek, which eventually empties into the Columbia River. At the Hoodspport Hatchery, sampling was timed to coincide with a low/ebb tide to minimize the influence of saltwater at the receiving water site.

CONCLUSION

In all of the hatcheries sampled for this study, the maximum concentration of formaldehyde measured did not exceed the FDA and EPA Region 10 level of concern of 10 ppm.

Based on data collected at the hatcheries that participated in this study, as well as the available toxicological data for threatened and endangered salmonids and an EPA risk assessment for formalin in Washington hatcheries, the EPA believes that current levels of formalin use are generally protective of aquatic life and ESA listed salmonids in Pacific Northwest waters.

TECHNICAL NOTES/RECOMMENDATIONS

EPA should consider repeat sampling and analysis at the Little White Salmon National Fish Hatchery to clarify the location of the permitted outfall in relation to where the effluent containing formaldehyde was being discharged. In addition, because the pump used to administer formalin failed during a power disruption, there is uncertainty about the highest concentration of formaldehyde in the effluent. Repeat sampling and analysis would result in more complete data for this site.

EPA should also consider repeat sampling and analysis at the Leavenworth National Fish Hatchery to help confirm the question of water temperature and how it relates to the concentration of formalin being administered to fish being treated at this facility. In addition, due to ease of access, this facility would be a good candidate for testing the concentration of formaldehyde downstream. An automated sampler could be used to collect discrete samples at the downstream location to provide data on the highest concentration present in the Wenatchee River downstream from the Leavenworth National Fish Hatchery outfall.

EPA should consider adjusting the analytical method parameters to better reflect the expected concentrations. At the same time the quality assurance could be modernized by adding a surrogate to monitor derivatization and extraction efficiency.

Appendices



PARASITE-S

Formalin (aqueous formaldehyde solution)

For control of External Protozoa and Monogenetic Trematodes on all Finfish and External Protozoans on Penaeid Shrimp; and for control of Fungi on all Finfish eggs.

DESCRIPTION

PARASITE-S is the aqueous solution of formaldehyde gas (this is equivalent to formalin 37% or 37 grams of formaldehyde in 100 mL of solution). U.S.P. grade PARASITE-S contains not less than 37% (by weight) of formaldehyde gas per weight of water and 6 to 14% methanol. In solution, formaldehyde is present chiefly as $\text{HO}(\text{CH}_2\text{O})\text{H}$. Its molecular weight is 30.93. PARASITE-S is readily miscible with water, methanol, and ethanol and is slightly soluble in ether. It is a clear, colorless liquid (Heyden Newport Chemical Corporation, 1961).

FISH AND SHRIMP TOXICITY STUDIES

The toxicity of PARASITE-S was measured by standard methods in laboratory bioassays with rainbow trout, Atlantic salmon, lake trout, black bullhead, channel catfish, green sunfish, bluegill, smallmouth bass, largemouth bass and striped bass. The 3,6,24 and 96-hour LC_{50} (lethal concentration for 50% of the animals) values for trout range from 1,230 to 100 $\mu\text{L/L}$ (455 to 37 ppm formaldehyde); for catfish, from 495 to 65.8 $\mu\text{L/L}$ (183 to 24 ppm formaldehyde); for bluegill, from 2,290 to 100 $\mu\text{L/L}$ (847 to 37 ppm formaldehyde); for largemouth bass, the values for 6 to 96-hour LC_{50} range from 1,030 to 143 $\mu\text{L/L}$ (381 to 53 ppm formaldehyde) (Bill et al. 1977) and for striped bass the values for 6 to 96-hour LC_{50} range from 940 to 30 $\mu\text{L/L}$ (347 to 11 ppm formaldehyde) (Bills, Marking & Howe-1993). The 24, 48, 72, and 96-hour LC_{50} values for penaeid shrimp range from 712 to 235 $\mu\text{L/L}$ (ppm) (Johnson, 1974 and Williams, 1980).

INDICATIONS FOR USE:

- Parasiticide for Finfish:** for the control of external protozoa (*Chilodonella* spp., *Ichthyophthirius* spp., *Epistylis* spp., *Ichthyophthirius* spp., *Ambiphris* spp. and *Trichodinella* spp.), and the monogenetic trematode parasites (*Cleaveliscus* spp., *Dactylogyrus* spp., and *Gyrodactylus* spp.).
- Parasiticide for Penaeid Shrimp:** for the control of external protozoan parasites (*Bodo* spp., *Epistylis* spp., and *Zoothamnium* spp.).
- Fungicide for Finfish Eggs:** for the control of fungi of the family Saprolegniaceae.

DIRECTIONS FOR USE:

1. Parasiticide for Finfish

Concentrations of Formalin

Aquatic species	Administer in Tanks & Raceways for up to 1 hr ($\mu\text{L/L}$)*	Administer in Earthen Ponds Indefinitely ($\mu\text{L/L}$)*
Salmon & Trout above 50°F below 50°F	up to 170 up to 250	15-25*** 15-25***
All other finfish	up to 250	15-25***

* Microliter per liter ($\mu\text{L/L}$) = parts per million (ppm).

** Use the lower concentration when ponds, tanks or raceways are heavily loaded with phytoplankton, or fish, to avoid oxygen depletion due to the biological oxygen demand created by decay of dead phytoplankton. Alternatively, a higher concentration might be used if dissolved oxygen is strictly monitored.

*** Although the indicated concentrations are considered safe for cold and warm water finfish, a small number of each lot or pond to be treated should always be used to check for any unusual sensitivity to formalin before proceeding.

2. Parasiticide for Penaeid Shrimp

Concentrations of Formalin

Aquatic species	Administer in Tanks & Raceways for up to 4 hours ($\mu\text{L/L}$)*	Administer in Earthen Ponds Indefinitely ($\mu\text{L/L}$)*
Shrimp	50 to 100**	25***

* Microliter per liter ($\mu\text{L/L}$) = parts per million (ppm).

** Treat for up to 4 hours daily. Treatment may be repeated daily until parasite control is achieved. Use the lower concentration when ponds, tanks or raceways are heavily loaded with phytoplankton, or shrimp, to avoid oxygen depletion due to the biological oxygen demand created by decay of dead phytoplankton. Alternatively, a higher concentration might be used if dissolved oxygen is strictly monitored.

*** Treatment may be repeated in 5 to 10 days, if needed

3. Fungicide for Finfish Eggs

Concentrations of Formalin

Aquatic species	Administer in Hatchery Systems ($\mu\text{L/L}$)*
Eggs of all finfish except Acipenseriformes	1000-2000 for 15 minutes**
Eggs of Acipenseriformes	up to 1500 for 15 minutes**

* Microliter per liter ($\mu\text{L/L}$) = parts per million (ppm).

** Apply in constant flow water supply of incubating facilities. A preliminary bioassay should be conducted on a small subsample of fish eggs to determine sensitivity before treating an entire group. This is necessary for all species because egg sensitivity can vary with species or strain and the unique conditions at each facility.

METHODS OF APPLICATION

APPLICATION TO TANKS AND RACEWAYS - Turn off water supply, provide aeration, apply appropriate amount of PARASITE-S, and thoroughly dilute and mix to assure equal distribution of PARASITE-S. Treat for up to 1 hour for fish and up to 4 hours for penaeid shrimp, then drain the solution and refill the tank with fresh, well-aerated water. While tank is under treatment, adequate oxygen must be present to maintain the fish or shrimp. If needed, aeration should be provided to prevent oxygen depletion. Treatments may be repeated daily until parasite control is achieved.

APPLICATION TO PONDS - Apply greatly diluted PARASITE-S to the pond evenly using a pump, sprayer, boat bailer, or other suitable device to assure even distribution. Allow PARASITE-S to dissipate naturally. Single treatments usually control most parasites, but may be repeated in 5 to 10 days if needed. Treatments for *Ichthyophthirius* should be made at 2-day intervals until control is achieved.

APPLICATION TO EGG INCUBATORS - Apply PARASITE-S into a constant water supply flowing around the eggs. A drip or pressure system should be used and timed. Apply PARASITE-S under the surface of the water flow.

WITHDRAWAL TIME

Zero days.

WARNING

Striped bass have been demonstrated to be hypersensitive to formalin; lethal toxicity has been noted to occur at levels approximately 2-3 times the recommended therapeutic concentration.

ANGER POISON

USER SAFETY WARNINGS

Exposure to high concentrations of formaldehyde vapor causes severe respiratory irritation which can be life-threatening. Lower vapor levels can cause irritation to the eyes, respiratory tract, and skin. Swallowing formaldehyde can be life-threatening. Formaldehyde is an irritant when splashed on skin or into the eyes. It can cause severe eye damage, even blindness.

Keep out of reach of children.

Use only with adequate ventilation.

Keep container tightly closed when not in use.

May aggravate a pre-existing asthmatic condition and allergic rhinitis.

Moderate fire and explosion hazard exists when exposed to heat or flame.

Contains methanol - cannot be made non-poisonous. Prolonged exposure to methanol has been associated with reproduction disorders.

Potential Cancer Hazard: Formaldehyde vapor may be carcinogenic if inhaled. Use applicable safety protection. (Note: This drug, used as labeled, does not cause formaldehyde tissue residues in fish).

Employers: Refer to Occupational Safety and Health Administration (OSHA) regulation 29 CFR 1910.1048 for human safety guidance that may be applicable to your specific operation. OSHA's "action level" concentration for airborne formaldehyde is 0.5 part per million (ppm), calculated as an 8 hour time-weighted average (TWA). Use respiratory, skin, and eye protection when needed (refer to OSHA's regulation 29 CFR 1910.1048). OSHA's airborne exposure limits (without use of a respirator) for formaldehyde shall not exceed 1) 0.75 part per million (ppm) as an 8-hour, time-weighted average (TWA) or 2) 2 parts per million (ppm) as a 15-minute, short term exposure limit (STEL). **NOTE:** The odor of formaldehyde in the air can generally be detected at about 0.5 to 0.8 ppm (range about 0.05 to 1 ppm).

USER EXPOSURE EMERGENCY AID

INHALATION (Breathing): Get medical aid immediately. Remove victim from exposure wearing protective clothing and respiratory protection appropriate to the type and degree of contamination. Move victim to fresh air immediately. If breathing is difficult, give oxygen. DO NOT use mouth-to-mouth respiration. If breathing has ceased, induce artificial respiration with the aid of a pocket mask equipped with a one-way valve or other proper respiratory medical device.

INGESTION (Swallowing): DO NOT induce vomiting. If the person is conscious, dilute, inactivate, or absorb the formaldehyde by giving milk, activated charcoal, or water. Get medical help immediately. If vomiting occurs, keep head lower than hips.

EYE CONTACT: Immediately flush eye(s) with large amounts of water for at least 15 minutes, lifting the lower and upper eyelids occasionally, until no evidence of chemical remains. Seek medical attention immediately. DO NOT allow victim to rub eyes or keep eyes closed for burns to eyes may have a delayed effect.

SKIN CONTACT: Remove contaminated clothing (including shoes) immediately. Wash affected area of body with soap and large amounts of water until no evidence of chemical remains (at least 15 minutes). If there are chemical burns, or appreciable eye or respiratory irritation, get medical help immediately.

PRECAUTIONS

Store PARASITE-S indoors away from direct sunlight, heat, sparks, and open flames, and ventilate storage area. Do not subject PARASITE-S to temperatures below 40°F (4.4°C). PARASITE-S subjected to temperatures below 40°F causes the formation of paraformaldehyde, a substance which is toxic to fish. Paraformaldehyde can be recognized as a white precipitate at the bottom or on the walls of the container.

Tolerance to PARASITE-S may vary with strain and species of fish, eggs and shrimp. While the indicated concentrations are considered safe for the indicated use, a small number of each lot to be treated should be used to check for any unusual sensitivity to PARASITE-S before proceeding.

Under some conditions, fish or penaeid shrimp may be stressed by normal treatment concentrations. Heavily parasitized or diseased fish or penaeid shrimp often have a greatly reduced tolerance to PARASITE-S. Such animals do not tolerate the normal tank treatment regimen the first time they are treated. Therefore, time and dosage may need to be reduced. If they show evidence of distress (by piping at the surface), the solution should be removed and replaced with fresh, well aerated water. Careful observations should always be made throughout the treatment period whenever tank or raceway treatments are made. Treatment should never exceed 1 hour for fish or 4 hours for penaeid shrimp (even if they show no sign of distress), nor should it exceed 15 minutes for fish eggs.

Do not apply PARASITE-S to fish ponds, tanks or raceways with water warmer than 80°F (27°C) when a heavy bloom of phytoplankton is present, or when the concentration of dissolved oxygen is less than 5 mg/L (ppm). Do not apply to penaeid shrimp ponds when the concentration of the dissolved oxygen is less than 3 to 4 mg/L (ppm). PARASITE-S may kill phytoplankton and can cause depletion of dissolved oxygen. If an oxygen depletion occurs, add fresh, well-aerated water to dilute the solution and to provide oxygen.

Because formalin may harm a biofilter, biofilters should be bypassed during treatment, and the system should be flushed and replaced with untreated water before reconnecting the biofilter.

Do not use PARASITE-S in a tank, pond or raceway in which methylene blue, or other dyes which are absorbed, have been recently used.

ENVIRONMENTAL PRECAUTIONS

Do not discharge the contents of fish treatment tanks into natural streams or ponds without thorough dilution (greater than or equal to 10X). Do not discharge the contents of egg treatment tanks without a 100X dilution. This will avoid damage to PARASITE-S sensitive phytoplankton, zooplankton, and fish populations and avoid depletion of dissolved oxygen.

Formaldehyde is identified by the U.S. Environmental Protection Agency (EPA) as a toxic pollutant and hazardous substance and is required by regulation (40 CFR, Part 122) to be identified as a discharge for NPDES permits for aquatic animal production facilities, aquaculture projects and other facilities. Formaldehyde is subject to SARA Title III, Section 313 reporting.

Use, storage, and disposal of this product must be handled in accordance with applicable local, state and Federal laws.

STORAGE

Recommended storage temperature 59°F (15°C). DO NOT EXPOSE TO DIRECT SUNLIGHT. Store PARASITE-S indoors away from direct sunlight, heat, spark, and open flame, and ventilate storage area. Do not subject PARASITE-S to temperatures below 40°F (4.4°C).



**Western
Chemical, Inc.**

Manufactured for:
Western Chemical Inc.
1269 Latimore Road, Ferndale, WA 98248
(360) 384-5898
ver. 020513

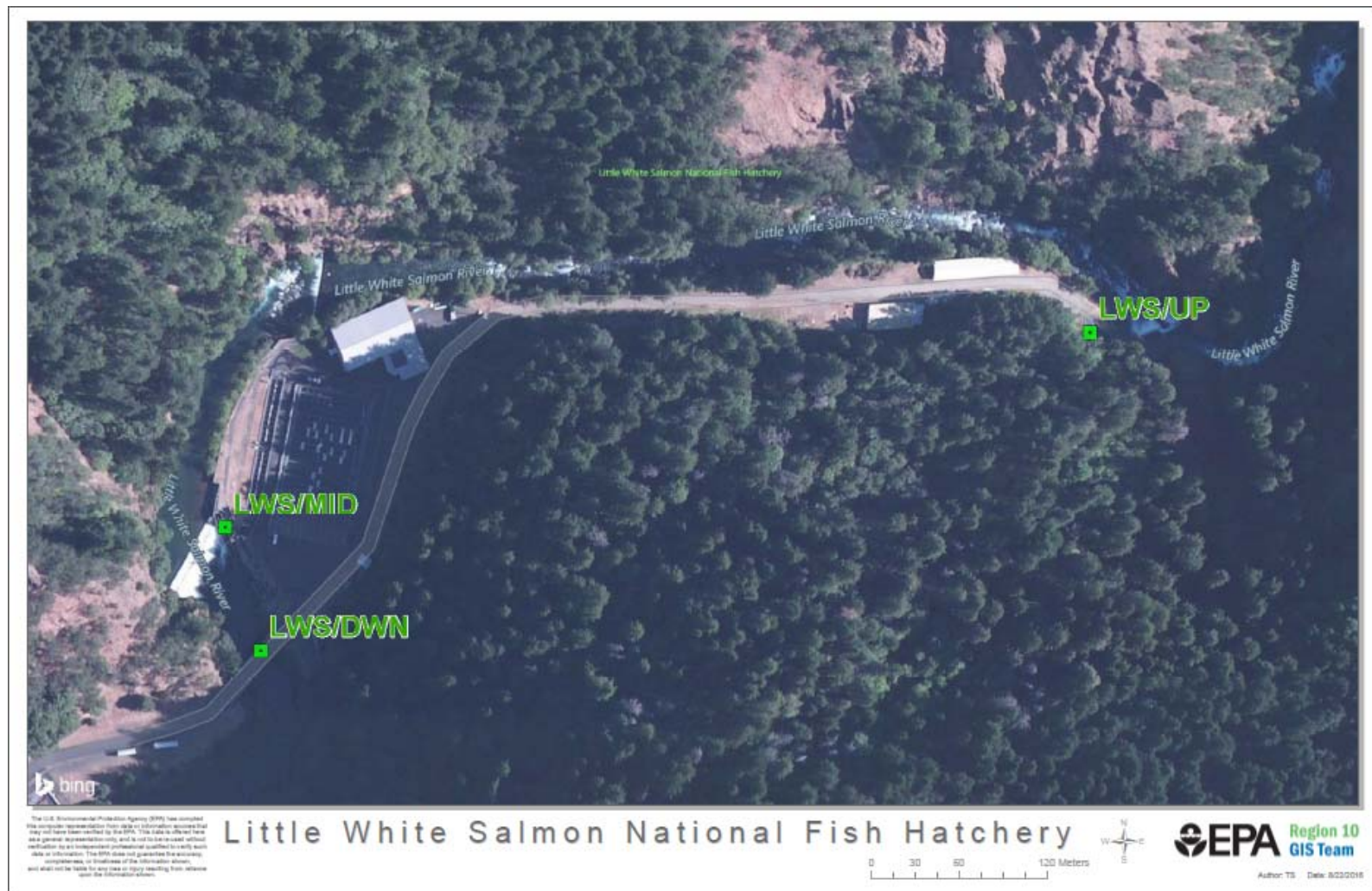
NADA 140-989, Approved by FDA

1. Little White Salmon National Fish Hatchery

56961 SR 14, Cook, WA 98605

Bob Turik

(509) 538-2755



2. Carson National Fish Hatchery

14041 Wind River Hwy. Carson, WA 98610

Larry Zeigenfuss (509) 427-5905



3. Dworshak National Fish Hatchery

4147 Ahsahka Rd. Ahsahka, ID 83520

Mark Drobish

(208) 512-9151



4. Clearwater Fish Hatchery

118 Hatchery Rd. Ahsahka, ID 83520

Tony Folsom

(208) 476-3331



5. Leavenworth National Fish Hatchery

12790 Fish Hatchery Rd. Leavenworth, WA 98826 Chris Foster (509) 548-7641



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Leavenworth National Hatchery



Author: TG Date: 8/22/2016

Little White Salmon National Fish Hatchery

July 11, 2016

Jed Januch

1 – LWSNFH Influent water location



2 – Automatic sampler deployed at LWSNFH influent location



Little White Salmon National Fish Hatchery

July 11, 2016

Jed Januch

3- Automatic sampler deployed at LWSNFH downstream



4- Automatic sampler deployed at LWSNFH outfall



Little White Salmon National Fish Hatchery

July 11, 2016

Jed Januch and Brent Richmond

5 – Brent Richmond measuring water quality parameters



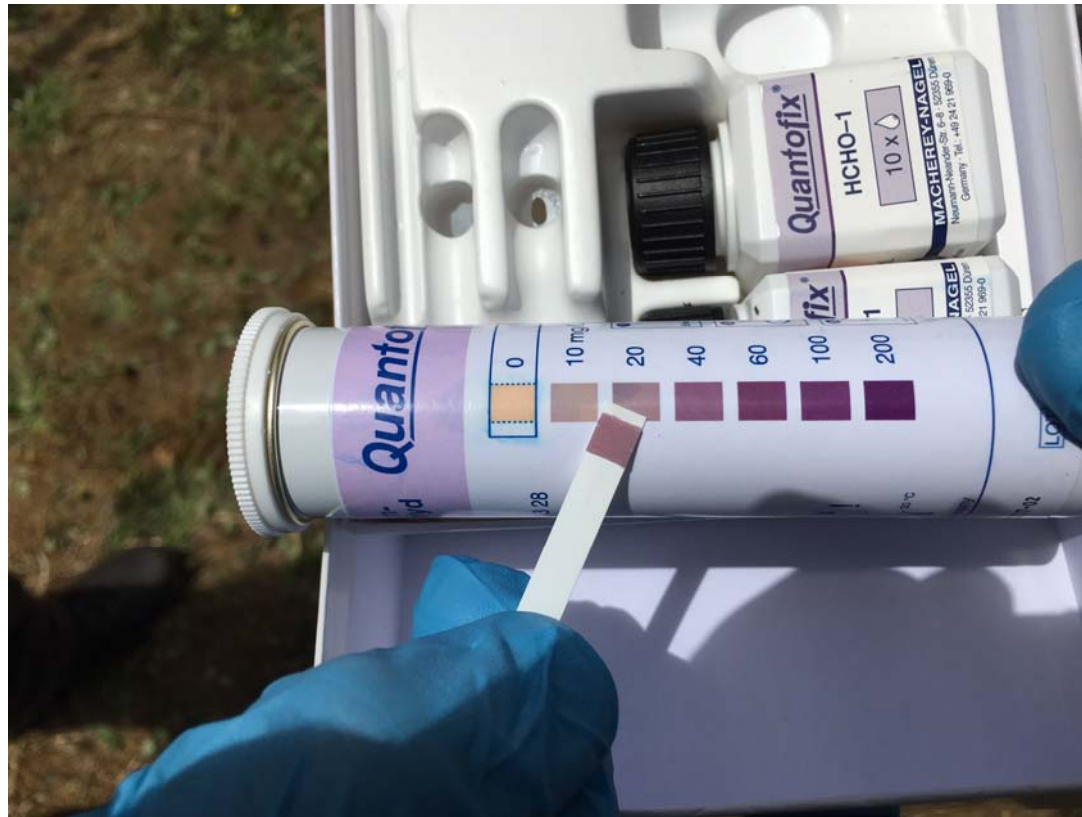
6 – Screening for formaldehyde with test kit downstream



Little White Salmon National Fish Hatchery

July 11, 2016, Jed Januch and Brent Richmond

7 – Screening effluent water with a formaldehyde test kit



Carson National Fish Hatchery

July 13, 2016

Jed Januch and Brent Richmond

8- Automatic sampler deployed at CNFH influent location



9- Automatic sampler deployed at CNFH outfall location



Carson National Fish Hatchery

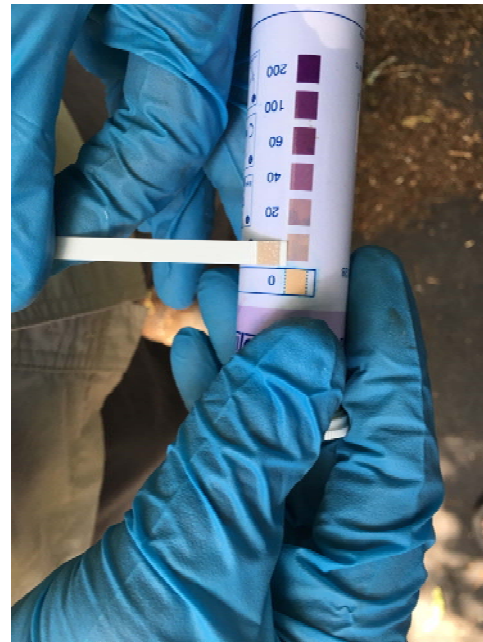
July 13, 2016

Jed Januch and Brent Richmond

10- Automatic sampler deployed at CNFH downstream location



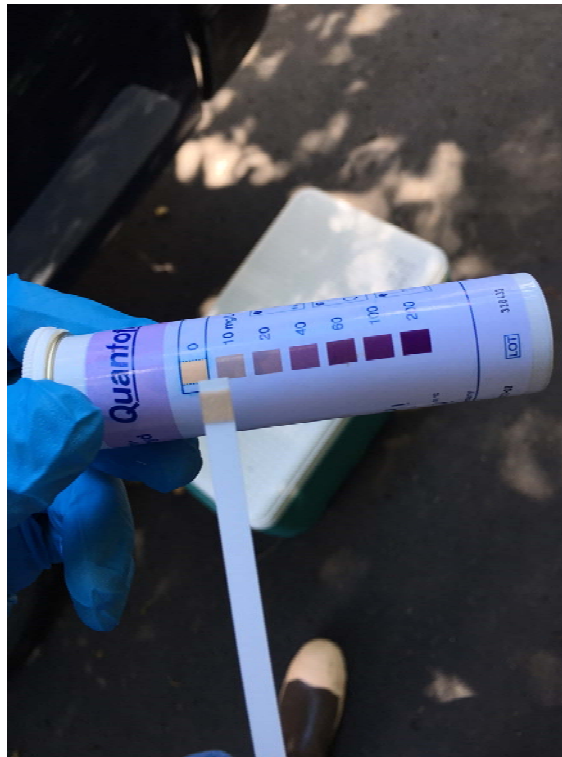
11- Screening for formaldehyde with test kit downstream



Carson National Fish Hatchery

July 13, 2016, Jed Januch and Brent Richmond

12 – Screening effluent water with a formaldehyde test kit



Dworshak National Fish Hatchery

July 18, 2016

Jed Januch and Lilian Herger

13- DNFH influent location



14- Automatic sampler deployed at CDNFH effluent location



Dworshak National Fish Hatchery

July 18, 2016

Jed Januch and Lilian Herger

15- Grab sample outfall location



16- Automatic sampler deployed at DNFH downstream location



Clearwater Fish Hatchery

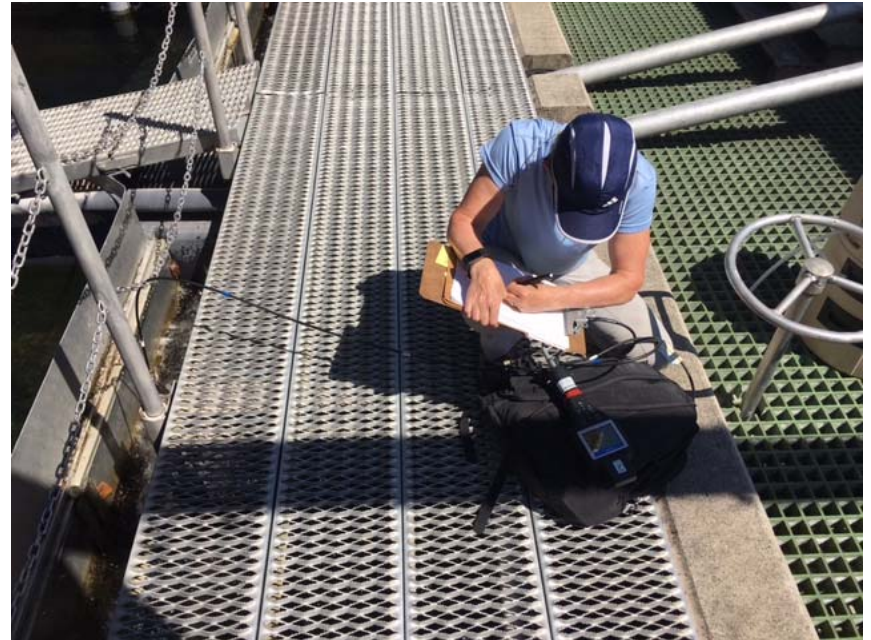
July 20, 2016

Jed Januch and Lilian Herger

17- Automatic sampler deployed at CIWFH influent location



18 – Lilian Herger measuring water quality parameters



Clearwater Fish Hatchery

July 20, 2016

Jed Januch and Lilian Herger

19- Chlorine test kit – effluent location



20- Automatic sampler deployed at CIWFH downstream location



Leavenworth National Fish Hatchery

August 17, 2016

Jed Januch and Raymond Wu

21- Automatic sampler deployed at LNFH influent location



22- Automatic sampler deployed at LNFH effluent location

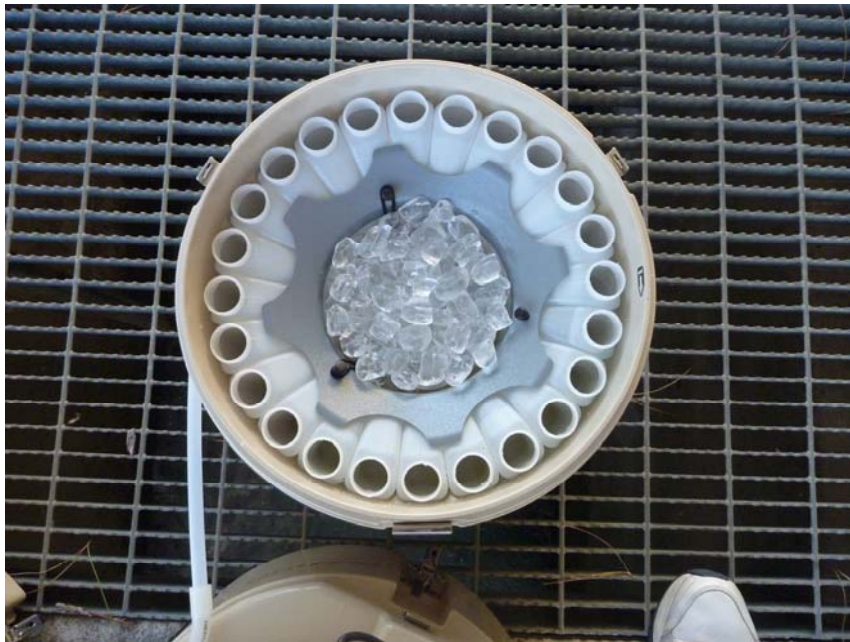


Leavenworth National Fish Hatchery

August 17, 2016

Jed Januch and Raymond Wu

23- Automatic sampler bottle configuration and wet ice



24- Automatic sampler deployed at LNFH downstream location



Leavenworth National Fish Hatchery

August 17, 2016

Jed Januch and Raymond Wu

23- Automatic sampler bottle configuration and wet ice



QUALITY ASSURANCE PROJECT PLAN (QAPP)

FOR

Water Sampling and Testing for Formaldehyde at Northwest Aquaculture Facilities

Date: June 2016

Prepared by:

Office of Environmental Review and Assessment
U.S. EPA Region 10

PROJECT MANAGEMENT

Title and Approval Page

Title: Water Sampling and Testing for Formaldehyde at Northwest Aquaculture Facilities

Approvals:

Filippini, Mark Digitally signed by Filippini,
Mark
Date: 2016.06.06 16:01:20 -07'00'

Date: _____

Mark Filippini, Manager
Environmental Services Unit
Office of Environmental Review and Assessment
U.S. EPA Region 10



Date: 6/8/16

Michael Lidgard, Manager
NPDES Permits Unit
Office of Water and Watersheds
U.S. EPA Region 10



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DN: c=US, o=U.S. Government, ou=USEPA,
ou=Staff, cn=JENNIFER CRAWFORD,
dnQualifier=0000024857
Date: 2016.06.07 09:15:28 -07'00'

Date: _____

Donald Brown, Regional Quality Assurance Manager
Office of Environmental Review and Assessment
U.S. EPA Region 10

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Acronyms

COC	Chain of Custody
DMP	Data Management Plan
DQO	Data Quality Objective
EPA	U.S. Environmental Protection Agency
IDOC	Initial Demonstration of Capability
GPS	Global Positioning System
LCS	Laboratory Control Sample
MEL	EPA Region 10 Manchester Environmental Laboratory
MS	matrix spike
MSD	matrix spike duplicate
NELAC	National Environmental Laboratory Accreditation Conference
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	relative percent difference
RSCC	Regional Sample Control Coordinator
SOP	Standard Operating Procedures
TNI	The NELAC Institute

Introduction

This Quality Assurance Project Plan (QAPP) was developed in accordance with the EPA QA guidance (EPA 240-R-02-009)⁽¹⁾ and data quality objective guidance (EPA 240-B-06-001)⁽²⁾. The sections herein describe the necessary planning elements for the EPA to conduct sampling and analytical activities at representative aquaculture facilities within the Region that utilize formaldehyde as a disease treatment for fish and eggs.

Distribution List

The list of project personnel and their respective contact information is provided in Table 1. The documentation generated in support of this study and its distribution is also indicated.

Table 1. Project Document Distribution List

Name Title / Project Role Organization-Affiliation	Address Phone Email	Document Distribution
Catherine Gockel, Permit Writer Principal Investigator NPDES Permits Unit Office of Water and Watersheds	1200 Sixth Ave., Suite 900, OWW-191 Seattle, WA 98101, (206) 553-0325 Gockel.Catherine@epa.gov	QAPP (e-copy) – maintains official signed e-copy in project file Data (QC reports & e-copy)
Dirk Helder, Permit Writer (ID) NPDES Permits Unit Office of Water and Watersheds	950 W Bannock, Suite 900 Boise, ID 83702, (208)378-5749 Helder.Dirk@epa.gov	QAPP (e-copy)
Jed Januch, Environmental Protection Specialist Principal Investigator; Scribe Project Manager Environmental Services Unit Office of Environmental Review and Assessment	7411 Beach Drive East Port Orchard WA 98366, (360) 871-8731 Januch.Jed@epa.gov	QAPP (e-copy) Data (QC reports & e-copy)
Donald M. Brown Regional QA Manager Office of Environmental Review and Assessment	1200 Sixth Ave., Suite 900, OERA-140 Seattle, WA 98101, (206) 553-0717 Brown.DonaldM@epa.gov	QAPP (e-copy)
Jennifer Crawford, Chemist Regional Sample Control Coordinator Office of Environmental Review and Assessment	1200 Sixth Ave., Suite 900, OERA-140 Seattle, WA 98101, (206) 553-6261 Crawford.Jennifer@epa.gov	QAPP (e-copy)

Don Matheny, Chemist QA; Alternate RSCC Office of Environmental Review and Assessment	1200 Sixth Ave., Suite 900, OERA-140 Seattle, WA 98101, (206) 553-2599 Matheny.Don@epa.gov	QAPP (e-copy)
Gerald Dodo Supervisory Chemist EPA Region 10 Laboratory (MEL)	7411 Beach Drive East Port Orchard WA 98366, (360) 871-8728 Dodo.Gerald@epa.gov	QAPP (e-copy)

Project/Task Organization

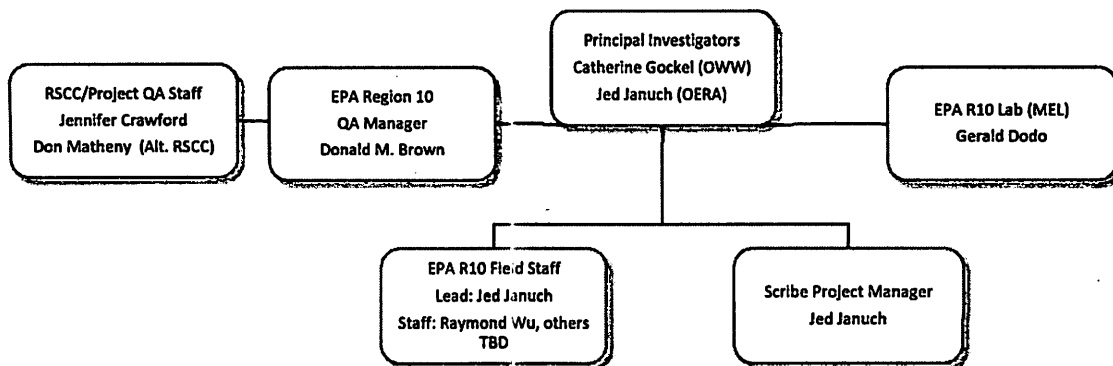
The project organization and lines of authority for this sampling event are provided in Figure 1 with the roles and responsibilities shown below in Table 2. The information produced by this project is limited to a summary report describing field observations, measurements, laboratory analytical data and interpretation for future permitting. Data obtained by this field work will be used to ascertain the concentration of formaldehyde in Pacific Northwest aquaculture effluent and receiving water. While evaluation of the final data is the responsibility of the OWW Principal Investigator, other project personnel may be consulted to provide a scientific perspective on its technical validity, usability and relevance.

Table 2. Roles & Responsibilities

Project Personnel	Responsibility	Authorities
Principal Investigator, Office of Water and Watersheds	Provides data interpretation from the perspective of a potential data user for the purpose of supporting NPDES permit decisions. Coordinates enrollment and study logistics with participating facilities.	OWW Programmatic Lead
Principal Investigator, Office of Environmental Review and Assessment	Develops QAPP, manages field operations, collects samples and performs direct field measurements. Coordinates logistics with participating facilities.	OERA Technical Lead
Regional QA Manager	Provides overall QA Program oversight. Delegates QAPP review/approval to EPA Project QA Staff.	Regional QA Program Authority
Regional Sample Control Coordinator (RSCC), Project QA Staff	Schedules EPA lab support services, coordinates sample shipments to labs, resolves issues with lab analyses, and consults on Scribe usage. Provides unique EPA Sample IDs and Regional Project Code. Reviews and approves QAPP. Reviews Scribe submissions for completeness.	Authorizes sample shipments to EPA R10 Lab, Delegated QAPP Approval

Scribe Project Manager	Data entry or upload into Scribe in accordance with the Region 10 DMP (EPA Region 10, 2014) and QAPP requirements. Coordinates with RSCC for sample shipment notification, prints sample labels, exports electronic COC records to lab. Finalizes and archives Scribe project file with lab data to Scribe.net.	Custodian of official Scribe Project File
Region 10 (MEL) Lab Chemistry Supervisor	Serves as the main contact between the laboratory and project personnel. Coordinates with lab team leaders on sample analysis, data review and reporting.	Authorizes acceptance of samples into MEL and the release of final reviewed data

Figure 1. Organization Chart



Problem Definition/Background

NPDES Aquaculture Permits

The U.S. Environmental Protection Agency (EPA) and the Washington State Department of Ecology (Ecology) issue permits under the National Pollutant Discharge Elimination System (NPDES) to establish conditions for the discharge of pollutants in wastewaters to waters of the United States. In order to ensure protection of water quality and human health, the NPDES permits place limits on the types and amounts of pollutants that can be discharged and places other conditions on such activity, such as monitoring and best management practices.

At 40 CFR §122.24, the U.S. Environmental Protection Agency (EPA) defines concentrated aquatic animal production (CAAP) facilities as point sources subject to the National Pollutant Discharge Elimination System (NPDES) permit program and further defines such a facility as a hatchery, fish farm, or other facility that contains, grows, or holds:

Cold water fish species or other cold water aquatic animals in ponds, raceways, or other similar structures which discharge at least thirty days per year, but does not include:

- a. *Facilities that produce less than 20,000 harvest weight pounds of aquatic animals per year, and*
- b. *Facilities that feed less than 5,000 pounds of food during the calendar month of maximum feeding.*

Warm water fish species or other warm water aquatic animals in ponds, raceways, or other similar structures which discharge at least 30 days per year, but does not include:

- a. *Closed ponds which discharge only during periods of excess runoff; or*
- b. *Facilities which produce less than 100,000 harvest weight pounds of aquatic animals per year.*

Cold water aquatic animals include, but are not limited to, the Salmonidae family of fish, e.g. trout and salmon. Warm water aquatic animals include, but are not limited to, the Ameiuride, Centrarchidae and Cyprinidae families of fish, e.g., respectively, catfish, sunfish and minnows.

EPA Region 10 is the NPDES permitting authority for CAAP facilities in Idaho^a. In Washington, EPA is the NPDES permitting authority for federal aquaculture facilities (i.e., National Fish Hatcheries), and for aquaculture facilities in Indian Country within the State of Washington^b. Ecology is the NPDES permitting authority for all other hatchery and aquaculture facilities^c.

Formalin

Formalin is a generic term that describes a solution of 37% formaldehyde gas dissolved in water. The Parasite-S formulation is administered as a treatment to control for external protozoa (*Chilodonella* spp., *Costia* spp., *Epistylis* spp., *Ichthyophthirius* spp., *Scyphidia* spp. and *Trichodina* spp.), and the monogenetic trematode parasites (*Cleidodiscus* spp., *Dactylogyrus* spp., and *Gyrodactylus* spp.) on all finfish. It is also used for the control of fungi of the family *Saprolegniaceae* on all finfish eggs (Western Chemical Label, no date)^d.

Formalin is administered to salmon and trout as a bath treatment for prolonged or short periods of time. The standard dosage recommended in the INAD #9013 Protocol to prevent or control fungus on fish and eggs is to administer formalin as a static-bath or flow-through treatment at 15 - 2000 µL/L (ppm) active

^a <https://yosemite.epa.gov/r10/water.nsf/npdes+permits/general+npdes+permits#Aquaculture>

^b <https://yosemite.epa.gov/r10/water.nsf/npdes+permits/general+npdes+permits#fedaquaculture>

^c http://www.ecy.wa.gov/Programs/wq/permits/fin_fish/index.html

^d <http://www.wchemical.com/products/fish-egg-treatments/parasite-s-formalin/parasite-s.html>

drug. Eggs are treated daily or every other day until hatch. Fish are treated every other day to weekly^e for 30 to 60 minutes, and then transferred to clean water. The formalin concentration is water temperature dependent and 50°F is the cutoff for the two treatment concentrations. Salmon and trout are treated up to 170 µL/L at water temperatures above 50°F and 250 µL/L at temperatures below 50°F. All other finfish are treated up to 250 µL/L regardless of temperature. Treatment is not recommended to exceed 1.0 hour.

In the finding of no significant impact for Parasite-S, the FDA requires a 10-fold dilution of finfish treatment water and a 100-fold dilution of finfish egg treatment water, which should lead to a discharge concentration of no more than 25 ppm.^f The FDA contended that additional in-stream dilution, infrequent use, and rapid degradation would render the discharged formalin below a level that causes significant environmental effects on aquatic animals (formaldehyde, the active ingredient in formalin, is oxidized in the aquatic environment into formic acid and ultimately into carbon dioxide and water; the estimated half-life of formaldehyde in water is approximately 36 hours). Directions for dilution of treatment water and additional environmental precautions are described on the labeling of the product. For product label information see:

http://www.wchemical.com/downloads/dl/file/id/45/parasite_s_package_insert.pdf.

As part of its Biological Evaluation in compliance with the Endangered Species Act^g, the EPA performed risk assessments to determine whether formalin use at EPA-permitted aquaculture facilities in Washington and Idaho have the potential to affect threatened or listed species or their critical habitat. These risk assessments used the best available science. The EPA's risk assessments likely resulted in unrealistically conservative assumptions, and did not account for in-stream dilution. Based on the available toxicological data for threatened and endangered salmonids, the EPA believes that the FDA's dilution requirement will be protective of aquatic life in Pacific Northwest waters.

However, calculating the maximum concentration of water-borne treatments in hatchery effluent can be challenging. Formalin presents a particularly complicated case because many facilities send their formalin-treated water to a holding tank, from which it is slowly metered out and mixed with hatchery water that does not contain formaldehyde. The EPA does not currently have the data inputs to calculate the formalin concentration in the effluent for individual facilities because we do not have information on holding tank size, flow and internal dilution rates, facility retention times, etc. for each treatment.

The EPA is undertaking this study to ascertain the concentration of formaldehyde in Pacific Northwest aquaculture effluent.

Based on many discussions with permittees, USFWS, Ecology, USGS, and the Northwest Indian Fisheries Commission, the EPA expects that this study will confirm that formalin use in Northwest aquaculture facilities does not present an ecological risk to listed species or the aquatic environment. If, however,

^e <http://www.fws.gov/fisheries/aadap/summaryHistoryFormalin.htm> accessed 8/12/2014

^f http://water.epa.gov/scitech/wastetech/guide/aquaculture/upload/2005_09_01_guide_aquaculture_EEBA_EEBA-Chapter-7.pdf accessed 12/5/2014

^g https://www3.epa.gov/region10/pdfs/permits/npdes/wa/WA_Hatchery_GP_WAG130000_BE.pdf

the study results suggest that formaldehyde estimated environmental concentrations are unacceptably high, the EPA will take steps to work with permittees to adjust their formalin use (e.g., treat a smaller subset of the hatchery at a time, provide more internal dilution prior to discharge, route formalin-treated water to an offline settling basin, or hold treated water for a longer period of time to allow for degradation). If the results of this field study demonstrate that formaldehyde estimated environmental concentrations are higher than allowed on the approved label or higher than acceptable levels to ESA listed species, formalin will be addressed in future NPDES permits.

Other Background Information/Literature Review

A study focused on measuring the formaldehyde concentration in effluent outfalls at freshwater aquaculture facilities in Nova Scotia was done by Environment Canada in 2011 and published in the Bulletin of Environmental Contamination and Toxicology in 2015. Formaldehyde was applied at an effective rate of 250 microliters per liter ($\mu\text{L/L}$) to a 10 meter diameter pond with a water temperature of 50° Fahrenheit (F). Samples were collected at the effluent outfall and at another location 100 meters downstream of the effluent outfall. Samples were collected with the aid of automated ISCO® samplers set to obtain 100 milliliters (ml) of water every 5 minutes over a 3-hour period. The ISCO® samples were composited in a glass container through Teflon tubing. Additional grab samples were also collected at three other aquaculture facilities at different times, with one effluent sample collected from an outfall 72 hours after treatment. The results of the study revealed the formaldehyde concentrations in samples collected at the outfall location during treatments ranged from 1.8 to 7.1 milligrams per liter (mg/L). The analysis of the sample collected 72 hours after treatment revealed 0.2 mg/L formaldehyde. The analysis of samples collected at the downstream locations varied in formaldehyde concentration of 0.9 to 1.9 mg/L. The study concluded that formaldehyde concentration downstream of land based aquaculture facilities could cause an adverse chronic impact to aquatic organisms.⁽⁴⁾

Project /Task Description and Schedule

The proposed study will monitor the concentration of formaldehyde discharged from fish hatchery outfalls during treatment of fish and eggs during the summer and fall of 2016. The following list of samples and field analyses are required:

Samples collected for laboratory analyses may be analyzed for:

- Formaldehyde

Sample analyses and field measurements may be conducted in the field for:

- Flow
- Temperature
- Chlorine (Total)
- Ammonia
- Dissolved Oxygen (DO)
- pH
- Formaldehyde Screening (test kits)

See Table 6 for the specific analytical methods, QA/QC, and container and preservation requirements applicable to this study. Table 3 includes a schedule for conducting tasks related to this project. It is intended as a guideline only as it is possible that unforeseen circumstances and conditions will require adjustment to some or all of the following dates and timeframes proposed. Site visits will be planned during a timeframe when facilities are performing formaldehyde treatments and able to accommodate EPA access to facilities. EPA will schedule visits with aquaculture facility managers prior to traveling to the site. In the event that changes are required to travel schedules, field staff will consult their manager on contingencies.

Table 3. Schedule of Tasks

Activities	Due Dates
QAPP review/approval	June 10, 2016
Sample Collection	July 1 through November 1, 2016
Analysis/data Review Completion	Analysis within the technical holding times required by 40 CFR Part 136. Generally, reviewed data will be provided within 8 weeks of sampling.
Report Preparation	Initial draft within 90 days of receiving the final reviewed analytical results.

Site Sampling Constraints

Time of sample collection is coordinated with the facility manager. Problems with coordinating the timing of facility treatment with the sample collection are not likely but are possible due to daily facility conditions. For example, EPA crew is available to do the sampling but the facility does not need to treat (discharge formaldehyde) on the assigned day. This could result in delay that would either require more travel time/funds or cause the site to not be sampled.

Quality Objectives and Criteria for Measurement Data

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. EPA uses a seven-step process for establishing DQOs and developing QAPPs to help ensure that data collected during a study will be adequate to support reliable decision-making. For this project the DQOs shown in Attachment 4 and their subsequent data quality indicators and acceptance criteria were developed.

Data will determine if the concentration of formaldehyde being discharged from Northwest aquaculture facility outfalls is significant enough to impact aquatic organisms downstream. The LC₅₀ for the most sensitive non-target aquatic species, such as certain crustaceans, is >1.0 mg/L formaldehyde based on 96-hour static LC₅₀ tests.⁽³⁾ The data should be of sufficient quality to aid in making NPDES permit decisions. The following is a compilation of the major data quality indicators used to evaluate data quality for this project.

Precision is the measure of agreement among repeated measurements of the same property under identical or substantially similar conditions. Approximately 5-10% (1 per facility with <20 field samples) of the field measurements will be made in duplicate by collecting co-located field duplicate samples (or by collecting well mixed field split samples from a butter churn type mixer) and analyzing both samples separately. In addition, approximately 5% of the samples will be analyzed in duplicate (analytical duplicate). For this project, precision will be measured by the relative percent difference of duplicates. The required precision is specified in Table 6. The calculation for relative percent difference (RPD) is given as follows:

$$RPD = \frac{(R_1 - R_2) \times 100\%}{(R_1 + R_2) / 2}$$

RPD = Relative percent difference
 R_1 = Matrix spike result
 R_2 = Matrix spike duplicate result

Accuracy is a measure of the overall agreement of a measurement to a known value; includes a combination of random error (precision) and systematic error (bias) components of both sampling and analytical operations. For this project accuracy will be evaluated based on the use of laboratory control samples (LCS) and matrix spike(s) recoveries. The required accuracy criteria are specified in Table 6. The calculation for percent recovery of matrix spikes is given as:

$$\% Rec = \frac{(Sm - N)}{Sa} \times 100\%$$

% Rec = Percent recovery
 Sm = Spike result
 N = Native concentration in the un-spiked sample
 Sa = Concentration of Spike Added

For laboratory control samples (LCS) the percent recovery calculation will be determined as follows:

$$\% Rec = \frac{Mv}{Tv} \times 100\%$$

% Rec = Percent recovery
 Mv = Measured Value in LCS
 Tv = True (certified) Value in LCS

Representativeness is a qualitative term that expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. For this project, samples intended for analysis in the laboratory will be both composites of influent and outfall waters collected at timed intervals and discrete grab samples that will be collected following the application of formaldehyde in the facility.

Comparability is a qualitative term that expresses the measure of confidence that one data set can be compared to another and can be combined for the decision(s) to be made. For this project sample processing, and the methods for extraction and analysis of Formaldehyde will utilize accepted EPA methodologies.

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest. For this project, the sensitivity for the measurement of formaldehyde will be 100 ug/L (ppb). This concentration range is expected to allow for dilution of the drug through the facility and still allow for a quantitative determination to be made. The reporting limits for all measurement parameters are provided in Table 6.

Completeness is the measure of the amount of valid data needed to be obtained from a measurement system. A complete data set will be free of field and laboratory errors and accidents such as broken sample containers, non-preserved samples when it is required, misidentified samples, and calibration errors. For this project, a completeness objective of 100% valid laboratory measurements is the goal. The completeness calculation is given as:

$$\% \text{ Completeness} = \frac{N_v}{N_m} \times 100\%$$

N_v = Number of Valid Measurements
 N_m = Total Number of Measurements

Special Training Requirements/Certification

Field samplers will have at a minimum 24-hour Basic Health and Safety training and/or the Hazardous Materials Incident Response Course and will be in compliance with regional health and safety requirements such as attending an 8-hour safety training refresher course every year after the basic training and participation in the medical monitoring program. The field sampling personnel involved with this project are senior EPA employees with extensive training and experience relevant to the type of sampling and field analysis required for this project.

Chemists/analysts performing the analytical work for this project have extensive knowledge, skill and demonstrated experience in the execution of the analytical methods being requested.

The Scribe Project Manager has been trained in the requirements for regional documentation as identified in the Region 10 Data Management Plan (2014) document and associated appendices.

Documentation and Records

A field log notebook, photos, GPS locational data, and chain of custody (COC) form generated by Scribe, will be used to document the sampling and inspection activities. Scribe is a software tool developed by the USEPA's Environmental Response Team (ERT) to assist in the process of managing environmental data. This includes sampling/laboratory, locational, observational, and monitoring field data. Scribe is required for all samples submitted to the EPA Region 10 Laboratory and the EPA CLP.

For each sample location, the following will be recorded in the notebook: facility name and address, sample numbers, date, time of each sample collection, physical description of each sample collection point, locational data, weather conditions, color, sample appearance, sample identifier, and measurements. Scribe documentation will include all required R10 template fields and valid values as defined in the R10 DMP, including locational data, sample collection information, COCs, labels, monitoring data, and final validated laboratory results when available. Field duplicates and any field QC (blanks) will be identified in the Scribe Sample Type field. For fixed laboratory analyses, field duplicates will be assigned a separate unique sample identifier. Analytical duplicate and matrix spike / matrix spike duplicate results will be reported with the appropriate laboratory identifier as defined in the lab SOP and the R10 Universal EDD Data Dictionary (DMP appendix B).

Photographs will be managed in accordance with the R10 SOP OEAFIELDSOP-022.

The laboratory will store all sample receipt, sample login, sample preparation documentation, and laboratory instrument documentation per the applicable SOPs.

Validated laboratory results and interpretation (if necessary) will be appended to the final report along with photographs and other supporting documentation.

MEASUREMENT/DATA ACQUISITION

Sampling Process Design (Experimental Design)

Sampling will be done at the following fish hatcheries shown in Table 4 which includes the name of the hatchery, the location, and a description of the treatment/outfall. Additional information for each facility will be provided in the site specific inspection plan (SSIP) included in Attachment 3.

Table 4. Hatchery Information

Name	Location	Estimated Gallons Formalin Used Per Year	Description of Treatment/Outfall
Leavenworth National Fish Hatchery	Leavenworth, WA	4,400	Flow through treatment, 1 outfall
Carson National Fish Hatchery	Carson, WA	516	1 outfall
Little White Salmon National Fish Hatchery	Cook, WA	370	Flow through treatment, 2 outfalls
Dworshak National Fish Hatchery	Ahsahka, ID	2,680	Flow through treatment, 1 outfall
Clearwater Fish Hatchery (Idaho Fish and Game)	Ahsahka, ID	1,650	Flow through treatment, 1 outfall

Sampling will be done at the following point sources: influent raw water, effluent and receiving water location. Analytical parameters for influent raw water and effluent, are applicable field measurements (flow, temperature, total chlorine, ammonia, DO, pH, and formaldehyde screening) and laboratory analysis for formaldehyde. Ammonia reacts with formaldehyde thus the presence of ammonia will result in a low bias for formaldehyde. Chlorine reacts with ammonia to create chloramines and will slowly oxidize the formaldehyde to formic acid. Dechlorination is required if chlorine is detected.

Sampling Methods Requirements

Field samplers visiting fish hatchery facilities need to be aware of and be sensitive to bio-security issues and/or procedures related to the potential disease transmission from one facility to another facility. Fish hatchery operators may deny access to a facility because of the existence of a disease or illness at the facility. In addition, there is a real potential that field samplers may be the vector that transmits a disease from one facility to another if proper precautions are not taken. To the extent possible, field samplers will adhere to facility specific bio-security measures recommended by hatchery operators. Minimal recommendations are that visitors to farms wear freshly laundered clothing and clean footwear, or disposable booties and gloves if appropriate. Some aquaculture facility operators may request that samplers not visit other aquaculture facilities on the day of the visit to their facility. Other options for decontamination of shoes, clothing, and vehicles (e.g., wearing rubber boots and cleaning them with hot water and disinfectant before and after an inspector visits a facility or adjacent property) should be used by field samplers. The USDA-APHIS Veterinary Service /Emergency Program maintain lists of disinfectants (such as Virkon®) that will help prevent the spread of disease causing bacteria, viruses, and other microorganisms. Field samplers may need to contact their local USDA-APHIS Office to obtain a copy of the latest list and follow the recommended disinfection and/or decontamination procedures before visiting an aquaculture facility.

Samples from the facility outfall will be collected as both grab and composite samples. A single grab sample will be manually collected after treatment at a period when the plume with the highest likely concentration of formaldehyde is discharging through the outfall. Three Sigma® composite samplers will be deployed to the facility influent, effluent and receiving water locations. Samplers will be programed to obtain composite samples from the influent and receiving water, and timed discrete samples from the effluent (outfall). Both discrete and composite samples will be collected at 10 minute intervals over

a 2 hour period following formaldehyde treatment. Table 5 below shows the number of samples by location and sample type. While significant concentrations of formaldehyde in the influent water is not expected, it is possible that in certain locations it occurs naturally or that other contaminants from upstream (including but not limited to pesticides and fertilizers used in agricultural or forestry operations) may interfere with formaldehyde analysis. The samples will be collected over a facility specific sampling period that will bracket the application of formalin. The sample collection containers will be iced during sample collection and will be preserved, stored and shipped with ice as the coolant according to Table 6. The temperature of each cooler containing samples will be checked with an infrared thermometer upon arrival at the laboratory.

Table 5. Sample Numbers, Types and Locations

Sampling Location	Sample Type	Number of Samples	Description
Influent	Composite	1	12:1 composite collected at 10 minute intervals with a Sigma® sampler over a 2 hour period
Effluent	Grab	1	Grab sample manually collected at a time when the formaldehyde concentration is expected to be highest
Effluent	Grab	12	12 discrete samples collected at 10 minute intervals with a Sigma® sampler over a 2 hour period
Receiving Water	Composite	1	12:1 composite collected at 10 minute intervals with a Sigma® sampler over a 2 hour period

Samples collected for both composites and grabs will be analyzed as follows:

- (1) Formaldehyde laboratory measurements,
- (2) One un-preserved container for total chlorine, ammonia, and formaldehyde screening field measurements.

The formaldehyde sample containers will be QC-Class glass jars with Teflon lids. Chlorine measurements will be made on site using a glass sample cell designed for use with a chlorine colorimeter. Ammonia measurements will be made in situ with ammonia test strips such as HACH Ammonia (Nitrogen) Test Strips, 0-6.0 mg/L, or equivalent. Where applicable, pH, temperature and dissolved oxygen will be measured in-situ with Horiba® model U53 water quality meters during collection of the physical samples. Total chlorine and formaldehyde screening measurements will be conducted at each facility, at the discretion of the field team lead in terms of frequency and location necessary. Flow measurements will be calculated from measurements taken on-site or will be documented from facility measurements.

At least one blank bottle sample will be obtained and submitted for selected analyses. One or more facilities will also have field rinsate blanks obtained and submitted for selected analyses. Sigma®

automatic composite samplers will require at least one rinsate blank be obtained for analysis. If composite samplers are used in more than one location, the Sigma® sampler will be rinsed with blank water by flushing approximately 5 gallons through the sampler. A field rinsate blank sample will be collected by running DI water through the composite sampler, prior to initial use and before moving to the next sample location.

Depending on the number of facilities to be sampled, the field duplicate ratio of one per facility. Lab QC will be assigned at a frequency of 5% (1/20 samples).

See the SSIP for approximate sampling numbers, specific analyses, containers, preservation, volumes, and holding times. All alterations or deviations from this QAPP shall be documented using attachment 1 - Sample Alteration Form.

Sample Handling and Custody Requirements

Sample custody is critical to establishing and maintaining the integrity of samples. Samples are determined to be in the custody of the designated EPA sample custodian when they are:

- in the physical possession,
- in plain sight,
- secured or locked in a manner that restricts access.

Samples will be kept in the custody of EPA project personnel. Region 10 Chain of Custody procedures and Chain of Custody forms generated by Scribe will be used. Custody seals (2) will be placed on all shipping containers/coolers.

Packaging, marking, labeling, and shipping of samples will comply with R10 requirements and all regulations promulgated by the U. S. Department of Transportation (DOT) in the Code of Federal Regulations, 49 CFR 171 -177 and International Air Transport Association (IATA) regulations.

Analytical Methods Requirements

Monitoring shall be conducted in accordance with accepted analytical procedures (Standard Methods or other EPA approved methods). See Table 6 for specific methods, reporting limits, etc. applicable to each facility.

Quality Control Requirements

Quality Control procedures will be implemented per the analytical methods and laboratory standard operating procedures (SOP's). Data quality indicators are provided in Table 6. Prior to analysis a full initial demonstration of capability will be performed by the lab.

Instrument/Equipment Testing, Inspection, and Maintenance Requirements

Field maintenance will be performed where appropriate prior to use of the instruments. The laboratory will follow their SOP's for any preventative maintenance required on laboratory instruments or systems used for this project.

Calibration Procedures and Frequency

Field calibration will be performed where appropriate prior to use of the instruments. The Horiba multi-meter will be calibrated and verified with each day of use in accordance with EPA SOP "Calibration and use of the Horiba U-53G Multi Water Quality Checker" - OEAFIELDSOP-100. Field measurement for total chlorine and formaldehyde screening will be conducted according to the manufacturer's procedure and EPA field SOP where applicable.

Laboratory instrumentation and other equipment will be calibrated in accordance with the applicable analytical procedure, laboratory quality manual and standard operating procedures. Laboratory instrumentation will be maintained in accordance with the instrument manufacturer's specifications and/or the Laboratory standard operating procedures.

Inspection/Acceptance Requirements for Supplies and Consumables

All sample jars used for this project will be new certified clean and supplied by MEL. These will consist of glass 125 mL containers for formaldehyde. The field samplers will make note of the information on the certificate of analysis that accompanies sample jars to ensure that they meet the specifications and guidance for contaminant free sample containers.

Data Acquisition Requirements (Non-Direct Measurements)

Where recent historical data for QAPP parameters exist from each facility inspected, it may be used for trends analysis and comparison of results. Facility generated flow data may be acquired at the time of sample setup and collection.

Data Management

Critical data for this project will consist of field logbook entries, field measurements, sample identification information (facility identification and sample IDs), locational data, and laboratory sample results. Field logbooks and photographs will be maintained by the OERA principal investigator and the information maintained in a project file. All required project data (EPA, 2014 DMP) will be managed in Scribe and archived to Scribe.net upon project completion. All supportive laboratory documentation will be kept at MEL in hardcopy format until archived to the federal records center in accordance with the

lab SOP. Prior to final release, an independent check of the laboratory results will be performed internal to the laboratory.

ASSESSMENT/OVERSIGHT

Assessments and Response Actions

An internal assessment of the data and results may be conducted by the appropriate supervisors and the Laboratory QA Coordinator. MEL routinely participates in EPA's water pollution performance evaluation studies (WP Studies). No U.S. EPA system audit is planned for the aquaculture monitoring activities.

Corrective action procedures that might be implemented from QA results or detection of unacceptable data will be developed if required (See Attachment 2- Corrective Action Form).

Reports to Management

Besides data verification reports to be provided to the principal investigators, no other QA reports are planned. If, for any reason, the schedules or procedures above cannot be followed, the appropriate person must complete the "Sample Alteration Form" (attachment 1) for each element changed and have the element(s) verified and reviewed by the Project Manager and the QA Office.

DATA VERIFICATION AND USABILITY

Data Review and Verification Requirements

The summary of all analytical results will be reported to the project managers and staff as identified in Table 1. The raw data for this project shall be maintained by the laboratory. Data verification will also be performed by MEL for all the analyses prior to the release of data at a level equivalent to 100% Stage 4 Validation Manual (S4VM, EPA 2009). The laboratory will archive the analytical data into their laboratory data management system.

Review and Verification Methods

All laboratory data generated will be reviewed in accordance with the analytical methods specified in the QAPP and the MEL data review procedures. All data generated by the laboratory will be reported to the principal investigators.

The OERA principal investigator will be responsible for reviewing field log notebooks for accuracy and completeness upon return from the field. Sample results will be appended to the final report. The Scribe data manager will spot check 10% of the electronic data against the hardcopy results prior to and after uploading the final laboratory data into Scribe.

Reconciliation with User Requirements

The principal investigators will review the formaldehyde and flow data in order evaluate if there is sufficient information to determine whether the dilution criteria for formalin usage within each facility has been achieved. This will involve evaluating if the reporting limit goals have been achieved and if the data collection approach was adequate.

References Cited

- (1) EPA 240-R-02-009, *Guidance for Quality Assurance Project Plans*, EPA QA/G-5, December, 2002
- (2) EPA 240-B-06-001, *Guidance on Systematic Planning Using the Data Quality Objectives Process* – EPA QA/G-4, February, 2006
- (3) U.S. EPA Region 10. *Biological Evaluation - Federal Aquaculture Facilities and Aquaculture Facilities Located in Indian Country within the Boundaries of Washington State*. December 2015
- (4) Lalonde BA, Earnest W, Garron C, (2015) *Formaldehyde Concentration in Discharge from Land Based Aquaculture Facilities in Atlantic Canada*. Bulletin of Environmental Contamination and Toxicology 94:444-447
- (5) EPA Region 10, 2014, *Data Management Plan for Environmental Monitoring and Associated Geospatial Data*, Appendix H, EPA Region 10 Quality Management Plan, January, 2014
- (6) EPA Region 10, 2015, *Quality Assurance Manual for the U.S. EPA Region 10 Environmental Laboratory*, April, 2015

Table 6. Sample Bottles, Preservation & Data Quality Indicators

Analytical Group	Number of Samples ¹	# of QA Samples: Field Dups/ Blanks ¹	Matrix	EPA Method	Reporting Limits	Accuracy	Precision (RPD)	Completeness	Preservation	Volume, Container	Holding Time ² (days)
Laboratory Measurements											
Formaldehyde	15	1/2	Water	1667A	100 µg/L	50 - 150%	30%	100%	Cool 4°C	1x125 mL Glass Tall / 4x125mL for one sample from each facility for lab QC	5 days
Field Measurements											
Flow	continuous	1/NA	Water	SOP	Cf/min	NA	NA	100%	NA	NA	NA
Chlorine Total ³	4	1	Water	DPD	0-4.5 mg/L	+/- 0.02 mg/L	NA	100%	NA	NA	Analyze Immed.
Ammonia	4	1	Water	Test Strip	0-6.0 mg/L	+/- 0.5 color block	NA	100%	NA	NA	Analyze Immed.
pH	4	1/NA	Water	150.1	NA	± 0.1 pH Unit	± 0.1 pH Unit	100%	None Required	Field Sample Container	Analyze Immed.
Temperature	4	1/NA	Water	170.1	NA	0.1°C	NA	100%	None Required		Analyze Immed.
Dissolved Oxygen (DO)	4	1/NA	Water	360.1	0.05 mg/L	± 2%	30%	100%	None Required		Analyze Immed.

¹ Number of field and QC samples represents the estimated number of samples for the influent, receiving water, and outfall per facility. The total number of samples including field QC will not exceed 20 per facility.

² For the timed discrete samples, the holding time begins at completion of the collection of the last sample. Formaldehyde derivatization must occur within 5 days of collection and analyzed within 3 days of derivatization. Field measurements indicated as 'analyze immediately' are within a holding time of 15 minutes from collection.

³ Where total chlorine is detected at a point of sample collection, the result must be indicated in the Scribe COC comments / special instructions so that the lab staff is alerted.

Attachment 1
Sample Alteration Form
(QAPP Addendum – SPAF # NN)

QAPP Title, Author (company), Revision, and Approval Date of standing 'parent' QAPP:

Project Name and assigned Region 10 Project Code:

Material to be Sampled:

Measurement Parameters:

Standard Procedure for Field Collection and Laboratory Analysis (cite references):

Reason for Change in Field Procedure or Analytical Variation:

Variation from Field or Analytical Procedure (reference specific QAPP sections):

Special Equipment, Materials, or Personnel Required:

CONTACT	APPROVAL SIGNATURE	DATE
Initiator: First and Last Name, Title		
EPA Project Manager: First and Last Name, Title		
EPA R10 QA Manager: First and Last Name		
Other project designee (title): First and Last Name		

Attachment 2

Corrective Action Form

QAPP Title, Author (company), Revision, and Approval Date of standing 'parent' QAPP:

--

Project Name and assigned Region 10 Project Code:

--

Sample Dates Involved:

--

Measurement Parameters:

--

Acceptable Data Range:

--

Problem Areas Requiring Corrective Action:

--

Measures Required to Correct Problem:

--

Means of Detecting Problems and Verifying Correction:

--

CONTACT	APPROVAL SIGNATURE	DATE
Initiator: First and Last Name, Title		
EPA Project Manager: First and Last Name, Title		
EPA R10 QA Manager: First and Last Name		
Other project designee (title): First and Last Name		

Attachment 3

Site Visit Information Form

Site Name/Facility Type:	
Address:	
NPDES Permit Number:	
Contact Person:	
E-mail Address /Phone Number:	
EPA Project Code:_____	Sample Numbers:_____

Facility Entry - Date: _____ Start Time: _____ End Time: _____

GPS Sample Location Influent Longitude: _____ Date/Time: _____

Datum: _____ Latitude: _____ Waypoint #: _____

Effluent Longitude: _____ Date/Time: _____

Latitude: _____ Waypoint #: _____

Receiving Water Longitude: _____ Date/Time: _____

Latitude: _____ Waypoint #: _____

Aquatic species treated: _____

Eggs ☐Juveniles ☐Adults ☐

Name of Chemical: _____ NADA Number: _____

Other Chemical Treatments with the Last 5 Days: No ☐ Yes ☐ If yes, list chemicals and concentrations.

Influent Sample

Sampler S/N: _____ Advanced Program – Number of Bottles: _____

Bottle Volume= _____ Time Between Samples _____

Tubing Length= _____ Number of Intake Rinses _____

In-Situ Measurements: pH _____ DO _____ Temperature _____

(record in Horiba
where applicable)

Cl _____

Flow _____

Formaldehyde _____

Effluent Samples

Sampler S/N: _____ Advanced Program – Number of Bottles: _____

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Bottle Volume=_____ Time between Samples_____

Tubing Length=_____ Number of intake rinses_____

In-Situ Measurements: pH_____ DO_____ Temperature_____

 Cl_____ Flow_____ Formaldehyde_____

Effluent Grab Samples Collected:_____

Receiving Water Sample

Sampler S/N:_____ Advanced Program – Number of Bottles:_____

Bottle Volume=_____ Time between Samples_____

Tubing Length=_____ Number of intake rinses_____

In-Situ Measurements: pH_____ DO_____ Temperature_____

 Cl_____ Flow_____ Formaldehyde_____

Treatment Type – Bath ☐ Duration: _____ minutes Flow-Through ☐ Duration: _____ minutes

Static Bath Treatment

Tank Volume	Liters
Desired Treatment Concentration	µg/L
Volume of Formalin Needed Per Treatment	Liters

Flow-Through Treatment

Tank Volume	Liters of water treated with formalin
Calculated Flow Rate	Liters/Minute
Duration of Treatment	Minutes
Flow-Through Concentration	µg/L
Amount of Formalin Added Initially	Liters
Amount of Formalin Added During Treatment	mL/Minute
Volume of Formalin Needed Per Treatment	Liters

Maximum percent Facility Discharge Treated:_____

Maximum Volume of Water Discharged per Day:_____

Attachment 4

Data Quality Objectives

The Environmental Protection Agency (EPA) has established policy which states that before information or data are collected on Agency-funded or regulated environmental programs and projects, a systematic planning process must occur during which performance or acceptance criteria are developed for the collection, evaluation, or use of these data. To meet this planning requirement the Agency has developed a 7 step planning process for the development of these data quality objectives. For this study the results of these planning efforts are documented in the following sections:

Step 1: State the problem

Describe the problem –

Formaldehyde (formalin) is currently used as a drug at specific aquaculture facilities with permitted discharges under the NPDES program. However even though there are labelling requirements that govern its use, there are no effluent limitations nor is there any monitoring data available on the formalin concentrations at these outfalls or receiving waters. The FDA recommends that the discharge concentration of formalin not exceed 25 mg/L and the receiving water concentration not exceed 1 mg/L after dilution. We need to determine if formaldehyde is being discharged from Northwest aquaculture facilities at concentrations of ecological concern.

Planning Team –

Planning team consists of Catherine Gockel, Program lead from the Office of Water and Watersheds and Jed Januch, Technical lead from the Office of Environmental Review Assessment. Other technical consultation was received from EPA Chemists, Steve Reimer and Theresa McBride (Manchester Lab), Don Matheny (Quality Assurance) and Jennifer Crawford (Quality Assurance & Sampling) Brent Richmond (Field Services), and Lillian Herger (Environmental Characterization Unit).

Data Needs and Use –

Data on formalin concentrations at the outfalls and receiving waters of aquaculture facilities who are actively applying the drug are needed. This information would be used to inform the aquaculture facility managers on the use and management of this drug in addition to informing permit writers on possible additions to the future permits.

Resources, Constraints, and Deadlines –

Data collection would be contingent on the timeframes when formalin is being applied at these facilities and the likely time that the drug would be reaching the facility outfall and receiving water.

Step 2: Identify the study goals and decisions

The goal of this study is to inform the EPA and Ecology NPDES permit authorities and aquaculture facility managers on the potential effects of formalin usage within the facilities that apply this drug.

Step 3: Identify the type of data needed

The type of data needed for this study are formaldehyde concentrations in water samples that are representative of permitted discharge locations at a time spanning after formalin application has occurred within the facility.

Step 4: Define the study boundaries

The boundaries of this study are limited to influent, permitted outfalls and downstream locations at aquaculture facilities that are actively applying formalin.

Step 5: Define the analytic approach

Collect samples that represent the highest concentration of formalin being flushed through the outfall and at a downstream location during and immediately following formalin treatment (depending on whether the treatment was flow-through or static bath). As a baseline check, co-collect water flowing into the facility at the same time period. Data on chemical interferences that are likely to be present include ammonia (via test strip) which also requires analysis of chlorine. Timeframes for sample collection are dependent on the timing and type of treatment (eggs, juveniles, etc.) being conducted. Water flow at the outfall during sample collection and the amount of formalin used for treatment are also needed to determine if the dilution requirement of the label is being achieved.

Step 6: Define the acceptance criteria

For this study, acceptance criteria for Formalin data are defined by the analytical method requirements. This includes testing for possible interferences (e.g., ammonia and chlorine). The Acceptance criteria are quantitative rules which specify how accurate or how precise the results of an analytical method must be to be considered acceptable.

Step 7: Optimize the study design

Number of facilities – 5

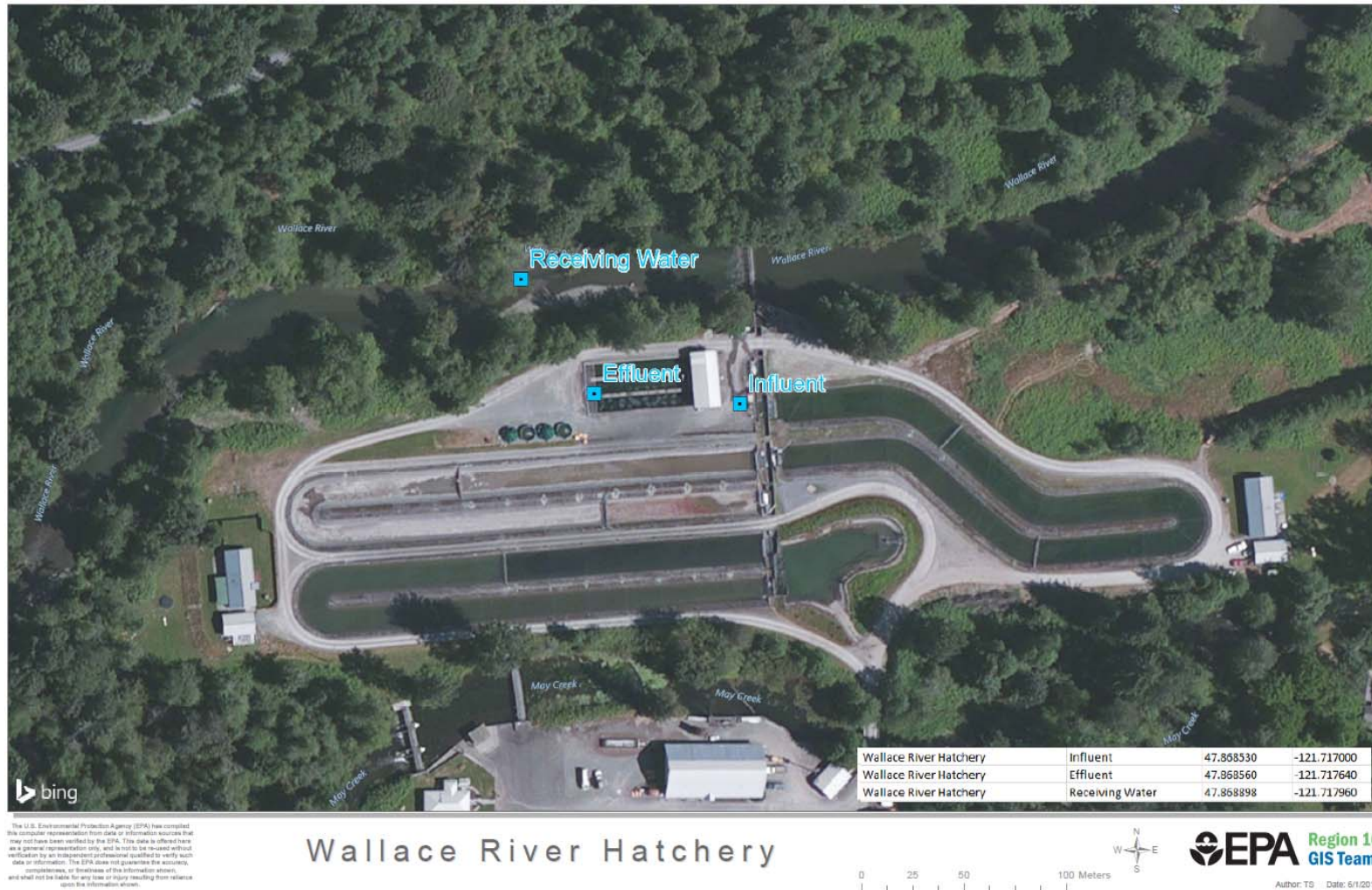
Number of samples – Total number anticipated will not exceed 90.

Sample specifications – A combination of automated sampling and hand collected grab samples will be done after treatment at a period when the plume with the highest likely concentration of formaldehyde is discharging. The estimated time it takes for water at each facility to move from the treatment area to the outfall was provided by hatchery managers.

1. Wallace River Fish Hatchery

14418 383rd Ave SE, Sultan, WA 98294

Bradley Hostetler (360) 793-1382

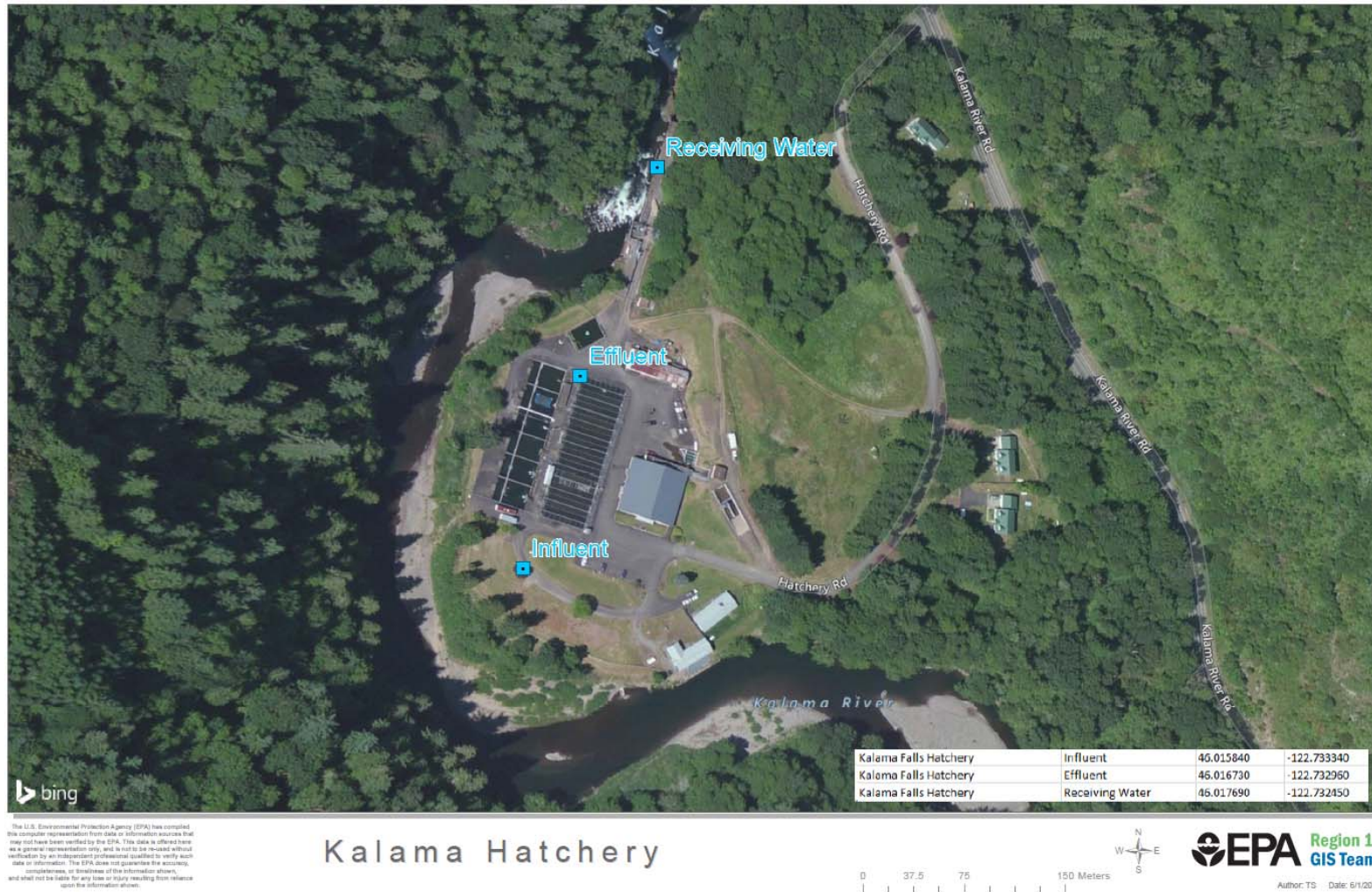


2. Kalama Falls Fish Hatchery

3900 Kalama River Rd, Kalama, WA 98625

Brian Gale

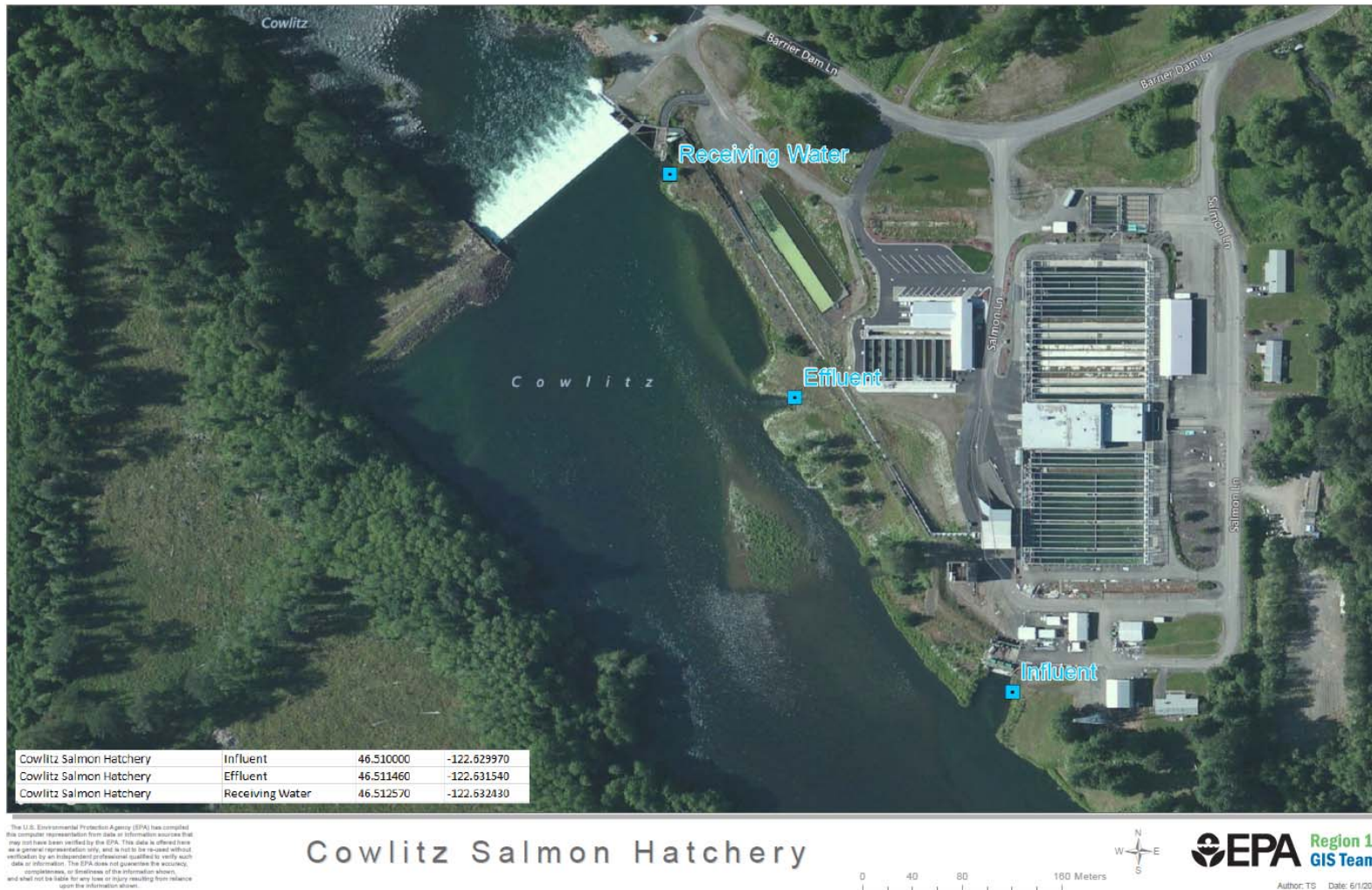
(360) 673-4825



3. Cowlitz Fish Hatchery

125 Salmon Ln, Salkum, WA 98582

Larona Newhouse (360) 985-7424



4. Priest Rapids Fish Hatchery

P.O. Box 937 Mattawa, WA 99349

Glen Pearson

(509) 932-4481



5. Hoodspport Fish Hatchery

P.O. Box 606, Hoodspport, WA 98548

Jorge Villarreal

(360) 877-6408





DEPARTMENT OF
ECOLOGY
State of Washington

Quality Assurance Project Plan

Water Sampling for Formaldehyde at Five Fish & Wildlife Hatcheries in Washington State: Screening Study

March 2017

Publication No. 17-03-108

Publication Information

This document is an addendum to the U.S. Environmental Protection Agency's Quality Assurance Project Plan (QAPP) entitled "*Quality Assurance Project Plan for Water Sampling and Testing for Formaldehyde at Northwest Aquaculture Facilities*" (EPA 2016). The QAPP describes the objectives of the study and the procedures to be followed to achieve those objectives.

This document, *Quality Assurance Project Plan: Water Sampling for Formaldehyde at Five Fish & Wildlife Hatcheries in Washington State: Screening Study*, is available at:
<https://fortress.wa.gov/ecy/publications/SummaryPages/1703108.html>

Ecology's Activity Tracker Code for this study is 17-013.

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Quality Assurance Project Plan

Water Sampling for Formaldehyde at Five Fish & Wildlife Hatcheries in Washington State: Screening Study

March 2017

Approved by:

Signature: Mike Hepp, Compliance Specialist, WQP, Eastern Regional Office	Date:
Signature: Donald Matheny, Chemist, EPA Region 10	Date:
Signature: Siana Wong, Author / Project Manager, EAP	Date:
Signature: Brandee Era-Miller, Author / EAP	Date:
Signature: Debby Sargeant, Author's Unit Supervisor, EAP	Date:
Signature: Jessica Archer, Author's Section Manager, EAP	Date:
Bill Kammin, Ecology Quality Assurance Officer	

Signatures are not available on the Internet version.

EAP: Environmental Assessment Program

WQP: Water Quality Program

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2.0 Abstract

The U.S. Environmental Protection Agency (EPA) is conducting a study to measure the concentrations of formaldehyde in effluent from aquaculture facilities in Washington and Idaho (EPA, 2016). In Washington State, EPA is the National Pollutant Discharge Elimination System (NPDES) permitting authority for federal aquaculture facilities and aquaculture facilities in Indian Country. The Washington State Department of Ecology (Ecology) is the NPDES permitting authority for all other aquaculture facilities.

EPA conducted sampling and field analysis at five National Fish Hatcheries as documented in the *EPA Formaldehyde in NW Aquaculture Facility Water Study Quality Assurance Project Plan* (QAPP) (EPA, 2016). In coordination with EPA, Ecology conducted sampling and field analysis at five Washington Department of Fish & Wildlife (WDFW) hatcheries that use formalin.

This report documents Ecology's sampling and field analysis for this project, which is adapted from EPA's QAPP for the study and the subsequent Addendum to the QAPP (*Sample Plan Alteration Form #2*) documenting the addition of Ecology-sampled facilities.

3.0 Background

3.1 Introduction and problem statement

Formalin, a 37% by mass aqueous solution of formaldehyde gas, is commonly used to treat and reduce introduction of external parasites in fish hatcheries. This study is being conducted to collect empirical data on formaldehyde concentrations in hatchery effluents in Washington State. The data will be used to determine if concentrations are at levels that pose ecological risk to threatened species or their critical habitat (EPA, 2016).

Ecology's sampling at state-operated hatcheries was intended to supplement EPA's sampling at federally operated hatcheries. The addition of state hatcheries provides further empirical data for the overall study. Details and background about the overall project can be found in EPA's QAPP (EPA, 2016).

3.2 Study area and surroundings

Five WDFW state fish hatcheries that use formalin during treatments were selected for this study (Table 1, Figure 1). These hatcheries were selected by Ecology based on their relatively high use of formalin. Formalin use is reported to Ecology by each NPDES-permitted facility in an Annual Disease Control Chemical Use report.

Table 1. Participating WDFW hatcheries for this study, estimated formalin used per year, WDFW contacts for sampling coordination, sampling dates, and treatment types.

Participating Hatchery	Estimated Formalin Used per Year (Gallons)*	WDFW Contact	Sampling Date	Treatment Type
Kalama Falls Hatchery	1554	Sam Gibbons Manager (360) 673-4825 Sam.Gibbons@dfw.wa.gov	Sept 12, 2016	Adults & eggs
Wallace River Hatchery	2575	Brad Hostetler FHS4 (360) 793-1382 Bradley.Hostetler@dfw.wa.gov	Sept 19, 2016	Adults & eggs
Cowlitz Salmon Hatchery	6608	Larona Newhouse FHS4 (360) 673-4825 Sam.Gibbons@dfw.wa.gov	Sept 30, 2016	Adults
Priest Rapids Hatchery	2310	Glen Pearson FHS4 (509) 932-4481 Glen.Pearson@dfw.wa.gov	Nov 4, 2016 Nov 30, 2016	Adults (Nov 4) Eggs (Nov 30)
Hoodspoint Hatchery	1155	Jorge Villarreal FHS4 (360) 877-2737 Jorge.Villarreal@dfw.wa.gov	Dec 7, 2016	Eggs

* Based on formalin use reported to Ecology by each facility in 2014.

3.2.3 Parameters of interest and potential sources

The parameter of interest is formaldehyde. The potential source is the hatchery performing formalin treatments as part of typical daily operations.

Ancillary data – including ammonia concentration, chlorine concentration, dissolved oxygen concentration, pH, and water temperature – were also collected.

3.2.4 Regulatory criteria or standards

Under Washington's Upland Fin-Fish Hatching and Rearing General NPDES Permit, aquaculture facilities that use formalin are required to follow chemical label instructions and report use and concentration information. Parasite-S by Western Chemical, Inc., is the commonly used chemical for formalin treatments. In the finding of no significant impact for Parasite-S, the Food and Drug Administration (FDA) requires a "10-fold dilution of finfish and penaeid shrimp treatment water and a 100-fold dilution of finfish egg treatment water." This should lead to a discharge concentration of no more than 25 ppm. According to the FDA, a

discharge concentration <25 ppm, instream dilution, infrequent use, and rapid degradation of formalin in water will cause no significant aquatic impact (EPA, 2015)

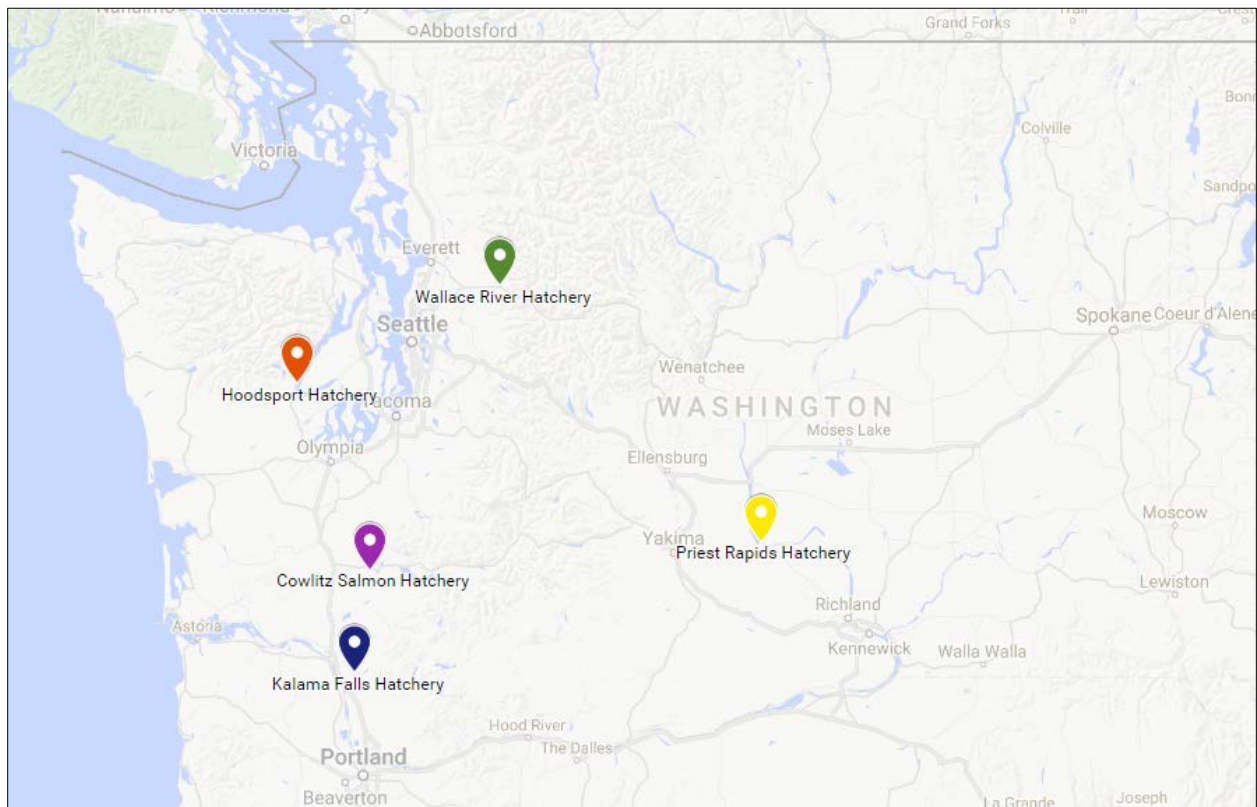


Figure 1. Location of participating WDFW hatcheries.

3.3 Water quality impairment studies

Not Applicable.

3.4 Effectiveness monitoring studies

Not Applicable.

4.0 Project Description

This section describes the specific objectives and tasks required for Ecology's sampling and field analysis at participating WDFW hatcheries. More information about the overall project can be found in EPA's QAPP (EPA 2016). No report or data interpretation will be provided by Ecology for this project.

4.1 Project goals

The goal of this study was to sample on behalf of EPA to provide an assessment of formaldehyde concentrations at WDFW hatcheries using the methods outlined in EPA (2016) as closely as possible.

4.2 Project objectives

The objectives for Ecology's sampling and field analysis were to:

- Collect water samples for laboratory analysis of formaldehyde concentration at each hatchery sampling site using an ISCO automated composite sampler (ISCO).
- Measure chlorine, ammonia, and formaldehyde concentration using test screening kits at each hatchery sampling site.
- Measure in-situ water chemistry (temperature, pH, dissolved oxygen) at each hatchery sampling site.

4.3 Information needed and sources

Not Applicable.

4.4 Tasks required

Pre-Sampling Tasks

- Coordinate with EPA staff prior to each sampling event.
- Schedule and verify sampling dates with hatchery managers.
- Print samples labels and obtain Chain of Custody form sent by EPA.
- Calibrate Hydrolab.
- Test ISCOs and charge ISCO batteries.
- Pre-wash sampling equipment.

Field Tasks

- Set up and program ISCOs to collect water samples for laboratory analysis of formaldehyde at each hatchery sampling site.
- Collect one grab sample per hatchery sampling site for measurement of chlorine, ammonia, and formaldehyde concentration using test screening kits.
- Measure water temperature, pH, and dissolved oxygen using Hydrolab at each hatchery sampling site.
- Collect information about formaldehyde treatment from hatchery staff.

Post-Sampling Tasks

- Send copy of Chain of Custody form, water quality data, and GPS coordinates to EPA staff.
- Ship samples to EPA Region 10 Laboratory.
- Perform Hydrolab post-calibration check.

Field Equipment Checklist

- ISCO water sample collection
 - ISCO automated samplers x 3
 - Pre-washed 1-L wedge-shaped polyethylene bottles x 24
 - Pre-washed 10-L glass jug x 2
 - 3/8" ID (1/2" OD) polyethylene/vinyl tubing for ISCOs (~50 feet total)
 - Clamps for attaching suction line to pump tube
 - 3 charged 12-volt batteries + chargers
 - 3 cables for connecting 12-volt batteries to ISCOs
 - Graduated cylinder for calibration (1000 mL)
 - 3 strainers for end of suction line
- GPS for documenting sample locations
- 125-mL glass jars for formaldehyde samples
- Hach Chlorine 46700-00 CL2 Test Kit with Pocket Colorimeter
- Hach AquaCheck Ammonia Test Strips
- QUANTOFIX® Formaldehyde Test Kit
- Calibrated Hydrolab, connection cable, & handheld meter
- Pre-washed 1-L polyethylene bottle for grab samples
- Extended pole for grab samples
- Cooler(s) with ice for water samples
- DI water for field equipment blank
- Field notebook
- EPA-Scribe preprinted labels and Chain of Custody forms (printed copy)
- Other
 - Writing utensils
 - Bubble wrap for sample jars
 - Packaging tape for sample labels
 - Ziploc bags- gallon size for water samples
 - Miscellaneous tools
 - Laboratory gloves
 - Boots, waders, rain gear

4.5 Systematic planning process used

This QAPP and the EPA QAPP (EPA, 2016) represent the systematic planning process for this project.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Key Ecology personnel (in addition to those EPA personnel identified in EPA's QAPP) are summarized in Table 2.

5.2 Special training and certifications

Not Applicable.

5.3 Organization chart

See Table 2.

Table 2. Organization of Ecology staff and project responsibilities.

Staff	Title	Responsibilities
Siana Wong Toxics Studies Unit Statewide Coordination Section Environmental Assessment Program Phone: 360-407-6432 Email: swon461@ecy.wa.gov	Project Manager	Leads field operations. Coordinates logistics and activities with EPA and participating WDFW hatcheries.
Brandee Era-Miller Toxics Studies Unit Statewide Coordination Section Environmental Assessment Program Phone: 360-407-6771 Email: bera461@ecy.wa.gov	Field Lead	Leads field operations. Reviews Ecology's QAPP.
Debby Sargeant Toxics Studies Unit Statewide Coordination Section Environmental Assessment Program Phone: 360-407-6775 Email: dsar461@ecy.wa.gov	Unit Supervisor for the Project Manager	Provides internal review, approves Ecology's final QAPP, manages budget and staffing needs
William R. Kammin Phone: 360-407-6964 Email: wkam461@ecy.wa.gov	Ecology Quality Assurance Officer	Reviews and approves the draft and final QAPP addendum.

5.4 Proposed project schedule

Five WDFW hatcheries that perform formalin treatments were selected for this study (Table 1). A one-time sampling event at each hatchery was coordinated with each of the hatchery facility managers during formalin treatment periods between September–December 7, 2016. The Priest Rapids Hatchery was sampled during two sampling events: once during adult treatments, and a second time during egg treatments. Contact information and sampling dates for each hatchery are shown in Table 1. Detailed schedules for each facility were determined in coordination with the EPA Region 10 lab staff, Principal Investigators, and Regional Sample Control Center prior to sample collection.

Ecology's sampling was also coordinated with EPA to ensure that similar sampling methods and procedures were used, and that samples were not submitted to EPA's laboratory during the same weeks in order to avoid overloading the laboratory's capacity. Ecology staff followed the EPA Region 10 sample shipment/delivery notification and coordination requirements with the EPA Regional Sample Control Center and lab staff.

Table 3. Proposed schedule for completing field work and data management tasks

Task	Due date	Lead staff
Field work completed	Dec 2016	Siana Wong
All in-situ and grab sample data and site coordinate information sent to EPA for entry into Scribe	Dec 2016	Siana Wong
Copies of field notes & files sent to EPA Project Manager	Jan 2017	Siana Wong

5.5 Budget and funding

Laboratory budget and funding for this project was provided by the EPA.

To complete Ecology's sampling, the EPA provided the following equipment:

- Pocket Colorimeter™ II, Chlorine (Free and Total).
- DPD Total Chlorine Reagent Powder Pillows for a 10 mL sample size.
- Free & Total Chlorine Test Strips, 0-10 mg/L.
- Ammonia (Nitrogen) Test Strips, 0-6.0 mg/L.
- Formaldehyde sample bottles, 125 mL.

All other equipment and staff time was provided by Ecology.

6.0 Quality Objectives

6.1 Data quality objectives

See EPA (2016).

6.2 Measurement quality objectives

See EPA (2016) for discussion on Measurement Quality Objectives (MQOs) for this study. MQOs for Ecology's Hydrolab measurements are shown in Table 4.

Table 4. Measurement quality objectives for Hydrolab calibration checks.

Parameter	Units	Accept	Qualify	Reject
pH	std. units	$< \text{or} = \pm 0.2$	$> \pm 0.2$ and $< \text{or} = \pm 0.8$	$> \pm 0.8$
Conductivity*	uS/cm	$< \text{or} = \pm 5$	$> \pm 5$ and $< \text{or} = \pm 15$	$> \pm 15$
Temperature	° C	$< \text{or} = \pm 0.2$	$> \pm 0.2$ and $< \text{or} = \pm 0.8$	$> \pm 0.8$
Dissolved Oxygen	mg/L	$< \text{or} = \pm 0.3$	$> \pm 0.3$ and $< \text{or} = \pm 0.8$	$> \pm 0.8$

* Criteria expressed as a percentage of readings; for example, buffer = 100.2 uS/cm and Hydrolab = 98.7 uS/cm; $(100.2-98.7)/100.2 = 1.49\%$ variation, which would fall into the acceptable data criteria of less than 5%.

6.2.1 Targets for precision, bias, and sensitivity

6.2.1.1 Precision

See EPA (2016).

6.2.1.2 Bias

See EPA (2016).

6.2.1.3 Sensitivity

See EPA (2016).

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

See EPA (2016).

6.2.2.2 Representativeness

See EPA (2016).

6.2.2.3 Completeness

See EPA (2016).

6.3 Acceptance criteria for quality of existing data

See EPA (2016).

6.4 Model quality objectives

Not Applicable.

7.0 Study Design

The study design follows EPA's study design as described in EPA (2016).

7.1 Study boundaries

See Section 3.2 of this document.

7.2 Field data collection

7.2.1 Sampling location and frequency

At each hatchery, sampling occurred at the influent, effluent (discharge), and receiving water. Table 5 shows the site location at each hatchery, analyte to be collected/measured at each site, sample/measurement type, and general collection description.

Table 5. Summary of water samples/measurements collected at each hatchery location.

Sampling Location at Each Hatchery	Analyte	Collection Type – # Samples/ Measurements	Collection Description
Influent	Formaldehyde	Composite – 1	Automated ISCO sampler set up to collect sample at 20-minute intervals for 4 hours following formaldehyde treatment (12:1 composite)
	Chlorine (Total)	Grab – 1	Hach Chlorine 46700-00 CL2 Test Kit with Pocket Colorimeter
	Ammonia	Grab – 1	Hach AquaCheck Ammonia Test Strips
	Temperature	Measurement – 1	Hydrolab MiniSonde
	Dissolved Oxygen	Measurement – 1	Hydrolab MiniSonde
	pH	Measurement – 1	Hydrolab MiniSonde
	Formaldehyde Screening	Grab – 1	QUANTOFIX® Formaldehyde Test Kit
Effluent	Formaldehyde	Grab – 13	Automated ISCO sampler set up to collect 12 discrete samples at 20-minute intervals for 4 hours following formaldehyde treatment. One grab sample will also be collected manually during estimated peak formalin discharge.
	Chlorine (Total)	Grab – 1	Hach Chlorine 46700-00 CL2 Test Kit with Pocket Colorimeter
	Ammonia	Grab – 1	Hach AquaCheck Ammonia Test Strips
	Temperature	Measurement – 1	Hydrolab MiniSonde
	Dissolved Oxygen	Measurement – 1	Hydrolab MiniSonde
	pH	Measurement – 1	Hydrolab MiniSonde
	Formaldehyde Screening	Grab – 1	QUANTOFIX® Formaldehyde Test Kit
Receiving water	Formaldehyde	Composite – 1	Automated ISCO sampler set up to collect sample at 20-minute intervals for 4 hours following formaldehyde treatment (12:1 composite)
	Chlorine (Total)	Grab – 1	Hach Chlorine 46700-00 CL2 Test Kit with Pocket Colorimeter
	Ammonia	Grab – 1	Hach AquaCheck Ammonia Test Strips
	Temperature	Measurement – 1	Hydrolab MiniSonde
	Dissolved Oxygen	Measurement – 1	Hydrolab MiniSonde
	pH	Measurement – 1	Hydrolab MiniSonde
	Formaldehyde Screening	Grab – 1	QUANTOFIX® Formaldehyde Test Kit

7.2.2 Field parameters and laboratory analytes to be measured

The following parameters were measured/sampled at each hatchery as documented in the EPA QAPP:

- Formaldehyde - Lab
- Chlorine (Total) - Field
- Ammonia - Field
- Water Temperature - Field
- Dissolved Oxygen - Field
- pH - Field
- Formaldehyde Screening (test kits) - Field

7.3 Modeling and analysis design

No modeling or data analysis were conducted for this project.

7.3.1 Analytical framework

Not Applicable.

7.3.2 Model setup and data needs

Not Applicable.

7.4 Assumptions in relation to objectives and study area

Not Applicable.

7.5 Possible challenges and contingencies

7.5.1 Logistical problems

Field work was coordinated with hatchery staff to ensure that sampling occurred on treatment days during peak formalin treatment periods. Field schedules were also coordinated with EPA laboratory staff to ensure that the lab could process the samples during the targeted sampling dates. Each hatchery was visited prior to sampling day to determine sampling sites and identify any logistical challenges.

7.5.2 Practical constraints

Not Applicable.

7.5.3 Schedule limitations

Not Applicable.

8.0 Field Procedures

8.1 Invasive species evaluation

Not Applicable.

8.2 Measurement and sampling procedures

Measurement and sampling procedures followed EPA's QAPP (EPA, 2016) as closely as possible to ensure that data are comparable. No deviations occurred, with the exception of the type of instruments used for collecting samples (ISCO vs. Sigma automated sampler; Hydrolab vs. Horiba multi-probe instrument). Calibration of the Hydrolab followed Ecology's SOP (Anderson, 2016).

Formaldehyde

Formaldehyde samples for laboratory analysis were collected using automated ISCO (Model 6712) samplers. Three ISCO samplers were programmed to collect samples at the influent, effluent, and receiving water at each hatchery. Prior to sampling, ISCO samplers were calibrated at each hatchery location to ensure that the accurate volumes were collected.

Each ISCO was programmed to collect the first sample ~15 minutes before treatment started and the last sample ~1 hour after treatment ended. This helped ensure that the pulse of formalin running through the system was captured.

At the influent and receiving water, a composite sample was collected at 20-minute intervals over a four-hour period during the formalin treatment application. The composite consisted of 12x250-mL aliquots collected into a single 10-L pre-washed (soap + water followed by deionized water rinse) glass jug. The internal compartment of the ISCO sampler was filled with ice to keep the 10-L jug cold during the sampling. After the composite was collected, water from the 10-L jug was mixed and poured into a single 125 mL glass sample jar, and then stored in a cooler on ice. The remaining water in the 10-L jug was discarded.

At the effluent, the ISCO sampler was set up to collect 12 discrete samples over a four-hour period during the formalin treatment. Each discrete sample was comprised of 450-mL water collected in a pre-washed wedge-shaped 1-L polyethylene bottle. At the end of discrete sampling, water from each polyethylene bottle was mixed and poured into a 125 mL glass sample jar (12 total samples). One grab sample for laboratory analysis of formaldehyde was also collected at the effluent during estimated peak formalin discharge.

All formaldehyde samples were stored in a cooler with ice in the field, and then shipped overnight to EPA's laboratory for further processing and analysis.

Chlorine (Total)

Chlorine samples were taken from an aliquot of a grab sample collected at each of the influent, effluent, and receiving water sites. Total chlorine was analyzed on-site using a Hach Chlorine 46700-00 CL2 Test Kit with Pocket Colorimeter. Results were recorded in the field notebook.

Ammonia

Ammonia samples were taken from an aliquot of a grab sample collected at each of the influent, effluent, and receiving water sites. Ammonia was analyzed on-site using Hach AquaCheck Ammonia Test Strips, 0-6.0 mg/L. Results were recorded in the field notebook.

Temperature, Dissolved Oxygen, and pH

A calibrated Hydrolab MiniSonde was used to measure in-situ water temperature, dissolved oxygen, and pH at the influent, effluent, and receiving water sites. The Hydrolab was calibrated on the day prior to sampling following Ecology's SOP (Anderson, 2016). A post-calibration check was performed following each sampling event. Table 4 shows the MQOs for post-calibration checks. Results were recorded in the field notebook.

Formaldehyde screening kit

Formaldehyde screening samples were taken from an aliquot of a grab sample collected at each of the influent, effluent, and receiving water sites. QUANTOFIX® Formaldehyde Test Strips were used to analyze formaldehyde concentrations on-site. Results were recorded in the field notebook.

Flow

Flow information was obtained from facility staff.

Other hatchery facility information

Hatchery facility and formalin treatment information will be obtained from hatchery staff:

- Static Bath Treatment
 - Tank volume
 - Desired treatment concentration
 - Volume of formalin needed per treatment
- Flow-Through Treatment
 - Tank volume
 - Calculated flow rate
 - Duration of treatment
 - Flow-through concentration
 - Amount of formalin added initially
 - Amount of formalin added during treatment
 - Volume of formalin needed per treatment
- Maximum percent facility discharge treated
- Maximum volume of water discharged per day

8.3 Containers, preservation methods, holding times

Table 6 summarizes the number of samples collected, QC samples, matrix, laboratory method, reporting limit, accuracy and precision, completeness, container, preservation, and holding time for each analyte collected. All formaldehyde samples will be sent to the EPA Region 10 Laboratory for analysis.

Table 6. Summary of sample collection information, laboratory and field methods, QC objectives, preservation, and holding times.

Table is adapted from EPA (2016).

Analyte	# Samples per Hatchery	# QC Samples: Field Dups/ Blanks per Hatchery	Matrix	Collection Type	Method	Reporting Limits	Accuracy	Precision (RPD)	Completeness	Container	Preservation	Holding Time
Formaldehyde	15	1/1	Water	Lab Sample	EPA 1667A	100 µg/L	50-150%	30%	100%	125 mL Certified Clean Glass with Teflon Lid Liner	Cool to 4 C	5 days
Chlorine (Total)	3	1/NA	Water	Field Measurement	EPA 330.5	0-4.5 mg/L	± 0.02 mg/L	NA	100%	NA	NA	Analyze Immediately
Ammonia	3	1/NA	Water	Field Measurement	NA (Test Strip)	0-6.0 mg/L	± 0.5 mg/L color block	NA	100%	NA	NA	Analyze Immediately
Temperature	3	1/NA	Water	Field Measurement	EPA 170.1	NA	0.1 C	NA	100%	NA	NA	Analyze Immediately
Dissolved Oxygen	3	1/NA	Water	Field Measurement	EPA 360.1	0.05 mg/L	± 2%	30%	100%	NA	NA	Analyze Immediately
pH	3	1/NA	Water	Field Measurement	EPA 350.1	NA	0.1 pH unit	0.1 pH unit	100%	NA	NA	Analyze Immediately
Formaldehyde Screening (Test Kits)	3	1/NA	Water	Field Measurement	NA (Test Strip)	NA	NA	NA	100%	NA	NA	Analyze Immediately

8.4 Equipment decontamination

Prior to sampling, equipment used for collecting samples was washed with hot tap water and Liquinox soap, followed by tap water rinse and de-ionized water rinse. The specific procedure for decontaminating equipment can be found in Ecology's SOP (Friese, 2014).

8.5 Sample ID

Ecology coordinated with EPA staff to assign unique sample IDs prior to sampling at each hatchery.

8.6 Chain-of-custody

Chain-of-custody was maintained for all samples throughout the project. As Ecology was acting as the sampling entity only, with EPA being the data recipient and data user, all samples and associated field/lab and locational data were managed in a single Scribe file by EPA Office of Environmental Review and Assessment Environmental Services Unit staff. At minimum, Ecology hand-entered the sample collection date and time onto pre-printed Scribe sample labels and Chain of Custody forms for each facility. Immediately after each sampling event, Ecology scanned the completed printed copy of the Chain of Custody form and completed field form (EPA, 2016) to the EPA Regional Sample Control Center so that field data could be entered into Scribe and the lab Chain of Custody XML generated for sample receipt/login.

8.7 Field log requirements

Field data was recorded in a bound, waterproof notebook on Rite in the Rain paper. Attachment 3 of the EPA's QAPP for this project will be used as Ecology's template (EPA, 2016).

8.8 Other activities

Not Applicable.

9.0 Laboratory Procedures

9.1 Lab procedures table

See Table 5 of this document.

9.2 Sample preparation method(s)

Methods for formaldehyde analysis in water samples are documented by EPA Method 1667A.

9.3 Special method requirements

Not Applicable.

9.4 Laboratories accredited for methods

All water sample analyses for formaldehyde concentration were performed by the EPA Region 10 Laboratory.

10.0 Quality Control Procedures

10.1 Table of field and laboratory quality control

Table 5 summarizes field duplicate and blank sample collections for each analyte.

Field Duplicates

One duplicate field sample was collected per sampling event for each grab sample analyte (ammonia, chloride, formaldehyde). One duplicate sample for laboratory analysis of formaldehyde was also collected from a sample taken from the automated discrete sampler during each sampling event.

Field Blanks

One field (rinsate) blank was collected per facility and analyzed for formaldehyde at the laboratory. This was performed by running de-ionized water through the ISCO sampler, collecting the water in the 10-L glass jug, then pouring the water into a formaldehyde sample jar.

Laboratory Matrix Spike

Following discrete sample collection at the effluent site, three formaldehyde sample jars were filled from the first ISCO bottle (representing a sample prior to treatment start). The samples were used by the EPA laboratory to conduct a laboratory matrix spike and matrix spike duplicate.

10.2 Corrective action processes

All data were submitted to the EPA. Corrective action procedures are described in EPA (2016), and will be determined by the EPA Principal Investigator for this project.

11.0 Management Procedures

11.1 Data recording and reporting requirements

See EPA (2016). All data will be submitted to EPA for data reduction and reporting.

11.2 Laboratory data package requirements

See EPA (2016). All data will be submitted to EPA.

11.3 Electronic transfer requirements

Not Applicable.

11.4 EIM/STORET data upload procedures

Not Applicable. All data will be submitted to EPA. The EPA Principal Investigator of this project will provide data upon request.

11.5 Model information management

Not Applicable.

12.0 Audits and Reports

12.1 Field, laboratory, and other audits

To ensure Ecology's sampling and field analysis followed EPA's procedures, Ecology staff accompanied and observed EPA field staff on one of their sampling dates for this project. In addition, EPA staff assisted Ecology at the first WDFW hatchery sampled.

12.2 Responsible personnel

Not Applicable.

12.3 Frequency and distribution of report

Not Applicable.

12.4 Responsibility for reports

The EPA is responsible for all data reduction and reporting for this project (EPA, 2016).

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

See EPA (2016).

13.2 Laboratory data verification

See EPA (2016).

13.3 Validation requirements, if necessary

See EPA (2016).

13.4 Model quality assessment

Not Applicable.

14.0 Data Quality (Usability) Assessment

14.1 Process for determining project objectives were met

EPA is responsible for all data quality determinations (EPA, 2016).

14.2 Treatment of non-detects

See EPA (2016).

14.3 Data analysis and presentation methods

All data will be submitted to the EPA for data analysis. Presentation methods will be determined by the principal investigator.

14.4 Sampling design evaluation

Not Applicable.

14.5 Documentation of assessment

See EPA (2016).

15.0 References

Anderson, P., 2016. Standard Operating Procedures for Hydrolab DataSonde® and MiniSonde® Multiprobes. Version 2.1. Washington State Department of Ecology, Olympia, WA. SOP Number EAP033.

http://www.ecy.wa.gov/programs/eap/qa/docs/ECY_EAP_SOP_Hydrolab_v2_1EAP033.pdf

EPA, 2015. Biological Evaluation- Federal Aquaculture Facilities and Aquaculture Facilities Located in Indian Country within the Boundaries of Washington State. U.S. Environmental Protection Agency.

https://www3.epa.gov/region10/pdf/permits/npdes/wa/WA_Hatchery_GP_WAG130000_BE.pdf

EPA, 2016. Quality Assurance Project Plan for Water Sampling and Testing for Formaldehyde at Northwest Aquaculture Facilities. U.S. Environmental Review and Assessment, U.S. Environmental Protection Agency, Region 10.

Friese, M., 2014. Standard Operating Procedures for Decontaminating Field Equipment for Sampling Toxics in the Environment. Version 1.0. Washington State Department of Ecology, Olympia, WA. SOP Number EAP090.

http://www.ecy.wa.gov/programs/eap/qa/docs/ECY_EAP_SOP_EquipmentDeconToxicsSampling_v1_0EAP090.pdf

16.0 Appendix. Glossaries, Acronyms, and Abbreviations

Glossary of General Terms

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.

Dissolved oxygen: A measure of the amount of oxygen dissolved in water.

Effluent: An outflowing of water from a natural body of water or from a human-made structure. For example, the treated outflow from a wastewater treatment plant.

National Pollutant Discharge Elimination System (NPDES): National program for issuing, modifying, revoking and reissuing, terminating, monitoring, and enforcing permits, and imposing and enforcing pretreatment requirements under the Clean Water Act. The NPDES program regulates discharges from wastewater treatment plants, large factories, and other facilities that use, process, and discharge water back into lakes, streams, rivers, bays, and oceans.

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.

Acronyms and Abbreviations

Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	U.S. Environmental Protection Agency
GPS	Global Positioning System
MQO	Measurement quality objective
NPDES	(See Glossary above)
QA	Quality assurance
QC	Quality control
SOP	Standard operating procedure
WDFW	Washington Department of Fish and Wildlife

Units of Measurement

mg/L	milligrams per liter (parts per million)
mL	milliliter
uS/cm	microsiemens per centimeter, a unit of conductivity

Quality Assurance Glossary

Accuracy: The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms precision and bias be used to convey the information associated with the term accuracy. (USGS, 1998)

Analyte: An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e.g., fecal coliform, Klebsiella. (Kammin, 2010)

Bias: The difference between the population mean and the true value. Bias usually describes a systematic difference reproducible over time, and is characteristic of both the measurement system, and the analyte(s) being measured. Bias is a commonly used data quality indicator (DQI). (Kammin, 2010; Ecology, 2004)

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. (USGS, 1998)

Calibration: The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured. (Ecology, 2004)

Comparability: The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator. (USEPA, 1997)

Completeness: The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator. (USEPA, 1997)

Data Quality Objectives (DQO): Qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions. (USEPA, 2006)

Data verification: Examination of a data set for errors or omissions, and assessment of the Data Quality Indicators related to that data set for compliance with acceptance criteria (MQOs). Verification is a detailed quality review of a data set. (Ecology, 2004)

Duplicate samples: Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis. (USEPA, 1997)

Field blank: A blank used to obtain information on contamination introduced during sample collection, storage, and transport. (Ecology, 2004)

Matrix spike: A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias due to interference or matrix effects. (Ecology, 2004)

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

Method: A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed. (EPA, 1997)

Parameter: A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all “parameters.” (Kammin, 2010; Ecology, 2004)

Precision: The extent of random variability among replicate measurements of the same property; a data quality indicator. (USGS, 1998)

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

Quality Assurance Project Plan (QAPP): A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives. (Kammin, 2010; Ecology, 2004)

Quality control (QC): The routine application of measurement and statistical procedures to assess the accuracy of measurement data. (Ecology, 2004)

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

$$[\text{Abs}(a-b)/((a + b)/2)] * 100$$

where “Abs()” is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Replicate samples: Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled. (USGS, 1998)

Representativeness: The degree to which a sample reflects the population from which it is taken; a data quality indicator. (USGS, 1998)

Sample (field): A portion of a population (environmental entity) that is measured and assumed to represent the entire population. (USGS, 1998)

Sensitivity: In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit. (Ecology, 2004)

Spiked sample: A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency. (USEPA, 1997)

Split sample: A discrete sample subdivided into portions, usually duplicates (Kammin, 2010)

Standard Operating Procedure (SOP): A document which describes in detail a reproducible and repeatable organized activity. (Kammin, 2010)

References for QA Glossary

Ecology, 2004. Guidance for the Preparation of Quality Assurance Project Plans for Environmental Studies. <https://fortress.wa.gov/ecy/publications/SummaryPages/0403030.html>

USEPA, 1997. Glossary of Quality Assurance Terms and Related Acronyms. U.S. Environmental Protection Agency. <http://www.ecy.wa.gov/programs/eap/quality.html>

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process EPA QA/G-4. U.S. Environmental Protection Agency. <http://www.epa.gov/quality/qs-docs/g4-final.pdf>

USGS, 1998. Principles and Practices for Quality Assurance and Quality Control. Open-File Report 98-636. U.S. Geological Survey. <http://ma.water.usgs.gov/fhwa/products/ofr98-636.pdf>

Appendix 7

EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 8/18/2016

CarrierName: Hand Delivered

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184A

1 of 1

No: 10-081116-165338-0005

20162017B10P202BD4X24

Contact Name: Jed Januch

Contact Phone: (360) 871-8731

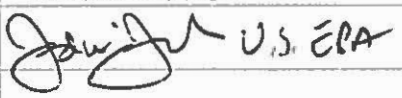
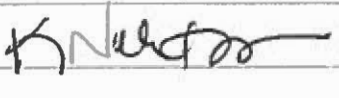
Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16334100		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	LNfH1influent	08/17/2016 12:34	Field Sample
16334101		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (3)	LNfH2effluent	08/17/2016 08:26	Field Sample
16334102		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 08:46	Field Sample
16334103		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 09:06	Field Sample
16334104		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 09:26	Field Sample
16334105		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 09:46	Field Sample
16334106		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 10:06	Field Sample
16334107		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 10:26	Field Sample
16334108		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 10:46	Field Sample
16334109		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 11:06	Field Sample
16334110		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNfH2effluent	08/17/2016 11:26	Field Sample

Shipment for Case Complete? Y

Sample(s) to be used for Lab QC: 16334101 Tag N1, 16334101 Tag N2, 16334101 Tag N3

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Sample Delivery	 U.S. EPA	8/18/16 1420		8/18/16 1420	

Appendix 7

EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 8/18/2016

CarrierName: Hand Delivered

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184A

1 of 1

No: 10-081116-165338-0005

20162017B10P202BD4X24

Contact Name: Jed Januch

Contact Phone: (360) 871-8731

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16334111		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNFH2effluent	08/17/2016 11:46	Field Sample
16334112		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNFH2effluent	08/17/2016 12:06	Field Sample
16334113		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNFH2effluent	08/17/2016 12:26	Field Sample
16334114		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNFH2effluent	08/17/2016 12:46	Field Sample
16334116		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	LNFH3rw	08/17/2016 13:45	Field Sample
16334117		Water/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	08/17/2016 08:05	QC Blank - Rinsate/Equipment
16334118		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LNFH2effluent	08/17/2016 09:46	Field Duplicate

Special Instructions:	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: Form=Formaldehyde	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Sample Delivery	Jed Januch U.S. EPA	8/18/16 1420	J. N. [Signature]	8/18/16 1420	

Appendix 7

Page 1 of 2

EPA R10 Lab (MEL) COC (REGION COPY)

07/14/16

Hand Carry

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184B

1 of 1

No: 10-070616-150810-0002


20162017B10P202BD4X24

Jed Januch

(360) 871-8731

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16284120		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	CNFH1influent	07/13/2016 02:15	Field Sample
16284121		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 12:20	Field Sample
16284122		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 12:30	Field Sample
16284123		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 12:40	Field Sample
16284124		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 12:50	Field Sample
16284125		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:00	Field Sample
16284126		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:10	Field Sample
16284127		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:20	Field Sample
16284128		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:30	Field Sample
16284129		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:40	Field Sample
16284130		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:50	Field Sample

Special Instructions:	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: Form=Formaldehyde	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Sample ship	 U.S. EPA	7/14/16 11:05	Karin Wood	7/14/16 11:05	

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EPA R10 Lab (MEL) COC (REGION COPY)

07/14/16

Hand Carry

CHAIN OF CUSTODY RECORD
Formaldehyde NW Aquaculture/WA
WTR-184B
1 of 1


No: 10-070616-150810-0002

20162017B10P202BD4X24

Jed Januch
(360) 871-8731

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16284131		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 02:00	Field Sample
16284132		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 02:10	Field Sample
16284133		Surface Water Total/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 01:21	Field Sample
16284134		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	CNFH3rw	07/13/2016 01:10	Field Sample
16284135		Surface Water Total/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	CNFH2effluent	07/13/2016 12:50	Field Duplicate
16284136		Water/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	07/13/2016 11:16	QC Blank - Rinsate/Equipment

Sample(s) to be used for Lab QC: 16284133 Tag N1	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: Form=Formaldehyde	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Samples 54, 1, 2	 U.S. EPA	7/14/16 11:05	Kim Wood	7/14/16 11:05	

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Page 1 of 2

EPA R10 Lab (MEL) COC (REGION COPY)

07/12/2016

UPS

1ZA4202V1596101284

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184C

1 of 1

No: 10-070616-144407-0001

20162017B10P202BD4X24

Jed Januch

(360) 871-8731


Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16284100		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:25	Field Sample
16284101		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 11:12	Field Sample
16284102		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 12:40	Field Sample
16284103		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 12:50	Field Sample
16284104		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:00	Field Sample
16284105		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:10	Field Sample
16284106		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:20	Field Sample
16284107		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:30	Field Sample
16284108		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:40	Field Sample
16284109		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 01:50	Field Sample
16284110		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influen t	07/11/2016 02:00	Field Sample

Special Instructions:

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Ship	 U.S. EPA	7/12/16 2:30P	Kim Wood	7/12/16 9:50	

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EPA R10 Lab (MEL) COC (REGION COPY)

07/12/2016

UPS

1ZA4202V1596101284

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184C

1 of 1

No: 10-070616-144407-0001

20162017B10P202BD4X24

Jed Januch

(360) 871-8731


Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16284111		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influent	07/11/2016 02:10	Field Sample
16284112		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influent	07/11/2016 02:20	Field Sample
16284114		Surface Water Total/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influent	07/11/2016 12:09	Field Sample
16284115		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influent	07/11/2016 02:00	Field Sample
16284116		Surface Water Total/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	LWSNFH1influent Ex	07/11/2016 01:30	Field Duplicate
16284117		Water/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	07/11/2016 10:50	QC Blank - Rinsate/Equipment

Sample(s) to be used for Lab QC: 16284114 Tag N1

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Ship	 U.S. EPA	7/12/16 2:30p	Kum Wood	7/13/16 9:50	

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Page 1 of 2

EPA R10 Lab (MEL) COC (REGION COPY)

07/19/16

UPS

1ZA4202V1596381919

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184D

1 of 1

No: 10-071216-151959-0003

20162017B10P202BD4X24

Jed Januch

(360) 871-8731

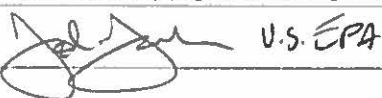
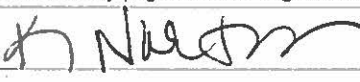
Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16294100		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	DNFH1influent	07/18/2016 14:04	Field Sample
16294101		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 11:03	Field Sample
16294102		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 11:13	Field Sample
16294103		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 11:23	Field Sample
16294104		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 11:33	Field Sample
16294105		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 11:43	Field Sample
16294106		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 N2 N3 (None) (3)	DNFH2effluent	07/18/2016 11:53	Field Sample
16294107		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 12:03	Field Sample
16294108		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 12:13	Field Sample
16294109		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 12:23	Field Sample
16294110		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 12:33	Field Sample

Sample(s) to be used for Lab QC: 16294106 Tag N3

Shipment for Case Complete? N

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Shipping	 U.S. EPA	7/19/16 1449	 Jed Januch	7/20/16 10:00	

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EPA R10 Lab (MEL) COC (REGION COPY)

07/19/16

UPS

1ZA4202V1596381919

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184D

1 of 1

No: 10-071216-151959-0003

20162017B10P202BD4X24

Jed Januch

(360) 871-8731

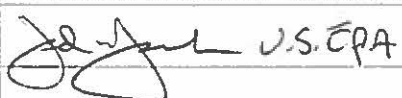

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16294111		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 12:43	Field Sample
16294112		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 12:53	Field Sample
16294113		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 13:03	Field Sample
16294114		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	DNFH3rw	07/18/2016 14:30	Field Sample
16294115		Surface Water Total/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	DNFH2effluent	07/18/2016 13:30	Field Sample
16294116		Water/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	07/18/2016 11:17	QC Blank - Rinsate/Equipm ent

Special Instructions:

Shipment for Case Complete? N

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Shipping	 U.S. EPA	7/19/16 1449		7/20/16 1000	

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EPA R10 Lab (MEL) COC (REGION COPY)

07/21/16

Hand Delivered

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184E

1 of 1

No: 10-071516-164155-0004

20162017B10P202BD4X24

Jed Januch

(360) 871-8731

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16294120		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N1 (None) (1)	CIWFH1influent	07/20/2016 11:35	Field Sample
16294121		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 09:20	Field Sample
16294122		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 09:30	Field Sample
16294123		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 09:40	Field Sample
16294124		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 09:50	Field Sample
16294125		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:00	Field Sample
16294126		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:10	Field Sample
16294127		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:20	Field Sample
16294128		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:30	Field Sample
16294129		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:40	Field Sample
16294130		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:50	Field Sample

Special Instructions:	Shipment for Case Complete? N
	Samples Transferred From Chain of Custody #
Analysis Key: Form=Formaldehyde	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Delivery	Jed Januch U.S. EPA	7/20/16 10:00 PM	Kim Wood	7/21/16 9:20	good hand delivered

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EPA R10 Lab (MEL) CDC (REGION COPY)

07/21/16

Hand Delivered

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

WTR-184E

1 of 1

No: 10-071516-164155-0004

20162017B10P202BD4X24

Jed Januch

(360) 871-8731

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16294131		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 11:00	Field Sample
16294132		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 11:10	Field Sample
16294133		Surface Water Total/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:36	Field Sample
16294134		Surface Water Total/ J Januch	Composite	Form(8 Weeks)	N3 (None) (3)	CIWFH3rw	07/20/2016 12:47	Field Sample
16294135		Water/ J Januch	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	07/20/2016 07:47	QC Blank - Rinsate/Equipment
16294136		Surface Water Total/ J Januch	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CIWFH2effluent	07/20/2016 10:30	Field Duplicate

Sample(s) to be used for Lab QC: 16294134 Tag N3	Shipment for Case Complete? N
Analysis Key: Form=Formaldehyde	Samples Transferred From Chain of Custody #

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
Delivery	Jed Januch J.S.EPA	7/21/16 10:30 PM	Kim Wood	7/21/16 9:20	good Hand Delivered

Appendix 8

Page 1 of 2

EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 9/12/2016

CarrierName: Hand Delivered

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184F

1 of 1

No: 10-090816-164101-0006

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16375100		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	KFH_influent	09/12/2016 14:38	Field Sample
16375101		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (3)	KFH_Effluent	09/12/2016 11:06	Field Sample
16375102		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 11:26	Field Sample
16375103		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 11:46	Field Sample
16375104		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 12:06	Field Sample
16375105		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 12:26	Field Sample
16375106		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 12:46	Field Sample
16375107		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 13:06	Field Sample
16375108		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 13:26	Field Sample
16375109		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 13:46	Field Sample
16375110		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 14:06	Field Sample

Sample(s) to be used for Lab QC: 16375101 Tag N1, 16375101 Tag N2, 16375101 Tag N3 - Special Instructions: Samples collected by Ecology and delivered by EPA/Januch. Questions regarding Scribe file - contact Jed Januch.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
			Kunni wood	9/13/16 8:05	good

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EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 9/12/2016

CarrierName: Hand Delivered

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184F

1 of 1

No: 10-090816-164101-0006

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16375111		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 14:26	Field Sample
16375112		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 14:46	Field Sample
16375113		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 13:07	Field Sample
16375114		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	KFH_rw	09/12/2016 14:57	Field Sample
16375115		Water/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	09/12/2016 09:52	QC Blank - Rinsate/Equipment
16375116		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	KFH_Effluent	09/12/2016 12:46	Field Duplicate

Special Instructions: Samples collected by Ecology and delivered by EPA/Januch. Questions regarding Scribe file - contact Jed Januch.	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: Form=Formaldehyde	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
			<i>Kim Wood</i>	<i>9/12/16 8:05</i>	<i>good</i>

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EPA R10 Lab (MEL) COC (REGION COPY)

Date Shipped: 9/19/2016

Carrier Name: FedEx

Airbill No.:

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184G

1 of 1

No: 10-091516-140459-0007

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16385200		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	WRFH_influent	09/19/2016 15:30	Field Sample
16385201	A	Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (3)	WRFH_Effluent	09/19/2016 11:35	Field Sample
16385202		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 11:55	Field Sample
16385203		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 12:15	Field Sample
16385204		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 12:35	Field Sample
16385205		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 12:55	Field Sample
16385206		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 13:15	Field Sample
16385207		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 13:35	Field Sample
16385208		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 13:55	Field Sample
16385209		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 14:15	Field Sample
16385210		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 14:35	Field Sample

Sample(s) to be used for Lab QC: 16385201 Tag N1, 16385201 Tag N2, 16385201 Tag N3 - Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
		9/20/16 09:10		9/20/16 07:15	2 CUSTODY TAGS Custody INITIALS
				9/20/16 9:10	

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EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 9/19/2016

CarrierName: FedEx

AirbillNo: :

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184G

1 of 1

No: 10-091516-140459-0007

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16385211		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 14:53	Field Sample
16385212		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 15:15	Field Sample
16385213		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 15:10	Field Sample
16385214		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	WRFH_rw	09/19/2016 16:15	Field Sample
16385215		Water/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	09/19/2016 10:45	QC Blank - Rinsate/Equipment
16385216		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_Effluent	09/19/2016 14:55	Field Duplicate
16385217		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_EggTreatmentEffluent	09/19/2016 12:45	Field Duplicate
16385218		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_EggTreatmentEffluent	09/19/2016 13:14	Field Duplicate
16385219		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	WRFH_EggTreatmentEffluent	09/19/2016 14:10	Field Duplicate

Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	Siana Wong	9-19-16	[Signature]	9/20/16 07:15	2 CUSTOM TAPES
	[Signature]	9/20/16 09:16	[Signature]	9/20/16 710	COOLERS INTACT

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EPA R10 Lab (MEL) COC (REGION COPY)

Date Shipped: 9/30/2016

Carrier Name: FedEx

Airbill No.:

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184H

1 of 1

No: 10-092816-174017-0008

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16395271		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 14:45	Field Sample
16395250		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	CSFH_influent	09/30/2016 15:00	Field Sample
16395251		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (3)	CSFH_Effluent	09/30/2016 10:00	Field Sample
16395253		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 10:30	Field Sample
16395254		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 11:00	Field Sample
16395255		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 11:30	Field Sample
16395256		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 12:00	Field Sample
16395257		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 12:30	Field Sample
16395258		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 13:00	Field Sample
16395259		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 13:30	Field Sample
16395260		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 14:00	Field Sample

Sample(s) to be used for Lab QC 16395271 Tag N1, 16395251 Tag N1, 16395251 Tag N2, 16395251 Tag N3 - Special Instructions. Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
			Kurn Wood	8:20	10/3/16

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Page 2 of 2

EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 9/30/2016

CarrierName: FedEx

AirbillNo: :

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184H

1 of 1

No: 10-092816-174017-0008

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16395261		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 14:30	Field Sample
16395262		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 15:00	Field Sample
16395263		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 15:30	Field Sample
16395264		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 16:00	Field Sample
16395265		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	CSFH_rw	09/30/2016 16:40	Field Sample
16395266		Water/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	09/30/2016 09:30	QC Blank - Rinsate/Equipment
16395267		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	CSFH_Effluent	09/30/2016 14:00	Field Duplicate

Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
			Kumi Ward	8:20	10/3/16

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EPA R10 Lab (MEL) COC (REGION COPY)

Date Shipped: 11/7/2016

Carrier Name: Hand Delivered

Airbill No.:

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-1841

1 of 1

No: 10-110216-135814-0009

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16444000		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	PRFH_influent	11/04/2016 1400	Field Sample
16444001		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (3)	PRFH_Effluent	11/04/2016 0940	Field Sample
16444002		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1000	Field Sample
16444003		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1020	Field Sample
16444004		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1040	Field Sample
16444005		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1100	Field Sample
16444006		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1120	Field Sample
16444007		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1140	Field Sample
16444008		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1200	Field Sample
16444009		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1220	Field Sample
16444010		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1240	Field Sample

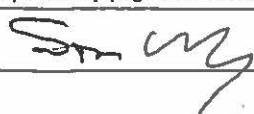
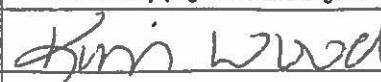
DLP

Sample(s) to be used for Lab QC: 16444001 Tag N1, 16444001 Tag N2, 16444001 Tag N3 - Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
		11-4-16 19:20		11/7/16 10:15	

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EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 11/7/2016

CarrierName: Hand Delivered

AirbillNo: :

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184I

1 of 1

No: 10-110216-135814-0009

20162017B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16444011		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1300	Field Sample
16444012		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1320	Field Sample
16444013		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1340	Field Sample
16444014		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	PRFH_rw	11/04/2016 1400	Field Sample
16444015		Water/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	11/04/2016 0900	QC Blank - Rinsate/Equipment
16444016		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1140	Field Duplicate
16444017		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/04/2016 1120	Field Sample
16444018		Surface Water Total/ S.Wong	GRAB	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent - DIRECT	1055	Field Sample

4007 dup

Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	Siana Wong	11/4/16 14:20	Kenn Wood	10:15	
				11/7/16	

Formaldehyde NW Aquaculture/WA
 Appendix 8
 FOR LEON-TAKE TO EIM LAB.
 Thanks

EPA R10 Lab (MEL) COC (REGION COPY)
 DateShipped: 12/1/2016
 CarrierName: Hand Delivered
 AirbillNo: NA

CHAIN OF CUSTODY RECORD
 Formaldehyde NW Aquaculture/WA
 Project Code: WTR-1841
 1 of 1

No: 10-112916-133120-0010
 20172018B10P202BD4X24
 Contact Name: Siana Wong
 Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16484000		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	PRFH_influent	11/30/2016 15:15	Field Sample
16484001		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (3)	PRFH_Effluent	11/30/2016 11:30	Field Sample
16484002		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 11:50	Field Sample
16484003		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 12:10	Field Sample
16484004		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 12:30	Field Sample
16484005		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 12:50	Field Sample
16484006		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 13:10	Field Sample
16484007		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 13:30	Field Sample
16484008		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 13:50	Field Sample
16484009		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 14:10	Field Sample
16484010		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 14:30	Field Sample

DUP

Sample(s) to be used for Lab QC: 16484001 Tag N1, 16484001 Tag N2, 16484001 Tag N3 - Special Instructions: Samples collected and shipped by Ecology. EGGS ONLY. RSCC needs ecology sample times and associated collection info emailed on day of shipment.	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	Siana W	11-30-16/20:00	walk in cooler at OC	11-30-16/20:00	
	OC walk in	12/01/16 07:30	[Signature]	12/01/16 07:30	CUSTODY TAG IN PLACE
	[Signature]	12/01/16 09:10	[Signature]	12/1/16 9:30	

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EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 12/1/2016

CarrierName: Hand Delivered

AirbillNo: NA

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-1841

1 of 1

No: 10-112916-133120-0010

20172018B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-8432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16484011		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 14:50	Field Sample
16484012		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 15:10	Field Sample
16484013		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 15:30	Field Sample
16484014		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	PRFH_rw	11/30/2016 15:45	Field Sample
16484015		Water/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	11/30/2016 09:00	QC Blank - Rinsate/Equipment
16484016		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 14:30	Field Duplicate
16484017		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent	11/30/2016 14:00	Field Sample
16484018		Surface Water Total/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	PRFH_Effluent Direct	11/30/2016	Field Sample

Dup of #10

Not collected

Special Instructions: Samples collected and shipped by Ecology. EGGS ONLY. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	<i>Siana Wong</i>	11-30-16/20:00	<i>WALKER</i>	11-30-16, 20:00	
	<i>OC WALKER</i>	12/01/16 01:30	<i>WALKER</i>	12/01/16 07:30	CUSTOMER TAKE INITIALS
	<i>WALKER</i>	12/01/16 09:10	<i>WALKER</i>	12/1/16	
				9:30	

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COPY

EPA R10 Lab (MEL) COC (REGION COPY)

DateShipped: 12/8/2016

CarrierName: Hand Delivered

AirbillNo: NA

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184K

1 of 1

No: 10-112916-204545-0011

20172018B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16494100		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	HFH_influent	12/07/2016 1430	Field Sample
16494101		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None), N2 (None), N3 (None) (2)	HFH_Effluent	12/07/2016 1720	Field Sample
16494102		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1430	Field Sample
16494103		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1440	Field Sample
16494104		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1450	Field Sample
16494105		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1500	Field Sample
16494106		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1510	Field Sample
16494107		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1520	Field Sample
16494108		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1530	Field Sample
16494109		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1540	Field Sample
16494110		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 1550	Field Sample

N2+N3 not collected

DUP

Sample(s) to be used for Lab QC: 16494101 Tag N1, 16494101 Tag N2, 16494101 Tag N3 - Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: Form=Formaldehyde	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	Stark Wong	12-7-16 18:50	CC-Walkin Locker	12-7-16 18:50	
			Turn Ward	12/8/16 9:35	

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EPA R10 Lab (MEL) COC (REGION COPY)

Date Shipped: 12/8/2016

Carrier Name: Hand Delivered

Airbill No: NA

CHAIN OF CUSTODY RECORD

Formaldehyde NW Aquaculture/WA

Project Code: WTR-184K

1 of 1

No: 10-112916-204545-0011

20172018B10P202BD4X24

Contact Name: Siana Wong

Contact Phone: (360) 407-6432

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
16494111		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 16:00	Field Sample
16494112		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 16:10	Field Sample
16494113		Surface Water Total/ S.Wong	Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 16:20	Field Sample
16494114		Surface Water Total/ S.Wong	Composite	Form(8 Weeks)	N1 (None) (1)	HFH_rw	12/07/2016 16:20	Field Sample
16494115		Water/ S.Wong	Grab	Form(8 Weeks)	N1 (None) (1)	Blank	12/07/2016 1300	QC Blank - Rinsate/Equipment
16494116		Surface Water Total/ S.Wong	Discrete Interval GRAB	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 15:15	Field Duplicate Field Sample
16494117		Surface Water Total/ S.Wong	Grab Discrete Interval	Form(8 Weeks)	N1 (None) (1)	HFH_Effluent	12/07/2016 15:30	Field Sample Field Duplicate

Special Instructions: Samples collected and shipped by Ecology. RSCC needs ecology sample times and associated collection info emailed on day of shipment.

Shipment for Case Complete? Y

Samples Transferred From Chain of Custody #

Analysis Key: Form=Formaldehyde

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt
	Siana Wong	12-7-16, 15:50	CC-Walk-in cooler	12-7-16	
			Kenn Wood	12-8-16 9:35	



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