

Chemical Residues in Surface and Ground Waters Following Rotenone Application to California Lakes and Streams

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Abstract.—Over the past 15 years, the California Department of Fish and Game has monitored applications of the rotenone formulations Nusyn-Noxfish® (synergized liquid formulation) and Pro-Noxfish® (powdered formulation) to lakes and streams. The monitoring studies were designed to limit liability and address environmental and public health issues. Results indicated that (a) the half-life ($t_{1/2}$) of rotenone increased inversely with temperature from 0.6 to 7.7 days, (b) the degradation product rotenolone was generally not found in the absence of rotenone, except in waters of low alkalinity (< 15 mg/L CaCO₃) temperature (< 11 °C), (c) Nusyn-Noxfish® contaminant trichloroethylene (up to 4.9 µg/L) and additive xylene (up to 6.7 µg/L) were found typically only in lakes, (d) Nusyn-Noxfish® additives naphthalene (up to 332 µg/L) and methylnaphthalenes (up to 390 µg/L) were found in both lakes and streams, (e) potassium permanganate can neutralize rotenone within a contact time of 30 minutes, (f) Nusyn-Noxfish® synergist piperonyl butoxide can persist in cold (< 10 °C) and deep (> 25 m) waters for up to nine months, and (g) ground water remained free of chemicals in both rotenone formulations. These studies demonstrated that (a) toxicity and other effects can be confined to the treatment and neutralization areas, (b) concentrations of chemicals in surface waters (with the exception of rotenolone and piperonyl butoxide in cold waters) persist for less than seven weeks, (c) rotenone, naphthalene, and methylnaphthalene persist in sediments for short periods, and (d) ground waters were not contaminated.

Introduction

The California Department of Fish and Game (CDFG) has used rotenone to (a) eradicate unwanted exotic fishes, (b) control fish diseases, (c) restore populations of threatened or endangered fishes, and (d) increase populations of desirable game fishes (CDFG 1994a). Rotenone use over the past 50 years has proceeded without serious incident although not without public controversy. CDFG interacts with regulatory agencies that have jurisdiction over pesticide use, maintenance of water quality, and protection of public health. The amount of interaction has generally paralleled concern by the general public. Many of the issues expressed by the regulatory agencies and the public can be addressed through carefully designed monitoring studies. These include (a) identification and effects of chemicals in the rotenone formulation, (b) containment of chemicals and effects to the project area, and (c) possible contamination of adjacent ground water with chemicals.

Since 1987, CDFG has monitored nine projects (Figure 1) in California lakes and streams treated with the synergized liquid rotenone formulation Nusyn-Noxfish® (USEPA Reg. No. 432-550) and the powdered rotenone formulation Pro-Noxfish® (USEPA Reg. No. 432-829). All of the studies have focused on the application of Nusyn-Noxfish®; only one application (Lake Davis) involved Pro-Noxfish®. The objectives of the studies were to address environmental and human health concerns. These studies monitored the distribution and persistence of rotenone and the degradation product rotenolone (Pro-Noxfish® and Nusyn-



Figure 1. Location of monitoring sites in California.

other semi-volatile and volatile organic compounds (Nusyn-Noxfish®) in surface and ground waters.

Methods

Study sites

Kaweah River – The Kaweah River drainage in Tulare County was treated with Nusyn-Noxfish® at 2 mg/L in fall 1987 to eradicate the unauthorized introduction of white bass *Morone chrysops* (CDFG 1987; Harrington and Finlayson 1988; Figure 1). The treatments lasted approximately four weeks. Rotenone toxicity was allowed to naturally dissipate. Kaweah Reservoir, Bravo Reservoir, several ground water recharge (percolation) ponds, the Kaweah River, and nine wells were monitored (Tables 1 and 2). Water was collected from the surface, mid-depth, and bottom of Kaweah Reservoir at three locations. Approximately 12,000 gallons of Nusyn-Noxfish® were used in the treatments.

Mill Creek – The upper reaches (11 km) of Mill Creek (Walker River Drainage) in the Toiyabe National Forest above a fish barrier in Mono County were treated with Nusyn-Noxfish® at 1 mg/L in fall 1988 (CDFG 1988a) and fall 1989 (CDFG 1989a) to remove brook trout *Salvelinus fontinalis* and rainbow trout *Oncorhynchus mykiss* (Figure 1). The treatments were done in preparation for the reestablishment of Lahontan cutthroat trout *Oncorhynchus clarki henshawi*, a threatened species (Gerstung 1986). The target fish compete (brook trout) and hybridize (rainbow trout) with the Lahontan cutthroat trout. Rotenone was neutralized with potassium permanganate at the fish barrier to limit the effects downstream. Mill Creek

and three wells located 2 km downstream of the fish barrier were monitored (Tables 1 and 2). Approximately 5 gallons of Nusyn-Noxfish® were used each year (10 gallons total).

Table 1. Characteristics of surface waters during monitoring studies.

Location (year)	Temperature (°C)	pH	Alkalinity (mg/L CaCO ₃)	Volume or Flow (AF or cfs)
Kaweah Rv. (1987)	18 – 21	7.8-9.1	50	25
Kaweah Reservoir	20 – 22	7.6-7.7	40-63	9,400
Bravo Reservoir	18 – 23	8.8	40	1,600
Lonestar Pond	13 – 16	---	---	845
Percolation Res. 5	14 – 27	9.4-9.5	18-20	8
Percolation Res. 12	14 - 26	9.3-9.5	90-96	7
U. Truckee Rv.(1988–1990)	8 - 20	8.3	20	1
Meiss Lake	10 - 21	9.4	14	45
Mill Cr. (1988 - 1989)	5 - 17	8.5	40	<1
Tule Rv. (1988)	18 - 20	8.4	170	15
Success Reservoir	18 - 20	7.7	170	5,000
Frenchman Lake (1991)	10 - 22	8.3	70	22,000
Silver King Cr. (1991–1993)	1 - 19	7.5	22	12
Wolf Cr. (1991 & 1992)	8 - 12	7.4	30	3
Wolf Creek Lake	5 - 11	7.7	7	33
Silver Cr. (1994-1996)	6 - 17	7.5	25	5
Lake Davis (1997)	1 - 12	7.5-9.2	31-42	48,000

Upper Truckee River – The upper reaches (9 km) of the Upper Truckee River in the Lake Tahoe Basin Management Unit above a fish barrier in Alpine County were treated with Nusyn-Noxfish® at 1 mg/L in fall 1988 (CDFG 1988b), fall 1989 (CDFG 1989b), and fall 1990 (CDFG 1990) to remove brook trout (Figure 1). The treatments were done in preparation for the reintroduction of Lahontan cutthroat trout (Gerstung 1986). Rotenone was neutralized with potassium permanganate at the fish barrier to limit effects. The Upper Truckee River, Meiss Lake, and three wells located 7 km downstream of the fish barrier were monitored (Tables 1 and 2). Approximately 50 gallons of Nusyn-Noxfish® were used each year (150 gallons total).

Tule River – Success Reservoir and 11 km of the Tule River upstream of the reservoir in Tulare County were treated with Nusyn-Noxfish® at 4 mg/L in fall 1988 to remove nongame fish species (CDFG 1989c; Figure 1). Rotenone toxicity was allowed to naturally dissipate. Success Reservoir, the Tule River and three wells adjacent to the Tule River were monitored (Tables 1 and 2). Water was collected from the surface and bottom of Success Reservoir at two locations. Approximately 4,300 gallons of Nusyn-Noxfish® were used in the treatment.

Frenchman Lake – Frenchman Lake and several miles of tributaries upstream (Feather River Drainage) in the Plumas National Forest in Plumas County were treated with Nusyn-Noxfish®

at 2 mg/L in spring 1991 to eradicate the exotic predatory northern pike *Esox lucius* (CDFG 1991a; 1991b; Figure 1). Rotenone toxicity was allowed to naturally dissipate in the lake but the discharge from the dam into Little Last Chance Creek was neutralized with potassium permanganate to limit effects. Frenchman Lake and three adjacent wells supplying water to Plumas National Forest campgrounds were monitored (Tables 1 and 2). Water was collected from the surface, mid-depth, and bottom of Frenchman Lake from four locations. Sediment was also collected. Approximately 15,000 gallons of Nusyn-Noxfish® were used in the treatment.

Silver King Creek – The upper reaches (10 km) of Silver King Creek (Carson River Drainage) in the Carson–Iceberg Wilderness Area of the Toiyabe National Forest above a fish barrier in Alpine County were treated with Nusyn-Noxfish® at 1 mg/L in summer 1991 (CDFG 1991c), summer 1992 (CDFG 1992a), and summer 1993 (CDFG 1994b) to remove rainbow trout and Lahontan cutthroat trout (Figure 1). The treatments were done in preparation for the reintroduction of Paiute cutthroat trout *Oncorhynchus clarki seleniris*, an endangered species (U.S. Fish and Wildlife Service 1985). The target fish hybridize with Paiute cutthroat trout. Rotenone was neutralized with potassium permanganate at the fish barrier to limit effects. Silver King Creek was monitored (Table 1). Sediment was also collected. Approximately 10 gallons of Nusyn-Noxfish® were used during each year of treatment (30 gallons total).

Wolf Creek – Wolf Creek Lake and the upper reaches (7 km) of Wolf Creek (Walker River Drainage) in the Toiyabe National Forest above a fish barrier in Mono County were treated with Nusyn-Noxfish® in fall 1991 (CDFG 1992b) and fall 1992 (CDFG 1992c) to remove brook trout and rainbow trout (Figure 1). The creek was treated at 1 mg/L and the lake received a 2 mg/L treatment. The treatments were done in preparation for the introduction of Lahontan cutthroat trout (Gerstung 1986). Rotenone was neutralized with potassium permanganate at the fish barrier to limit effects. Wolf Creek, Wolf Creek Lake, and the West Fork Walker River were monitored (Table 1). Sediment was also collected. Approximately 45 gallons of Nusyn-Noxfish® were used each year (90 gallons total).

Silver Creek – The upper reaches (13 km) of Silver Creek (Walker River Drainage) in the Toiyabe National Forest above a fish barrier in Mono County were treated with Nusyn-Noxfish® at 1 mg/L in summer 1994 (CDFG 1994c), summer 1995 (CDFG 1995), and summer 1996 (CDFG 1996) to remove brook trout (Figure 1). The treatments were done in preparation for the introduction of Lahontan cutthroat trout (Gerstung 1986). Rotenone was neutralized with potassium permanganate at the fish barrier to limit effects. Silver Creek and the West Fork Walker River were monitored (Table 1). Sediment was also collected. Approximately 5.5 to 8.5 gallons of Nusyn-Noxfish® were used each year (20 gallons total).

Lake Davis – Lake Davis (Feather River Drainage) and several miles of tributaries upstream of the lake in Plumas County were treated with Nusyn-Noxfish® at 1 mg/L and Pro-Noxfish® at 1 mg/L in fall 1997 (2 mg/L total) to eradicate exotic predatory northern pike (CDFG 1997; CDFG 1999; Siepmann and Finlayson 1999). Rotenone toxicity was allowed to naturally dissipate in the lake, but the discharge from the dam into Big Grizzly Creek was neutralized with potassium permanganate to limit effects. Lake Davis, Big Grizzly Creek, and several adjacent wells supplying water to private parties were monitored (Tables 1 and 2). Water was collected from the surface, mid-depth, and bottom of Lake Davis from ten locations (36 sites total). Sediment was also collected. Approximately 16,000 gallons of Nusyn-Noxfish® and 64,000 pounds of Pro-Noxfish® were used in the treatment.

Table 2. Characteristics of ground waters monitored.

Location (Year)	Well Type ^a	Well Depth (m)	Horizontal Distance ^b (m)
Kaweah Rv. (1987)			
Corps of Engineers Well	D	61	0
Lemon Cove Well 1	D	46	183
Lemon Cove Well 2	D	11	400
Woodlake Well 1	M	66	400
Woodlake Well 2	D	24	45
Woodlake Well 3	D	21	18
Woodlake Well 4	I	---	183
Woodlake Well 5	M	49	30
Kaweah Percolation Res. 5 Well	D	43	45
U. Truckee Rv. (1988-1990)			
Christmas Valley Well 1	D	8	20
Christmas Valley Well 2	D	1	3
Christmas Valley Well 3	D	17	10
Mill Cr. (1988-1989)			
Walker Well 1	D	34	200
Walker Well 2	D	62	200
Walker Well 3	D	34	200
Tule Rv. (1988)			
Springville Well	D	22	30
Country Club Well	I	10	200
Corps of Engineers Well	D	30	100
Frenchman Lake (1991)			
Big Cove Campground Well	D	17	30
Spring Creek Campground Well	D	35	400
Cottonwood Springs Well	D	12	1,000
Lake Davis (1997)			
South Davis Well 1	D	61	450
South Davis Well 2	D	73	640
South Davis Well 3	D	52	760
South Davis Well 4	D	26	670
Grasshopper Flat Campground Well	D	55	670

^a D = Domestic use; I = Irrigation use; and M = Municipal use.

^b Distance from surface water containing Nusyn-Noxfish®.

Sampling

Frequency –All of the surface and ground water monitoring sites were sampled prior to treatment to establish environmental levels of rotenone, rotenolone, and other organic chemicals found in the rotenone formulations. Surface water sites were sampled during (flowing waters) or immediately following (standing water) rotenone application at previously established sites and intervals until rotenone and the other chemicals had dissipated below the limits of detection. Ground water sites were sampled up to 456 days following treatment. Sediment samples were also taken from several study locations. Samples of undiluted Nusyn-Noxfish® were also analyzed to determine chemical levels.

Collection – Surface water was grab sampled at a depth of 0.5 m, and water from mid-depth and the bottom from Kaweah Reservoir, Frenchman Lake, Davis Lake, and Success Reservoir was collected using a Kemmerer bottle. Ground water was sampled as close to the wellhead as possible, sampling from an Schrader valve or faucet before the storage tank using standard procedures (Sava 1986). Well pumps were turned on for a minimum of 15 minutes to purge standing water in the well casing. Water samples for rotenone and rotenolone analysis were collected in 500-ml amber glass bottles with Teflon®-lined caps. Water samples for volatile organic chemicals (VOC) analyses were collected in 20-ml or 40-ml glass vials with Teflon®-lined silicone-septa screw caps. Water samples for semivolatile organic (semiVOC) chemicals analyses were collected in 1-L amber glass bottles with Teflon®-lined or foil-lined caps. Water samples for analysis of the synergist piperonyl butoxide (PB) were collected in 500-ml or 1-L amber glass bottles with Teflon®-lined caps. Sediment samples were collected in chemically clean 500-ml polycarbonate jars (rotenone and rotenolone) and amber glass jars (VOC, PB, and semiVOC) with Teflon®-lined lids. Sediment samples were collected by depositing at least 100 ml of sediment from the bottom, in shallow areas. The remainder of the jar was filled with overlying water. All containers were filled to capacity and carefully capped as to avoid trapped air in the sample container. Generally, samples were collected in replicate; only one of the two replicates was analyzed. The other replicate served as insurance against analytical anomaly or breakage during transit.

Storage – All water samples were placed on ice immediately after collection and transported to a laboratory refrigerator and kept at a temperature of 4 °C until analyzed. Sediment samples for rotenone analysis were generally frozen for up to three months prior to analysis. The samples for VOC and semiVOC analyses were extracted and analyzed within time periods allowed by the method. Samples for rotenone and rotenolone analysis were extracted and analyzed within seven days. The water and sediment samples for VOC analyses were extracted within 14 days, and the sediment and water samples for semiVOC analyses were extracted within seven days.

Harrington and Finlayson (1988) conducted experiments to determine the effect of sample storage on rotenone residues in water. Surface water samples from several sites containing rotenone were split in the field among 10 duplicate samples. One set of five duplicate samples was analyzed before and the other set was analyzed after six days storage at a temperature of 4° C in the absence of light.

Chemical analyses

Nusyn-Noxfish® - Nusyn-Noxfish®, in addition to the active ingredient rotenone and the synergist PB, contains emulsifiers, carriers, and solvents (VOC and semiVOC) to disperse rotenone in water. Sixteen lots of Nusyn-Noxfish® were analyzed to determine the concentrations of each formulation constituent (Table 3). Rotenone was determined using the method of Dawson et al. (1983). VOC concentrations (Table 3) were determined using U.S. Environmental Protection Agency (USEPA) methods 624 (USEPA 1984a) or 8240 (USEPA 1994a), and semiVOC concentrations (Table 3) were determined using USEPA methods 625 (USEPA 1984b) or 8270 (USEPA 1994b). VOC and semiVOC analyses were completed to address public health issues (i.e., carcinogenicity) associated with some of these compounds (i.e., trichloroethylene) and to identify water quality monitoring needs. Quality assurance for the analyses was provided by systematic analysis of blanks, replicates, and spiked samples.

Water – Water samples from study sites were analyzed for Nusyn-Noxfish® constituents (Table 3). Concentrations of rotenone and the degradation product rotenolone were determined using the method described by Dawson et al. (1983). VOC concentrations were determined using USEPA methods 601 and 602, 502.2 or 8260 (USEPA 1984c; USEPA

1984d; USEPA 1989; USEPA 1994c respectively), and semiVOC concentrations were determined using USEPA methods 610 or 8310 (USEPA 1984e; USEPA 1986). After 1991, USEPA methods 502.2 or 8260 were substituted for USEPA methods 601 and 602, and USEPA method 8310 was substituted for USEPA method 610. The PB was measured using USEPA method 8270 (USEPA 1994b). The detection limits for rotenone and rotenolone were 2.0 µg/L, for VOC typically varied from 0.2 µg/L (USEPA methods 8260 and 502.2) to 0.5 µg/L (USEPA methods 601 and 602), for semiVOC typically varied from 0.2 µg/L (USEPA method 8270) to 2.5 µg/L (USEPA method 610), and for PB typically varied from 2 to 8 µg/L. Quality assurance for the analyses was provided by systematic analysis of blanks, replicates, and spike samples. Recovery rates for samples spiked with rotenone and rotenolone were typically 78 to 80%. Recovery rates for samples spiked with VOC (USEPA methods 8260, 502.2, 601 and 602) were typically 86 to 116 % and for samples spiked with semiVOC (USEPA methods 8270 and 610) were typically 60 to 92%.

Sediment – Sediment samples from study sites were analyzed for Nusyn-Noxfish® constituents (Table 3). Sediment samples were analyzed for rotenone and rotenolone (Dawson 1986), VOC using USEPA method 8260 (USEPA 1994c), and semiVOC using USEPA method 8270 (USEPA 1994b). PB was measured using USEPA method 8270 (USEPA 1994b). The detection limits for rotenone and rotenolone were 30 µg/kg (dry weight), for semiVOC were 6 to 70 µg/kg (dry weight), for VOC were 5 to 6 µg/kg (dry weight), and for PB was 70 µg/kg PB (dry weight).

Table 3. Matrix for analytical methods used in analysis of Nusyn-Noxfish®, water, and sediment for rotenone, volatile organic compounds (VOC), semivolatile organic compounds (semiVOC), and piperonyl butoxide (PB).

Media	Rotenone	VOC	SemiVOC	PB
Nusyn-Noxfish®	Dawson et al. (1983)	624 (USEPA 1984a) 8240 (USEPA 1994a)	625 (USEPA 1984b) 8270 (USEPA 1994b)	--
Water	Dawson et al. (1983)	601 & 602 (USEPA 1984c & USEPA 1984d) 502.2 (USEPA 1989) 8260 (USEPA 1994c)	610 (USEPA 1984e) 8310 (USEPA 1986)	8270 (USEPA 1994b)
Sediment	Dawson (1986)	8260 (USEPA 1994c)	8270 (USEPA 1994b)	8270 (USEPA 1994b)

Water quality – Water temperature was recorded at the time of sample collection using a Yellow Springs Instruments® Model 57 oxygen meter. Water samples for pH and alkalinity determinations were collected in 500-ml high-density polyethylene bottles, stored on ice, and transported with the other samples to the laboratory. The pH was determined using a Hach® Model 1 pH meter, and total alkalinity was determined using the standard titration method (American Public Health Association 1985).

Results and Discussion

Constituents of Nusyn-Noxfish®

Nusyn-Noxfish® contains petroleum hydrocarbons as solvents and emulsifiers to disperse the rotenone in water (Penick Bio UCLAF 1987). The analyses of 16 different lots of Nusyn-Noxfish® (Table 4) for VOC and semiVOC during the past 15 years found mean concentrations of 73,502 mg/L for naphthalene, 95,055 mg/L for 2-methylnaphthalene, 1,925

mg/L for xylenes, and 565 mg/L for trichloroethylene. The Nusyn-Noxfish® manufactured in 1997 (Lots 32365, 32367, and 32369) contained significantly less (an order of magnitude) TCE and xylenes (means = 17.3 and 151 µg/L, respectively) than the material manufactured prior (mean = 692 and 2,334 µg/L, respectively) to 1997 (Table 4). Other compounds that have been detected, but usually at less than the quantifiable limits, include acetone, benzene, ethylbenzene, methylene chloride, toluene, phenanthrene, anthracene, and bis (2-ethylhexyl) phthalate. None of these chemicals are known ingredients in the Nusyn-Noxfish® formulation (Roussel Bio Corporation 1991). No explanation is known for the presence of these “foreign” materials except that these are common ingredients in industrial fluids including gasoline and several are plasticizers. These may be contaminants in the industrial solvents used during product formulation or may have resulted from the use of plastics in the sampling process. Regardless, their occurrence was sporadic and at insignificant levels.

Table 4. Identified volatile organic compounds (VOC) and semivolatile organic compounds (semiVOC) in Nusyn-Noxfish® (in mg/L).

Lot Number	TCE ^a (mg/L)	Xylenes (mg/L)	Naphthalene (mg/L)	Methyl Naphthalenes (mg/L)
10883	77	690	110,000	100,000
6425 HJA6-2	484	2,020	76,300	86,300
6425 HJA9-2	532	1,320	68,200	85,300
6425 HJA10-2	633	1,930	71,300	87,600
6425 HJA11-2	554	1,280	62,900	78,300
6425 HJA12-2	540	1,330	70,100	86,300
6425 HJA13-A	575	2,720	68,000	84,900
4974 ALM-02	710	3,234	74,240	74,180
4674 ALM-08	910	1,400	44,000	94,000
4674 AMM-06	910	5,400	81,000	100,000
4674 AMM-05	910	1,790	73,000	97,000
4674 AMM-08	1,200	1,830	80,000	97,000
32367	28	112	60,000	110,000
32365	16	193	58,000	110,000
32369	8	148	59,000	110,000
- ^b	960	5,400	120,000	120,000
mean	565	1,925	73,502	95,055
SD	373	1,611	18,782	12,841
range	8-1,200	112-5,400	44,000-120,000	74,180-120,000

^a TCE = Trichloroethylene

^b No lot number given

Stability of rotenone

Typically a delay of one to seven days (three-day average) occurred between sample collection and analysis for rotenone and rotenolone while the samples when stored at a temperature of 4 °C in the absence of light. This delay could have a significant impact on the analytical results because rotenone is a short-lived compound. The degradation of rotenone in water from several locations was determined under normal storage conditions. Four of the six sites investigated showed significant ($P \leq 0.05$) differences in rotenone concentrations after six days of storage (Table 5). Water with higher alkalinity (>170 mg/L CaCO_3) and pH (>9.0) had higher degradation of rotenone (-24% and -25%) than did water with lower alkalinity (40 mg/L CaCO_3) and pH (7.7) that had lower degradation (no change to -16%). These results are

in agreement with Clemens and Martin (1953) who found rotenone degradation increased with increased alkalinity.

Table 5. Rotenone concentrations (in µg/L) before and after six days storage at a temperature of 4 °C in the absence of light. Asterisks denote significant changes ($p > 0.05$) using the Kruskal-Wallis test; mean values with SD in parentheses.

Alkalinity (mg/L CaCO ₃)	pH	Rotenone				Percent Change
		Before		After		
40	7.8	91.0	(7.1)	93.0	(6.1)	+2
180	9.2	68.0	(8.0)	52.0	(8.8)	-24*
40	7.7	31.6	(1.9)	28.2	(1.6)	-11*
40	7.7	47.8	(1.6)	40.0	(1.0)	-16*
40	9.3	238.0	(29.5)	238.0	(8.4)	0
172	9.6	14.0	(2.8)	10.5	(0.7)	-25*

Surface waters

Rotenone – Nusyn-Noxfish® was applied in lakes and reservoirs at target concentrations from 2 to 4 mg/L. At this application rate, rotenone generally degraded to non-detectable levels within one to three weeks (Table 6). The estimated half-life ($t_{1/2}$) of rotenone averaged 2.3 days and varied from 0.58 days in Meiss Lake to 7.7 days in Lake Davis. Half-life values appeared to increase with increased water depth indicating photolysis may be an important route of rotenone decomposition. Kaweah Reservoir, Success Reservoir, Lake Davis, and Frenchman Lake had respective $t_{1/2}$ values of 1.7, 2.4, 7.7, and 3.5 days (average depths of 8 to 12 m) and Percolation Reservoir 12 and Meiss Lake had respective $t_{1/2}$ values of 0.94 and 0.83 days (average depths of 0.8 to 1.0 m). Rotenone appeared to have a longer half-life in colder water; Kaweah Reservoir (temperature of 20 to 22 °C), Frenchman Lake (temperature of 10 to 22 °C), and Lake Davis (temperature of 1 to 12 °C) have similar average water depths but respective $t_{1/2}$ values of 1.7, 3.5, and 7.7 days. These values are in agreement with Gilderhaus et al. (1988) who found a $t_{1/2}$ value of 0.94 days for a warm (temperature of 23 to 27 °C), shallow (<1 m deep) pond and those of Dawson et al. (1991) who found $t_{1/2}$ values of 1.8 to 0.7 days for average water temperatures of 15 to 24 °C, respectively. Rotenone had a longer $t_{1/2}$ of 10.3 days in a colder (temperature of 0 to 5 °C), shallow pond (Gilderhaus et al. 1988).

Rotenolone – Rotenolone concentrations appeared to parallel rotenone concentrations and typically were not found in the absence of rotenone. Exceptions to this were Meiss Lake in 1988 and 1990 (CDFG 1988b; 1989b) and Wolf Creek Lake in 1991 (CDFG 1992b). A decrease in water temperature below 11 °C in 1988 coincided with rotenolone residues persisting in Meiss Lake for three weeks, two weeks longer than rotenone residues (CDFG 1988b). Rotenolone persisted for at least six weeks past rotenone in Wolf Creek Lake (CDFG 1992b). The increased persistence of rotenolone in Meiss Lake and Wolf Creek Lake may be reflective of the low water alkalinity (< 15 mg/L CaCO₃), cooler temperatures (≤ 11 °C), high solar radiation at high elevations (> 2,500 m), and the relatively sterile granitic soils of the Sierra Nevada Mountain Range. Rotenone may be more susceptible to photolysis than rotenolone. The persistence of rotenolone could delay stocking fish because rotenolone is reported to have

toxicity comparable to that of rotenone in mammals (Yamamoto 1970). However, anecdotal observations indicate that rotenolone is approximately one-tenth as lethal as rotenone to salmonids.

Table 6. Rotenone and rotenolone concentrations (in $\mu\text{g/L}$) at various time intervals (days in parentheses) and corresponding half-life ($t_{1/2}$) values (in days) of rotenone decay assuming first-order kinetics.

Location (Year)	Rotenone/Rotenolone Concentrations ($\mu\text{g/L}$)				$t_{1/2}$
Kaweah Reservoir (1987)	76/17 (1)	55/27 (3)	43/23 (5)	<2/<2 (12)	1.7
Bravo Reservoir (1987)	254/236 (1)	46/42 (2)	<2/<2 (6)	---	0.65
Lonestar Pond (1987)	310/46 (1)	49/10 (2)	24/13 (6)	<2/<2 (14)	1.8
Percolation Res. 5 (1987)	370/65 (1)	150/160 (3)	120/190 (8)	<2/<2 (15)	1.7
Percolation Res. 12 (1987)	200/70 (1)	27/61 (3)	<2/<2 (8)	---	0.94
Success Reservoir (1988)	122/64 (1)	39/20 (2)	22/34 (6)	<2/<2 (30)	4.6 ^a
Meiss Lake (1988)	64/220 (0.13)	30/70 (1)	8.2/52 (3)	<2/23 (6.2)	0.96
Meiss Lake (1989)	47/20 (0.08)	41/27 (0.17)	30/28 (0.5)	18/16 (1)	0.96
Meiss Lake (1990)	11/36 (0.04)	5.9/37 (2.9)	3.8/24 (0.92)	<2/13 (1.9)	0.58
Frenchman Lake (1991)	90/42 (1)	39/35 (2)	28/34 (3)	6/21 (14)	3.5
Wolf Creek Lake (1992)	16/70(8)	<2/90(21)	<2/70(28)	<2/55(51)	2.9 ^b
Lake Davis (1997)	44/14(1)	32/18(3)	29/20(7)	11/20(21)	7.7

^a A value of 2.4 days is computed without the 30-day value

^b Assumes initial concentration of 100 $\mu\text{g/L}$ rotenone

Neutralization - Rotenone was allowed to naturally degrade in the Tule River and Kaweah River drainages, Frenchman Lake, Meiss Lake, Wolf Creek Lake, and Lake Davis. Rotenone can be neutralized with potassium permanganate, however, potassium permanganate is toxic to fish at relatively low (2-4 mg/L) concentrations (Finlayson et al. 2000). Rotenone at 2 mg/L formulation (50 $\mu\text{g/L}$ rotenone) was neutralized with potassium permanganate at 4 mg/L in the discharge from Frenchman Lake into Little Last Chance Creek and in the discharge from Lake Davis into Big Grizzly Creek. Rotenone applied at 1 mg/L formulation (25 $\mu\text{g/L}$) was neutralized with potassium permanganate at 3 mg/L at the fish barriers on Mill Creek, Upper Truckee River, Silver King Creek, Silver Creek and Wolf Creek. A 30-minute contact (travel) time was utilized as the neutralization area. Flow rates of potassium permanganate were checked every 30 to 120 minutes. Stress of caged rainbow trout placed upstream and downstream was used to initiate the neutralization and judge the effectiveness of neutralization on-site, respectively. No direct on-site method for measuring rotenone concentrations exists. Results from water samples analyzed for rotenone in the laboratory indicated that potassium permanganate was very effective in oxidizing rotenone and rotenolone to concentrations below detection limits (2 $\mu\text{g/L}$). If potassium permanganate levels are in balance with rotenone levels, then toxic levels of potassium permanganate should be quickly reduced through the oxidation of organic components and rotenone in water. Typically, successful neutralization of rotenone with potassium permanganate occurs within the 30-minute neutralization zone.

However, there have been several failures of the neutralization process in California. In 1992, fish were killed below the 30-minute neutralization zone in Silver King Creek probably due to lower than anticipated rotenone concentrations, in turn causing excessive (and probably toxic) potassium permanganate concentrations (CDFG 1992a). In 1997, fish were killed below the 30-minute neutralization zone in Big Grizzly Creek when the flow of potassium permanganate had been inadvertently decreased from 4 mg/L to 2 mg/L, in turn causing toxic concentrations of rotenone (Siepmann and Finlayson 1999). In both these instances, cold-water temperatures (< 10 °C) may have contributed to the ineffective neutralization. The CDFG has experimented successfully with maintaining a nontoxic 1 mg/L potassium permanganate concentration at the end of the 30-minute neutralization zone with the aid of a calibrated field spectrophotometer. The results of the spectrophotometer were used to adjust the potassium permanganate flows accordingly (Parmenter and Fujimura 1994).

VOC and semiVOC – The four known non-rotenoid organic compounds found in undiluted Nusyn-Noxfish® (xylene, trichloroethylene, naphthalene, and 2-methylnaphthalene) have been found in surface water (Table 7). The concentrations of these in standing waters were generally close to expected values based on dilution. Maximum residues detected have been 4.9 µg/L for trichloroethylene, 6.7 µg/L for xylene, 332 µg/L for naphthalene, and 390 µg/L for 2-methylnaphthalene. Standing waters have contained higher concentrations of these components than flowing waters because of higher treatment rates of Nusyn-Noxfish® (2 to 4 mg/L versus 1 mg/L) and lack of conditions (i.e., agitation) conducive to volatility. Flowing waters allow for chemicals to more easily volatilize through agitation in riffles, rapids, and waterfalls.

Neither trichloroethylene (<0.5 µg/L) nor xylene (<0.5 µg/L) have been found in flowing waters (Table 7), except for one sample collected immediately below a drip station (CDFG 1994a). Concentrations of trichloroethylene have never exceeded the USEPA drinking water standard (Maximum Contaminant Level) of 5 µg/L (USEPA 1985). Similarly, the concentrations of xylene have never exceeded the drinking water standard (Health Advisory) of 620 µg/L (USEPA 1981). Drinking water standards have not been developed for naphthalene and methylnaphthalenes, but acute toxicity values to mammals are >1,500 mg/kg (Verschueren 1983). These short-lived VOC and semiVOC occur at levels that do not appear to be threats to public health. All four chemicals dissipated within two to three weeks.

PB – The synergist PB has been found to be a persistent chemical in deep and cool waters. It persisted for almost nine months in Lake Davis (Siepmann and Finlayson 1999). PB was present at about 32 µg/L one week after application in mid-October 1997. PB remained at 20 to 30 µg/L until the lake iced-over in late December 1997, and degraded to about 5 µg/L by the time the ice melted the following spring 1998. PB remained detectable (> 2 µg/L) in the cold (< 10 °C), deep water (>25 m) until early July 1998 (Siepmann and Finlayson 1999).

Ground waters

Twenty-six wells have been monitored since 1987 for the presence of Nusyn-Noxfish® constituents. Samples for analysis of rotenone, rotenolone, VOC, and semiVOC were collected between 1 and 456 days following treatment (Table 8). All samples were negative for all compounds with the exception of a 1.5 µg/L xylene detection that was found in a single sample collected from the Corps of Engineers Well at Kaweah Reservoir, 59 days after the treatment. This finding was believed to be an anomaly. A subsequent sample collected at 185 days after treatment was negative for xylene.

Residues of rotenone or rotenolone were never found in any of the wells monitored. This was expected because the wells were a minimum of 1 m deep and at least 3 m horizontally from the rotenone-treated water bodies. Rotenone leaches vertically less than 2 cm in most soil types, less than 8 cm in sandy soil, and binds readily to sediment (K_{oc} of 1,060 to 1,810; Dawson 1986). Additionally, none of the other VOC or semiVOC constituents of the Nusyn-Noxfish® formulation have been detected in any of the wells monitored. This is probably due to the chemicals' volatility and lack of persistence in surface water.

Table 7. Maximum concentrations (in µg/L) of volatile organic compounds (VOC) and semivolatile organic compounds (semiVOC) detected (maximum duration of detection for standing waters in days in parentheses) following application of Nusyn-Noxfish® at 1 mg/L to flowing waters and 2 to 4 mg/L in standing waters. Expected concentrations based on dilution are in brackets. Samples were collected during application to flowing waters and within one day following application to standing waters.

Location (Year)	Trichloroethylene (µg/L)	Xylene (µg/L)	Naphthalene (µg/L)	2-Methylnaphthalene (µg/L)
Standing waters (2-4 mg/L)				
Kaweah Reservoir (1987)	2.1 (<21)	<1.0	78 (<21)	NA ^a
Bravo Reservoir (1987)	4.9 (<14)	2.2 (<14)	<2.0	NA
Success Reservoir (1988)	<0.5	<0.5	<2.5	NA
Meiss Lake (1989) ^c	1.0	6.7	332	NA
Meiss Lake (1990) ^c	<0.5	0.9	24	21
Frenchman Lake (1991)	1.6 (<14)	4.5 (<7)	16 (<14)	57 (<7)
Lake Davis (1997)	0.8 (<7) [1.1-2.2]	2.0 (<7) [3.8-7.6]	210 (<14) [147-294]	390 ^b (<14) [190-380]
Flowing waters (1 mg/L)				
U. Truckee River (1989)	<0.5	<0.5	14	NA
U. Truckee River (1990)	<0.5	<0.5	4.2	<2.5
Mill Creek (1989)	<0.5	<0.5	19	NA
Silver King Creek (1991)	<0.2	<0.2	57	35
Silver King Creek (1992)	<0.5	<0.5	<0.5	<2
Silver King Creek (1993)	0.76	0.56	52	50
Wolf Creek (1991)	<0.2	<0.2	<0.2	0.7
Wolf Creek (1992)	<0.5	<0.5	<0.5	<2
Silver Creek (1994)	<0.2	<0.2	1.7	<2
Silver Creek (1995)	<0.2	<0.2	9.1	5.1
Silver Creek (1996)	<0.2 [0.55]	<0.2 [1.9]	5.2 [74]	<2 [95]

^a NA = Not analyzed

^b 1-methylnaphthalene was also detected at maximum concentration of 210 µg/L and degraded with 2-methylnaphthalene concentrations.

^c Measurements in Meiss Lake taken only once.

Sediment

The presence of rotenone formulation constituents in sediment has been monitored at five locations since 1991 (Table 9). The majority of samples from sediments in flowing waters did not contain detectable residues (> 30 µg/kg, dry weight) of either rotenone or rotenolone. Only one sample contained a detectable rotenone concentration (37 µg/kg, dry weight). In one case, rotenolone was detected at an unusually high concentration (440 µg/kg, dry weight). This detection, however, is believed to be an analytical anomaly due to the magnitude of the detection, its presence in the absence of the parent compound, and its brief persistence (< 24h). The other rotenolone residue detected in sediment from flowing water (60 µg/kg, dry weight)

may also be anomalous. In no case did rotenone or rotenolone residues persist in flowing water sediments longer than seven days. Detections of rotenone and rotenolone in sediments in standing waters were more common than in flowing waters. The maximum concentrations detected were 522 and 890 µg/kg (dry weight) for rotenone and rotenolone, respectively. In no case did these residues persist in sediments from standing waters for longer than 60 days.

Table 8. Days after Nusyn-Noxfish® application that samples were collected from wells. All samples contained less than detectable concentrations of rotenone, rotenolone, volatile organic compounds (VOC) and semivolatile organic compounds (semiVOC).

Basin (Year)	Rotenone/Rotenolone (days)	VOC and semiVOC (days)
Kaweah River (1987)		
Corps of Engineers Well	24, 39, and 59	24, 39, 59 ^a , and 185
Lemon Cover Well 1	7, 28, and 49	49
Lemon Cover Well 2	7, 28, and 49	50
Woodlake Well 1	8, 28, and 50	50
Woodlake Well 2	7, 28, and 50	50
Woodlake Well 3	15, 37, and 58	58
Woodlake Well 4	7, 28, and 49	49
Woodlake Well 5	7, 28, and 49	7, 28, and 49
Kaweah Percolation Res. 5 Well	8, 30, and 51	51
Upper Truckee Rv. (1988 & 1989)		
Christmas Valley Wells 1,2 & 3	2 and 30	NS ^b
Upper Truckee River (1990)		
Christmas Valley Wells 1 & 3	7 and 49	NS
Christmas Valley Well 2	7 and 49	7 and 49
Mill Creek (1988)		
Walker Wells 1, 2 & 3	2 and 30	NS
Mill Creek (1989)		
Walker Wells 1, 2 & 3	2 and 30	2 and 30
Tule River (1988)		
Springville Well	1 and 30	30 and 456
Country Club Well	1 and 30	30 and 456
Corps of Engineers Well	6 and 30	30 and 456
Frenchman Lake (1991) ^c		
Big Cove Campground Well	1 and 30	1 and 30
Spring Creek Campground Well	1 and 30	1 and 30
Cottonwood Campground Well	1 and 30	1 and 30
Lake Davis (1997) ^c		
South Davis Wells 1,2,3 & 4	5,14,90,194, and 324	5,14,90,194, and 324
Grasshopper Campground Well	5,14,90,194, and 324	5,14,90,194, and 324

^aXylene was detected at 1.5 µg/L at day 59 and believed to be an anomaly; sample taken on day 185 was free of xylene.

^bNS = Not sampled.

^cAll of the Lake Davis wells were also analyzed for PB.

Table 9. Maximum concentrations (in $\mu\text{g}/\text{kg}$, dry weight) of rotenone, rotenolone, and semivolatile (semiVOC) organic compounds detected in sediment from formulated rotenone use sites. No volatile organic compounds (VOC) were detected. The durations of detectable residues (in days) are indicated in parentheses.

Location (Year)	Rotenone ($\mu\text{g}/\text{kg}$)	Rotenolone ($\mu\text{g}/\text{kg}$)	Naphthalene ($\mu\text{g}/\text{kg}$)	Methyl Naphthalene ($\mu\text{g}/\text{kg}$)
Silver King Creek (1991)	<30	<30	<30	<30
Silver King Creek (1992)	<30	440 ^b (<1)	<30	<30
Silver King Creek (1993)	<30	<30	<30	<30
Silver Creek (1994)	<30	<30	<70	<70
Silver Creek (1995)	37 (<7)	<30	<30	<30
Silver Creek (1996)	<30	<30	<30	<30
Wolf Creek (1991)	<30	60 ^b (<7)	<30	<30
Wolf Creek (1992)	310 ^a (<14)	890 ^a (<14)	<30	<30
Frenchman Lake (1991)	180 (<14)	560 (<21)	24 (<180)	218 (<180)
Lake Davis (1997)	522 (<60)	134 (<60)	91 (<60)	231 (<60)

^a Results are from Wolf Creek Lake. No residues above the reporting limit (30 $\mu\text{g}/\text{kg}$, dry weight) were found for Wolf Creek.

^b Probable analytical anomaly.

All sediment samples were negative (< 30 $\mu\text{g}/\text{kg}$, dry weight) for VOC constituents of formulated rotenone (xylene and trichloroethylene) in both flowing and standing waters. Likewise, the semiVOC naphthalene and methylnaphthalene were not detected in sediments in flowing waters. In standing waters, the maximum concentrations of naphthalene and methylnaphthalene in sediments were 91 and 231 $\mu\text{g}/\text{kg}$ (dry weight), respectively. In no case did detectable residues of semiVOC persist in standing water sediments for longer than 180 days.

Conclusions

Fifteen years of monitoring Nusyn-Noxfish® applications and one Pro-Noxfish® application indicate that rotenone and the other organic compounds in surface and ground waters behave as expected based on dilution and known physicochemical properties. All chemicals with the exception of PB and rotenolone can be expected to dissipate from surface water within six weeks. The persistence of PB and rotenolone increases inversely with temperature, but neither has persisted for greater than nine months. None of the constituents in Nusyn-Noxfish® or Pro-Noxfish® have contaminated ground water. Only rotenone, rotenolone, and semiVOC were found in sediment, and none persisted longer than 180 days.

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