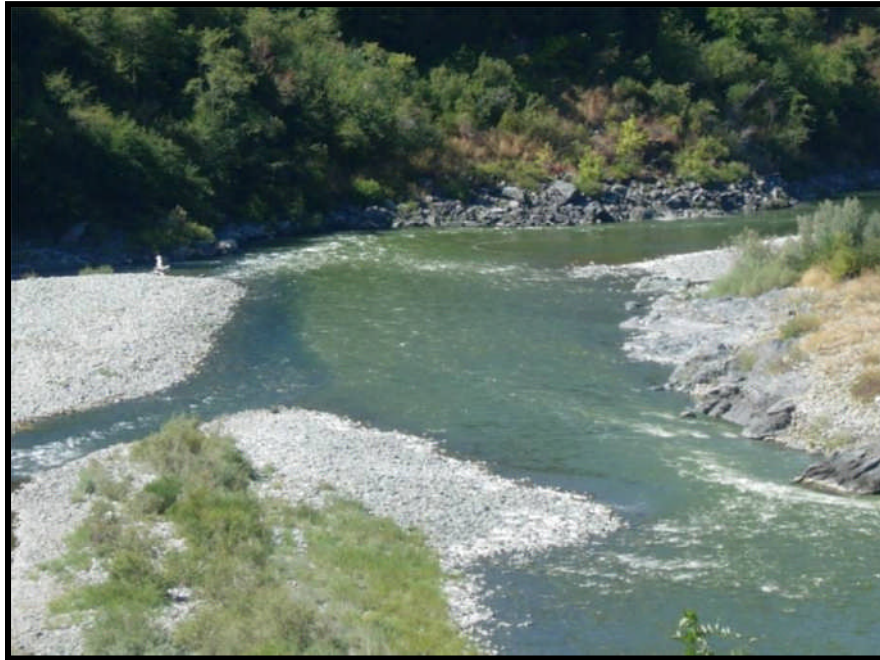


**Preliminary 2010 Microcystin Bioaccumulation Results for
Klamath River Salmonids** (Updated 4-7-2011).



Prepared By

Jacob Kann, Ph.D. and Lisa Bowater

Aquatic Ecosystem Sciences LLC

295 East Main St., Suite 7

Ashland, OR97520

and

Grant Johnson and Crystal Bowman

Karuk Tribe Department of Natural Resources

Prepared For

Karuk Tribe Department of Natural Resources

PO Box 282

Orleans, CA95556

INTRODUCTION

Copco and Iron Gate Reservoirs (the lowermost projects of PacifiCorp's Klamath Hydropower Project-- KHP) experienced extensive blooms of toxigenic *Microcystis aeruginosa* (MSAE) from 2004-2009 (Kann and Corum 2009; 2010; Jacoby and Kann 2007). These blooms have been associated with high levels of the cyanotoxin microcystin, a potent hepatotoxin capable of causing chronic liver damage and acting as a tumor promoter (Carmichael 1995; Chorus et al. 1999; Chorus 2001).

The results of the 2005-2009 sampling program demonstrated widespread and abundant seasonal blooms of toxigenic MSAE in Copco and Iron Gate reservoirs and in the Klamath River downstream. These yearly bloom events consistently exceed World Health Organization (WHO 1999) and California State Water Resources Control Board (SWRCB 2010) public health threshold levels for both MSAE cell density and microcystin toxin by 10 to over 1000 times. The blooms vary in duration and severity in the free flowing-section of the River but have generally been present at some level during the August through October period (Kann and Corum 2009; Fetcho 2009).

Due to the overlap in timing of the toxic algal blooms and run-timing of salmonids that serve as a food source, the potential for bioaccumulation of microcystins exists both as a public health concern and as a contributor to fish stress and disease. For example, the Yurok Tribe fishes for fall Chinook starting in August, and fall Chinook reach the Karuk fishery in September. Fall steelhead enter the Klamath River in late summer; are in the mid-Klamath River by September or October, and reach Iron Gate hatchery by November. Salmonids are also caught and consumed by recreational fishermen and are sold in the Yurok commercial fishery.

Initial field sampling of salmonid fish tissue for public health was conducted by the Yurok Tribe in 2005, when a small number of fish livers and fillets were collected from the Klamath River between mid-September and early October (Fetcho 2006). Of the 5 Chinook livers, 4 Chinook fillets, 2 steelhead livers, and 2 steelhead fillets sampled, a trace amount of microcystin was detected in one steelhead liver, and 0.54 µg/g microcystin was found in the other steelhead liver (Fetcho 2006). In addition, bioaccumulation studies undertaken in 2007 showed accumulation of microcystin toxin in muscle and/or liver tissues of yellow perch, Irongate hatchery salmon, and freshwater mussels (Mekebri et al. 2009; Kann 2008; Kanz 2008). Microcystin levels in many of these samples exceeded public health threshold values for safe consumption (Kann 2008; OEHHA 2008).

Although other studies of Klamath River salmon and steelhead in 2007, and yellow perch in 2008 and 2009, did not show microcystin bioaccumulation in tissues (e.g., CH2MHILL 2009; Prendergast and Foster 2010), histological examination of liver tissues determined that lesions were present in liver tissue from both salmonid species (CH2M HILL 2009). Substantial bioaccumulation continued to be shown in freshwater mussels throughout the Klamath River below Irongate Dam in 2009 (Kann et al. 2010).

Given previous results showing the presence of microcystin in salmon and steelhead livers, and that in the mainstem of the Klamath River adult salmonids are an important subsistence food for Tribal people, additional salmonid sampling for microcystin bioaccumulation was conducted in

2010 by the Karuk Tribe. Although histological results have not yet been received from the laboratory, the following memo provides results for the presence of microcystin in salmonid tissues during the fall of 2010. The memo will be updated when completed histology results are received.

METHODS

Klamath River fish samples were collected at five locations during September through November; Orleans, Ishi Pishi Falls, Weitchpec, Happy Camp, and Irongate hatchery (Figure 1). Fish were collected using hook and line at Weitchpec, Orleans, and Happy Camp and by traditional dip net at Ishi Pishi Falls. Fish from Irongate hatchery were collected after being spawned. Samples for *Microcystis* and microcystin toxin were also collected at a series of stations as part of the Karuk Tribe's public health monitoring program (Figure 2). In September, 10 livers and fillets were collected from fall steelhead and 7 livers and fillets were collected from fall Chinook (Figure 3). In October, 15 livers and fillets were collected from steelhead, and 7 livers and fillets from Chinook. In November, 6 liver and fillets were collected from Chinook and 3 livers and 2 fillets from Coho. Fillet and liver samples were sent to the California Department of Fish and Game (CDFG) lab for microcystin analysis and various organs were sent to the University of California, Davis for histological examination.

CDFG Protocol

Samples of five fish were collected per sample period. Fish tissue and liver samples consisted of 5-10 grams of tissue and 5-10grams of liver. Samples were placed in aluminum foil and Ziploc bags and then frozen. The samples were shipped with ice overnight to Dr. Abdou Mekebri at the Fish and Wildlife Water Pollution Control Lab (WPCL) in Rancho Cordova, CA for microcystin analysis by LCMS/MS (Mekebri et al. 2009). Chain of Custody forms are shown in Appendix I. Two of the September Chinook liver samples were split at the lab for quality assurance purposes and are labeled with "Dup" following the WPCL Lab number (Appendix II).

Histology Protocol

Fish samples were refrigerated for a maximum of three days. Photos were taken of whole fish and organs; ocular examination was performed for anomalies in tissues and gills to look for *Columnaris*. Various tissue types (e.g., gills, heart, liver, etc.) were grouped into separate containers. These containers were filled with a 10% formalin solution using a minimum 1:10 ratio by volume of tissues to formalin to ensure preservation of tissues.

Organ removal occurred as follows: fish guts were removed whole (GI tract, spleen, heart); gill arches removed (at least 1 side); liver removed (sampled at least 2 sections from 2 different locations assuring not to cut into the gallbladder). The fillet and kidney were removed as an x-section cut perpendicular to the backbone. This piece of kidney, backbone, and muscle was no more than 1cm thick to insure saturation of preservative. The tissue samples were rinsed with saline solution to minimize superficial blood. The presence of blood on tissue samples will darken upon exposure to formalin reducing the quality of slide mounts. The head was cut off (after the gill arches were removed) and shipped fresh for lab removal of the brain for September

samples only. Samples were shipped weekly on ice to Melissa A. Miller, DVM, PhD at the Marine Wildlife Veterinary Care and Research Center Department of Fish and Game and University of California, Davis in Santa Cruz, CA. Histological results are pending and are not included here.



Figure 1. 2010 Fish sampling locations: Clockwise from top left; Iron Gate hatchery, Weitchpec, Ishi Pishi Falls, and Orleans (Happy Camp location not shown).

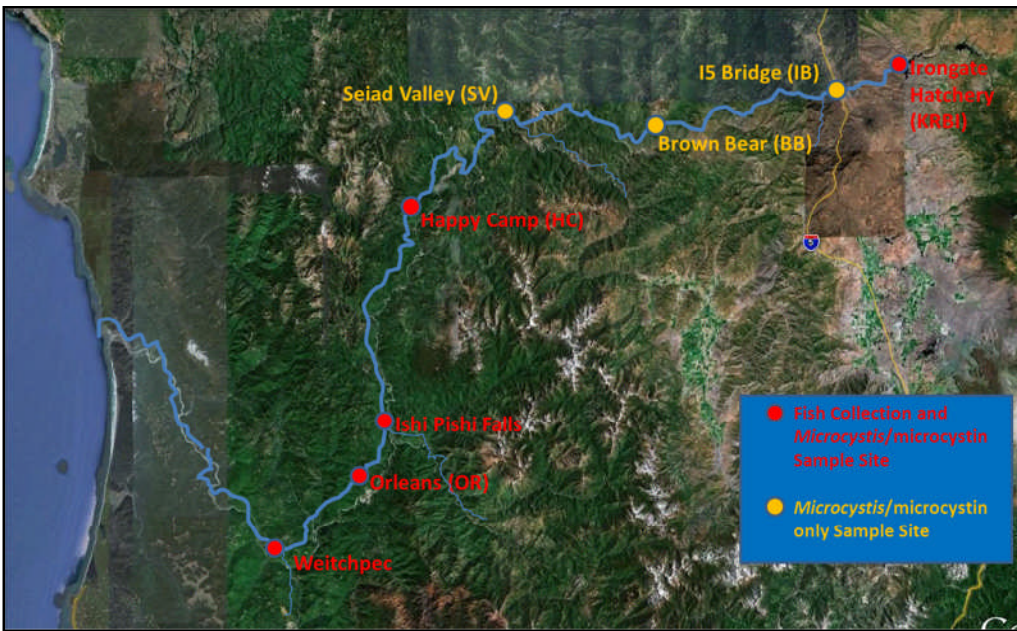


Figure 2. Klamath River fish and *Microcystis* sampling locations, 2010.



Figure 3. Example of 2010 Fish samples: clockwise from top left: Chinook from Isbi Pishi- IP092810_1C, Chinook liver from Isbi Pishi- IP092910_1C_Liver, Steelhead from Orleans- OR092310_1S and Chinook from Isbi Pishi- IP092910_1C.

RESULTS/DISCUSSION

Results from salmonid tissue samples collected by the Karuk Tribe in September, 2010 showed that 3 of 7 Chinook livers collected below Happy Camp at Ishi Pishi Falls had detectable levels of microcystin-RR (Table 1; Figure 4). Microcystin was not detected in any of the other September fish samples. Samples collected on the 14th and 15th of October showed that 1 of 7 Chinook livers had a high level of microcystin-RR (121 ppb), and 1 of 15 steelhead livers had a high level of microcystin-LR (152 ppb), both exceeding public health guideline levels (Table 1; Figure 4). Microcystin was not detected in fish tissue samples during any of the other October or November samples. Aside from microcystin, none of the other measured algal toxins were detected in any of the Klamath River fish samples (Table 1; i.e., anatoxin-a, domoic acid, or okadaic acid). Duplicate samples analyzed on two of the September fish showed good agreement (Table 1), and internal lab QA recoveries were generally 100% \pm 20%, although a few were closer to 75% (Figure 5; Appendix II).

During the period the Chinook were collected, the 2010 longitudinal microcystin (total microcystin as determined by ELISA) and *Microcystis* public health sampling data showed that ambient levels of both *Microcystis* cells and microcystin toxin increased in the Klamath River during mid-September, exceeding public health guideline values at nearly all stations (Figures 6 and 7). Although microcystin values declined somewhat during the third week in September, they then rebounded in late September and into early October (Figures 6 and 7). These results indicate that microcystin was being transported downstream to areas where Chinook and steelhead were migrating upstream, and that fish collected during the September and October efforts were likely exposed to microcystin either prior to or during the collection period. Microcystin levels then declined to levels that were below detection during the November fish sampling period.

In addition to total microcystin as determined by ELISA during the course of regular public health sampling (e.g., Figure 7), samples were also periodically collected to specifically determine the presence of various microcystin congeners in Klamath River water samples (Table 2). These data show only two congeners to be detected, microcystin-LR early in the season at station IB, and microcystin-LA during August through October at various stations (Figure 8).

These data indicate that congeners bioaccumulated in Klamath River salmonids do not match the ambient data with respect to detected microcystin congeners or variants. For example, of the five fish showing positive bioaccumulation, four showed the presence of microcystin-RR, and one microcystin-LR. Moreover, even though microcystin-LR was detected in ambient water earlier in the season, only -LA was detected during the period bracketing the October 15th steelhead that showed 152 ppb of microcystin -LR. The reason for this difference between ambient and bioaccumulated microcystin is unclear. However, a similar trend was noted in Klamath River freshwater mussels (Kann et al. 2010). A potential explanation is differential uptake, whereby even though the concentrations of -RR and -LR were below detection in ambient water, they could still have been present at low levels and were then differentially accumulated through the bioaccumulation process.

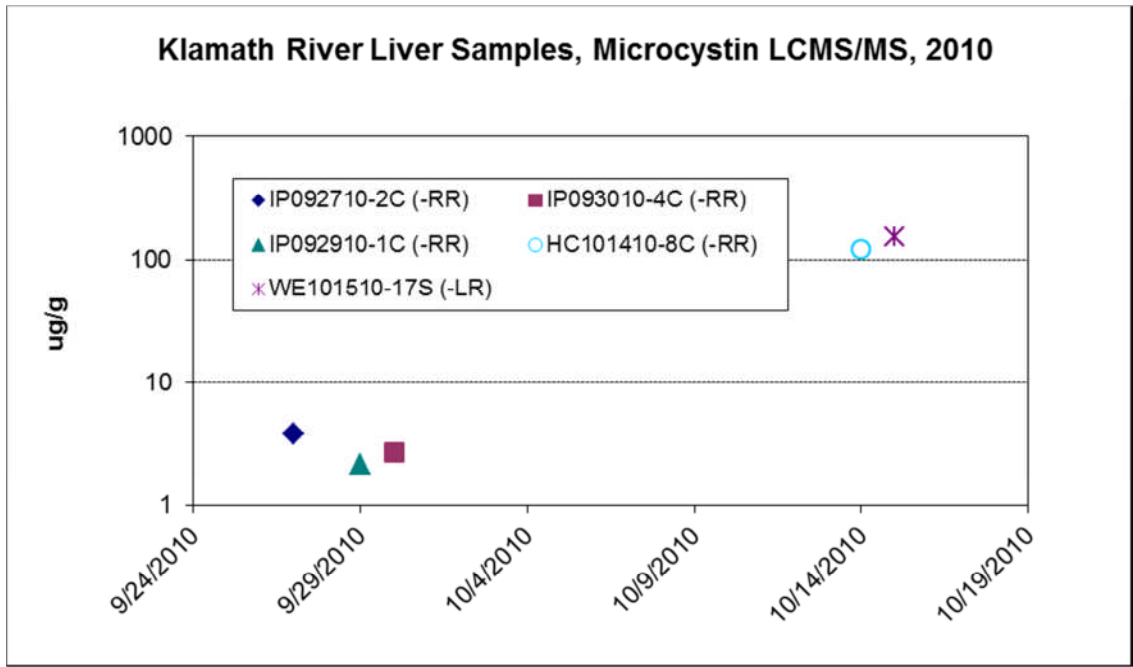


Figure 4. Microcystin concentration in Klamath River Salmonid Liver Samples, 2010 (showing occurrence of positive hits only—see data in Table 1).

Table 1. 2010 Fish and Wildlife Water Pollution Control Lab Toxin Results for microcystin bioaccumulation in Klamath River Salmonids.

2010 Adult Salmonid Toxin Results			Estimated MDL (ppb)	0.500	0.500	0.500	0.500	0.500	0.500	0.500	0.500	5.00	2.00	1.00	
			Reporting Limit (ppb)	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	10.0	5.00	2.00
WPCL Lab#	Sample Identification	Date Collected	Matrix (Fresh Weight)	MC-RR (ppb)	MC-Desmethyl-RR* (ppb)	MC-LR (ppb)	MC-Desmethyl-LR (ppb)	MC-YR (ppb)	MC-LA (ppb)	MC-LW (ppb)	MC-LF (ppb)	MC-LY (ppb)	Anatoxin A (ppb)	Domoic acid (ppb)	Okadaic acid (ppb)
L-620-10-1	OR092310-1S	9/23/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-2	OR092310-1S	9/23/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-3	OR092310-2S	9/23/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-4	OR092310-2S	9/23/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-5	OR092610-3S	9/26/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-6	OR092610-3S	9/26/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-7	OR092610-4S	9/26/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-8	OR092610-4S	9/26/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-9	OR092610-5S	9/26/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-10	OR092610-5S	9/26/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-13	IP092710-2C	9/27/2010	fish liver	3.80	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-13Dup	IP092710-2C	9/27/2010	fish liver	2.93	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-14	IP092710-2C	9/27/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-15	IP092710-3C	9/27/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-16	IP092710-3C	9/27/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-11	IP092810-1C	9/28/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-620-10-12	IP092810-1C	9/28/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-1	IP093010-4C	9/30/2010	fish liver	2.71	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-2	IP093010-4C	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-3	IP092910-1C	9/30/2010	fish liver	2.17	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-3Dup	IP092910-1C	9/30/2010	fish liver	2.10	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-4	IP092910-1C	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-5	WE092910-5S	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-6	WE092910-5S	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-7	IP093010-3C	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-8	IP093010-3C	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-9	IP092910-2C	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-10	IP092910-2C	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-11	WE092910-1S	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-12	WE092910-1S	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-13	WE092910-2S	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-14	WE092910-2S	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-15	WE092910-4S	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-16	WE092910-4S	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-17	WE092910-3S	9/30/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-641-10-18	WE092910-3S	9/30/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-1	HC101410-8C	10/14/2010	fish liver	121.20	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-2	HC101410-8C	10/14/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

WPCL Lab#	Sample Identification	Date Collected	Matrix (Fresh Weight)	MC-RR (ppb)	MC-Desmethyl-RR* (ppb)	MC-LR (ppb)	MC-Desmethyl-LR (ppb)	MC-YR (ppb)	MC-LA (ppb)	MC-LW (ppb)	MC-LF (ppb)	MC-LY (ppb)	Anatoxin A (ppb)	Domoic acid (ppb)	Okadaic acid (ppb)
L-678-10-3	HC101410-11S	10/14/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-4	HC101410-11S	10/14/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-5	HC101410-12S	10/14/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-6	HC101410-12S	10/14/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-7	HC101410-13S	10/14/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-8	HC101410-13S	10/14/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-9	HC101410-14S	10/14/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-10	HC101410-14S	10/14/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-11	HC101410-15S	10/14/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-12	HC101410-15S	10/14/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-13	WE101510-16S	10/15/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-14	WE101510-16S	10/15/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-15	WE101510-17S	10/15/2010	fish liver	ND	ND	152.40	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-16	WE101510-17S	10/15/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-17	WE101510-18S	10/15/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-18	WE101510-18S	10/15/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-19	WE101510-19S	10/15/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-20	WE101510-19S	10/15/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-21	WE101510-20S	10/15/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-22	WE101510-20S	10/15/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-23	OR101710-21S	10/17/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-24	OR101710-21S	10/17/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-25	OR101710-22S	10/17/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-26	OR101710-22S	10/17/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-27	OR101710-23S	10/17/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-28	OR101710-23S	10/17/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-29	OR101710-24S	10/17/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-30	OR101710-24S	10/17/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-31	OR101710-25S	10/17/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-32	OR101710-25S	10/17/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-33	IG101810-9C	10/18/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-34	IG101810-9C	10/18/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-35	IG101810-10C	10/18/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-36	IG101810-10C	10/18/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-37	IG101810-11C	10/18/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-38	IG101810-11C	10/18/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-39	IG101810-12C	10/18/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-40	IG101810-12C	10/18/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-41	IG101810-13C	10/18/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-42	IG101810-13C	10/18/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-43	IG101810-14C	10/18/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-678-10-44	IG101810-14C	10/18/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-1	IG110110-15C	11/1/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-2	IG110110-15C	11/1/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-3	IG110110-16C	11/1/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-4	IG110110-16C	11/1/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-5	IG110110-17C	11/1/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-6	IG110110-17C	11/1/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-7	IG110110-18C	11/1/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-8	IG110110-18C	11/1/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-9	IG110110-19C	11/1/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-10	IG110110-19C	11/1/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-11	IG110110-20C	11/1/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-711-10-12	IG110110-20C	11/1/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-742-10-1	IG112910-1CO	11/29/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-742-10-2	IG112910-1CO	11/29/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-742-10-3	IG112910-2CO	11/29/2010	fish fillet	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-742-10-4	IG112910-2CO	11/29/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
L-742-10-5	IG112910-3CO	11/29/2010	fish liver	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

* Desmethyl-RR quantified as parent analog compound.

**Sample ID ending in 'S' denotes Steelhead , Sample ID ending in 'C' denotes Chinook and Sample ID ending in 'CO' denotes Coho.

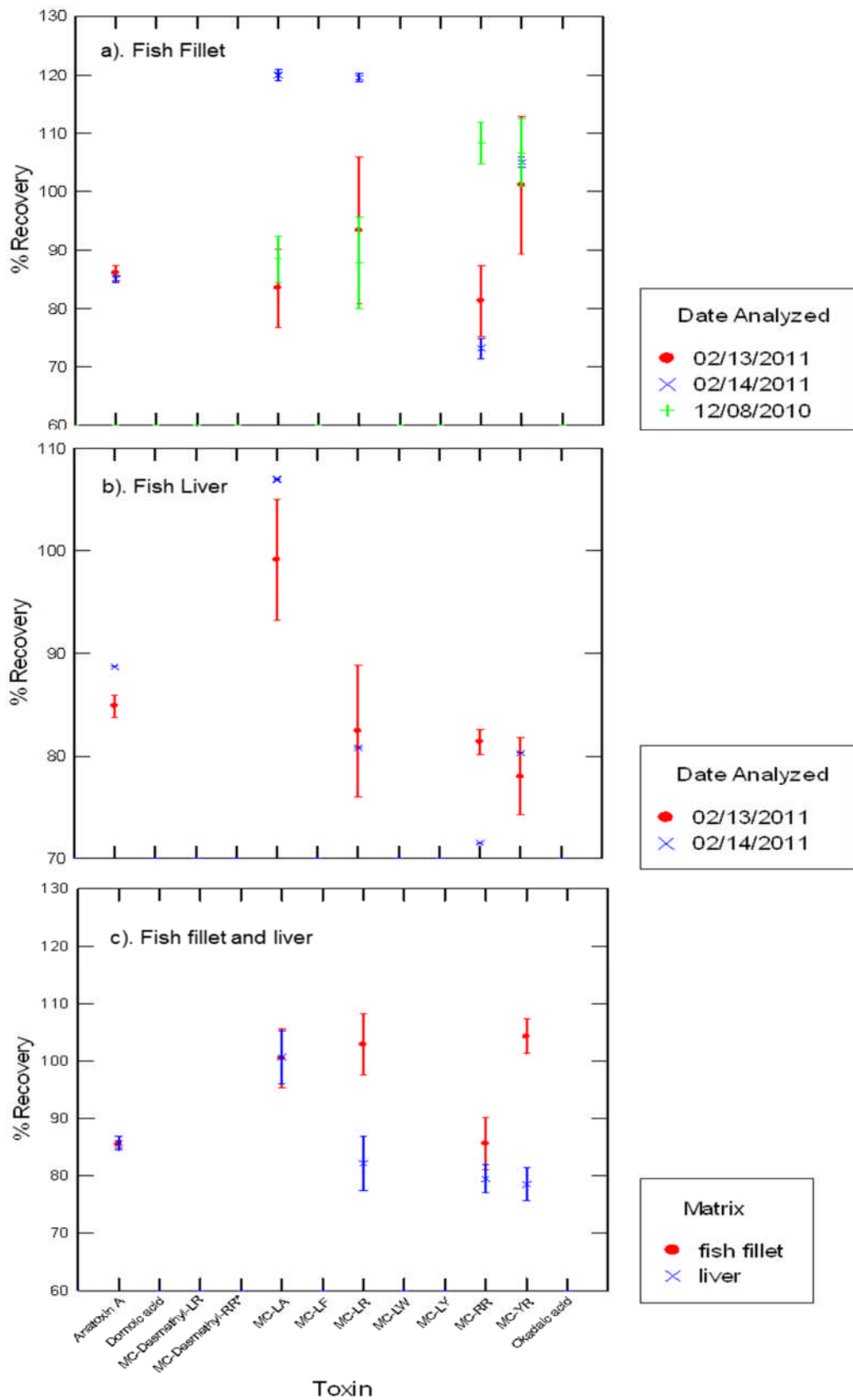


Figure 5. 2010 Fish and Wildlife Water Pollution Control Lab; Quality control analysis, percent recovery of spiked toxin in fish fillets (a), fish livers (b) and fillets and livers combined (c). Data plotted by date samples were analyzed; shown with mean and standard error bars.

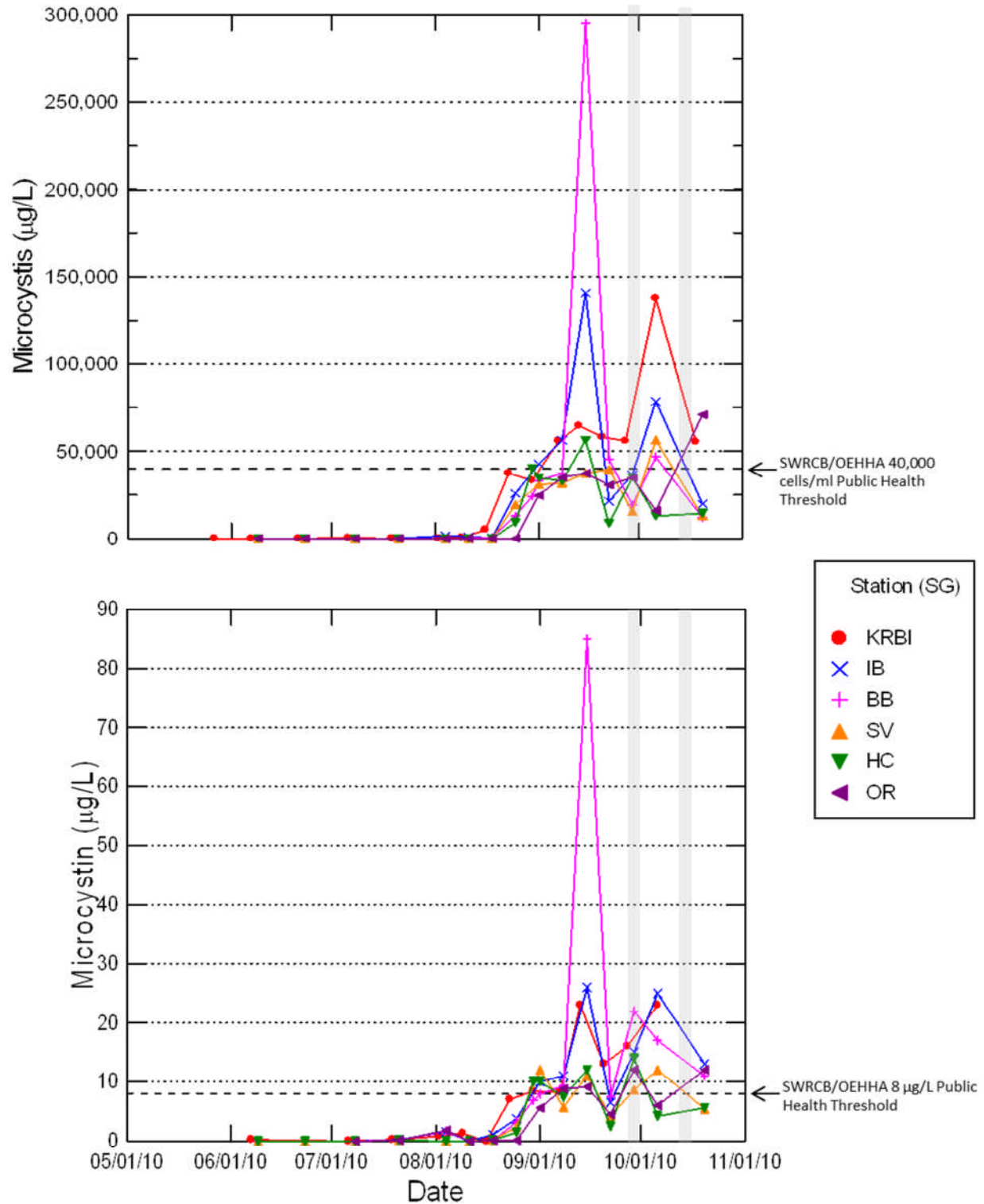


Figure 6. *Microcystis aeruginosa* cell density (top panel) and microcystin toxin concentration (bottom panel) in Klamath River SG (surface grab near shoreline) water samples during 2010. Samples collected as part of the Karuk Tribes public health monitoring program; SWRCB/OEHHA limit line indicates the public health guideline value and the shaded bars indicate when fish samples with positive toxin results were taken.

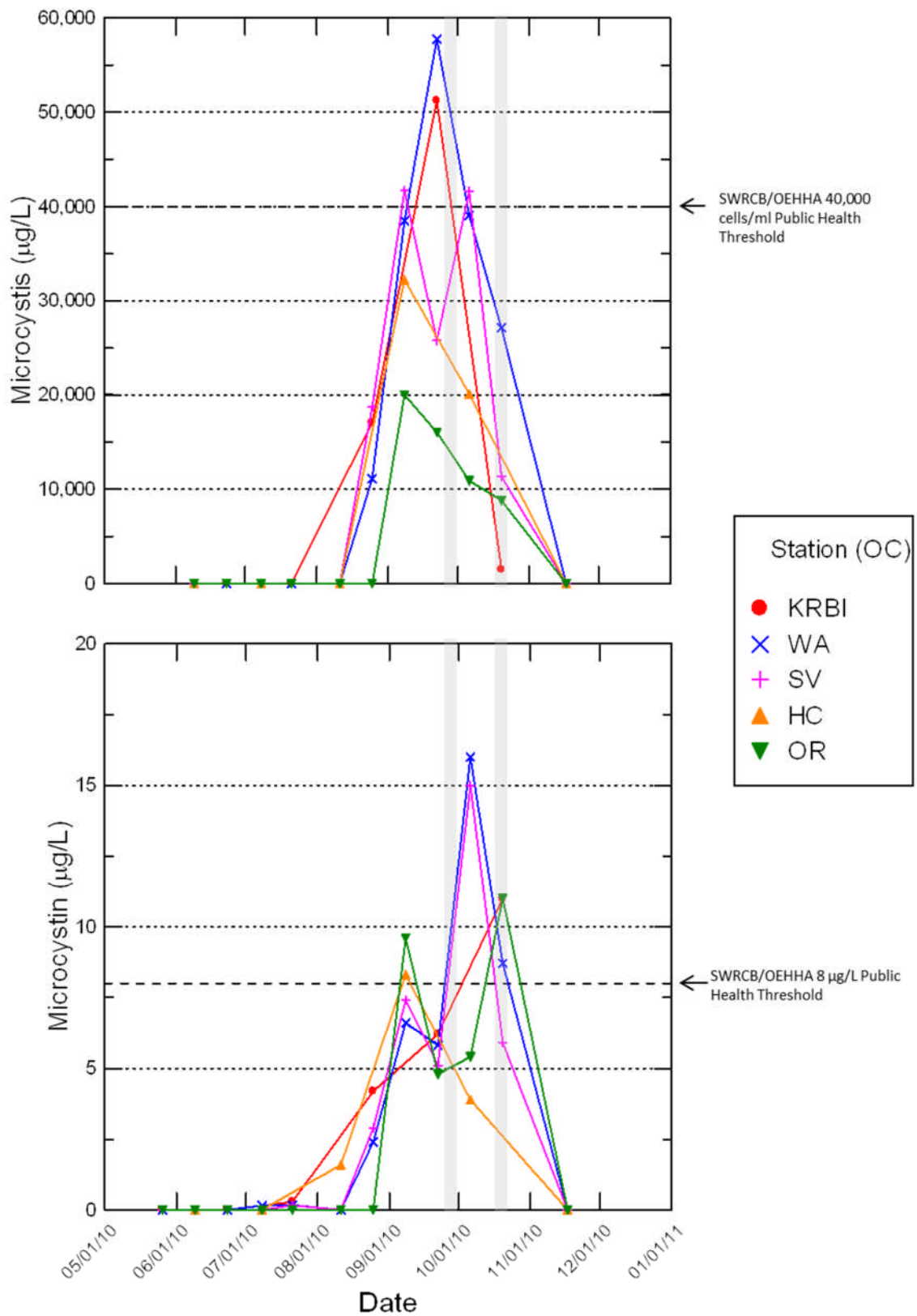


Figure 7. *Microcystis aeruginosa* cell density (top panel) and microcystin toxin concentration (bottom panel) in Klamath River OC (near mid-channel) water samples during 2010. Samples collected as part of the Karuk Tribes public health monitoring program; SWRCB/OEHHA limit line indicates the public health guideline value and the shaded bars indicate when fish samples with positive toxin results were taken.

Table 2. 2010 Fish and Wildlife Water Pollution Control Lab Ambient Microcystin Toxin Results (site codes as above in Figure 1, with the addition of LE and LES which are the Lower Estuary and the Lower Estuary Surface as collected by the Yurok Tribe).

Sample ID	Matrix	Date	Site Name	MC-RR	MC-Desmeth yl-RR*	MC-LR	MC-Desmeth yl-LR	MC-YR	MC-LA	MC-LW	MC-LF	MC-LY	Anatoxin A	Domoic acid	Okadaic acid
SV060910-SG	Water	6/9/2010	SV	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
IB062310-SG	Water	6/23/2010	IB	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SV070810-SG	Water	7/8/2010	SV	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
IG072110-SG	Water	7/21/2010	IG	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SV072110-SG	Water	7/21/2010	SV	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
IB081110-SG	Water	8/11/2010	IB	ND	ND	0.0478	ND	ND	0.517	ND	ND	ND	ND	ND	ND
SV081110-OC	Water	8/11/2010	SV	ND	ND	ND	ND	ND	0.125	ND	ND	ND	ND	ND	ND
IB082510-SG	Water	8/25/2010	IB	ND	ND	0.467	ND	ND	1.82	ND	ND	ND	ND	ND	ND
SV090810-SG	Water	9/8/2010	SV	ND	ND	ND	ND	ND	2	ND	ND	ND	ND	ND	ND
SV090810-OC	Water	9/8/2010	SV	ND	ND	ND	ND	ND	1.95	ND	ND	ND	ND	ND	ND
WE090810-OC	Water	9/8/2010	WE						1.74						
LES090810-OC	Water	9/8/2010	LES						3.74						
BB092210-SG	Water	9/22/2010	BB	ND	ND	ND	ND	ND	2.58	ND	ND	ND	ND	ND	ND
SV092210-SG	Water	9/22/2010	SV	ND	ND	ND	ND	ND	1.8	ND	ND	ND	ND	ND	ND
LES092210-OC	Water	9/22/2010	LES						1.93						
HC100610-OC	Water	10/6/2010	HC	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
SV100610-SG	Water	10/6/2010	SV	ND	ND	ND	ND	ND	6.82	ND	ND	ND	ND	ND	ND
WE100610-OC	Water	10/6/2010	WE						2.24						
LES100610-OC	Water	10/6/2010	LES						3.26						
IG102010-OC	Water	10/20/2010	IG	ND	ND	ND	ND	ND	2.13	ND	ND	ND	ND	ND	ND
SV102010-SG	Water	10/20/2010	SV	ND	ND	ND	ND	ND	1.93	ND	ND	ND	ND	ND	ND

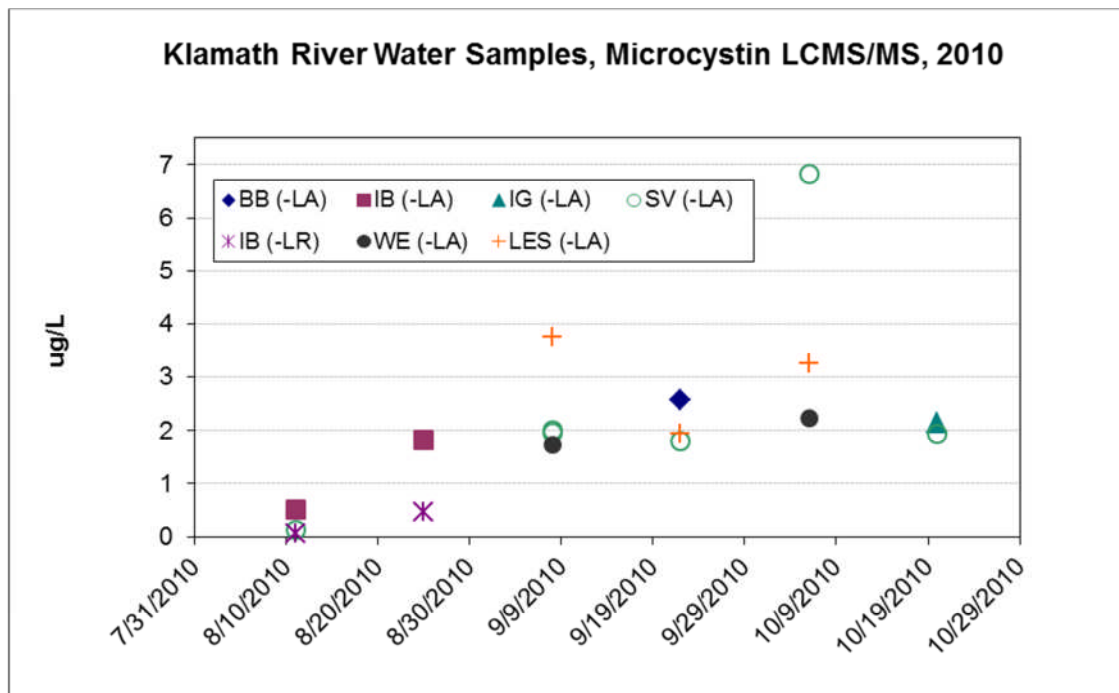


Figure 8. Microcystin congeners detected in Klamath River ambient water samples, 2010 (CFG Lab data shown in Table 2).

Because concentrations of microcystin-RR in the September Chinook livers were below public health guideline values (e.g. Kann 2008; OEHHA 2008) and livers are not typically consumed, those tested fish did not likely pose a public health concern with respect to consumption. They do, however, indicate that fish were exposed to microcystin, and that direct effects to fish health in terms of stress and/or disease are a possibility. The lack of consistent microcystin bioaccumulation among the sampled fish likely reflects variable exposure time due to spatial differences in toxin distribution, as well as temporal and spatial differences with respect to migration timing and habitat use

Although, as noted above, fish livers are not typically consumed, the level of microcystin-RR in the October Chinook and –LR in the October steelhead did exceed public health guideline values (e.g. OEHHA 2008; Ibelings and Chorus 2007). The State of California recommends that internal organs should be removed from fish exposed to *Microcystis* blooms and specifically that the viscera (e.g., liver, kidney, etc.) of the fish should not be eaten. This would be especially important given the demonstrated exceedance of specific public health guideline values for microcystin in liver tissue of salmonids from the Klamath River.

Aside from fish consumption issues with respect to public health, the positive detection of microcystin in Klamath River Chinook and steelhead may indicate an impact to the health of these fish in terms of stress and/or disease. For example, fish exposed to typical microcystin producing blooms may experience sublethal toxic effects such as liver damage (OEHHA/CEP 2009). In addition, laboratory and field studies from elsewhere have also demonstrated the toxic effects of microcystin on salmonids (Anderson et al. 1993, Tencalla et al. 1994; Bury et al. 1997, Landsberg 2002) and other fish (Smith et al. 2008). Based on these studies, and the documented presence of microcystin in the Klamath River and in Klamath River salmonid organs, the potential clearly exists for sublethal (e.g., stress and disease) effects on salmonids from exposure to algal toxins. Pending histology results will allow a further examination of this possibility.

REFERENCES

- Andersen RJ, Luu HA, Chen D.Z., Holmes CF, Kent ML, Le Blanc M, Taylor F.J.R., and DE Williams. 1993. Chemical and biological evidence links microcystins to salmon netpen liver disease. *Toxicol* 31(10):1315-1323.
- Bury N.R., McGeer J.C., Eddy FB, and Codd GA. 1997. Liver damage in brown trout, *Salmo trutta* L., and rainbow trout, *Oncorhynchus mykiss* (Walbaum), following administration of the cyanobacterial hepatotoxin microcystin-LR via the dorsal aorta. *Journal of Fish Diseases* 20(3):209-215.
- CH2M HILL. 2009. Occurrence of microcystin in salmon and steelhead fish tissue in the Klamath River in 2007. Prepared by CH2M HILL, Inc. Prepared for PacifiCorp Energy. December 2009.
- Fetcho, K. 2006. Klamath River Blue-Green Algae Bloom Report, Water Year 2005. Yurok Tribe Environmental Program, Klamath, CA.
<http://www.yuroktribe.org/departments/ytep/documents/YurokBGARReport032106.pdf>
- Jacoby, J.M., and J. Kann. 2007. The occurrence and response to toxic cyanobacteria in the Pacific Northwest, North America. *Lake and Reserv. Manage.* 23:123-143.
- Ibelings, B.W., and I. Chorus. 2007. Accumulation of cyanobacterial toxins in freshwater “seafood” and its consequences for public health: A review. *Environ. Pollut.* 150: 177-192.
- Kann J, Corum S, and Fetcho K. 2010. Microcystin Bioaccumulation in Klamath River Freshwater Mussel Tissue: 2009 Results. Prepared by Aquatic Ecosystem Sciences, LLC., the Karuk Tribe Natural Resources Department, and the Yurok Tribe Environmental Program:23 pp. + appendices.
<http://www.klamathwaterquality.com/documents/2009_Klamath_River_FreshwaterMussel_%20Microcystin_%20Bioaccumulation.pdf>
- Kann, J. 2008. Technical Memorandum: Microcystin Bioaccumulation in Klamath River Fish and Mussel Tissue: Preliminary 2007 Results. Aquatic Ecosystem Sciences, Ashland, OR.13 pp. + appendices.<http://www.klamathwaterquality.com/documents/2009/Kann_2008_Mussell_Bioaccumulation_.pdf>
- Kann, J. and S. Corum. 2009. Toxigenic *Microcystis aeruginosa* bloom dynamics and cell density/chlorophyll a relationships with microcystin toxin in the Klamath River, 2005-2008 Prepared For: Karuk Tribe Department of Natural Resources, P.O. Box 282 Orleans, CA.
- Kanz, R. 2008. Final Report to the U.S. Environmental Protection Agency on Cyanotoxin Accumulation in Fish and Freshwater Mussels of the Klamath River. Water Quality Cooperative Agreement CP 96941301-2. State Water Resources Control Board.
- Landsberg J.H. 2002. The effects of harmful algal blooms on aquatic organisms. *Reviews in Fisheries Science* 10(2):113–390.
- Office of Environmental Health Hazard Assessment (OEHHA). 2008. Applying Recommendations by the World Health Organization to the issue of fish consumption from the Klamath River. Memorandum from OEHHA to NCWQCB, June 10, 2008.
- OEHHA and CEP. 2009. Microcystins: A brief overview of their toxicity and effects, with special reference to fish, Wildlife, and livestock. January, 2009. Prepared by: Integrated Risk Assessment Branch Office of Environmental Health Hazard Assessment California Environmental Protection. Prepared for: Department of Water Resources Agency.
- Prendergast, L. and K. Foster. 2010. Analysis of Microcystin in Fish in Copco and Iron Gate Reservoirs in 2009. Technical Memorandum, PacifiCorp, Portland Oregon.

Smith J.L., Boyer GL, and Zimba PV. 2008. A review of cyanobacterial odorous and bioactive metabolites: Impacts and management alternatives in aquaculture. *Aquaculture* 280(1-4):5-20.

SWRCB and OEHHA. 2010. *Cyanobacteria in California Recreational Water Bodies Providing Voluntary Guidance about Harmful Algal Blooms, Their Monitoring, and Public Notification (July 2010 draft)*. Blue Green Algae Work Group of the State Water Resources Control Board (SWRCB), the California Department of Public Health (CDPH), and Office of Environmental Health and Hazard Assessment (OEHHA). <http://www.cdph.ca.gov/healthinfo/environhealth/water/Pages/bluegreenalgae.aspx>

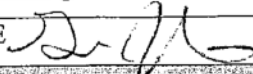
Tencalla, F.G., D.R. Dietrich, and C. Schlatter, 1994. Toxicity of *Microcystis aeruginosa* peptide toxin to yearling rainbow trout (*Oncorhynchus mykiss*). *Aquatic Toxicology*. 30(3): p. 215-224

WHO, 1999. *Toxic Cyanobacteria in Water. A Guide to Public Health Consequences, Monitoring and Management*. E & FN Spon, London.

APPENDIX I: Chain of custodies for Klamath River Tissue Study, 2010.

L-620-10

Chain of Custody for Klamath River Tissue Study

Karuk DNR	39051 Hwy 96
PHONE 530-469-3258	Orleans, CA 95556
CONTACT Grant Johnson	EMAIL cbowman@karuk.us, gjohnson@karuk.us
Collected By <u>Grant Johnson</u>	SIGNATURE 

	Sample ID	Date	Time	Lab ID	Sample Description	Microcystin ELISA	Microcystin LCMS/MS	Anatoxin
1	OR092310-15	09/23	liver fillet	CDG	liver & Fillet (Steelhead) Klamath		X	
2	OR092310-2S	09/23					X	
3	OR092610-3S	09/26					X	
4	OR092610-4S	1					X	
5	OR092610-5S	09/26			liver & fillet (Steelhead) Klamath		X	
6	IP092810-1C	09/28			liver & fillet (Chinook) Klamath			
7	IP092710-2C	09/27			1			
8	IP092710-3C	09/27			liver & fillet (Chinook) Klamath			
9								
10								
11								
12								
13								
14								

Date Shipped: 09/29/10 Carrier/ Shipping # UPS
 Date Received 9-30-10
 Received by Jan Riley
 Notes liver & fillet in separate bags, please return coolers & ice packs

Ship to: **For microcystin ELISA:**
 Sample Custodian
 USEPA Region 9 Lab
 1337 S. 46th Street
 Building 201
 Richmond, CA 94804
 510-412-2389

For anatoxin-a and microcystin LCMS/MS:
 Dr. Abdou Mekebri
 Fish and Wildlife Water Pollution Control Lab
 2005 Nimbus Road
 Rancho Cordova, CA 95670
 (916) 358-4396

Send Results To:
 Grant Johnson
 Karuk Tribe
 Dept of Natural Resources
 PO Box 282
 Orleans, CA 95556
 (530) 469-3258

Chain of Custody for Klamath River Tissue Study L-641-10

Karuk DNR	39051 Hwy 96
PHONE 530-469-3258	Orleans, CA 95556
CONTACT Grant Johnson	EMAIL: chowman@karuk.us , gjohnson@karuk.us
Collected By <i>Grant Johnson</i>	SIGNATURE <i>[Signature]</i>

	Sample ID	Date	Time	Lab ID	Sample Description	Microcystin ELISA	Microcystin LCMS/MS	Anatoxin
1	IP093010-4C	09/30		CDFG	Klamath River, Tissue filet & liver		X	
2	IP092910-1C							
3	WE092910-2S							
4	IP093010-3C							
5	IP092910-2C							
6	WE092910-1S							
7	WE092910-2S							
8	WE092910-4S							
9	WE092910-3S	09/30		CDFG	Klamath River, Tissue filet & liver		X	
10								
11								
12								
13								
14								

Date Shipped: 10-06-10 Carrier/ Shipping # _____
 Date Received 10-7-10 0930
 Received by [Signature]
 Notes _____

Ship to:

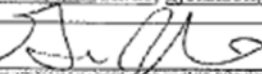
For microcystin ELISA:
 Sample Custodian
 USEPA Region 9 Lab
 1337 S. 46th Street
 Building 201
 Richmond, CA 94804
 510-412-2389

For anatoxin-a and microcystin LCMS/MS:
 Dr. Abdou Mekebri
 Fish and Wildlife Water Pollution Control Lab
 2005 Nimbus Road
 Rancho Cordova, CA 95670
 (916) 358-4396

Send Results To:

Grant Johnson
 Karuk Tribe
 Dept of Natural Resources
 PO Box 282
 Orleans, CA 95556
 (530) 469-3258

Chain of Custody for Klamath River Tissue Study L-678110

Karuk DNR	39051 Hwy 96
PHONE 530-469-3258	Orleans, CA 95556
CONTACT Grant Johnson	EMAIL cbowman@karuk.us , gjohnson@karuk.us
Collected By <u>Grant Johnson</u>	SIGNATURE 

	Sample ID	Date	Time	Lab ID	Sample Description	Microcystin ELISA	Microcystin LCMS/MS	Anatoxin
1	HC101410-8C	10/14		CDFG	Fish Tissue, Klamath		X	
2	HC101410-11S							
3	HC101410-12S							
4	HC101410-13S							
5	HC101410-14S							
6	HC101410-15S	10/14						
7	WE101510-16S	10/15						
8	WE101510-17S							
9	WE101510-18S							
10	WE101510-19S							
11	WE101510-20S	10/15						
12	OR101710-21S	10/17						
13	OR101710-22S							
14	OR101710-23S	10/17		CDFG	Fish Tissue, Klamath		X	

Date Shipped: 10/20/10 Carrier/ Shipping # _____
 Date Received 10-21-10 0930
 Received by Dot Harris
 Notes Frozen 30%

Ship to:

Send Results To:

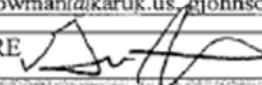
For microcystin ELISA:
 Sample Custodian
 USEPA Region 9 Lab
 1337 S. 46th Street
 Building 201
 Richmond, CA 94804
 510-412-2389

For anatoxin-a and microcystin LCMS/MS:
 Dr. Abdou Mekebri
 Fish and Wildlife Water Pollution Control Lab
 2005 Nimbus Road
 Rancho Cordova, CA 95670
 (916) 358-4396

Grant Johnson
 Karuk Tribe
 Dept of Natural Resources
 PO Box 282
 Orleans, CA 95556
 (530) 469-3258

Chain of Custody for Klamath River Tissue Study

L. 678-10

Karuk DNR	39051 Hwy 96
PHONE 530-469-3258	Orleans, CA 95556
CONTACT Grant Johnson	EMAIL cbowman@karuk.us , gjohnson@karuk.us
Collected By <u>Grant Johnson</u>	SIGNATURE 

	Sample ID.	Date	Time	Lab ID	Sample Description	Microcystin ELISA	Microcystin LCMS/MS	Anatoxin
15	CR101710-24S	10/17		CDFG	Fish Tissue, Klamath		X	
16	CR101710-25S	10/17						
17	IG101810-9C	10/18						
18	IG101810-10C							
19	IG101810-11C							
20	IG101810-12C							
21	IG101810-13C							
22	IG101810-14C	10/18		CDFG	Fish Tissue, Klamath		X	
	9							
	10							
	11							
	12							
	13							
	14							

Date Shipped: 10/20/10 Carrier/ Shipping # _____
 Date Received 10-21-10 11:30
 Received by Scott Hamill
 Notes FROZEN 30°F

Ship to:

For microcystin ELISA:
 Sample Custodian
 USEPA Region 9 Lab
 1337 S. 46th Street
 Building 201
 Richmond, CA 94804
 510-412-2389

For anatoxin-a and microcystin LCMS/MS:
 Dr. Abdou Mekebri
 Fish and Wildlife Water Pollution Control Lab
 2005 Nimbus Road
 Rancho Cordova, CA 95670
 (916) 358-4396

Send Results To:

Grant Johnson
 Karuk Tribe
 Dept of Natural Resources
 PO Box 282
 Orleans, CA 95556
 (530) 469-3258

Chain of Custody for Klamath River Tissue Study L-711-10

Karuk DNR	39051 Hwy 96
PHONE 530-469-3258	Orleans, CA 95556
CONTACT Grant Johnson	EMAIL cbowman@karuk.us , gjohnson@karuk.us
Collected By <i>Grant Johnson</i>	SIGNATURE <i>[Signature]</i>

	Sample ID	Date	Time	Lab ID	Sample Description	Microcystin ELISA	Microcystin LCMS/MS	Anatoxin
1	IG110110-15C	11/01		CDFG	Tissue, Liver & Fillet		X	
2	IG110110-16C						X	
3	IG110110-17C						X	
4	IG110110-18C						X	
5	IG110110-19C						X	
6	IG110110-20C	11/01		CDFG	Tissue, Liver & Fillet		X	
7								
8								
9								
10								
11								
12								
13								
14								

Date Shipped: 11/08/10 Carrier/ Shipping # _____
 Date Received: 11-9-10
 Received by: [Signature]
 Notes: 7-20C PB

Ship to:

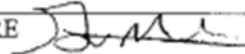
For microcystin ELISA:
 Sample Custodian
 USEPA Region 9 Lab
 1337 S. 46th Street
 Building 201
 Richmond, CA 94804
 510-412-2389

For anatoxin-a and microcystin LCMS/MS:
 Dr. Abdou Mekebri
 Fish and Wildlife Water Pollution Control Lab
 2005 Nimbus Road
 Rancho Cordova, CA 95670
 (916) 358-4396

Send Results To:

Grant Johnson
 Karuk Tribe
 Dept of Natural Resources
 PO Box 282
 Orleans, CA 95556
 (530) 469-3258

Chain of Custody for Klamath River Tissue Study L-742.10

Karuk DNR	39051 Hwy 96
PHONE 530-469-3258	Orleans, CA 95556
CONTACT Grant Johnson	EMAIL cbowman@karuk.us , gjohnson@karuk.us
Collected By <u>Grant Johnson</u>	SIGNATURE 

	Sample ID	Date	Time	Lab ID	Sample Description	Microcystin ELISA	Microcystin LCMS/MS	Anatoxin
1	IG112910-100	11/29		CDFG	Fish tissue, Fillet		X	
2	IG112910-100				Fish Tissue, Liver			
3	IG112910-200				Fish Tissue, Fillet			
4	IG112910-200				Fish Tissue, Liver			
5	IG112910-300	11/29		CDFG	Fish Tissue, Liver		X	
6								
7								
8								
9								
10								
11								
12								
13								
14								

Date Shipped: 12-06-10 Carrier/ Shipping # UPS
 Date Received 12-7-10 1600
 Received by Grant Johnson
 Notes _____

Ship to:

For microcystin ELISA:
 Sample Custodian
 USEPA Region 9 Lab
 1337 S. 46th Street
 Building 201
 Richmond, CA 94804
 510-412-2389

For anatoxin-a and microcystin LCMS/MS:
 Dr. Abdou Mekebri
 Fish and Wildlife Water Pollution Control Lab
 2005 Nimbus Road
 Rancho Cordova, CA 95670
 (916) 358-4396

Send Results To:

Grant Johnson
 Karuk Tribe
 Dept of Natural Resources
 PO Box 282
 Orleans, CA 95556
 (530) 469-3258

APPENDIX II: Fish and Wildlife Water Pollution Control Lab Sheets September, 2010.

WPCL Lab#	Estimated MDL	Reporting Limit	L-620-10-1	L-620-10-2	L-620-10-3	L-620-10-4	L-620-10-5	L-620-10-6	L-620-10-7	L-620-10-8	L-620-10-9	L-620-10-10	L-620-10-11	L-620-10-12	L-620-10-13	L-620-10-13Dup	L-620-10-14	L-620-10-15	L-620-10-16	L-620-10-MBLK	L-620-10-LCS	L-620-10-6MS	L-620-10-6MSD	
Sample Identification			OR092310-1S	OR092310-1S	OR092310-2S	OR092310-2S	OR092610-3S	OR092610-3S	OR092610-4S	OR092610-4S	OR092610-5S	OR092610-5S	IP092810-1C	IP092810-1C	IP092710-2C	IP092710-2C	IP092710-2C	IP092710-3C	IP092710-3C			OR092610-3S	OR092610-3S	
Date Collected			23/Sep/2010	23/Sep/2010	23/Sep/2010	23/Sep/2010	26/Sep/2010	26/Sep/2010	26/Sep/2010	26/Sep/2010	26/Sep/2010	26/Sep/2010	28/Sep/2010	28/Sep/2010	27/Sep/2010	27/Sep/2010	27/Sep/2010	27/Sep/2010	27/Sep/2010			26/Sep/2010	26/Sep/2010	
Time Collected			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA			NA	NA	
Date Received			30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010			30/Sep/2010	30/Sep/2010	
Date Extracted			04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010	04/Dec/2010			04/Dec/2010	04/Dec/2010	
Date Analyzed			08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010			08/Dec/2010	08/Dec/2010	
Matrix			fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet	fish liver	fish fillet			fish fillet	fish fillet
			fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight	fresh weight			fresh weight	fresh weight
Biotoxin Analytes	ppb	ppb	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	% Recovery	% Recovery	% Recovery	
MC-RR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	3.80	2.93	ND	ND	ND	ND	109	101	106	
MC-Desmethyl-RR*	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA	
MC-LR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	106	75.1	78.0	
MC-Desmethyl-LR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA	
MC-YR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	117	99.3	99.4	
MC-LA	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	112	81.4	84.4	
MC-LW	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA	
MC-LF	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA	
MC-LY	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA	
Anatoxin A	5.00	10.0	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	
Domoic acid	2.00	5.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	
Okadaic acid	1.00	2.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	

* Desmethyl-RR quantified as parent analog compound.

Quality Control Results

WPCL Lab#	Estimated MDL	Reporting Limit	L-641-10-1	L-641-10-2	L-641-10-3	L-641-10-3Dup	L-641-10-4	L-641-10-5	L-641-10-6	L-641-10-7	L-641-10-8	L-641-10-9	L-641-10-10	L-641-10-11	L-641-10-12	L-641-10-13	L-641-10-14	L-641-10-15	L-641-10-16	L-641-10-17	L-641-10-18	L-641-10-MBLK	L-641-10-LCS	L-641-10-AMS	L-641-10-MSD	
Sample Identification			IP093010-4C	IP093010-4C	IP092910-1C	IP092910-1C	IP092910-1C	WE092910-5S	WE092910-5S	IP093010-3C	IP093010-3C	IP092910-2C	IP092910-2C	WE092910-1S	WE092910-1S	WE092910-2S	WE092910-2S	WE092910-4S	WE092910-4S	WE092910-3S	WE092910-3S			IP092910-1C	IP092910-1C	
Date Collected			30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010	30/Sep/2010			30/Sep/2010	30/Sep/2010	
Time Collected			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA			NA	NA	
Date Received			07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010	07/Oct/2010			07/Oct/2010	07/Oct/2010	
Date Extracted			07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010			07/Dec/2010	07/Dec/2010	
Date Analyzed			08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010	08/Dec/2010			08/Dec/2010	08/Dec/2010	
Matrix			fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight	fish liver fresh weight	fish fillet fresh weight			fish fillet fresh weight	fish fillet fresh weight	
Biotoxin Analytes	ppb	ppb	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	% Recovery	% Recovery	% Recovery
MC-RR	0.500	1.00	2.71	ND	2.17	2.10	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	104	113	113
MC-Desmethyl-RR*	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
MC-LR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	104	98.5	99.4
MC-Desmethyl-LR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
MC-YR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	125	120	108
MC-LA	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	107	92.6	95.3
MC-LW	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
MC-LF	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
MC-LY	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
Anatoxin A	5.00	10.0	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
Domoic acid	2.00	5.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA
Okadaic acid	1.00	2.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	NA	NA	NA

* Desmethyl-RR quantified as parent analog compound.

Quality Control Results

WPCL Lab#	Estimated MDL	Reporting Limit	L-711-10-1	L-711-10-2	L-711-10-3	L-711-10-4	L-711-10-5	L-711-10-6	L-711-10-7	L-711-10-8	L-711-10-9	L-711-10-10	L-711-10-11	L-711-10-12	L-678-10-MBLK3	L-678-10-LCS3	L-711-10-4MS	L-711-10-4MSD
Sample Identification			IG110110-15C	IG110110-15C	IG110110-16C	IG110110-16C	IG110110-17C	IG110110-17C	IG110110-18C	IG110110-18C	IG110110-19C	IG110110-19C	IG110110-20C	IG110110-20C			IG110110-16C	IG110110-16C
Date Collected			01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010	01/Nov/2010			01/Nov/2010	01/Nov/2010
Time Collected			NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA			NA	NA
Date Received			09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010	09/Nov/2010			09/Nov/2010	09/Nov/2010
Date Extracted			06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011			06/Feb/2011	06/Feb/2011
Date Analyzed			14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011			14/Feb/2011	14/Feb/2011
Matrix			liver	tissue	liver	tissue	liver	tissue	liver	tissue	liver	tissue	liver	tissue			liver	tissue
			wet weight	wet weight	wet weight	wet weight	wet weight	wet weight	wet weight	wet weight	wet weight	wet weight	wet weight	wet weight			wet weight	wet weight
Biotxin Analytes	ppb	ppb	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)			ppb (ng/g)	% Recovery
MC-RR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	83.1
MC-Desmethyl-RR*	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA
MC-LR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	75.2
MC-Desmethyl-LR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA
MC-YR	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	76.8
MC-LA	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	105
MC-LW	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA
MC-LF	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA
MC-LY	0.500	1.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA
Anatoxin A	5.00	10.0	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	85.5
Domoic acid	2.00	5.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA
Okadaic acid	1.00	2.00	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND			ND	NA

Quality Control Results

WPCL Lab#	Estimated MDL	Reporting Limit	L-742-10-1	L-742-10-2	L-742-10-3	L-742-10-4	L-742-10-5	Quality Control Results	L-742-10-MBLK	L-742-10-LCS	L-711-10-4MS	L-711-10-4MSD	
Sample Identification			IG112910-1CO	IG112910-1CO	IG112910-2CO	IG112910-2CO	IG112910-3CO					IG110110-16C	IG110110-16C
Date Collected			29/Nov/2010	29/Nov/2010	29/Nov/2010	29/Nov/2010	29/Nov/2010					01/Nov/2010	01/Nov/2010
Time Collected			NA	NA	NA	NA	NA					NA	NA
Date Received			07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010	07/Dec/2010					09/Nov/2010	09/Nov/2010
Date Extracted			06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011	06/Feb/2011			06/Feb/2011	05/Feb/2011	06/Feb/2011	06/Feb/2011
Date Analyzed			14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011			14/Feb/2011	14/Feb/2011	14/Feb/2011	14/Feb/2011
Matrix			tissue	liver	tissue	liver	liver			liver	liver	tissue	tissue
			wet weight	wet weight	wet weight	wet weight	wet weight			wet weight	wet weight	wet weight	wet weight
Biotoxin Analytes	ppb	ppb	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)	ppb (ng/g)			ppb (ng/g)	% Recovery	% Recovery	% Recovery
MC-RR	0.500	1.00	ND	ND	ND	ND	ND			ND	71.5	76.7	69.7
MC-Desmethyl-RR*	0.500	1.00	ND	ND	ND	ND	ND			ND	NA	NA	NA
MC-LR	0.500	1.00	ND	ND	ND	ND	ND			ND	80.8	118	121
MC-Desmethyl-LR	0.500	1.00	ND	ND	ND	ND	ND			ND	NA	NA	NA
MC-YR	0.500	1.00	ND	ND	ND	ND	ND		ND	80.3	103	107	
MC-LA	0.500	1.00	ND	ND	ND	ND	ND		ND	107	118	122	
MC-LW	0.500	1.00	ND	ND	ND	ND	ND		ND	NA	NA	NA	
MC-LF	0.500	1.00	ND	ND	ND	ND	ND		ND	NA	NA	NA	
MC-LY	0.500	1.00	ND	ND	ND	ND	ND		ND	NA	NA	NA	
Anatoxin A	5.00	10.0	ND	ND	ND	ND	ND		ND	88.7	86.2	83.8	
Domoic acid	2.00	5.00	ND	ND	ND	ND	ND		ND	NA	NA	NA	
Okadaic acid	1.00	2.00	ND	ND	ND	ND	ND		ND	NA	NA	NA	

* Desmethyl-RR quantified as parent analog compound.